

Handbook of Materials Failure Analysis

With Case Studies from the Electronic
and Textile Industries



Edited by
Abdel Salam Hamdy Makhlouf
Mahmood Aliofkhazraei



Handbook of Materials Failure Analysis

With Case Studies From the
Electronic and Textile
Industries

This page intentionally left blank

Handbook of Materials Failure Analysis

With Case Studies From the
Electronic and Textile
Industries

Edited by

Abdel Salam Hamdy Makhoul

*Vice President & Engineering Consultant:
Integrated Mechanical Material Corrosion Consulting (IM2C),
Texas, United States*
and

*Full Professor: Central Metallurgical
Research and Development Institute,
Cairo, Egypt*

Mahmood Aliofkhazraei

*Department of Materials Science, Tarbiat Modares University,
Tehran, Iran*



Butterworth-Heinemann
An imprint of Elsevier

Butterworth-Heinemann is an imprint of Elsevier
The Boulevard, Langford Lane, Kidlington, Oxford OX5 1GB, United Kingdom
50 Hampshire Street, 5th Floor, Cambridge, MA 02139, United States

Copyright © 2020 Elsevier Ltd. All rights reserved.

No part of this publication may be reproduced or transmitted in any form or by any means, electronic or mechanical, including photocopying, recording, or any information storage and retrieval system, without permission in writing from the publisher. Details on how to seek permission, further information about the Publisher's permissions policies and our arrangements with organizations such as the Copyright Clearance Center and the Copyright Licensing Agency, can be found at our website: www.elsevier.com/permissions.

This book and the individual contributions contained in it are protected under copyright by the Publisher (other than as may be noted herein).

Notices

Knowledge and best practice in this field are constantly changing. As new research and experience broaden our understanding, changes in research methods, professional practices, or medical treatment may become necessary.

Practitioners and researchers must always rely on their own experience and knowledge in evaluating and using any information, methods, compounds, or experiments described herein. In using such information or methods they should be mindful of their own safety and the safety of others, including parties for whom they have a professional responsibility.

To the fullest extent of the law, neither the Publisher nor the authors, contributors, or editors, assume any liability for any injury and/or damage to persons or property as a matter of products liability, negligence or otherwise, or from any use or operation of any methods, products, instructions, or ideas contained in the material herein.

British Library Cataloguing-in-Publication Data

A catalogue record for this book is available from the British Library

Library of Congress Cataloging-in-Publication Data

A catalog record for this book is available from the Library of Congress

ISBN: 978-0-08-101937-5

For Information on all Butterworth-Heinemann publications
visit our website at <https://www.elsevier.com/books-and-journals>

Publisher: Matthew Deans
Acquisition Editor: Christina Gifford
Editorial Project Manager: Lindsay Lawrence
Production Project Manager: Nirmala Arumugam
Cover Designer: Victoria Pearson

Typeset by MPS Limited, Chennai, India



Contents

List of contributors	xiii
About the editors	xvii
Preface	xix

Part 1 Electronics industries 1

CHAPTER 1 Failures of electronic devices: solder joints failure modes, causes and detection methods.....3

*Mohammad A. Gharaibeh and
Abdel Salam Hamdy Makhlouf*

1.1 Introduction	3
1.2 Thermal cycling.....	4
1.3 Shock and vibration	8
1.4 Failure detection methods in electronics industry	12
1.5 Conclusion.....	14
1.6 Recommendations	15
References.....	16

CHAPTER 2 Electron beam radiation and its impacts to failure analysis in semiconductor industry 19

*Binghai Liu, Xiaoming Li, Younan Hua, Nan Cho,
Zhili Dong, Yuzhe Zhao, Kenny Ong and Zhiqiang Mo*

2.1 Introduction	19
2.2 Impact of electron beam radiation damage during SEM failure analysis	22
2.2.1 SEM physical FA and low- <i>k</i> /ultralow- <i>k</i> dielectrics	22
2.2.2 Electron beam radiation damage to low- <i>k</i> and ultralow- <i>k</i> dielectric materials	25
2.2.3 Control of electron beam radiation damage to low- <i>k</i> and ultralow- <i>k</i> dielectric materials	30
2.3 Impact of electron beam radiation during FIB and TEM failure analysis: radiation damage to LK and ULK dielectrics	35
2.3.1 Electron beam radiation damage during electron beam survey before focus ion beam milling	36
2.3.2 Electron beam radiation damage during electron beam coating before focus ion beam milling	37
2.3.3 Electron beam radiation damage during focus ion beam milling for transmission electron microscopy sample preparation	38

2.3.4	Electron beam radiation damage during transmission electron microscope analysis	40
2.4	Impact of electron beam radiation damage during TEM failure analysis: radiation damage to silicon nitride	45
2.5	Impact of electron beam radiation damage during TEM FA: boron diffusion and segregation induced phase and microstructure changes in CoFeB material	55
2.5.1	Stage-I: The electron radiation—induced unilateral amorphization of Co ₃ Fe thin film	57
2.5.2	Stage-II: The electron radiation—induced recrystallization in the amorphized Co ₃ Fe thin film.....	60
2.6	Conclusion	65
	References.....	66

CHAPTER 3 Failure of intermetallic solder ball due to stress shielding and amplification effects

E.P. Ooi, R. Daud, N.A.M. Amin, M.S. Abdul Majid, M. Afendi, A. Mohamad and A.K. Ariffin

3.1	Introduction	71
3.2	Methodology.....	72
3.2.1	Finite element modeling	72
3.3	Results and discussion.....	77
3.3.1	The effect of distance (B) between two parallel edge cracks.....	77
3.3.2	Multiple crack analysis—coplanar cracks.....	78
3.4	Conclusion	82
	Acknowledgment.....	83
	References.....	83
	Further reading	84

CHAPTER 4 Assessment of failure of consumer electronics due to indoor corrosion in subtropical climates.....

Armando Ortiz, Víctor Hugo Jacobo and Rafael Schouwenaars

4.1	Introduction	87
4.2	Methods	89
4.3	Damage analysis.....	90
4.4	Discussion.....	98
4.5	Conclusion	103
	Acknowledgments	103
	References.....	103

CHAPTER 5	Pb-free solder—microstructural, material reliability, and failure relationships	107
	<i>Guang Ren, Maurice N. Collins, Jeff Punch, Eric Dalton and Richard Coyle</i>	
5.1	Introduction	107
5.1.1	Development of Pb-free solder alloys	107
5.1.2	Failure and microstructure	108
5.1.3	An overview of the chapter	109
5.2	Case study I—Pb-doped solder alloys	113
5.2.1	Forward compatible mixing	113
5.2.2	Backward compatible mixing	115
5.2.3	Lessons learnt from case study I	126
5.3	Case study II—First- and second-generation Sn—Ag—Cu solder alloys	128
5.3.1	Effect of ball grid array component	130
5.3.2	Effect of Ag content	130
5.3.3	Effect of dwell time	131
5.3.4	Effect of accelerated temperature cycling profile	131
5.3.5	Microstructural evolution and failure mechanisms	131
5.3.6	Lessons learnt from case study II	135
5.4	Case study III—High-performance solders (third generation)	135
5.4.1	Effect of micro-alloying on Sn—Ag—Cu	136
5.4.2	Two commercialized alloys	136
5.4.3	Lessons learnt from case study III	141
5.5	Case study IV—Low-temperature solders	141
5.5.1	Effect of substrate	142
5.5.2	Effect of micro-alloying	142
5.5.3	Lessons learnt from case study IV	145
5.6	Conclusion	145
	References	145
 CHAPTER 6	 The role of contamination in the failure of electronics—case studies	 153
	<i>W. John Wolfgang, Joseph Colangelo and Jason Wheeler</i>	
6.1	Introduction	153
6.2	Case studies	154
6.2.1	Example 1—Contamination as a primary cause of motor failures	154
6.2.2	Example 2—Electrolyte contamination	165

6.3 Discussion.....	175
References.....	177

CHAPTER 7 Analytical solutions for electronic assemblies subjected to shock and vibration loadings 179

Mohammad A. Gharaibeh

7.1 Introduction	179
7.2 Test assembly details	180
7.3 Experimental modal analysis	180
7.4 Finite element modeling	180
7.5 Analytical solution details.....	182
7.5.1 Free vibration	182
7.5.2 Forced vibration: harmonic loading	185
7.5.3 Forced vibration: shock loading	187
7.6 Results and discussions.....	190
7.6.1 Free vibration: natural frequencies and mode shapes....	190
7.6.2 Forced vibration: harmonic loading	190
7.6.3 Forced vibration: impact loading.....	195
7.7 Conclusion	200
Nomenclature.....	201
References.....	201

CHAPTER 8 Stress analysis of stretchable conductive polymer for electronics circuit application..... 205

*N.A. Aziz, A.A. Saad, Z. Ahmad, S. Zulfiqar,
F.C. Ani and Z. Samsudin*

8.1 Introduction	205
8.2 Experimental procedure	206
8.2.1 Sample preparation	206
8.2.2 Printing process of circuits	207
8.2.3 Universal tensile testing.....	207
8.3 Stress–strain analysis of substrate and conductive ink	208
8.3.1 Neo-Hookean model	209
8.3.2 Multilinear plastic model.....	209
8.4 Finite element analysis.....	209
8.4.1 Modeling and meshing of different printing shapes models	210
8.4.2 Boundary conditions	212
8.4.3 Analysis using the simulation process.....	214

8.5	Results and discussion.....	215
8.5.1	Material properties of stretchable electronic circuit material	215
8.5.2	Deformation behavior of stretchable electronics circuit	216
8.5.3	Equivalent stress analysis of a thermal sensor circuit design up to 10% strain	219
8.5.4	Effect of width in reducing the equivalent stress in a thermal sensor circuit	220
8.5.5	Equivalent stress limitation when the load is applied up to 10% strain	221
8.6	Future recommendations	222
8.7	Conclusion	222
	Acknowledgments	223
	References.....	223

CHAPTER 9 New methodology for qualification, prediction, and lifetime assessment of electronic systems..... 225

Bey Temsamani Abdellatif

9.1	Introduction	225
9.2	Improved reliability assessment method	226
9.2.1	Prediction handbooks	227
9.2.2	Life data analysis	228
9.2.3	Accelerated life testing	228
9.2.4	Improved reliability estimation methods.....	229
9.2.5	Prediction handbooks: FIDES rather than MIL-217F	229
9.2.6	Intelligent life data analysis rather than real life data averaging.....	232
9.2.7	[HALT + ALT] rather than ALT	234
9.2.8	Reliability block diagram tools and fault tree analysis for complex systems	236
9.3	Application examples.....	239
9.3.1	Electrolytic capacitors reliability analysis.....	239
9.3.2	Demonstration of the combined methodology on front light module	244
9.3.3	Supercapacitors reliability analysis	245
9.4	New trends to improve reliability analysis.....	259
9.4.1	Mission profile	259
9.4.2	Online condition monitoring—case study.....	260

9.5	Summary.....	267
9.6	General observations and conclusion	269
	Acknowledgments	270
	References.....	270

Part 2 Textiles industries 275

CHAPTER 10 Failure of yarns in different textile applications..... 277

Radostina A. Angelova

10.1	Introduction	277
10.2	Staple yarn failure depending on the spinning method.....	281
10.3	Yarn failure depending on the gauge length	285
10.4	Yarn failure depending on the strain rate	286
10.5	Modeling of the yarn failure	287
10.6	Yarn failure in fabrics and composite structures.....	288
	10.6.1 Yarn failure in fabrics.....	288
	10.6.2 Yarn failure in composite structures	290
	10.6.3 High-strength yarn failure.....	293
	10.6.4 Failure of yarns from brittle high-performance fibers.....	294
	10.6.5 Carbon nanotubes yarn failure.....	295
	10.6.6 Yarn failure in electronic textiles	296
10.7	Conclusion and future trends	297
	References.....	298

CHAPTER 11 Textile failure analysis and mechanical characterization using acoustic emission technique..... 303

Carlos Rolando Rios-Soberanis

11.1	Introduction	303
	11.1.1 Textiles architecture.....	304
	11.1.2 Textiles-reinforced composites	306
	11.1.3 Acoustic emission technique	307
	11.1.4 Textiles mechanical and damage characterization	309
11.2	Conclusion.....	324
11.3	Future trends.....	325
	References.....	326
	Further reading	327

CHAPTER 12 Treatment effect on failure mode of industrial carbon textile at elevated temperature	329
<i>Manh Tien Tran, Xuan Hong Vu and Emmanuel Ferrier</i>	
12.1 Introduction	329
12.2 Experimental work	332
12.2.1 Equipment used	332
12.2.2 Specimens	334
12.2.3 Loading paths	335
12.3 Results	337
12.3.1 Elevated temperature behavior of industrial textiles	337
12.3.2 Evolution of the thermomechanical properties as a function of temperature	340
12.3.3 Discussion	342
12.4 Conclusions	350
12.5 Future trends	350
References	351
Index	355

This page intentionally left blank

List of contributors

Bey Temsamani Abdellatif

Flanders Make—The Strategic Research Center in Manufacturing Industry,
Leuven, Belgium

M. Afendi

Fracture and Damage Mechanic Research Group, School of Mechatronic
Engineering, Universiti Malaysia Perlis, Arau, Malaysia

Z. Ahmad

Universiti Sains Malaysia, Engineering Campus, Nibong Tebal, Malaysia

N.A.M. Amin

Fracture and Damage Mechanic Research Group, School of Mechatronic
Engineering, Universiti Malaysia Perlis, Arau, Malaysia

Radostina A. Angelova

Technical University of Sofia, Sofia, Bulgaria

F.C. Ani

Jabil Circuit Sdn Bhd, Bayan Lepas Industrial Park, Bayan Lepas, Malaysia

A.K. Ariffin

Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia,
Bangi, Malaysia

N.A. Aziz

Universiti Sains Malaysia, Engineering Campus, Nibong Tebal, Malaysia

Nan Cho

Wintech Nano-Technology Services Pte Ltd, Singapore

Joseph Colangelo

Raytheon Company, McKinney Failure Analysis Laboratory, McKinney, TX,
United States

Maurice N. Collins

Stokes Labs, Bernal Institute, University of Limerick, Limerick, Ireland

Richard Coyle

Nokia Bell Labs, Murray Hill, NJ, United States

Eric Dalton

Stokes Labs, Bernal Institute, University of Limerick, Limerick, Ireland

R. Daud

Fracture and Damage Mechanic Research Group, School of Mechatronic
Engineering, Universiti Malaysia Perlis, Arau, Malaysia

Zhili Dong

School of Materials Science & Engineering, College of Engineering, Nanyang
Technological University, Singapore

Emmanuel Ferrier

University of Lyon, University Claude BERNARD Lyon 1, Laboratory of Composite Materials for Construction LMC2, Villeurbanne, France

Mohammad A. Gharaibeh

Department of Mechanical Engineering, The Hashemite University, Zarqa, Jordan

Younan Hua

Wintech Nano-Technology Services Pte Ltd, Singapore

Víctor Hugo Jacobo

Departamento de materiales y manufactura, Facultad de ingeniería, Edificio O, Universidad Nacional Autónoma de México, Coyoacán, Ciudad de México, México

Xiaoming Li

Wintech Nano-Technology Services Pte Ltd, Singapore

Binghai Liu

Wintech Nano-Technology Services Pte Ltd, Singapore; Product, Test and Failure Analysis, Department of Technology Development, GLOBALFOUNDRIES Singapore Pte Ltd, Singapore

M.S. Abdul Majid

Fracture and Damage Mechanic Research Group, School of Mechatronic Engineering, Universiti Malaysia Perlis, Arau, Malaysia

Abdel Salam Hamdy Makhlouf

Integrated Mechanical Material Corrosion Consulting (IM2C), Texas, United States; Central Metallurgical Research and Development Institute, Cairo, Egypt

Zhiqiang Mo

Product, Test and Failure Analysis, Department of Technology Development, GLOBALFOUNDRIES Singapore Pte Ltd, Singapore

A. Mohamad

Fracture and Damage Mechanic Research Group, School of Mechatronic Engineering, Universiti Malaysia Perlis, Arau, Malaysia

Kenny Ong

Product, Test and Failure Analysis, Department of Technology Development, GLOBALFOUNDRIES Singapore Pte Ltd, Singapore

E.P. Ooi

Fracture and Damage Mechanic Research Group, School of Mechatronic Engineering, Universiti Malaysia Perlis, Arau, Malaysia

Armando Ortiz

Departamento de materiales y manufactura, Facultad de ingeniería, Edificio O, Universidad Nacional Autónoma de México, Coyoacán, Ciudad de México, México

Jeff Punch

Stokes Labs, Bernal Institute, University of Limerick, Limerick, Ireland

Guang Ren

Stokes Labs, Bernal Institute, University of Limerick, Limerick, Ireland

Carlos Rolando Rios-Soberanis

Centro de Investigación Científica de Yucatán (CICY), Mérida, Mexico

A.A. Saad

Universiti Sains Malaysia, Engineering Campus, Nibong Tebal, Malaysia

Z. Samsudin

Jabil Circuit Sdn Bhd, Bayan Lepas Industrial Park, Bayan Lepas, Malaysia

Rafael Schouwenaars

Departamento de materiales y manufactura, Facultad de ingeniería, Edificio O, Universidad Nacional Autónoma de México, Coyoacán, Ciudad de México, México

Manh Tien Tran

University of Lyon, University Claude BERNARD Lyon 1, Laboratory of Composite Materials for Construction LMC2, Villeurbanne, France; Hanoi University of Mining and Geology (HUMG), Hanoi, Vietnam

Xuan Hong Vu

University of Lyon, University Claude BERNARD Lyon 1, Laboratory of Composite Materials for Construction LMC2, Villeurbanne, France

Jason Wheeler

Raytheon Company, McKinney Failure Analysis Laboratory, McKinney, TX, United States

W. John Wolfgang

Raytheon Company, McKinney Failure Analysis Laboratory, McKinney, TX, United States

Yuzhe Zhao

Wintech Nano-Technology Services Pte Ltd, Singapore; Product, Test and Failure Analysis, Department of Technology Development, GLOBALFOUNDRIES Singapore Pte Ltd, Singapore

S. Zulfiqar

Universiti Sains Malaysia, Engineering Campus, Nibong Tebal, Malaysia

This page intentionally left blank

About the editors

Professor Abdel Salam Hamdy Makhlouf is the vice president of Integrated Mechanical Material Corrosion Consulting (IM2C), Texas, United States, and a full professor at the Central Metallurgical Research and Development Institute. He has 26 years of experience working in research and development as well as industrial and academic leadership as a professor of materials science, advanced “nano-bio” manufacturing engineering, and as a materials engineering consultant. He is the recipient of numerous national and international prizes and awards, including the Humboldt Research Award for Experienced Scientists from the Max Planck Institute, Germany; Fulbright, NSF; and Department of Energy fellowships, United States; Shoman Award in Engineering Science; and the State Prize of Egypt in Advanced Science and Technology. His research has focused on five critical areas: energy, health, environment, advanced manufacturing, and advanced materials. Dr. Makhlouf has published over 200 journal articles, as well as 17 books and handbooks for Springer and Elsevier in a broad range of cross-disciplinary research fields, including advanced multifunctional materials, nanotechnology, smart coatings, corrosion, biomaterials, waste/water treatment, and materials for energy applications. Dr. Makhlouf has served as a senior editor and board member of many international journals, as well as reviewer for several international funding agencies in the United States, Germany, United Kingdom, Qatar, Belgium, European Union, and Kazakhstan. He is also a consultant and reviewer for several universities worldwide.



Abdel Salam Hamdy Makhlouf

*Integrated Mechanical Material Corrosion Consulting (IM2C), Texas, United States
Central Metallurgical Research and Development Institute, Cairo, Egypt*

Dr. Mahmood Aliofkhazraei earned his PhD in materials science (corrosion, coatings, and surface engineering) from Tarbiat Modares University. In 2012, he joined in the Department of Materials Engineering at Tarbiat Modares University and then established coatings laboratory. Totally he has published more than 15 books, 150 journal papers, and 20 patents. He has given some invited talks including several keynotes in several countries. He has received several awards including the Khwarizmi award, IMES medal, INIC award, best thesis award (multiple times), best book award (multiple times), and the best young nanotechnologist award of Iran (two times). He is on the advisory editorial board of several materials science and nanotechnology journals. He is a member of the International Association of Corrosion Engineers (NACE International) and International Society of Electrochemistry (ISE).



Mahmood Aliofkhazraei

Department of Materials Engineering, Tarbiat Modares University, Tehran, Iran

Preface

This handbook provides a thorough understanding of the reasons materials fail in certain situations, covering important scenarios, including material defects, mechanical failure as a result of improper design, corrosion, surface fracture, and other environmental causes. The handbook was divided into two main parts. Part I covers the failure analysis in electronics industries and contains nine chapters. Part II explores the failure analysis in textile industries and contains three chapters.

Part I begins with a general overview of the main failure modes of electronic devices and focusing on three main parts of the electronic packages, printed circuit board, integrated circuit, and solder interconnects. A full discussion on solder failure modes and causes was presented as well as common solder crack failure detection approaches were presented. Then, the handbook proceeds from a discussion of the failure analysis process, types of failure analysis, and specific tools and techniques, to chapters on analysis of materials failure from various causes. In [Chapter 2](#), Electron beam radiation and its impacts to semiconductor failure analysis by SEM, FIB, and TEM, the issues of the electron-beam radiation damage that are commonly encountered during physical failure analysis in the modern semiconductor industry by SEM, FIB, and TEM were discussed. The effects of electron-beam radiation on the phase, microstructure, and compositions of some typical electron-beam sensitive materials utilized in semiconductor devices, such as low κ and ultra-low k dielectrics, silicon nitrides and CoFeB ferromagnetic materials were discussed. Comprehensive technical solutions were proposed in order to minimize the electron-beam radiation damages during the physical failure analysis of these special types of materials.

[Chapter 3](#), Stress shielding and amplification effect on intermetallic solder ball, discusses the stress shielding and amplification effect on intermetallic solder ball. Shielding and amplification effect between microcracks have been the unsolved problem in intermetallic solder ball failure. The interaction between multiple edge cracks in solder ball was investigated to quantify the effect of shielding and amplification on the crack driving force based on stress singularity approach. The effect of crack length between two parallel edge cracks on stress intensity factor was evaluated. The distance between the two parallel edge cracks plays an important role to reduce the shielding effect. It is concluded that when compare both shielding and amplification, the distance between the two crack-tip must be shorter to give better effect.

In [Chapter 4](#), Assessment of failure of consumer electronics due to indoor corrosion in subtropical climates, the failure of electronics due to indoor corrosion in telephones subject to normal use in various climate zones was studied. High humidity combined with strong day–night variations in temperature was the most important factor in promoting corrosion, together with the presence of urban and industrial air pollution. Materials selection, with an abundance of galvanic cells in the electronic assembly, also promote rapid deterioration.

Chapter 5, Pb-free solder—microstructural, material reliability, and failure relationships, introduces Pb-free solder and the relationship between its microstructural, material reliability, and failure for electronic packaging applications. The establishment of near-eutectic Sn–Ag–Cu (SAC) alloys as replacements for eutectic Sn–Pb marked the beginning of lead-free solder alloy development in electronic packaging industry. Second generation lead-free alloys with lower Ag content were introduced to address the shortcomings such as poor mechanical shock performance and higher cost. Yet the evolution has not stopped. In response to higher reliability requirements, third generation lead-free alloys are being developed to serve the applications operated in increasingly aggressive environments. In this chapter, case studies on thermal fatigue performance of various SAC-based lead-free solders and Sn–Zn-based low-temperature solders have been made. The effect of solder size, solder composition, Sn grain morphology, PCB surface finish, and thermal cycling profile on solder joint microstructure and reliability was evaluated. The relationship between microstructural evolution and thermal fatigue failure mechanism was discussed.

Chapter 6, The role of contamination in the failure of electronics—case studies, provides several case studies about the role of contamination such as water, dust, or metallic debris in failures of electronics. Some examples were given which highlight the role of electrolyte contamination (ionic salts and related chemicals) leading to electronics failures. Remediation was also discussed with regards to resolution of the cited examples.

Chapter 7, Analytical solutions for electronic assemblies subjected to shock and vibration loadings, introduces an analytical solution to solve for the dynamic problem of electronic assemblies subjected to shock and vibration loadings. The solution was first formulated to obtain the free vibration characteristics, that is, first natural frequency and mode shape, of the electronic assembly. Consequently, it was used to solve for the forced vibration problems of shock/impact and harmonic base excitations. The results of this analytical model were correlated with experimentally measured and finite element analysis data. Finally, the results of this solution were employed to examine the effect of the geometric and material configurations of the electronic structure on the fatigue performance of electronic products subjected to mechanical shock and harmonic vibrations.

Because of the lack of information regarding the reliability of stretchable electronic circuits, **Chapter 8**, Stress analysis of stretchable conductive polymer for electronics circuit application, presents the stress analysis of these circuits using a polymer material of polydimethylsiloxane (PDMS) as the substrate and a new formulated Ag flakes blends with PDMS (Ag–PDMS) conductive ink. The mechanical properties were characterized using Neo–Hookean model for substrate and multilinear plastic model for conductive ink. Different geometries of stretchable electronic circuit were modeled and analyzed under static structural analysis in simulation. The structural analyses were conducted on a real prototype of thermal sensor circuit application. The structural integrity of the circuit under different

geometries, loadings, and materials was assessed by investigating the deformation behavior of the circuit. The obtained results show that the critical area for stress concentration depends on the loading direction to the circuit printing.

Chapter 9, New methodology for qualification, prediction, and lifetime assessment of electronic systems, introduces new methodology for qualification, prediction, and lifetime assessment of electronic systems. The chapter addresses the physics of failure of some critical electronic components in modern industrial systems and assess their effects on system's reliability. A new methodology combining "conventional" reliability analysis and physics of failure analysis was illustrated for overall reliability and lifetime analysis in components and system level. Some examples of how such analysis could be used by industries to optimize a design or select optimal components maximizing system's reliability was provided. The new trends to improve reliability analysis, such as online mission profile measurements, online condition monitoring were discussed.

Part II is dedicated to discussing the failure analysis in textiles industries. **Chapter 10**, Failure of yarns in different textile applications, provides a comprehensive discussion about the failure of yarns in different textile applications. The yarns made of fibers or filaments can be subject of different bending forces, extreme load or dynamic elongation stress. Thus the failure of the yarns can influence the reliability of the textile macrostructures. The chapter presents state of the art in research in yarn failure. The problems of the spinning method applied to consolidate the fiber bundle, the gauge length, and the strain rate on the yarn failure were discussed. Problems of the yarn failure in different types of textile macrostructures, applied as technical textiles, composites, e-textiles, were also discussed.

In **Chapter 11**, Textile failure analysis and mechanical behavior characterization by using acoustic emission technique, damage mechanisms and mechanical performance in structural applications textiles have been a major concern in textiles industry. The chapter presents deep discussion about the use of acoustic emission technique for textile failure analysis and characterization of their mechanical behavior. This chapter discussed the actual cases in which textiles of different architecture were used to manufacture epoxy-based composites in order to study failure events under different external load such as tension and bending. The effects of the textile architecture/geometry on the mechanical behavior and damage process were discussed.

Chapter 12, Treatment effect on failure mode of industrial carbon textile at elevated temperature, provides a detailed study on the effect of treatment using epoxy resin and amorphous silica on the failure mode of industrial carbon textile at high temperature. The chapter presents an experimental study on the thermo-mechanical tensile behavior of three different industrial carbon textiles at high temperatures ranging from 25°C to 600°C. Three industrial carbon textiles were treated in manufacturing chain with different products in nature as coating (epoxy resin with different ratios and amorphous silica).

This handbook explores many real-world failure cases and case studies covering a wide spectrum of materials failure in electronics and textiles applications. The editors thank all the contributors for their excellent chapter contributions to this handbook, their hard work and patience during preparation and production of the book. We sincerely hope that the publication of this handbook will help people from industry and academia to get the maximum benefits from the experience contained in the published chapters.

PART

Electronics
industries

1

This page intentionally left blank

Failures of electronic devices: solder joints failure modes, causes and detection methods

Mohammad A. Gharaibeh¹ and Abdel Salam Hamdy Makhlouf^{2,3}

¹*Department of Mechanical Engineering, The Hashemite University, Zarqa, Jordan*

²*Integrated Mechanical Material Corrosion Consulting (IM2C), Texas, United States*

³*Central Metallurgical Research and Development Institute, Cairo, Egypt*

1.1 Introduction

Electronics nowadays come in smaller, thinner, and lighter specifications and can be found in different applications, such as computers, laptops, and handheld devices. Consequently, this brings up the importance of designing fully functional, reliable, and cheap electrical components. An electronic package can be defined as a complex set of materials with specific properties designed and assembled into electronic paths which interconnect devices to perform a function at a specific performance level. Generally, a typical electronic package consists of three main parts: printed circuit board (PCB), component, and solder joints, as shown in Fig. 1.1.

In electrical components, there are numerous number of interconnects, solder joints, which mainly allow the electrical current to flow smoothly between the PCB and the integrated circuit (IC) component. Fig. 1.2 shows an example of a ball grid array (BGA) solder joint cross-section [1]. Relevant research shows that 70% of the electronic equipment failures are caused by the failure of solder joint [2].

During manufacturing, shipping and service life electronics are prone to various harmful loading conditions that cause solder failures. Mainly, three loading conditions that are cyclic in nature can be experienced by an electronic assembly:

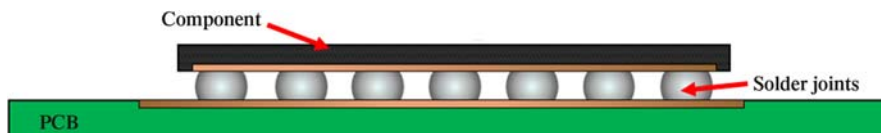
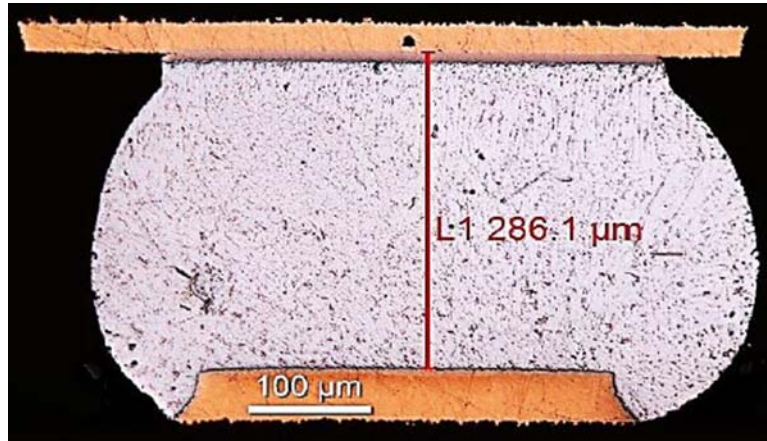


FIGURE 1.1

Parts of a typical electronic package. *PCB*, Printed circuit board.

**FIGURE 1.2**

Typical ball grid array solder joint cross-section.

1. Thermal cycling,
2. Mechanical shock, and
3. Vibration loadings.

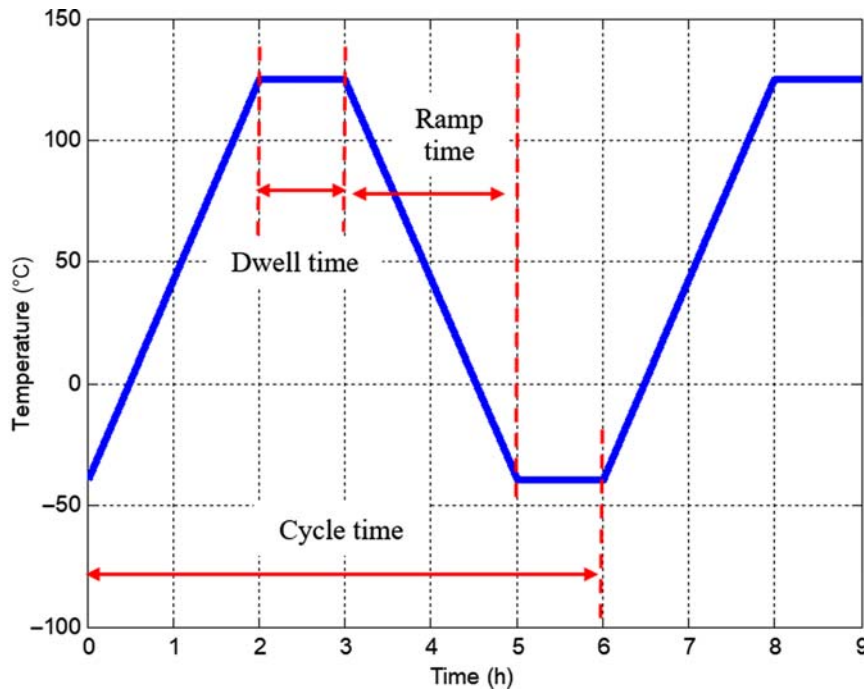
The purpose of this study is to understand the mechanical failures of solder interconnects caused due to the effect of each loading condition on electronic devices.

1.2 Thermal cycling

The term thermal cycling mainly refers to the periodic change in the surrounding temperature of the electronic package. Usually, one loading cycle can be divided into four consecutive steps: dwell at high temperature, ramp to low temperature, and dwell at this low level, then ramp to the high temperature. This sequence is repeated for each thermal cycle. Common cycling profile in electronics reliability tests ranges from -40°C to 125°C with different dwell and ramp times. Fig. 1.3 presents one thermal cycle profile.

Standardized accelerated thermal cycling (ATC) tests are commonly used to evaluate the thermomechanical reliability of electronic assemblies. During ATC, assemblies are uniformly heated up and cooled down in order to induce thermomechanical strains and stresses in interconnections and interfaces of the electronic assemblies according to the previously described thermal cycle step.

The main driving force for solder interconnection failure in thermal cycling is due to the coefficient of thermal expansion (CTE) mismatch between the PCB and the component. Specifically, when the assembly experiences high temperature,

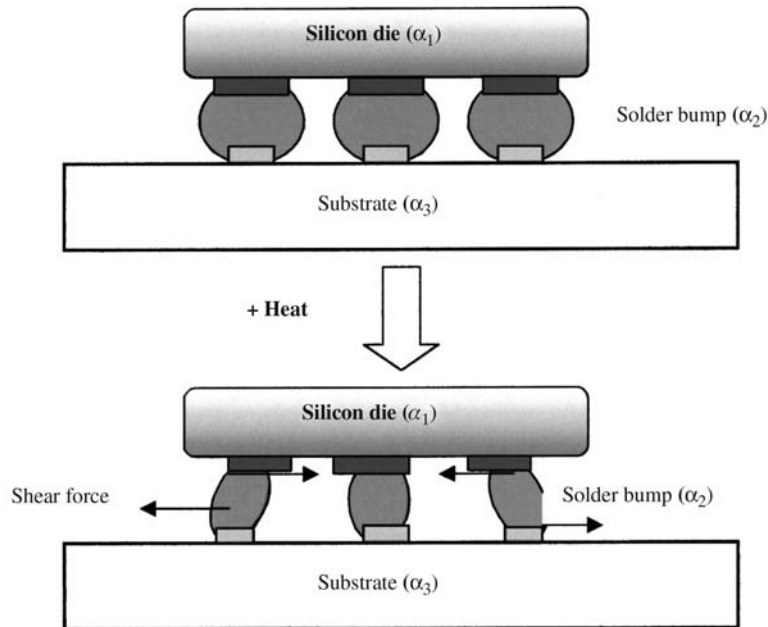
**FIGURE 1.3**

One thermal cycle profile.

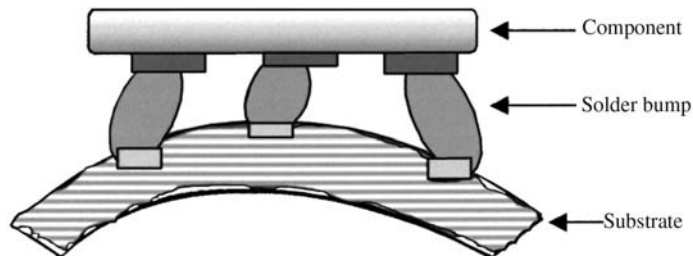
the component will extend in a dissimilar amount from the PCB or the chip. Moreover, at low temperature, the shrinkage is not the same for both parts. Consequently, this will lead to cyclic deformation in the solder joints of the assembly. Therefore during thermal cycling, the solder joints will produce periodic stress and strain effects, the part where stress concentration occurs produces plasticity and creep crack initiation, crack propagation, so that the whole joint completely breaks, causing electronic equipment failure or malfunction. Failure due to thermal cycling loading is considered one of the most common failures in the electronics industry [3].

The type and magnitude of strains in solder joints under conditions of thermomechanical fatigue are often quite complex. For surface mount applications, the strain is nominally in shear, as shown in Fig. 1.4. However, tensile and mixed-mode strains can occur due to bending of the IC component or board, as shown in Fig. 1.5.

The combination of strain and temperature during thermomechanical fatigue has a large effect on the microstructure and microstructural evolution of solder joints [5,6]. Strain concentration enhances diffusion, leading to microstructural coarsening at elevated temperatures [7]. It has been observed that typically only a

**FIGURE 1.4**

Deformation of solder joint subjected to shear strain during thermal cycling due to coefficient of thermal expansion mismatch [4].

**FIGURE 1.5**

Deformation of solder joint subjected to tensile due to printed circuit board flexing [4].

fraction of the solder joint cross-section actually participates in cyclic deformation because strain distribution inside solder joints is seldom uniform. Deformation of the most highly strained areas of solder joints leads to localized deformation. The recrystallization or grain coarsening takes place first in the regions where the microstructure is the most heavily deformed plastically and then gradually

expands. Failure eventually occurs due to cracks that form in the coarsened regions of a joint. The thermal anisotropy of the recrystallized grains enhances the nucleation of microcracks along their boundaries [8]. The failure mechanism under thermal cycling has been widely studied by many researchers [9–11].

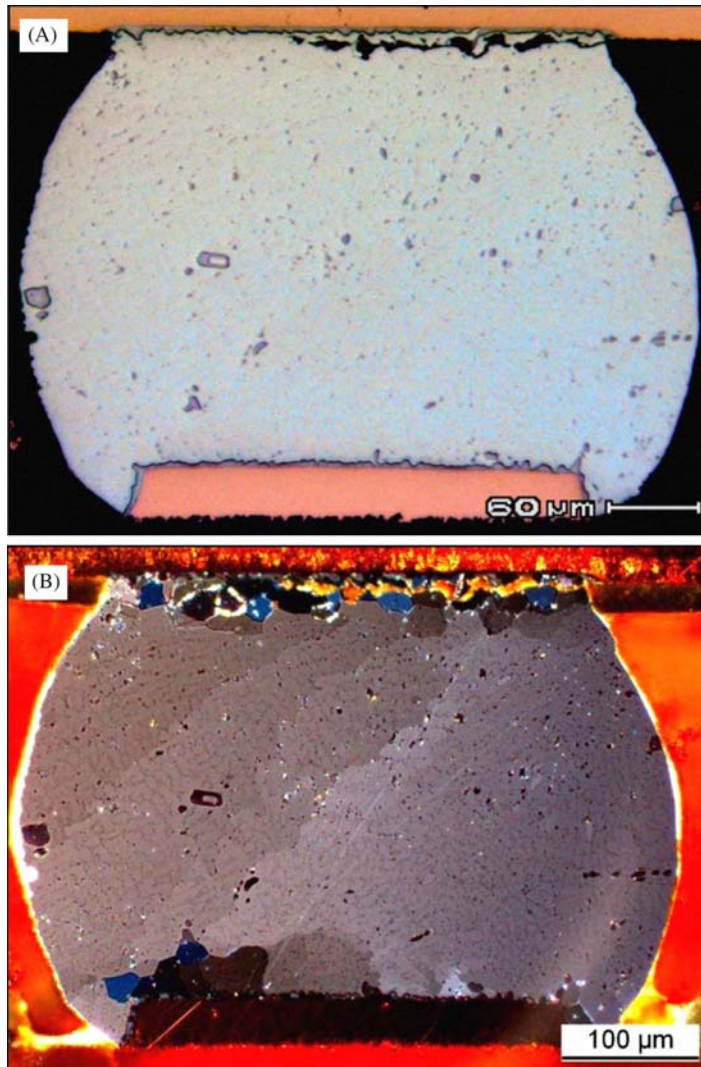


FIGURE 1.6

Thermal cycling failure: (A) the bright light micrograph shows the crack path of a failed solder interconnection during thermal cycling and (B) a cross-polarized light image of the same interconnection shows the change of microstructure as caused by the cyclic deformation [13].

It was observed that cracks always take place inside the matrix of solder along or close to intermetallic layers closely parallel to the direction of imposed shear strain, as shown in Fig. 1.6. The propagation path of the crack shown in Fig. 1.6A is enclosed entirely within the recrystallized region of the interconnection shown in Fig. 1.6B. The propagation of cracks, and therefore the reliability of interconnection, relies on the properties of the solder matrix. The solder alloy with low strength facilitates plastic deformation of the solder alloy by external stress of solder joint and cracks are generated and grows more easily within the solder and shows poor fatigue resistance [12].

1.3 Shock and vibration

The second common loading exerted on electronics is drop, shock, or impact loading. In such harsh conditions, high strains are produced in solder interconnects which cause the solder materials to deform plastically. According to regulations of the Joint Electron Device Engineering Council (JEDEC), the best way to assess the damage of electronic devices due to impact loading is by the use of drop test experiment, as shown in Fig. 1.7. This experiment is performed by dropping the test specimen freely from a certain height to achieve certain acceleration, usually measured in gravity accelerations (g 's), level. Each drop is counted as one cycle. These drops produce high solder strains at low loading frequencies.

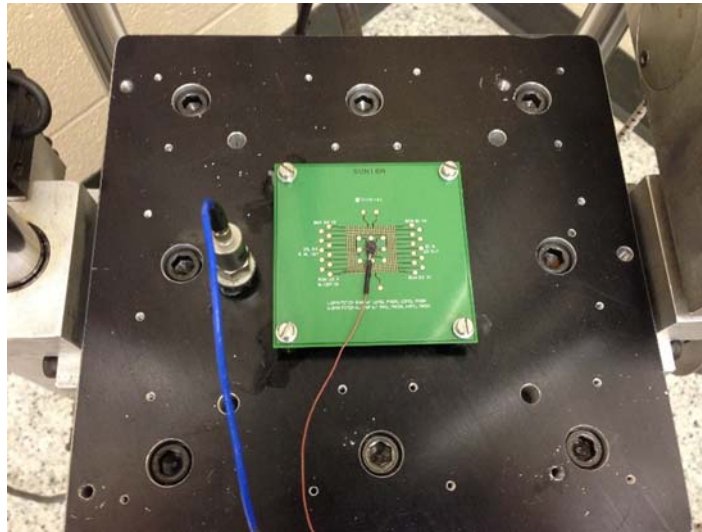


FIGURE 1.7

Drop test experiment: an assembly (face down) mounted to the drop table with two reference accelerometers are shown.

Mechanical vibrations are usually applied on electronic devices during product shipping and service life. During such loading conditions, low solder strains at high frequencies are produced in solder balls which assumingly limit solder material deformations to elastic region. Acceleration vibration reliability tests are widely used in the reliability assessment of electronic packages. In these tests, low acceleration (g) levels at high fixed frequencies are applied. A typical vibration reliability experiment is shown in Fig. 1.8.

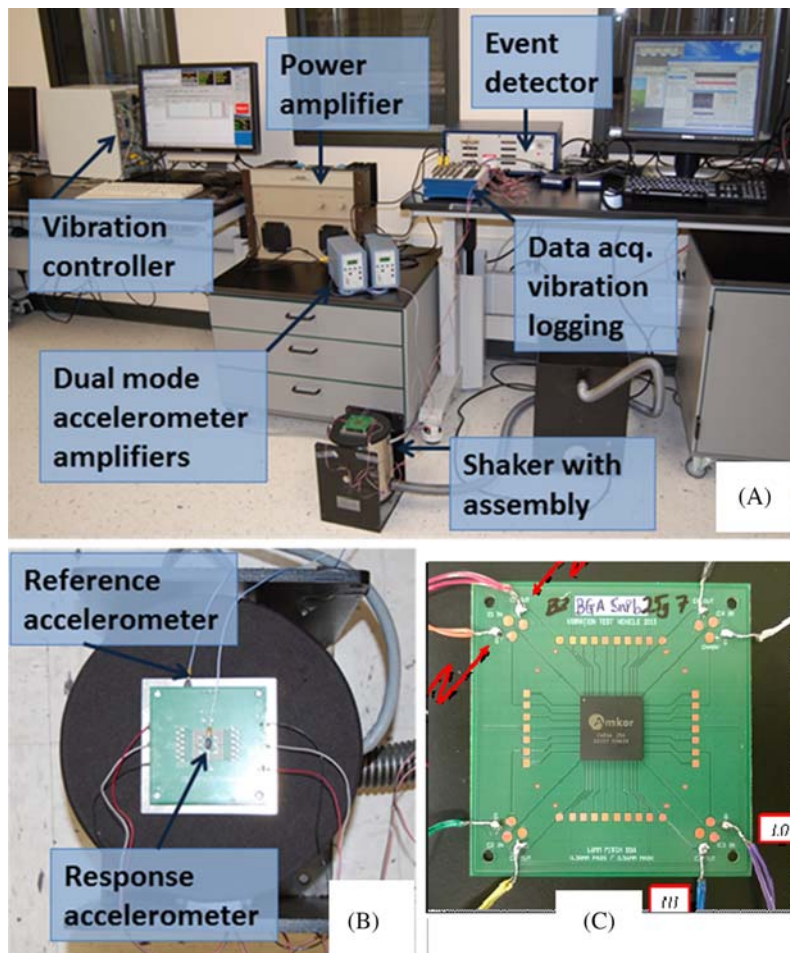
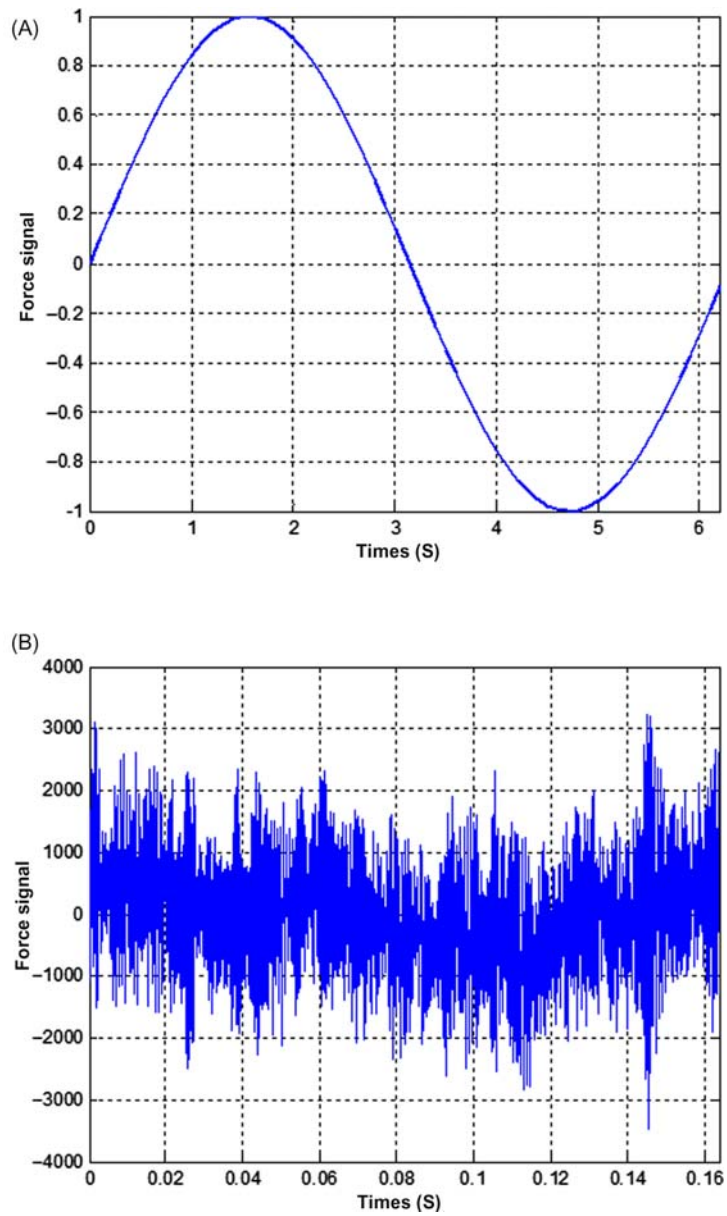


FIGURE 1.8

Photographs of a vibration test setup. (A) Physical test setup, (B) assembly mounted component side down to standoffs on shaker head with accelerometers mounted to the shaker head and circuit board center, and (C) component side of assembly.

**FIGURE 1.9**

(A) Harmonic (sinusoidal) signal and (B) random vibration signal.

Vibrations can be categorized into two types: harmonic and random. In harmonic vibrations, the loading is cyclic in nature. Specifically, the cycle time is well-defined and the loading signal is deterministic at any time instance. One common harmonic vibration profile is the sinusoidal signal, as shown in Fig. 1.9A. However, in random vibrations the loading cycles cannot be easily counted and the force signal is nondeterministic, as shown in Fig. 1.9B. Random vibrations are more common in real-life applications.

The primary driver of solder joint failure under vibration and drop is the mechanical bending of the PCB [14]. The mechanism causing the failure of interconnects under PCB bending is shown in Fig. 1.10A. The PCB bending leads to differential flexing between the PCB and IC component which must be accommodated by deformation of the interconnects. The deformation mechanics of a single interconnect is illustrated in Fig. 1.10B. The interconnect experiences (1) axial deformation associated with axial force, F_a , (2) shear deformation associated with shear force, F_s , (3) moment, M_f that is required to balance the shear couple, and (4) moment, M_ϕ due to relative rotation between the IC component and PCB.

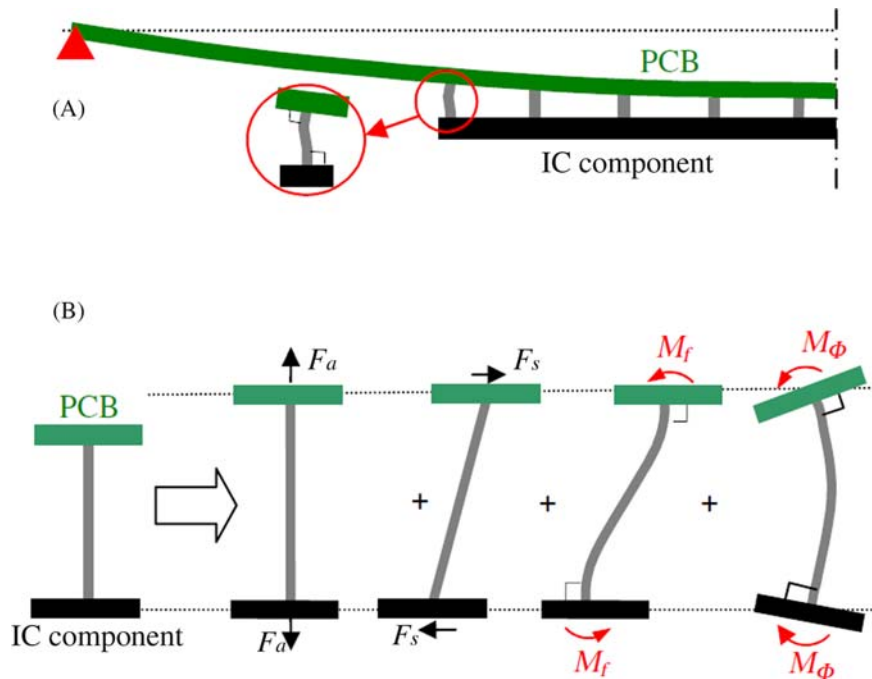
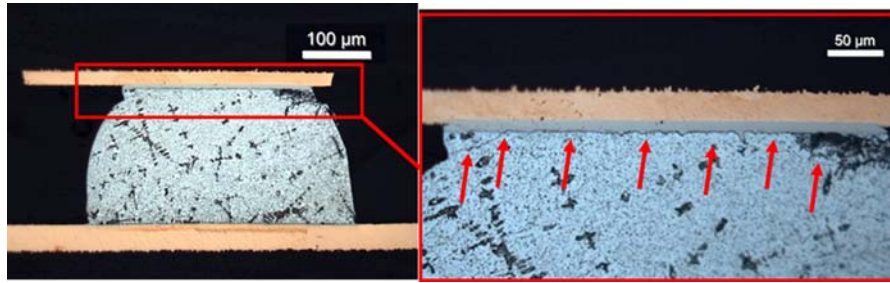
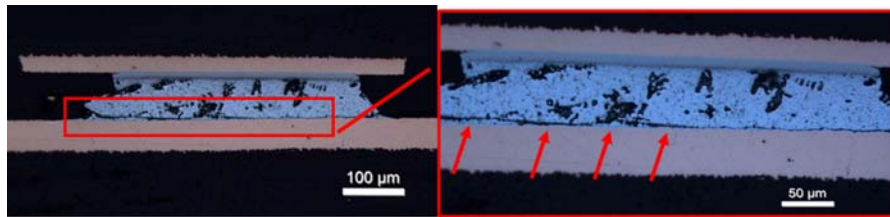


FIGURE 1.10

(A) Deformation of interconnect due to differential deflection between the PCB and IC component and (B) deformation mechanics of the interconnect [14]. *IC*, Integrated circuit; *PCB*, printed circuit board.

**FIGURE 1.11**

Drop and vibration failure (interfacial crack along solder joint at the integrated circuit component side) in a ball grid array.

**FIGURE 1.12**

Drop and vibration failure (interfacial crack along solder joint at the printed circuit board side) in a land grid array.

The failure mode during drop impact loading is manifested in interfacial cracking along the solder joint either on the package or board side [15–17], as shown in Figs. 1.11 and 1.12. In either case, shock failure is characterized by a lack of solder deformation and an absence of solder bulk cracking. This is due to the strain-rate sensitivity of metallic materials. Metallic materials including solders typically become stronger with increasing strain rates. Thus the robustness of a solder joint is influenced by a complex combination of bulk solder and intermetallic properties [18]. Ductile failures through bulk solder typically progress slowly, but crack through brittle intermetallic progress much faster [19].

1.4 Failure detection methods in electronics industry

One of the important challenges in the experimental studies and failure analysis of electronic products, is to determine when the crack occurs in a solder interconnect. A common nondestructive solder failure detection method is to monitor the electrical resistance during a reliability test. This method basically relies on the assumption that an electrical discontinuity will occur if there is a crack or multiple

cracks that cause complete fracture through the cross section of the solder joint. In general, this method involves the use of event detectors and data loggers for the in situ measurements of a solder joint electrical resistance during a test. For this approach, failure may be identified based on the sudden increase in the electrical resistance relative to an initial value, resistance threshold, or electrical discontinuities.

Electronics industry is currently adopting four common standards to specify solder interconnect failure criteria of surface mount technology solder attachments during qualification and reliability tests. These standards are:

- IPC-SM-785 published in November 1992 [20],
- IPC-9701 published in January 2002 [21],
- JESD22-B111 published in July 2003 [22], and
- IPC/JEDEC-9702 published in June 2006 [23].

In IPC-SM-785 standard, the failure criteria are defined based on three key parameters of a solder joint failure in thermal cycling reliability test: the electrical resistance increases magnitude due to an electrical discontinuity, the duration of electrical change, and the frequency of this electrical event occurrence. In this standard, there are two common specified resistance thresholds: 300 Ohms or greater and 1000 Ohms or greater. It is important to mention here that both threshold values are normally much higher than the initial resistance values of the daisy-chained IC package. False failure indications due to electrical noise can be a problem for threshold values lower than 1000 Ohms. Therefore the use of the 1000-Ohms approach is most commonly recommended. As mentioned previously, the IPC-SM-785 states that the solder failure results in electrical discontinuities that are defined as resistance increase of short durations. Specifically, it defines the solder joint failure should be associated with a resistance transient of 1 μ s or longer duration.

The third key parameter of solder failure criteria in IPC-SM-785 is the frequency of electrical discontinuity event occurrence to distinguish between interconnect failure from electrical noise. According to this standard, the first electrical interruption should be confirmed with nine additional interruption events with additional 10% of the cyclic life. For example, if the time of the first failure event is 3000 cycles, then there must be nine additional electrical events between 3000 and 3300 cycles in order to consider this as a solder interconnect failure.

For failure detection, the IPC-SM-785 standard generally recommends the use of continuous resistance monitoring of the daisy-chain connectivity loops. Also, the standard emphasizes that the polling time (the maximum amount of time has elapsed before a monitor, i.e., event detector, detects a change in status for one particular parameter) should be 2 seconds or shorter. The shorter polling time is the better in having more recent detected failure data. In addition, the IPC-SM-785 does not particularly identify the use of an event detector. However, it states that the use of a multiplexer and data logger only does not permit the continuous surveillance which will result in solder failure detection that is usually later than when it actually occurs.

The second widely used standard, the IPC-9701, defines the failure criteria based on the chosen measurement technique in accelerated thermal cycling reliability test. Similar to IPC-SM-785, the IPC-9701 recommends the use of an event detector and/or data logger. However, the event detector approach is preferred. If the event detector approach was used, the failure is defined very similar to that in the IPC-SM-785. Specifically, the failure here is designated as the first increase in the electrical resistance to a 1000 Ohms or higher for a 1 μ s or longer duration along with the confirmation of nine or more additional events within 10% of the failure cycles. For the data logger approach, the solder failure is set to relative to the initial resistance value of the IC package, instead of the use of a preset threshold resistance value. Specifically, the solder failure is characterized as an increase in the electrical resistance by 20% or more of the initial resistance value for six or more consecutive electrical events. In this standard, there is no information on the scan interval (polling time) or how the 20% criterion was elected.

The JESD22-B111 failure criteria are widely used in the failure definition of solder interconnects in board-level drop tests. As in all previously described standards, the JESD22-B111 uses the in situ continuous electrical surveillance for failure detection in each drop. This standard recommends that the electrical discontinuity is to be detected by an event detector or by a high-speed data acquisition system. If the latter is adopted, the measurement device sampling rate has to be 50,000 samples per second (50 kHz) or greater. In this case, the solder joint failure is detected if the resistance exceeds 100 Ohms or it increased by 20% above the initial resistance if the initial value is greater than 85 Ohms. Also, this electrical event should be followed by three more events during five consecutive drops. If the event detector approach is considered, the failure is defined as the first resistance rise for greater than 1000 Ohms. As in the high-speed acquisition system approach, this electrical event has to be confirmed with three additional events within five consecutive drops.

For the failure identification in bending reliability tests, the IPC/JEDEC-9702 standard is widely adopted in electronics industry. This standard defines the failure as a 20% increase in the initial resistance of the daisy-chained IC package. Generally, IPC/JEDEC-9702 standard grants some modifications to this limit based on the capabilities of the used test tools and on the daisy-chain engineering design. Also, it is important to mention that this failure criterion is commonly used in the identification of solder failures in the accelerated board-level vibration reliability testing.

1.5 Conclusion

In this chapter, the three main parts of the electronic packages PCB, IC, and solder interconnects are presented. In real-life, three cyclic in nature loading

conditions are extensively prone to electronic devices: thermal cycling, shock and vibration loadings. In thermal cycling, the main driver of solder joints failure is due to the mismatch in CTE between solder alloy and PCB and/or IC component. In such type of loading, tensile and mixed-mode strains can occur in the solder body due to bending of the IC component or board. In shock and vibrations, the differential bending between the component and the PCB which results in axial as well as shear deformations of the solder interconnects is the main driver of electronic failure in such harsh mechanical loading atmospheres. This chapter also provided several solder failures due to thermal, shock and vibration loadings. Finally, this chapter presented the most common solder interconnects failure detection approaches for each reliability test.

1.6 Recommendations

In electronic packaging, there are some design guidelines used to prevent solder interconnects failures. The first and important design factor is the oriented circuit board stiffness. Generally, the use of stiffer board could significantly improve the fatigue life of solder joints. Stiffer PCB structure could be achieved by the choice of thicker board design. In addition, smaller PCB size would also result in stiffer design. Moreover, the selection of stiffer laminate (PCB laminate with high modulus of elasticity value) is also a viable design option. Furthermore, the stiffer PCB structure could be achieved by the attachment of some stiffeners, such as heat sinks.

Another design factor that could have a significant effect on the prevention of solder interconnect failure is the rigidity of the IC component. Normally, the rigid one is the better. Rigid IC package could be accomplished by the selection of smaller and thicker IC package. In addition, the engineered choice of the IC package location of the circuit board might also have a significant effect on solder joint failures. For example, avoiding the placement of the component at the expected high PCB deformation regions will result in lower solder stresses and strains. Hence, it is less likely to allow solder fatigue failures especially in bending loading environments.

The solder geometric design plays an important role in solder failure prevention. In general, shorter solder joints, for example, land grid array solders have longer fatigue life in thermal cycling loading conditions. However, taller interconnects, for example, BGA solders, do better in bending and vibration loading environments. Slimmer (cylinder-like) solder design is also preferred in bending. In general, stiffer solder design is recommended in engineering applications with thermal cycling primary loading conditions. However, compliant solder structures are more likable in impact loading environments.

References

- [1] M.A. Gharaibeh, Finite Element Modeling, Characterization and Design of Electronic Packages Under Vibration, State University of New York at Binghamton, 2015.
- [2] B. Mirman, Tools for stress analysis of microelectronic structures, *J. Electron. Packag.* 122 (3) (2000) 280–282.
- [3] L. Fang, J. Bo, T. Wei, Review of board-level solder joint reliability under environmental stress, *Prognostics and System Health Management Conference (PHM-Chengdu)*, 2016, IEEE, 2016, pp. 1–6.
- [4] M. Abtew, G. Selvaduray, Lead-free solders in microelectronics, *Mater. Sci. Eng.: R: Rep.* 27 (5–6) (2000) 95–141.
- [5] D.R. Frear, No. SAND-91-1461C; CONF-911003-8 Microstructural Evolution During the Thermomechanical Fatigue of Solder Joints, Sandia National Labs, Albuquerque, NM, 1991.
- [6] D. Frear, D. Grivas, J.W. Morris, Parameters affecting thermal fatigue behavior of 60Sn-40Pb solder joints, *J. Electron. Mater.* 18 (6) (1989) 671–680.
- [7] K.C.R. Abell, Y.-L. Shen, Deformation induced phase rearrangement in near eutectic tin–lead alloy, *Acta Mater.* 50 (12) (2002) 3193–3204.
- [8] L. Garner, S. Sane, D. Suh, T. Byrne, A. Dani, T. Martin, et al., Finding solutions to the challenges in package interconnect reliability, *Intel Technol. J.* 9 (4) (2005).
- [9] T. Hirano, K. Fukuda, K. Ito, T. Kiga, Y. Taniguchi, Reliability of lead free solder joint by using chip size package, in: *Electronics and the Environment*, 2001. Proceedings of the 2001 IEEE International Symposium on, IEEE, 2001, pp. 285–289.
- [10] S.-W. Ricky Lee, B.H. Wai Lui, Y.H. Kong, B. Baylon, T. Leung, P. Umali, et al., Assessment of board level solder joint reliability for PBGA assemblies with lead-free solders, *Solder. Surf. Mount Technol.* 14 (3) (2002) 46–50.
- [11] J.E. Sohn, Are lead-free solder joints reliable?-Judge for yourself. A NEMI team found that lead-free manufacturing can be implemented without degrading solder joint reliability, *Circuits Assembly* 13 (6) (2002) 32–34.
- [12] F.X. Che, E.C. Poh, W.H. Zhu, B.S. Xiong, Ag content effect on mechanical properties of Sn-xAg-0.5 Cu solders, in: *Electronics Packaging Technology Conference*, 2007. EPTC 2007. 9th, IEEE, 2007, pp. 713–718.
- [13] T.T. Mattila, M. Mueller, M. Paulasto-Kröckel, K.-J. Wolter, Failure mechanism of solder interconnections under thermal cycling conditions, in: *Electronic System-Integration Technology Conference (ESTC)*, 2010 3rd, IEEE, 2010, pp. 1–8.
- [14] E.-H. Wong, S.K.W. Seah, V.P.W. Shim, A review of board level solder joints for mobile applications, *Microelectron. Reliab.* 48 (11–12) (2008) 1747–1758.
- [15] D. Suh, D.W. Kim, P. Liu, H. Kim, J.A. Weninger, C.M. Kumar, et al., Effects of Ag content on fracture resistance of Sn–Ag–Cu lead-free solders under high-strain rate conditions, *Mater. Sci. Eng. A460* (2007) 595–603.
- [16] A. Syed, T.S. Kim, Y.M. Cho, C.W. Kim, M. Yoo, Alloying effect of Ni, Co, and Sb in SAC solder for improved drop performance of chip scale packages with Cu OSP pad finish, in: *Electronics Packaging Technology Conference*, 2006. EPTC'06. 8th, IEEE, 2006, pp. 404–411.

- [17] A. Syed, J. Scanlan, S.W. Cha, W.J. Kang, E.S. Sohn, T.S. Kim, C.G. Ryu, Impact of package design and materials on reliability for temperature cycling, bend, and drop loading conditions, in: Electronic Components and Technology Conference, 2008. ECTC 2008. 58th, IEEE, 2008, pp. 1453–1461.
- [18] J. Graf Reliability and Quality Aspects of FBGA Solder Joint, *Forschung & Technology Revue*, 2008, pp. 2224–2234.
- [19] D.R. Frear, L.N. Ramanathan, J-W. Jang, N.L. Owens, Emerging reliability challenges in electronic packaging, in: Reliability Physics Symposium, 2008. IRPS 2008. IEEE International, IEEE, 2008, pp. 450–454.
- [20] Guidelines for Accelerated Reliability Testing of Surface Mount Solder Attachments, IPC-SM-785, The Institute for Interconnecting and Packaging Electronic Circuits, Northbrook, IL, 1992.
- [21] Performance Test Methods and Qualification Requirements for Surface Mount Solder Attachments, IPC-9701, The Institute for Interconnecting and Packaging Electronic Circuits, 2002.
- [22] Monotonic Bend Characterization of Board-Level Interconnects, IPC/JEDEC-9702, IPC/JEDEC Association Connecting Electronics Industries, 2004.
- [23] Board Level Drop Test Method of Components for Handheld Electronic Products, JESD22-B111, JEDEC Solid State Technology Association, 2003.

This page intentionally left blank

Electron beam radiation and its impacts to failure analysis in semiconductor industry

Binghai Liu^{1,2}, Xiaoming Li¹, Younan Hua¹, Nan Cho¹, Zhili Dong³,
Yuzhe Zhao^{1,2}, Kenny Ong² and Zhiqiang Mo²

¹Wintech Nano-Technology Services Pte Ltd, Singapore

²Product, Test and Failure Analysis, Department of Technology Development,
GLOBALFOUNDRIES Singapore Pte Ltd, Singapore

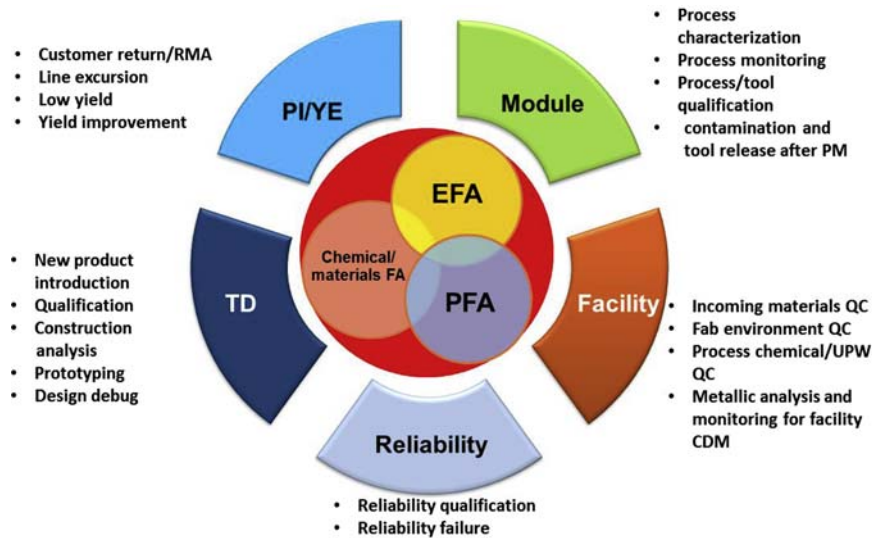
³School of Materials Science & Engineering, College of Engineering, Nanyang Technological
University, Singapore

2.1 Introduction

For semiconductor manufacturing, failure analysis (FA) is always an important support function and plays an indispensable role in various activities involved in daily fab production. The task of FA is to identify the failure mechanism of semiconductor devices, providing valuable information and guidance to the issues related to fab production, such as low yield and yield improvement. In general, FA is typically classified into three major functional groups: electrical failure analysis (EFA), physical failure analysis (PFA), and chemical FA.

In the FA processes, PFA and EFA are well integrated with each other. While EFA is responsible for fault isolation and the characterization of failure mode, the task of PFA is to identify physical failure signatures (e.g., phase, composition, and microstructures of semiconductor materials and structures) that were intimately correlated with the failure modes identified by electrical characterization. By correlating these physical failure signatures with wafer fabrication processes, PFA is responsible for the final root cause identification and failure mechanism understanding of integrated circuit (IC) devices, addressing the issues related to IC design, manufacturing processes, and field application of IC devices.

Besides the role in fault isolation and defect analysis, PFA has become increasingly important in process development, optimization, and qualification in the semiconductor industry. Hence, in modern semiconductor industry, PFA marks its presence in different cycles of IC products, from initial product tape out, to process development and qualification, to mass manufacturing, and to final

**FIGURE 2.1**

The correlation of failure analysis with different stages of integrated circuit device production. *EFA*, Electrical failure analysis; *PFA*, physical failure analysis; *PI*, process integration; *QC*, quality control; *RMA*, return material analysis; *TD*, technology development; *YE*, yield enhancement.

field application of products. Together with EFA and chemical FA, PFA activities are involved in different stages of IC device production, as shown in Fig. 2.1.

Over the past decades, the semiconductor industry has been undergoing fast development, continuously moving toward the fabrication of smaller and more complex microelectronic devices with better performance. The continuing shrinkage in device dimension and the application of new semiconductor materials lead to great challenges for not only electrical FA but also physical FA. To characterize nanosized defects and structures in IC devices, physical analysis with a high spatial resolution is generally required. Therefore electron beam–based FA techniques, such as transmission electron microscope (TEM) and scanning electron microscope (SEM), are playing more and more important roles in the PFA due to their high spatial resolution such as subangstrom level in TEM. In addition, modern electron microscopes can be equipped with a wide variety of analytical attachments, such as energy-dispersive X-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS). Thus they are able to offer comprehensive solutions to the PFA of semiconductor devices.

However, for physical analysis by TEM and SEM, electron beam radiation damage–induced structure modification, material diffusion, and phase transformation brings issues for those electron beam–sensitive materials used in semiconductor devices, such as low-*k* (LK) and ultralow-*k* (ULK) dielectrics, nitride,

and even single-crystal Si. As it is well known, the electron beam radiation has been the subject of tremendous research efforts, especially in the field of biology imaging by electron microscopy since 1950s [1–3]. In the field of materials science and semiconductor FA, the electron beam radiation and its associated impacts are also the unavoidable topics although not so much efforts have been devoted as for biological science. For PFA of those electron beam–sensitive semiconductor structures or defects, it is crucial to understand the underlying radiation damage mechanism and take corresponding technical measures in order to prevent or minimize radiation-induced structure damage and phase modification.

In general, the radiation damage refers to any changes in physical structure and chemical composition which occur as a result of exposure to electron beam. Fig. 2.2 shows different types of electron beam radiation damage. As it is well recognized, the electron beam radiation damage involves complex physical and chemical processes during the interaction between high energetic electron beam and materials under analysis [1–7]. As shown in Fig. 2.2, electron beam radiation damage can be generated by both the elastic electron scattering and inelastic electron scattering [3–7]. The knock-on damage and electron bombardment–induced sputtering are attributed to elastic scattering events with high-energy electrons. The knock-on damage refers to the direct atom displacement by high-energy electrons due to large momentum transferring, normally occurring only during TEM

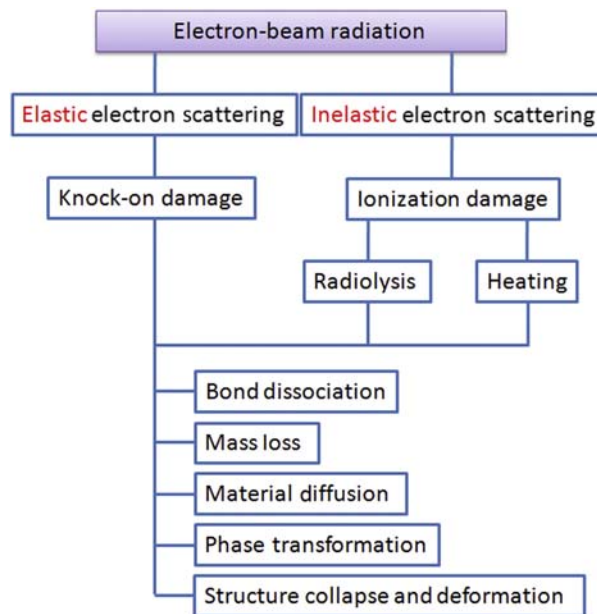


FIGURE 2.2

The classification of electron beam radiation damage.

analysis owing to the high acceleration voltages of up to several hundreds of kilovolts. The knock-on damage is the primary cause of radiation damage in metals and some other inorganic materials. It can also happen in the organic compounds, but is not considered as the major contributor to radiation damage of organics because of the faster and significant ionization damage before such knock-on damage could occur [4]. The radiation-induced ionization effects might be the most severe cause of the radiation damage to various materials [3]. The ionization damage can lead to diverse physical and chemical changes in materials, depending on the composition and chemical bonding nature of the materials. It may occur in both organic and inorganic compounds, resulting in chemical bond dissociation, atomic displacement, loss of crystallinity, mass loss (sublimation of materials), enhanced material diffusion, and phase transformation [3–15]. The electron beam ionization can also result in specimen heating. Different models were proposed to estimate the rise in local temperature inside the specimen during SEM and TEM analyses [5,16–19]. It is generally accepted that the temperature increment by electron beam radiation is not significant except the beam current is high enough up to μA range. During normal SEM and TEM analyses, in general beam current as measured is in the nA range. Therefore radiation-induced heat damage is not significant especially for those materials with good thermal conductivity, like metallic materials.

As far as semiconductor PFA is concerned, the above-mentioned electron beam radiation–induced damage all could occur during SEM, dual-beam focus ion beam (FIB) with electron beam imaging system and TEM FA of semiconductor devices. In this chapter, we will discuss the impacts of electron beam radiation on some typical beam-sensitive materials in semiconductor devices. Based on case studies, detailed analysis was performed in order to understand the process related to electron beam radiation–induced changes in microstructure, phase and composition of these beam-sensitive semiconductor materials, and how these changes would affect FA by SEM, FIB, and TEM. Then corresponding technical solutions will be proposed in order to avoid or to minimize the electron beam radiation–induced damage in these beam-sensitive semiconductor materials. The electron beam–sensitive semiconductor materials to be discussed in this chapter include LK dielectric, ULK dielectric, nitride, and its associated semiconductor structures, and CoFeB materials in magnetic random access memory (MRAM) devices.

2.2 Impact of electron beam radiation damage during SEM failure analysis

2.2.1 SEM physical FA and low- k /ultralow- k dielectrics

As an important PFA tool, SEM is widely used in daily FA to support fab production and technology development in the semiconductor industry. With the

continuous advances in SEM techniques, nowadays modern SEMs can offer sub-nanometer spatial resolution, and thus have been playing active roles in various FA activities. In general, the application of SEM in semiconductor FA can be summarized as follows:

1. Profile measurement and analysis of semiconductor structures and process layers, which is mainly for process development, optimization and qualification and so on.
2. Defect characterization, usually with the combination of FIB cross-sectional milling and elemental analysis by EDS, for the issues related to IC device failure, and process drift or environmental contamination-induced defects and so on.
3. Electron beam–based fault isolation, such as passive voltage contrast, electron beam–induced contrast, electron beam–induced absorption contrast. These fault isolation analyses are normally performed in the SEM platforms and are widely used in the wafer-level and die-level failure analyses of semiconductor IC devices.

The SEM physical analysis, as discussed in this chapter, essentially refers to the first two applications in the above list. Compared with other physical failure techniques such as TEM, the advantages of SEM are (1) large depth of focus and simple tool operation procedure, (2) fast and simple sample preparation, (3) high throughput with short job cycle time, and (4) cost-effective. The limitation of the SEM technique lies in its limited imaging contrast modes and lower spatial resolution when compared with the TEM technique. As it is well known, the primary signal carriers for SEM imaging are secondary electrons (SE) and backscattered electrons (BSE), as shown in Fig. 2.3A. SE are those electrons with the energy less than 50 eV, while BSE have the energy close to the energy of the primary incident electrons. SE signals have relatively shallow escape depth of around several tens of nanometers, while BSE signals have much larger escape depth of up to several micrometers or tens of micrometers depending on the type of materials, as illustrated in Fig. 2.3B. Therefore the high-resolution SEM imaging has to rely on the SE signals, which offers topographic contrast in SEM analysis. For BSE signals, their large escape depth leads to large signal delocalization during SEM imaging, and thus BSE imaging has poor spatial resolution, but it can offer mass contrast.

During PFA by SEM, we always need to take note of both spatial resolution and signal-to-noise ratio. As it is well known, the spatial resolution of SEM is determined by the probe size. Increasing acceleration voltage of electron beam can help to improve SEM imaging resolution by not only shortening the wavelength of electron waves, but also reducing the chromatic aberration of the SEM electron optical system. On the other hand, in order to obtain sufficient signal-to-noise ratio in SEM images, it is necessary to acquire images with a large beam current. However, higher acceleration voltage and higher electron beam current will bring in the issues of electron beam radiation damage to those beam-sensitive

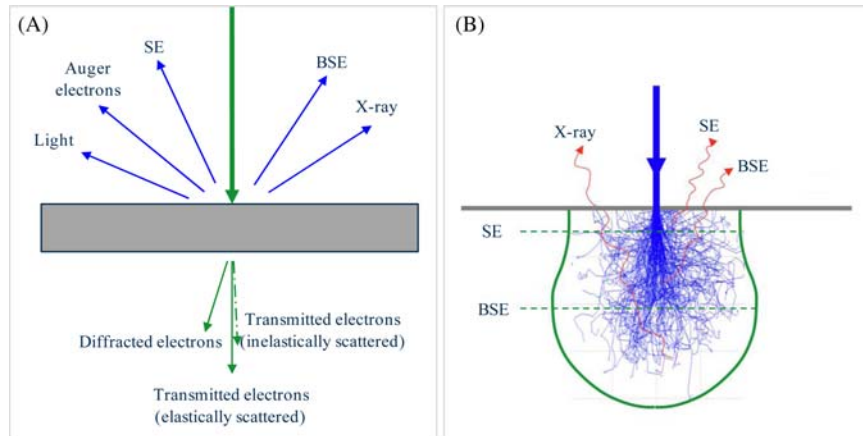
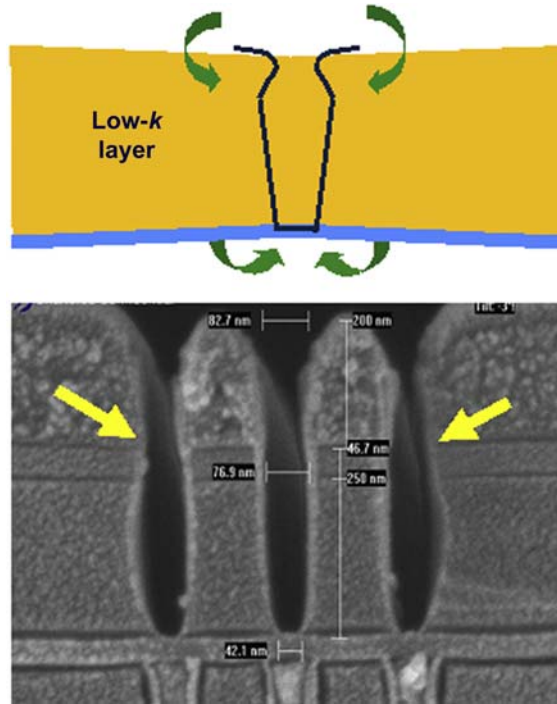
**FIGURE 2.3**

Illustration of (A) the interaction between the electron beam and specimen; (B) the interaction volume of the electron beam in a thick specimen. *BSE*, Backscattered electrons; *SE*, secondary electrons.

materials. High acceleration voltage of the electron beam directly results in large electron interaction volume in SEM samples, while large beam current leads to more intensive inelastic scattering events in the samples, and thus more severe radiation-induced damage.

During the semiconductor FA by SEM, the most frequently encountered issue could be structural damage such as deformation of LK and ULK dielectric materials, as shown in Fig. 2.4. The specimen in Fig. 2.4 was cut by mechanical cleaving from a wafer after via the etch process. As shown in Fig. 2.4, the LK dielectric process layer suffered from severe structure deformation, appearing in bowl shape. Such SEM profile is definitely not the authentic one after via the etch process, and thus the associated measurements are no more accurate and useful for the fab process development or monitoring.

To solve the issue of the electron radiation damage to LK and ULK dielectrics, we need to understand the physical and chemical properties of this group of beam-sensitive materials. As it is well known, the use of LK and ULK materials to replace SiO_2 as intermetal dielectrics (IMD) is to reduce cross-talk and the resistance–capacitance delay of the global wiring in semiconductor devices. With the continuous device scaling, chip wiring (interconnect) is scaled as well, which leads to the performance degradation as both resistance and current density increase due to the smaller cross-sectional area of the scaled interconnect metal lines. An additional consequence of scaling is an increase in sidewall capacitance as conductors are placed in closer proximity to one another [20,21]. Therefore the introduction of LK dielectrics as IMD in between Cu interconnect metal lines provides a good material solution for the above-mentioned issues.

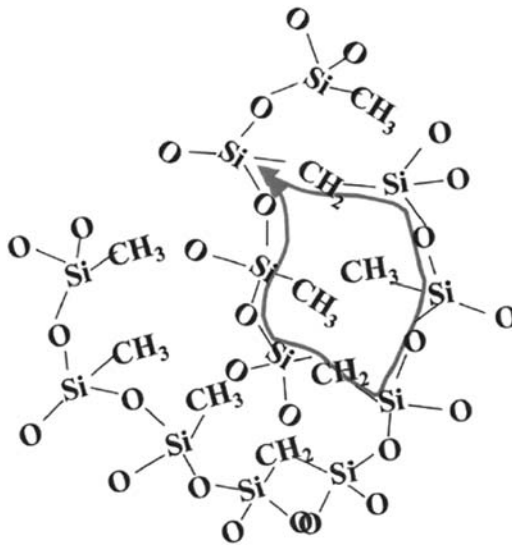
**FIGURE 2.4**

The electron beam radiation–induced structure deformation of low- k dielectrics during scanning electron microscope analysis.

Essentially, LK dielectric is a type of Si-based polymer materials. Fig. 2.5 shows a typical elementary unit of an LK material [22]. It consists of the networks of Si, O, C, and H elements, and thus is known by different names, such as SiCOH, carbon-doped oxide, or organosilicate glass. The dielectric constant of typical LK SiCOH materials is around 2.5–3.1, lower than ~ 4.0 of SiO_2 dielectric materials [23,24]. For the ULK SiCOH materials, porous structure was introduced into their elementary units by tuning the deposition chemistry and processes. The dielectric constant of the ULK SiCOH materials is typically lower than 2.5 [23,24]. Normally the LK and ULK dielectric materials have low thermal conductivity. The low thermal conductivity and high content of organic radicals in LK and ULK SiCOH materials render them to be very electron-beam sensitive, which will be discussed in the following sections.

2.2.2 Electron beam radiation damage to low- k and ultralow- k dielectric materials

As it is well recognized, most organic materials are extremely electron-beam sensitive showing severe structure and phase degradation under electron dose

**FIGURE 2.5**

Elementary unit of carbon-doped silica glass and schematic bonding structure with cross-linking [22].

© 2003 American Institute of Physics.

above $\sim 100 \text{ e/nm}^2$ [25]. For LK and ULK dielectrics, high-dose electron beam—induced radiation damage could be induced by either elastic knock-on damage or inelastic scattering—induced ionization damage or both during SEM FA.

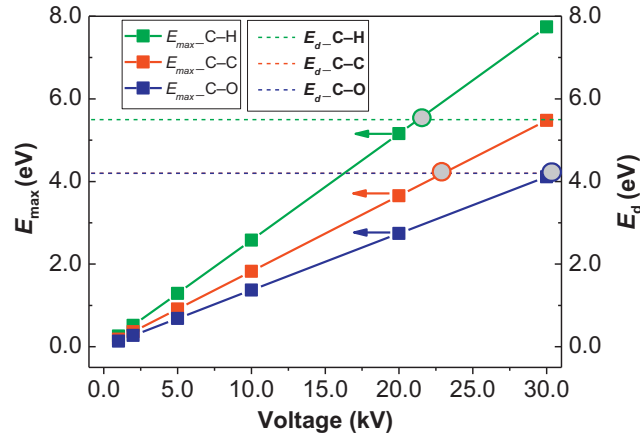
2.2.2.1 Electron beam—induced knock-on damage

Under the elastic collision model, the maximum knock-on energy transfer to an atom can be estimated by [26,27]:

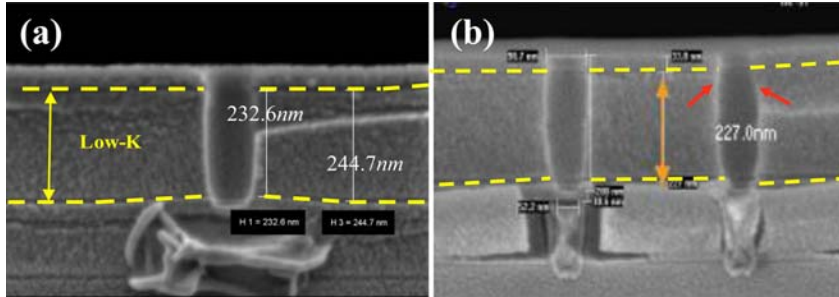
$$E_{\max} = \frac{4M_i M_a E_0}{(M_i + M_a)^2} \quad (2.1)$$

where $E_{\max}$ is the maximum energy transferred from electrons to a target atom, M_i is the mass of the incident electron, and M_a is the mass of the target atom in the material, and E_0 , the energy of the incident electrons. Based on Eq. (2.1), the calculated $E_{\max}$ of different organic radicals is plotted as shown in Fig. 2.6.

In the case of energetic elastic electron head-on collision on target atoms, when the maximum transferred energy $E_{\max}$ exceeds the threshold energy E_d of the target materials, the atomic displacement damage or knock-on damage occurs. In SiCOH materials, the C–H, C–C, and C–O are the organic radicals with the chemical bonding, with their E_d of 5.5, 4.2, and 4.2 eV, respectively [17]. Based on the calculation shown in Fig. 2.6, the threshold displacement voltages for

**FIGURE 2.6**

The dependent of E_{max} (the maximum energy transferred to target organic radicals) on the acceleration voltage of electron beam.

**FIGURE 2.7**

Cross-sectional scanning electron microscope micrographs of the via holes patterned in low- k carbon-doped oxide (SiCOH) intermetal dielectrics layers ($k = 2.75$, and the standard SiCOH layer thickness of 270 nm by inline measurement) taken with 1 nm Pt coating and probe current of 220 pA at (A) 1 kV and (B) 3 kV.

C–H, C–C, and C–O radicals are 21.4, 23.0, and 30.9 keV, respectively. If acceleration voltage used in SEM analysis is lower than 21 keV, the knock-on damage should not take place in SiCOH dielectrics. Therefore SEM analysis at low voltage is preferable for SiCOH materials.

Fig. 2.7A and B shows SEM micrographs of the cross section of via holes patterned in an LK SiCOH IMD layer at 3 and 1 kV, respectively. The cross-sectional samples were prepared by the mechanical cleave method without using FIB. Before SEM analysis, the samples were sputtering coated with an

anti-charging Pt layer of ~ 1 nm in thickness. The probe current used was 220 pA. As shown in Fig. 2.7, large shrinkage was observed in SiCOH IMD layer at both 1 and 3 kV, more than 40 nm at 3 kV. Such electron beam radiation—induced SiCOH deformation occurred very fast, within several seconds depending on the imaging conditions. In general, higher working voltage led to more severe and faster shrinkage in SiCOH layer. As shown in Fig. 2.7A, the radiation-induced SiCOH shrinkage resulted in not only the profile deformation of via holes, but also the delamination and cracks between different process layers. Since the working voltage of 1–3 kV was far below the calculated threshold displacement voltage, E_d , the results above indicated that the electron beam radiation damage to LK SiCOH was not due to elastic knock-on damage mechanism at such low-kV SEM imaging conditions.

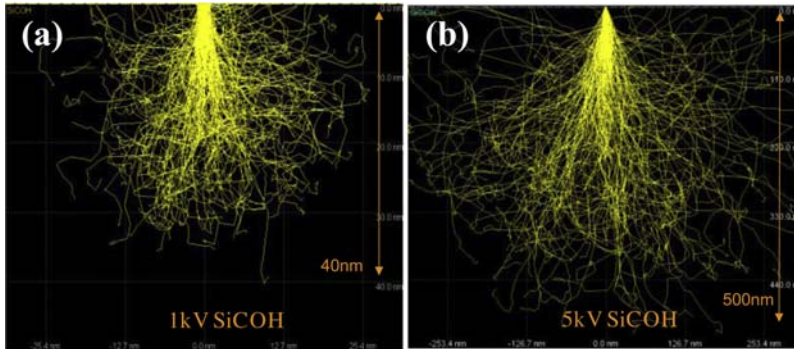
2.2.2.2 Electron beam—induced thermal damage

As discussed in Section 2.1, the inelastic scattering—induced ionization effect also involves significant energy transfer from the incident energetic electrons to the target atoms. Most of the energy ends up as heat within the specimen, leading to the local temperature increase in the beam illuminated area [5,17]. Such electron beam radiation—induced heat damage could occur in both SEM and TEM FA depending on the beam illumination condition and the properties of the materials. Based on the model by Reimer, the temperature increment under stationary probe illumination mode can be estimated by Eq. (2.2) [16,17]:

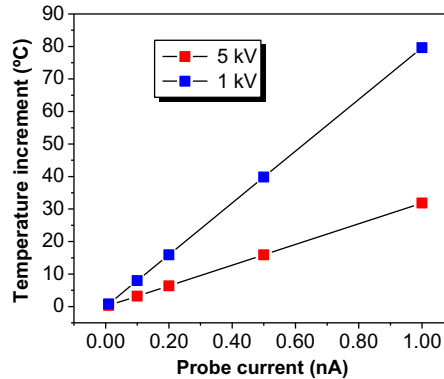
$$\Delta T = \frac{3V \cdot I}{2\pi k \cdot R} \quad (2.2)$$

where V is the acceleration voltage, I is the probe current, k is the thermal conductivity, and R is the electron range. According to the study by Delan et al. [28], thermal conductivity of LK materials is around 0.11 W/m·K for porous ULK, and 0.19 W/m·K for LK materials. Based on the Monte Carlo simulation, the electron ranges in SiCOH LK materials at 1 and 5 kV are around 40 and 500 nm, respectively, as shown in Fig. 2.8A and B. Using the average thermal conductivity of 0.15 W/m·K for SiCOH materials, the temperature increment inside LK SiCOH materials can be estimated by Eq. (2.2) and plotted out as shown in Fig. 2.9.

As shown in Fig. 2.9, the temperature rise induced by electron beam radiation is not significant when electron probe current is less than 1 nA. In terms of our FA work, typical probe current was usually in pico-ampere (pA) range for SEM analysis of SiCOH LK materials. As shown in Fig. 2.7A and B, the probe current was only 220 pA. Based on Eq. (2.2) and Fig. 2.9, the temperature increment at 220 pA was less than 20°C at both 1 and 3 kV, which was too low to induce any structure damage or deformation. Therefore the structure deformation and shrinkage in SiCOH materials should not be ascribed to electron beam radiation—induced heating effects unless the electron probe current is up to several tens of nano-ampere and above.

**FIGURE 2.8**

Electron interaction volume inside low- k carbon-doped oxide material at (A) 1 kV and (B) 5 kV (by Monte Carlo simulation program Casino V2.42 based on Rutherford scattering model).

**FIGURE 2.9**

The dependence of temperature rise in carbon-doped oxide materials on the current of electron probe during scanning electron microscope analysis.

In addition, as shown in Fig. 2.9, the radiation-induced heating effect is more obvious at a lower acceleration voltage. The reason may lie in the fact that radiation generated heat flow is dissipated across three dimensions (3D) in bulk SEM specimen. At the same level of probe current, higher acceleration voltage gives rise to larger interaction volume, and thus larger heat dissipation space for which local temperature increment under beam radiation is less significant. This is in contrary to what we observed during SEM FA of LK SiCOH materials. As shown in Fig. 2.7, at similar probe current of ~ 220 pA, SiCOH shrinkage was apparently larger and faster at 3 kV than at 1 kV. Higher working voltage always

brought faster and larger structure deformation of the SiCOH layer. The results provide further evidence that heating effect by electron beam radiation does not account for the radiation damage of SiCOH materials during SEM FA.

2.2.2.3 Electron beam—induced radiolysis damage

The radiolysis damage is one type of ionization damage as a result of inelastic electron scattering. The energy lost from the primary incident electrons is transferred to the atoms under beam illumination, by which single-electron transition or collective single-electron transition processes take place, namely, electron excitation from valence band to conduction band occurs [7]. For those materials with low density of electrons in conduction band like LK SiCOH materials, the lifetime of the minority carriers is long enough, for which before electron–hole combination in valence band, large amount of the excitation energy will be stored as potential energy inside materials. This leads to the chemical bond rupture and mass loss with the escape of light elements such as H, N, and O, the so-called radiolysis damage [7]. The radiolysis damage is reported to be most severe in organic materials such as biological samples containing covalent bonds and other type of weaker chemical bonds. The as-mentioned radiolysis damage well applies to LK SCOH materials which contains substantial amount of C–O, C–H, and C–C radicals with covalent bonds [29].

2.2.3 Control of electron beam radiation damage to low- k and ultralow- k dielectric materials

2.2.3.1 The effects of acceleration voltage

As mentioned in Section 2.2.2.1, knock-on damage does not happen to LK and ULK dielectrics under SEM FA due to low acceleration voltage. However, as shown in Fig. 2.7A and B, higher working voltage did induce larger and faster radiation damage to LK and ULK dielectrics. The reason behind may lie in the fact that higher acceleration voltage gives rise to larger interaction volume of electron beam, for which inelastic electron scattering events can occur in deeper volume of the materials. For instance, at 1 kV, the electron range is just around 40 nm into the sample surface, while it can be up 500 nm at 5 kV, as indicated in Fig. 2.8A and B. Giving the same probe current, the radiation damage at 5 kV will occur deeper into sample surface for 500 nm, while the radiation damage at 1 kV only affect 40 nm in depth, near surface region of the sample. Therefore the higher the acceleration voltage, the deeper the radiation-induced radiolysis damage inside the sample takes place, for which larger and faster structure deformation or shrinkage will appear for LK and ULK dielectrics. This is evidenced by more severe deformation of via holes as shown in Fig. 2.7B.

Modern SEMs now have good imaging capability with reasonable high resolution at a voltage lower than 1 kV. There is a good review of the issues related to low-voltage SEM imaging by Müllerová and Frank [30]. Essentially, these issues

are mainly related to the effects of chromatic aberration, and diffraction effects to imaging resolution, which can be expressed by Eqs. (2.3) and (2.4):

$$d_c = K_c C_c \frac{\Delta E}{E} \alpha \quad (2.3)$$

$$d_D = K_D \lambda \alpha^{-1} \quad (2.4)$$

where d_c and d_D are the disk of confusion (i.e., minimum probe size) due to chromatic aberration and diffraction limits, K_c and K_D , the numeric factors; C_c , the chromatic aberration constant; ΔE , the energy spread; E , the incident beam energy; λ , the wavelength of electron beam; and α , the angular aperture related to primary electron beam. As given in Eqs. (2.3) and (2.4), the imaging resolution decreases with decrease in acceleration voltage, that is, both d_c and d_D increase with voltage decrease.

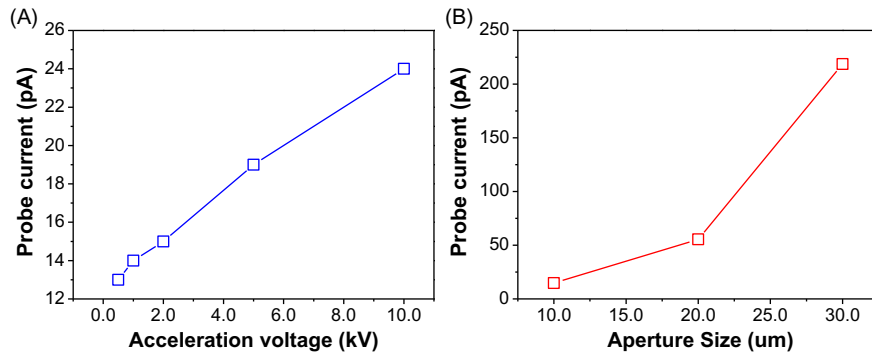
To overcome or decrease the impacts of chromatic aberration and diffraction limits, advanced SEM systems have different electron optics designs, such as electron beam booster system and the beam deceleration designs. Essentially all these designs try to maintain a relatively high acceleration voltage of electron beam inside the SEM column before the electron beam strikes on the specimen. Therefore for SEM FA associated with LK and ULK dielectrics, it is advised to use the lowest working voltage if the resolution at such low voltage can meet the analysis requirements.

2.2.3.2 The effects of probe current

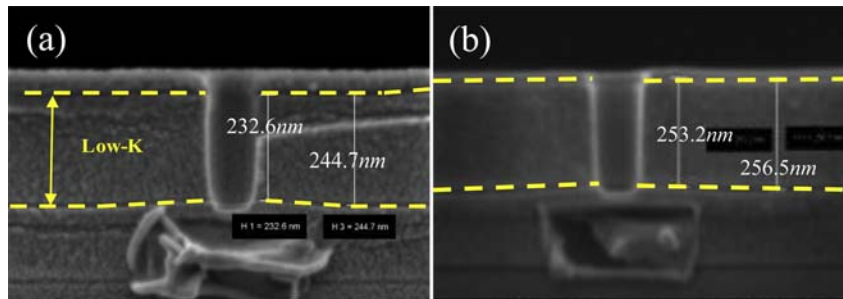
Since the radiation damage in LK and ULK dielectrics mainly arises from the radiolysis damage during SEM analysis, electron dose control is thus the key to minimize such radiation damage by tuning electron beam current. We should use the lowest electron beam current provided that the signal-to-noise ratio in SEM images is reasonable to give sufficient contrast of the target structures. In general, the electron beam current of SEM is dependent on the type of electron source, and the setting of beam illumination system [gun lens and condenser lens (CL)], as well as the size of CL apertures. At the common user level, the beam current can be directly tuned by selecting the size of CL aperture at a certain voltage.

Fig. 2.10A and B shows, respectively, the effects of acceleration voltage and the size of CL apertures on the probe current of Zeiss Ultra55 SEM, which was measured by a Faraday cup. It is clear that the probe current increases with both acceleration voltage and size of CL aperture. Normally the electron current extracted from the electron source is proportional to the acceleration voltage [30]. Compared with the effects of acceleration voltage, the CL aperture has a larger impact on the probe current. During SEM operation, when selecting different probe currents at a fixed acceleration voltage, the users are actually selecting different CL apertures at a fixed acceleration voltage.

Fig. 2.11 shows the cross-sectional SEM micrographs of post-etch via hole profiles imaged with 220 and 15 pA at 1 kV, respectively. The maximum

**FIGURE 2.10**

The dependence of probe current of the Zeiss Ultra55 scanning electron microscope on (A) the acceleration voltage and (B) the size of the condenser lens aperture.

**FIGURE 2.11**

Cross-sectional scanning electron microscopy micrographs of the postetch via holes patterned in low- k carbon-doped oxide (SiCOH) intermetal dielectrics layers ($k = 2.75$, and the standard SiCOH layer thickness of 270 nm by inline measurement) taken at 1 kV with 1 nm Pt surface coating and the probe current of (A) 220 pA and (B) 15 pA.

shrinkage of the LK dielectric layer was up to ~ 38 nm under 220 pA, while it was only ~ 17 nm under 15 pA. The results indicated that the small probe current can effectively reduce the beam radiation damage to LK dielectrics. However, there was still appreciable structure shrinkage at the smallest beam current of 15 pA at 1 kV. Therefore further reduction in the radiation damage is necessary in order to obtain the authentic via hole profile after via etch process, which is essential for the etch process development, qualification, and monitoring.

2.2.3.3 The effects of Pt anticharging coat layer

To further reduce the electron beam radiation-induced structure deformation in LK dielectrics, we may consider using a relatively thick surface coating materials

such as Pt and Au. As it is well recognized that the electron ranges of materials are strongly dependent on the atomic mass of the materials, and heavy metallic materials such as Pt normally have smaller electron ranges than those materials with low atomic mass such as carbon and LK dielectrics. It was reported that the electron range of carbon was just around 33 nm while it was just around 3 nm for Au at 1 kV [31].

By using Monte Carlo simulation, we can appreciate the large difference in the interaction volume of Pt and SiCOH LK dielectrics, as shown in Fig. 2.12A–C. At 1 kV, the electron range of Pt is just around 4 nm, 10 times smaller than that of SiCOH LK dielectrics, 40 nm. The results indicate that a thin layer of Pt coating on SiCOH LK dielectric layer can effectively reduce the penetration depth of electron beam for about 10 times. This helps to reduce radiation damage in two aspects. First, the significant reduction in the electron penetration depth implies the inelastic scattering only occurs near the surface region of the

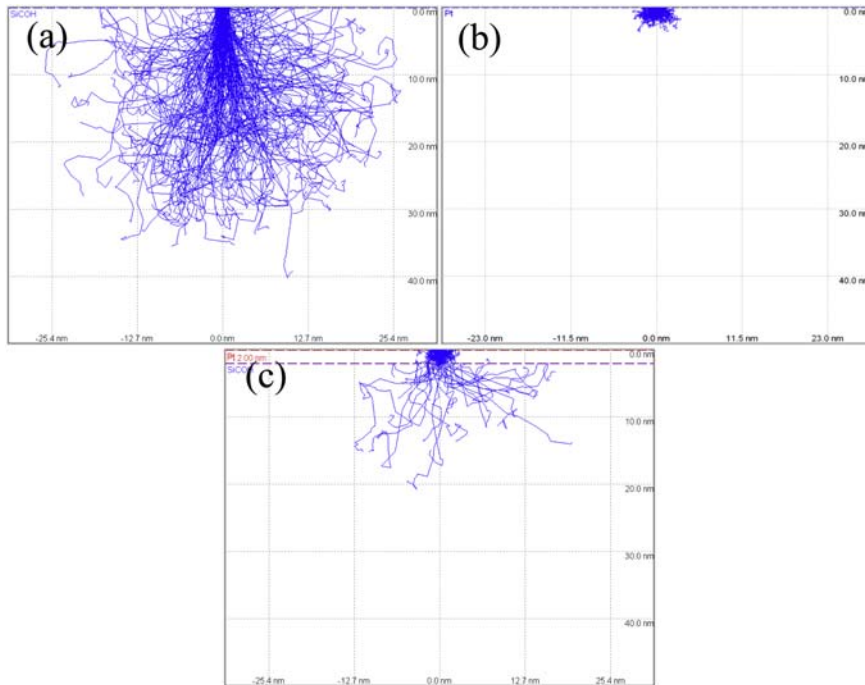


FIGURE 2.12

The effects of Pt surface coating layer on the size of interaction volume (using Monte Carlo simulation program Casino V2.42 based on Rutherford scattering model): (A) low-*k* carbon-doped oxide (SiCOH) at 1 kV with 2 nm probe; (B) Pt at 1 kV with 2 nm probe; and (C) low-*k* SiCOH with 2 nm Pt top coating layer at 1 kV with 2 nm probe.

© 2008 ASM International [32].

LK SiCOH process layer, for which the radiation damage is confined to a shallow depth of ~ 2 nm SiCOH layer. Even with significant radiation damage to SiCOH, such shallow surface damage would not lead to a large structure deformation in the thick SEM specimen. On the other hand, the small penetration depth of electron beam also indicates the less electron flux striking into the SiCOH LK process layer, and thus less inelastic electron scattering events take place for which the radiation-induced structure damage is greatly inhibited.

Fig. 2.13A–C verified the effects Pt surface coating layer on the electron radiation damage in LK SiCOH materials. As shown in Fig. 2.13B, 3 nm thick Pt surface coating layer effectively reduced the electron radiation damage, the thickness of SiCOH layer was around 265 nm, which was close to the inline measurement of 270 nm with only around 2% deformation. With further increase in Pt coating layer thickness to 6 nm, the SiCOH layer shrinkage was further reduced, for which the as-measured SiCOH thickness of 269 nm was close to the inline data. Therefore the profile and measurement shown in Fig. 2.13B and C can be

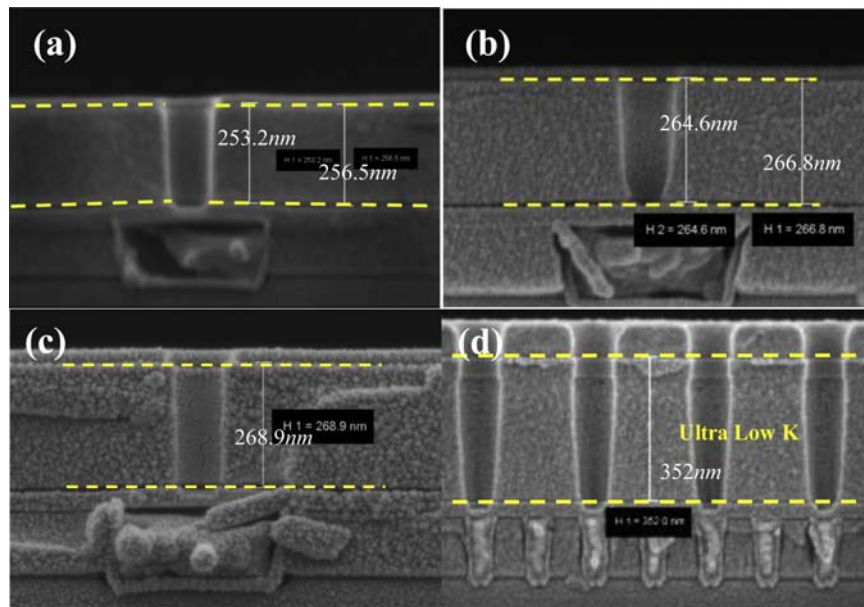


FIGURE 2.13

Cross-sectional scanning electron microscopy micrographs of the via holes patterned in low- k carbon-doped oxide (SiCOH) intermetal dielectrics layers ($k = 2.75$, and the standard SiCOH layer thickness of 270 nm by inline measurement) taken at 1 kV with a probe current of 15 pA and (A) 1 nm Pt coating, (B) 3 nm Pt coating and (C) 6 nm Pt coating; (D) the via holes patterned in ultralow- k SiCOH intermetal dielectrics layers ($k = 2.45$, and the thickness of 350 nm by inline measurement) imaged with 15 pA and 3 nm Pt surface coating at 1 kV.

considered as the authentic profile of the via holes after via etch processes. Only with such level of SiCOH deformation can SEM FA results be used for the purposes of fab process development, monitoring, and qualification.

The abovementioned low-kV and low-dose with Pt coating technical solutions were successfully applied to the SEM FA of ULK process layers or their associated structures. As shown in Fig. 2.13D, there was no appreciable shrinkage in the ULK process layer, and no deformation in via holes during SEM analysis.

2.3 Impact of electron beam radiation during FIB and TEM failure analysis: radiation damage to LK and ULK dielectrics

For the physical FA of LK and ULK SiCOH IMD process layers and the associated semiconductor structures, although SEM FA can provide good imaging resolution up to nanometer size range, there are some FA cases that require TEM FA with a higher resolution and complex chemical analysis for the nanoscale microstructure features. Fig. 2.14A and B shows two typical examples that required TEM physical FA. In Fig. 2.14A, the fab requested clear TEM analysis in order to understand the via etch gouging into bottom NBLK (N-rich block layer, mainly silicon nitride) layer which is only of several tens of nanometers in thickness. Under SEM, it is difficult to get the clear profile of NBLK layer because of almost no contrast in-between NBLK and LK IMD process layer. Therefore TEM FA is needed in order to understand the exact etch gouging into the NBLK process layer. However, due to electron radiation damage to LK IMD layer, the

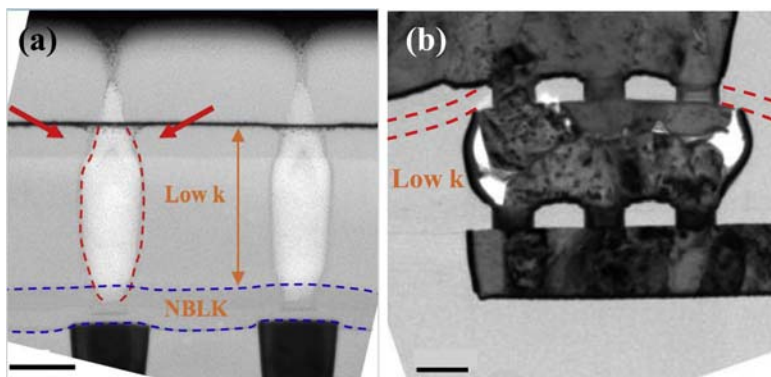


FIGURE 2.14

Transmission electron microscopy micrographs of (A) via holes and (B) Cu metal voids in low-k (LK) intermetal dielectrics layers, showing the severe profile deformation due to electron beam–induced radiation damage to LK process layer.

severe deformation in via holes and NBLK layer rendered the questionable FA results. Fig. 2.14B shows another typical TEM FA case related to Cu voiding issue, and the fab wished to understand the mechanism related to the formation of the Cu voids. Therefore detailed TEM FA with elemental analysis was required to find out the root cause and associated process drift. However, as shown in Fig. 2.14B, there was strong deformation of NBLK layer [by *red dash lines* (gray in print version)] above the LK IMD layer, indicating large structure deformation of LK layer during TEM analysis. In addition, in terms of the profile of the Cu voids, it was hard to understand how the production processes would lead to such abnormal metal profile with Ta/TaN linear bowed, leaving behind big voids inside the metal. Therefore such TEM FA results were doubtful, and the profile obtained TEM FA was not authentic one and thus could not be correlated to the manufacturing processes. All these abnormal FA results require FA engineers to solve the electron beam radiation damage to LK dielectrics before the valuable TEM FA results can be delivered for fab production supports.

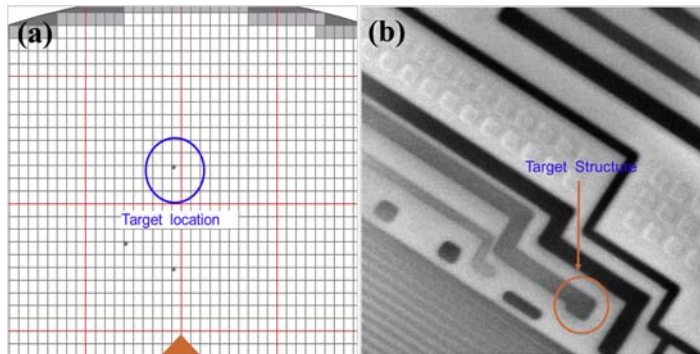
Compared with SEM physical FA, TEM FA faces more challenges when we deal with the issue of electron beam radiation damage to LK and ULK dielectrics. For TEM FA, the electron beam radiation damage could occur in the following TEM FA process steps:

- Electron beam survey before FIB milling for locating target location or structures if LK or ULK process layer is the top process layer or close to the top process layer.
- Surface coating by electron beam deposition method before FIB milling if LK or ULK process layer is the top process layer or close to the top process layer.
- FIB slice and electron beam view before milling to the target structures.
- TEM imaging analysis.

To understand how the above-mentioned processes would induce the electron beam radiation damage to LK and ULK dielectrics, in the following sections we will give detailed description of the electron radiation-induced structure deformation and the associated technical solutions during different TEM FA process steps. Cu/ULK ($k = 2.55$) structures with different metal densities after chemical mechanical polishing (CMP) were chosen for evaluating the electron radiation damage to ULK materials during different process steps of TEM FA work.

2.3.1 Electron beam radiation damage during electron beam survey before focus ion beam milling

For wafer-level site-specific FA work, the general procedure is first to cut out the die with the target location from the wafer based on the wafer map (Fig. 2.15A), and then mark the target structure by using laser mark under optical microscope if the target structure is large enough to be visible under optical microscope. If the target structure is too small to be visible under optical microscope, electron

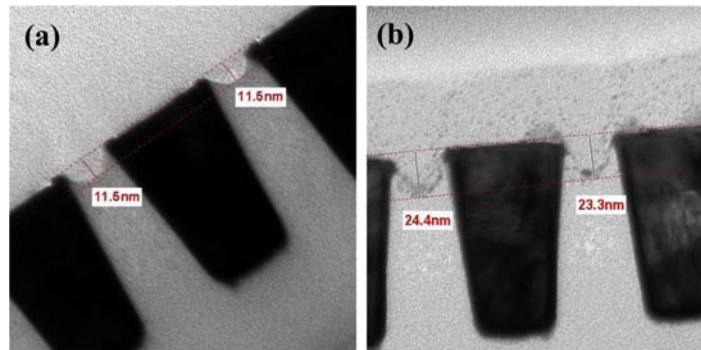
**FIGURE 2.15**

(A) A schematic wafer map showing the location of the target structure for transmission electron microscopy failure analysis (TEM FA). (B) A typical top-down scanning electron microscope micrograph showing the target structure to be analyzed by TEM FA.

beam survey is needed to locate and mark the target structure by using SEM or electron beam viewing in dual-beam FIB (with both ion beam and electron beam systems). Fig. 2.15B is a typical top-down SEM micrograph of Cu metal layers of one semiconductor device, and the target structure as circled is just around 200 nm, and thus site survey and target localization need to be done with electron beam viewing by a SEM or a dual-beam FIB. Once using electron beam site survey, it is important to take note of electron radiation damage issue when LK and ULK dielectrics is the top process layer or close to the top process layer. To minimize the radiation damage, the site survey should be performed under low-kV and low-dose electron beam imaging conditions, and meanwhile a thin layer of Pt coating on the sample surface is suggested in order to further reduce the radiation damage, as discussed in Section 2.2.

2.3.2 Electron beam radiation damage during electron beam coating before focus ion beam milling

In general, before FIB milling, a surface protection layer needs to be deposited on sample surface in order to avoid the surface damage by ion beam during FIB milling. Normally, such protection layer can be different types of materials such as oxide, Pt, and carbon-based materials. It can be deposited either by the ex situ coating methods before sample loading to FIB or by the in situ electron beam or ion beam assisted chemical vapor deposition processes inside FIB. However, for the post-CMP Cu/ULK structure, ion beam surface coating should be avoided due to ion beam milling and bombardment on the sample surface. Thus two methods were used for the surface coating, namely in situ electron beam coating in FIB tool and ex situ photoresist (PR) spin coating.

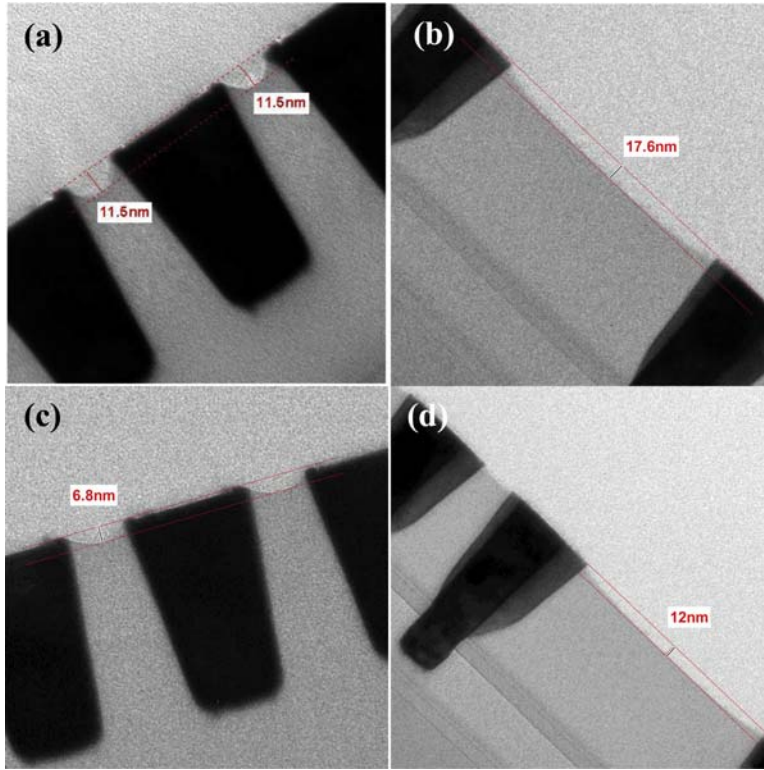
**FIGURE 2.16**

Transmission electron microscopy (TEM) micrographs of Cu/ultralow- k ($k = 2.55$) structures: (A) with electron beam carbon coating (under electron beam of 5 kV and 13 nA) and (B) with photoresist spin coating (focus ion beam milling and TEM imaging were done under the same conditions).

Fig. 2.16A and B shows, respectively, the TEM micrograph of Cu/ULK structures with electron beam carbon coating and PR spin coating. For both samples, the FIB milling and TEM imaging were performed under the same conditions and thus they underwent similar level of electron dose except the additional electron beam exposure in case of electron beam coating. Apparently, the sample with electron beam coating had much larger ULK shrinkage of ~ 24 nm, two times as large as that of PR-coated sample. The large ULK shrinkage can be attributed to the enhanced radiation damage during electron-coating process in which an intensive electron beam of 13 nA continuously scanned across the sample surface. Therefore in order to minimize the radiation damage to LK and ULK dielectrics, electron beam coating should be avoided when the top process layer contains LK or ULK dielectrics.

2.3.3 Electron beam radiation damage during focus ion beam milling for transmission electron microscopy sample preparation

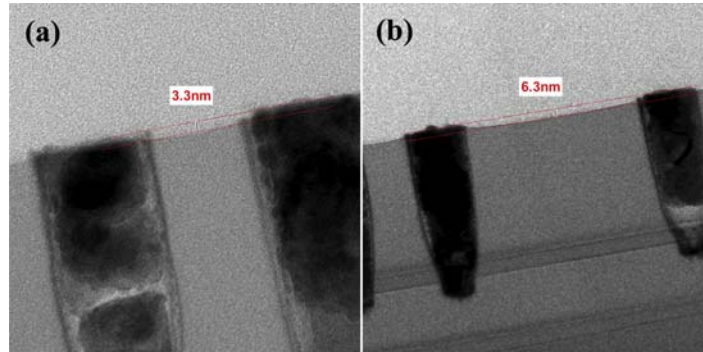
The TEM sample preparation can be performed using either single-beam or dual-beam FIB tools. Single-beam FIB has only ion beam column and thus no electron beam imaging system is available for cross-sectional viewing during FIB milling. For those FIB work related to the nonsite-specific and big structures, single-beam FIB can accomplish the jobs without electron beam viewing. However, for the site-specific small structures, FIB milling by a dual-beam FIB is needed because cross-sectional electron beam viewing is needed to aim for the small target structure and to achieve the accurate FIB cut for the purpose of TEM analysis. This is

**FIGURE 2.17**

The TEM micrographs of Cu/ultralow- k ($k = 2.55$) structures by FIB milling with different electron beam slice/view processes: (A) dense Cu metal structure; (B) loose Cu metal structures with electron beam viewing at 10 kV and 340 pA; (C) dense Cu metal structure; and (D) loose Cu metal structures with electron beam viewing at 2 kV and 11 pA (all samples were analyzed under a typical bright-field TEM imaging conditions with Spot-1 and condenser lens aperture-50 μm). *FIB*, Focus ion beam; *TEM*, transmission electron microscope.

the so-called FIB slice and view processes. During these FIB FA processes, if the electron dose is not properly controlled, large structure deformation of LK/ULK dielectrics will occur. Fig. 2.17A and B shows, respectively, the TEM micrographs of dense and loose Cu/ULK structures after FIB milling with high kV (10 kV) and high-dose (340 pA probe current) electron beam viewing. Clearly, large shrinkage of 11.5 nm was observed in the ULK layers of the dense Cu/ULK structure (Fig. 2.17A); while for the loose metal structures, the deformation was even larger, up to 17.6 nm (Fig. 2.17B).

In contrast, when using low-kV and low-dose electron beam viewing at 2 kV and 11 pA, the ULK deformation was reduced to 6.8 and 12 nm, respectively, as

**FIGURE 2.18**

The TEM micrographs of Cu/ULK ($k = 2.55$) structures by single-beam FIB milling without electron beam viewing: (A) dense Cu/ULK structure and (B) loose Cu/ULK structure. *FIB*, Focus ion beam; *TEM*, transmission electron microscope; *ULK*, ultralow- k .

shown in Fig. 2.17C and D. If the target structure is large enough and FIB slice/view process is not required, the electron beam illumination before and during FIB milling can be completely avoided. Fig. 2.18A and B shows Cu/ULK structures that were cut by single-beam FIB. Without electron beam viewing, the deformation of ULK was further reduced to 3.3 and 6.3 nm for dense and loose Cu/ULK structures, respectively. Table 2.1 summarizes the ULK shrinkage under different electron beam viewing conditions during FIB milling for TEM sample preparation. In order to minimize the electron beam radiation damage to ULK, electron beam surface coating should be avoided, and the FIB slice and view process needs to be performed with low-kV and low-dose electron beam viewing mode. In addition, different Cu/ULK metal stacks may have different levels of structure deformation under the same beam illumination conditions. Loose metal structure always had larger shrinkage in ULK than that of dense structure, as indicated in Table 2.1. Such phenomena were commonly observed during FIB and TEM FA work, for which FA engineer should be always aware of such abnormality due to electron beam radiation damage, and thus be cautious of the interpretation of FA results associated.

2.3.4 Electron beam radiation damage during transmission electron microscope analysis

With even proper pre-FIB surface coating and low-kV/low-dose FIB milling and single-beam sample preparation techniques, appreciable shrinkage of ULK dielectrics still occurred, as indicated in Table 2.1 and Figs. 2.16–2.18. Such level of ULK shrinkage was due to the radiation damage generated during TEM analysis under the conventional bright-field imaging mode with a high electron dose.

Table 2.1 A summary of ULK shrinkage under different pre-focus ion beam (FIB) surface coating and electron beam viewing conditions during FIB milling processes.

	ULK shrinkage under dual-beam FIB slice/view with electron beam at (10 kV, 340 pA)	ULK shrinkage under dual-beam FIB slice/view with electron beam at (2 kV, 11 pA)	ULK shrinkage without electron beam viewing (single-beam FIB)
PR coating, dense Cu/ULK structure (nm)	11.5	6.8	3.3
PR coating, loose Cu/ULK structure (nm)	17.6	12.0	6.3

Fig. 2.19A–D shows the time-dependent ULK shrinkage under normal bright-field TEM imaging conditions with electron dosage of $\sim 1012 \text{ e}/\text{\AA}^2 \cdot \text{s}$. The results indicated that large and fast ULK shrinkage occurred during TEM analysis, and electron dose control is needed to minimize the radiation damage to LK and ULK dielectrics.

As mentioned previously, for the cryo-TEM imaging of those beam-sensitive biological samples, low-dose TEM technique has been well established to minimize the electron beam radiation damage and thus to maintain the native structures of biomolecules such as proteins [33,34]. Therefore in the field of biological TEM image, TEMs usually have the low-dose imaging capability with the associated software control during sample search, image focus, and capture, such as SerialEM in JEOL TEMs and low dose in FEI TEMs. In addition, during TEM analysis, the dosage of the electron beam can be real-time monitored, and thus the total electron dose to specimen be precisely controlled, especially in some complex TEM analysis such as 3D cryo-electron tomography TEM analysis [35].

Normally in semiconductor physical FA labs, TEMs are rarely equipped with any low-dose and cryo-TEM imaging facilities. However, there are still other technical approaches that can be used to control the electron dose for LK and ULK associated TEM analysis. Table 2.2 listed the electron dose rates at the different TEM beam illumination settings and CL aperture size.

For cryo-TEM analysis of biological samples, generally the total electron dose limit that cryo-samples can withstand is around $100 \text{ e}/\text{\AA}^2$. For LK and ULK dielectric materials, our experimental studies also revealed similar dose limits. Therefore at a magnification of $\times 97\text{K}$, if the TEM search, focus and image capture can be done within 5 seconds, the feasible beam illumination conditions can only be those with spot size of 6 or smaller and the CL aperture of $30 \mu\text{m}$ or smaller. If the whole TEM imaging process takes longer than 10 seconds, then the

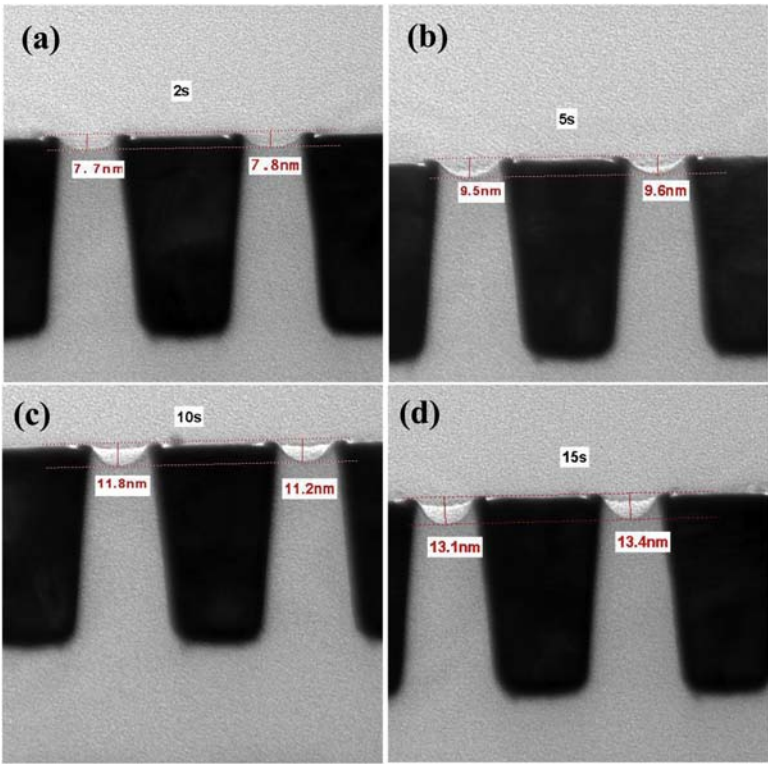


FIGURE 2.19
The dependence of ultralow- k (ULK) shrinkage of Cu/ULK ($k = 2.55$) structures with beam illumination time (focus ion beam slice/view with electron beam viewing at 2 kV and 11 pA, and transmission electron microscope imaging conditions with Spot-1 and condenser lens aperture of 50 μm).

Table 2.2 The electron dose rates at the different beam illumination settings (FEI Tecnai F20 TEM at 200 kV).

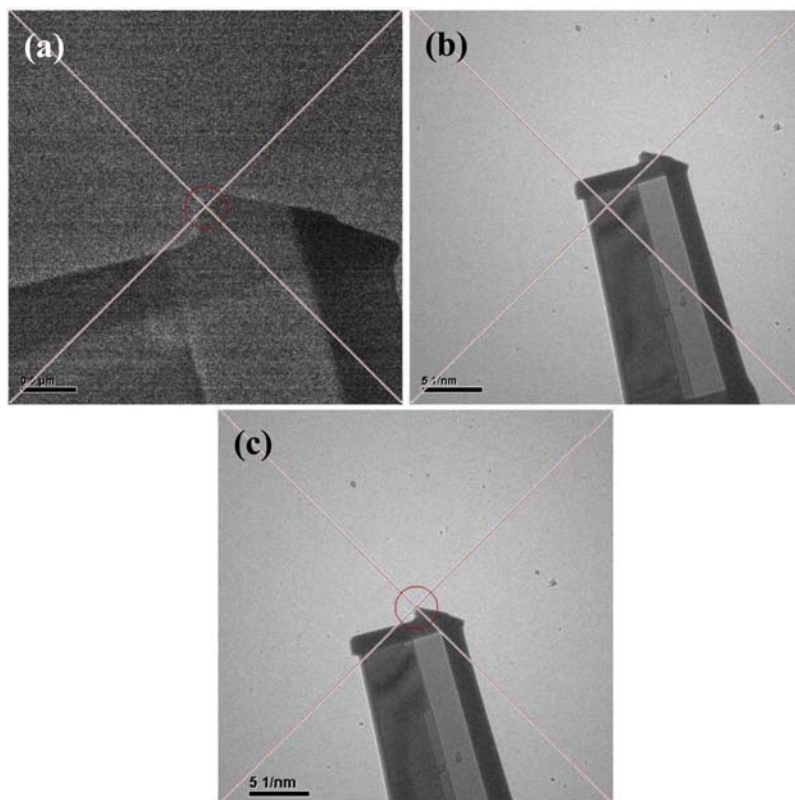
	Spot-1, CL aperture (50 μm)	Spot-3, CL aperture (50 μm)	Spot-6, CL aperture (50 μm)	Largely defocused diffraction imaging mode, Spot-9, CL aperture (30 μm)
Electron dosage ($\text{e}/\text{A}^2 \cdot \text{s}$) at magnification of $\times 97\text{K}$	1012	278	24	Search mode: 0.0013 Exposure mode: 3

CL, Condenser lens.

feasible illumination setting will be Spot-9 and condenser aperture of 30 μm or smaller. Therefore such level of dose control is quite challenging in terms of normal TEM operation. To overcome the issues related to extended sample exposure to the electron beam during sample search, and imaging focus, we proposed an alternative low-dose TEM imaging technique using a conventional TEM, that is, sample search and imaging focusing in the largely defocused diffraction imaging mode, and image capture at low-dose bright-field TEM imaging mode. The whole procedures are described in an FEI Tecnai F20 TEM:

1. Set the beam illumination condition to Spot-9, the smallest CL aperture-1 (30 μm) before starting TEM viewing.
2. Setting and image alignment in the largely defocused diffraction imaging mode:
 - a. Fully defocusing CL to set it at the lowest excitation level under which the imaging brightness becomes very weak. Tuning the condense lens a little bit until some weak contrast can be seen at the defined magnification. Find a typical small feature near the target structure, and center it to the screen center by using TEM stage, as shown in Fig. 2.20A.
 - b. Switch to diffraction mode and largely defocus the intermediate lens so that sufficient image contrast can be obtained in the diffraction mode, as shown in Fig. 2.20B.
 - c. Use diffraction shift to align the same feature as illustrated in Fig. 2.20A to the center of the screen, as shown in Fig. 2.20C. With such alignment, whatever is centered in defocused diffraction image mode will also be at the center in the bright-field TEM imaging mode.
 - d. Search through the interested TEM sample in the defocused diffraction imaging mode, and centered the target structure to the center of the screen and record the x/y physical position of the target structure.
3. Separate image focus and image capture:
 - a. Using stage control to move to a structure that is a bit far away from the target one.
 - b. Switch to bright-field TEM imaging mode, and do all the necessary optical alignments, and adjust magnification, electron doses and image focus properly.
 - c. Switch to the largely defocused diffraction imaging mode again and load the as-saved physical position of the target structure (by clicking x/y movement only).
 - d. After image centering, switch to TEM imaging mode, and immediately capture the image of the target structure at the predefined magnification (exposure time 1–3 seconds).

With abovementioned low-dose TEM imaging technique, the electron dose that the sample received during TEM search and focus can be greatly minimized due to very low-dose rate of $0.0013 \text{ e}/\text{\AA}^2\cdot\text{s}$ in the diffraction imaging mode, as indicated in Table 2.3. Therefore the radiation damage to ultralow- k can be reduced effectively.

**FIGURE 2.20**

Low-dose imaging techniques in a conventional transmission electron microscope: (A) image under bright-field mode with the lowest brightness; (B) misaligned image; and (C) well-aligned image in the largely defocused diffraction imaging mode.

Fig. 2.21 shows the TEM micrographs of Cu/ULK structures under different TEM imaging conditions. With the same CL aperture ($50\ \mu\text{m}$), when the spot size was reduced from Spot-1 to Spot-6, the electron dose rate was reduced from $10,120$ to $24\ \text{e}/\text{\AA}^2\cdot\text{s}$, and thus ULK shrinkage was reduced from 6.8 to $3.6\ \text{nm}$, as shown in Fig. 2.21A–C. By using the low-dose imaging in diffraction mode, the electron dose was further reduced, and thus ULK shrinkage was just around $1.8\ \text{nm}$.

The above results indicate that, even with low-dose imaging techniques, there still exists some ULK shrinkage, as shown in Fig. 2.21D and Table 2.3. Such a small ULK shrinkage could be induced by low-dose dual-beam FIB milling process before TEM analysis. To verify this, a combination of single-beam FIB milling and a low-dose TEM imaging technique were employed. Fig. 2.22A and B

Table 2.3 The electron dose rates and the associated ultralow- k shrinkage at the different focus ion beam and transmission electron microscopy beam illumination conditions (FEI Tecnai F20 TEM at 200 kV).

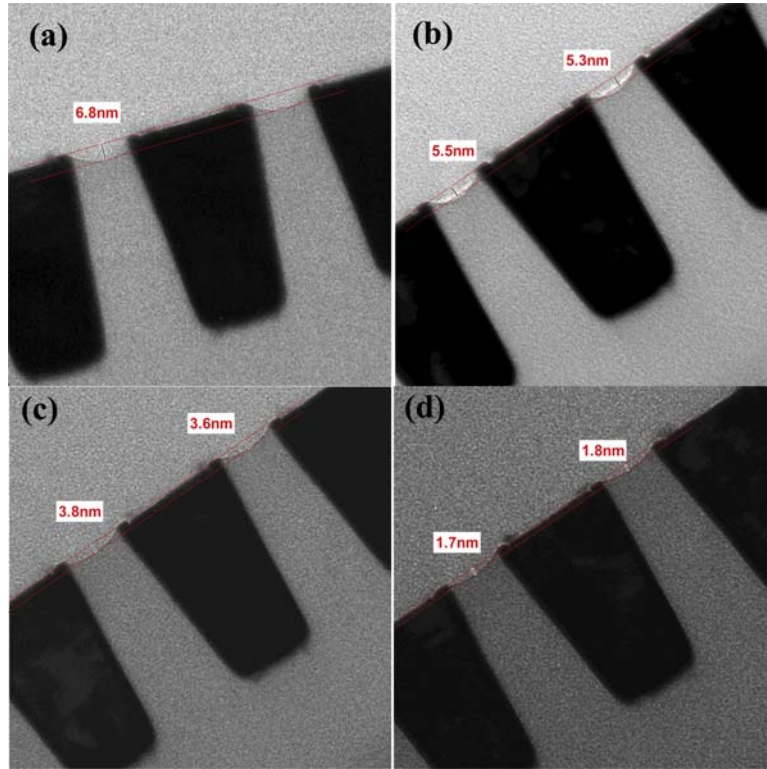
	Spot-1, CL aperture (50 μm)	Spot-3, CL aperture (50 μm)	Spot-6, CL aperture (50 μm)	Largely defocused diffraction imaging mode, Spot-9, CL aperture (30 μm)
Electron dosage ($\text{e}/\text{Å}^2\cdot\text{s}$) at magnification of $\times 97\text{K}$	1012	278	24	Search mode: 0.0013 Exposure mode: 3
ULK shrinkage (dense Cu/ULK) (FIB electron beam view: 2 kV and 11 pA)	6.8 nm	5.4 nm	3.7 nm	1.8 nm
ULK shrinkage(FIB electron beam view: 2 kV and 11 pA)	3.3 nm (dense) 6.3 nm (loose)	—	—	~ 0 nm (both dense and loose structures)

shows the TEM micrograph of Cu/ULK, and clearly the radiation-induced shrinkage to ULK layer was almost zero.

In summary, for FIB/TEM physical FA of LK/ULK dielectrics and their associated semiconductor structures, it is important to take note of the electron radiation damage in every possible FA steps, namely, pre-FIB sample surface coating, sample navigation, FIB slice and view, and TEM imaging. In the FA process that needs electron beam viewing, the electron dose rate should be kept as low as possible. On the other hand, for such low-dose FIB and TEM FA work, normally the FA engineers need to have relatively high technical skills in terms of FIB and TEM operation so that they can perform high-quality FA work as fast as possible to minimize unnecessary extended sample exposure to electron beam radiation.

2.4 Impact of electron beam radiation damage during TEM failure analysis: radiation damage to silicon nitride

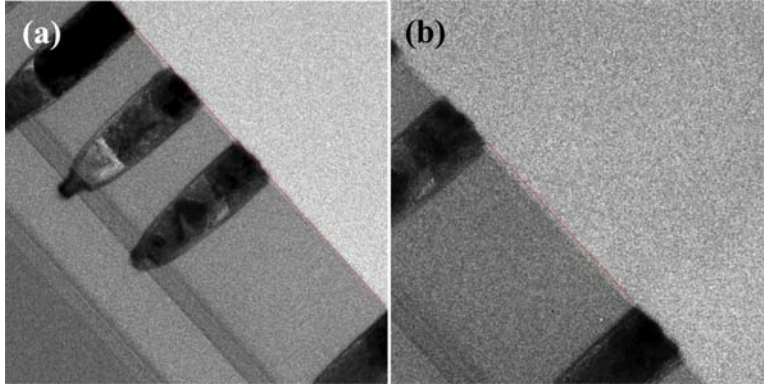
Since the early semiconductor development, silicon nitride has been commonly used for various process layers and for various purposes in semiconductor devices. Silicon nitride has intrinsic impermeability to most impurities, which qualifies its primary use as a passivation layer, especially as a diffusion barrier to moisture and sodium [23]. With its high dielectric constant of 6–9, it is an ideal dielectric material for capacitor devices, such as metal–insulator–metal capacitors. By using the large difference in etch rate of SiO_2 and Si_3N_4 , silicon nitride can be used as an etch hard mask for oxide dielectric etch processes. By tuning

**FIGURE 2.21**

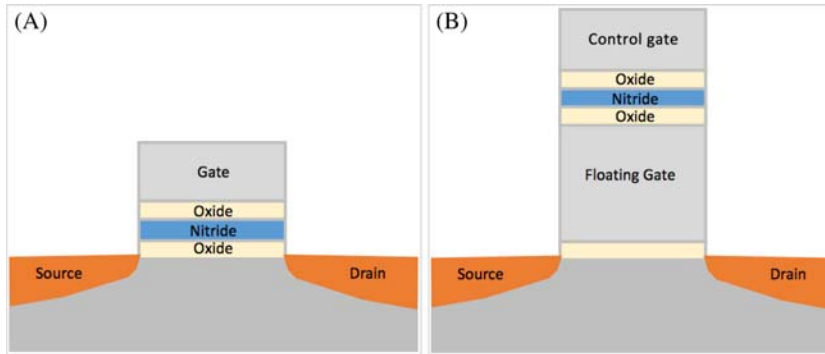
Transmission electron microscopy (TEM) micrographs of Cu/ultralow- k ($k = 2.55$) structure under different imaging conditions: (A) low-kV/low-dose focus ion beam (FIB), TEM Spot-1, condenser lens (CL) aperture-50 μm ; (B) low-kV/low-dose FIB, TEM Spot-3, CL aperture-50 μm ; (C) low-kV/low-dose FIB, TEM Spot-6, and CL aperture-50 μm ; and (D) low-kV/low-dose FIB, largely defocused diffraction imaging with Spot-9, and CL aperture-30 μm .

the deposition processes and film composition, silicon nitride can be used as tensile stress linear to enhance the carrier mobility of N-channel MOSFET devices [36,37]. Furthermore, silicon nitride has important applications in nonvolatile memory (NVM) devices either in the floating gate-based or nitride-based charge-trapping memory devices, as shown in Fig. 2.23.

In both floating gate-based and nitride-based NVM devices, oxide-nitride-oxide (ONO) layers are the indispensable structures. In a typical silicon-oxide-nitride-oxide-silicon NVM device, silicon nitride in the ONO layers is used for charge storage with its intrinsic charging trapping characteristics. While in a typical floating gate-based NVM device, ONO is used as the interpoly dielectrics for blocking any leakage during the programming and erase

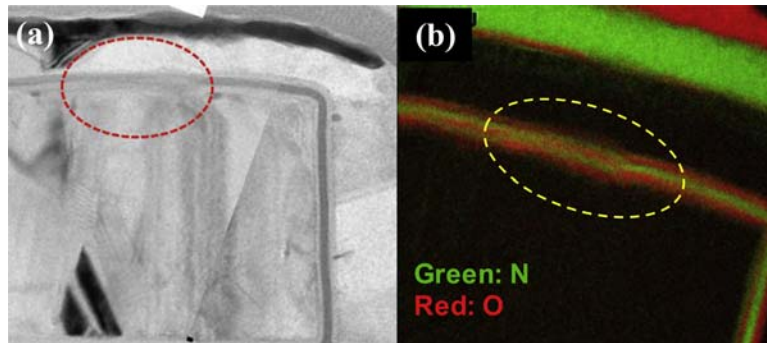
**FIGURE 2.22**

Transmission electron microscopy (TEM) micrographs of Cu/ultralow- k ($k = 2.55$) structure under focus ion beam (FIB)/TEM imaging conditions: single-beam FIB, and largely defocused diffraction imaging with Spot-9, condenser lens aperture-30 μm .

**FIGURE 2.23**

Typical (A) nitride-based silicon—oxide—nitride—oxide—silicon and (B) floating-gate nonvolatile memory devices.

operations. With the high dielectric constant of nitride, ONO also helps to ensure reliable charge retention in the floating gates and to provide good capacitive coupling of the control gate to the floating gate so as to lower the voltages needed for read, program and erase operations [38]. Therefore the ONO dielectric layer plays a crucial role in affecting the performance and the data retention reliability of NVM devices under harsh programming and erasing conditions. With NVM continuously scaling down, the thickness of ONO dielectric films is also reduced to obtain enough storage capacitance. As a consequence, the degradation of the ONO breakage down characteristics is becoming a critical problem for NVM

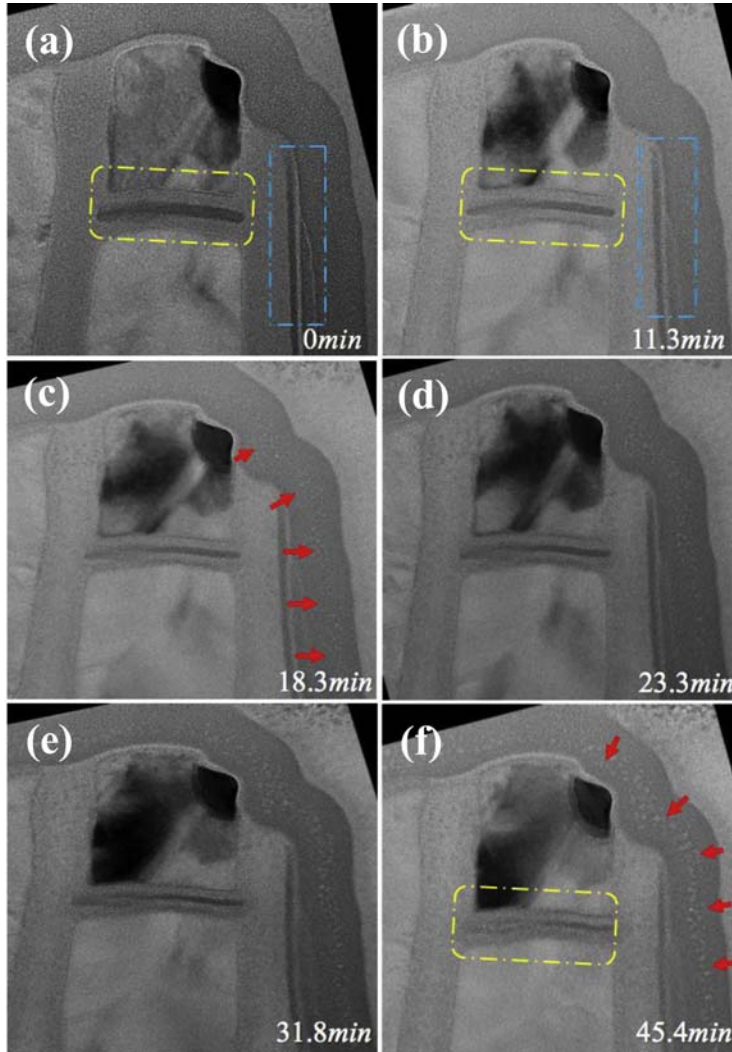
**FIGURE 2.24**

(A) Transmission electron microscopy micrograph of the cross-sectional view of a failed electrically erasable random access memory (EEPROM) memory cell, showing abnormal oxide–nitride–oxide (ONO) layers and (B) electron energy loss spectroscopy mapping revealing the fused nature of the ONO structure.

memory devices. In terms of FA of NVM devices, many failure cases are associated with breakdown or degradation of the ONO dielectric layers, in which the physical characterization of ONO structure is constantly required in order to understand if there is any abnormality associated with the ONO film stacks. Various studies have shown that time-dependent dielectric breakdown characteristics of ONO dielectric film is strongly dependent on the quality and morphology of nitride film stacks, such as film roughness, thickness uniformity [39–41].

Fig. 2.24A shows the cross-sectional TEM micrograph of a typical NVM cell with data retention issue, in which abnormal contrast in ONO layers was observed. Furthermore, EELS mapping analysis revealed that nitride and oxide layers were fused together, as shown in Fig. 2.24B. Such physical signature well agreed with the EFA, which showed the current leakage between the floating gate and control gate. Such fused ONO dielectric film in turn accounted for the failure of the data retention reliability test of the NVM device.

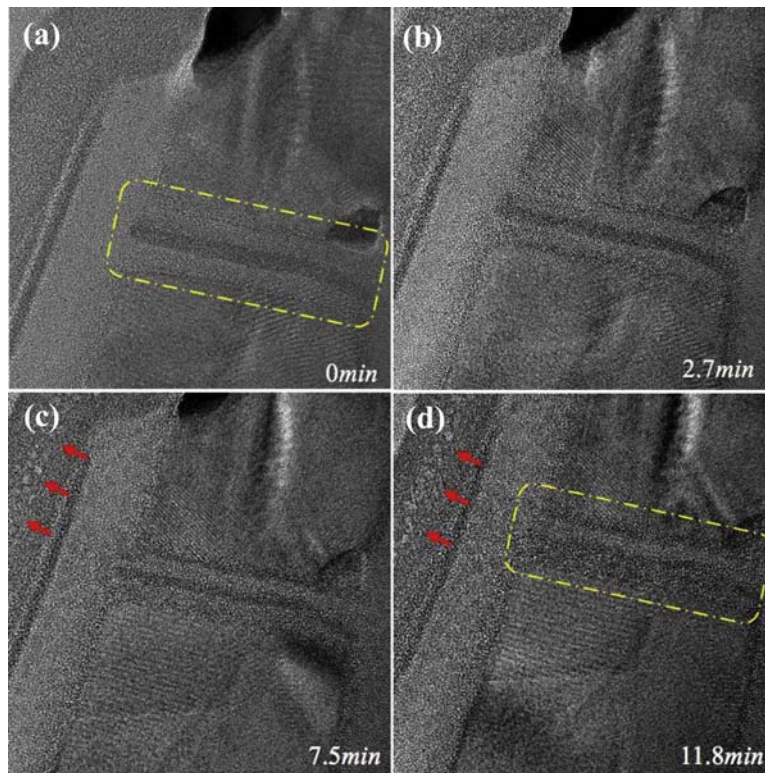
However, for such PFA by FIB and TEM, we should always be aware of the electron beam radiation damage to nitride film. Although silicon nitride is not so sensitive to electron radiation as LK and ULK dielectrics, the radiation damage to silicon nitride by high-dose electron beam has been reported, such as radiation-induced hole drilling [42], the changes in luminescence behavior [43] and the nature of chemical bonding [44]. Figs. 2.25 and 2.26 show microstructure evolution of nitride process layers under the electron beam radiation damage for a typical floating-gate NVM device. As shown in Fig. 2.25, under electron beam current of 2.11 nA, 11.3 minutes electron radiation led to apparent changes in nitride process layers, that is, the thinning-down of nitride layer in ONO dielectric layers [marked by the yellow rectangle (white in print version) in Fig. 2.25] and the interface fusing between different nitride spacer layers [marked by blue rectangle (gray in print version) in Fig. 2.25].

**FIGURE 2.25**

Transmission electron microscopy micrographs of a typical floating-gate nonvolatile memory cell after electron beam radiation for different periods of time with beam current of 2.11 nA at 200 kV: (A) ~ 0 min; (B) 11.3 min; (C) 18.3 min; (D) 23.3 min; (E) 31.8 min; and (F) 45.4 min [45].

© 2018 American Institute of Physics.

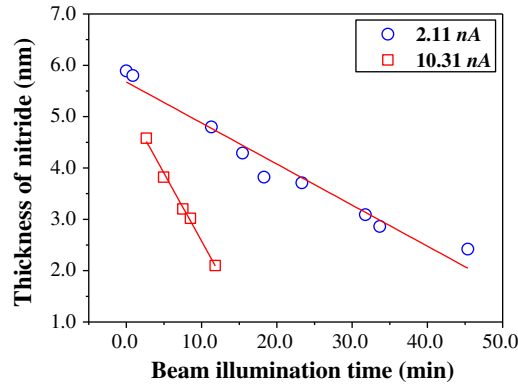
Further electron radiation for around 18.3 minutes resulted in the further thinning-down of the nitride in ONO layers, and the formation of the white-contrasted pore-like defects in the nitride spacer as indicated by the *red arrows*

**FIGURE 2.26**

Transmission electron microscope micrographs of a typical floating-gate nonvolatile memory cell after electron beam radiation for different periods of time with beam current of 10.31 nA at 200 kV: (A) ~ 0 min; (B) 2.7 min; (C) 7.5 min; and (D) 11.8 min [45].

© 2018 American Institute of Physics.

(gray in print version) in Fig. 2.25C. After electron radiation for 45 minutes, the ONO dielectric layers showed the diffused contrast with barely visible nitride layer, and the white-contrasted pore defects became larger and formed a continuous bubble-like layer in the middle of the nitride spacer layer. At a larger electron beam current of 10.31 nA, the electron radiation damage to nitride process layers became much faster, as shown in Fig. 2.26. A short beam illumination of just 2.7 minutes directly resulted in the contrast blurring of nitride layer in ONO dielectric layers as indicated in Fig. 2.26B. The pore like started to form after beam illumination for around 7 minutes, and meanwhile the nitride in ONO was thinning down and its contrast became invisible as indicated in Fig. 2.26C. After electron radiation for 11.8 minutes, the ONO layers were completely invisible with the large and continuous porous-like defects in the middle of the nitride spacer, as shown in Fig. 2.26D.

**FIGURE 2.27**

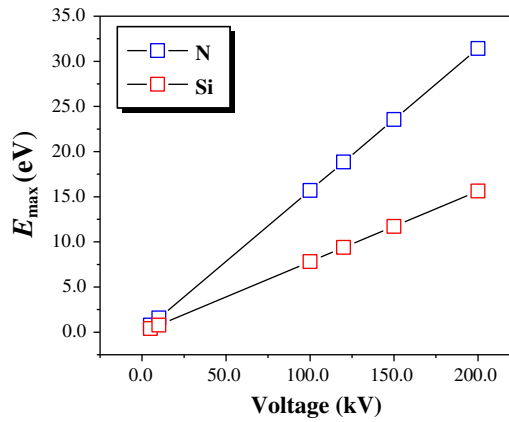
The dependence of nitride layer thickness in oxide–nitride–oxide on the electron beam illumination time under two different electron beam currents at 200 kV (nitride thinning speed: 0.7 nm/min at 2.11 nA; 2.6 nm/min at 10.31 nA) [45].

© 2018 American Institute of Physics.

Fig. 2.27 shows the radiation time-dependent nitride thickness in ONO dielectric layers. As seen from it, under the electron radiation with the beam currents of both 2.11 and 10.31 nA, the nitride thinning-down speed showed approximate linear dependence on the electron beam radiation damage time. Such a linear time dependence suggests that the nitride thinning-down is a diffusion-controlled process [15,46]. The fitting of the experiment data revealed that the nitride thinning-down speeds were 0.7 and 2.6 nm/min at 2.11 and 10.31 nA, respectively.

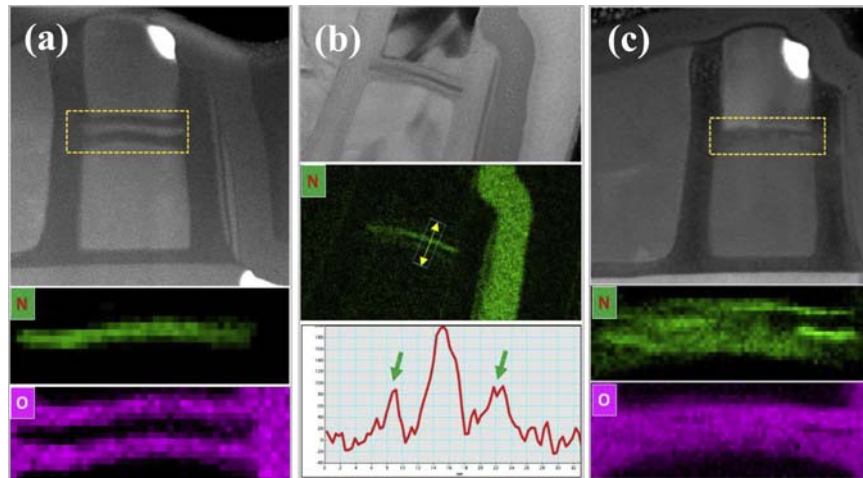
To understand the underlying mechanism associated with the electron radiation–induced nitride thinning-down in the ONO structure and the formation of pore-like defects in the middle of nitride spacer, more detailed analysis is needed.

Under the elastic collision model, according to Eq. (2.1), the maximum knock-on energy transferred to N and Si atoms ($E_{\max}$) were estimated and plotted in Fig. 2.28. At 200 kV, $E_{\max}$ are 15.63 and 31.41 eV for Si and N atoms, respectively. It was reported that the threshold displacement energies of Si and N in silicon nitride were around 11–13 and 21–27 eV for Si and N atoms, respectively [42]. Therefore electron radiation at 200 kV can directly lead to the knock-on damage with Si and N atoms displaced from nitride film. However, such atomic displacement damage should be of isotropic nature and would occur globally across the whole nitride process layers, and thus simple displacement knock-on damage cannot explain the continuous thinning-down of the nitride layer in ONO and the appearance of pore-like defects in the middle of nitride spacer layers in Figs. 2.25 and 2.26. Instead, such nitride thinning-down and the formation of pore-like defects may be associated with the material diffusion during high-dose electron beam radiation, which was verified by the results shown in Fig. 2.29. At the beginning of the beam radiation, ONO dielectric layers were well

**FIGURE 2.28**

The dependent of $E_{\max}$, the maximum energy transferred to Si and N atoms on the acceleration voltage of electron beam [45].

© 2018 American Institute of Physics.

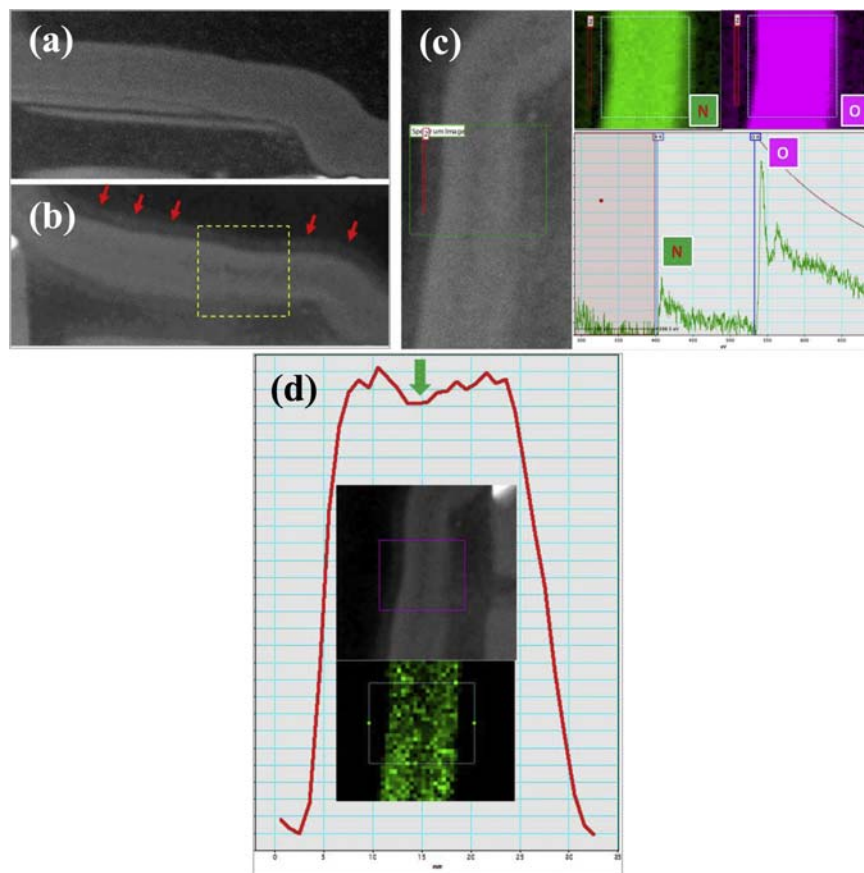
**FIGURE 2.29**

Scanning transmission electron microscopy micrographs and electron energy loss spectroscopy mapping analysis of one nonvolatile memory cell at (A) ~ 0 min beam radiation; (B) around 7.5 min beam radiation; and (C) 11.8 min beam radiation under electron beam current of 10.31 nA at 200 kV [45].

© 2018 American Institute of Physics.

distinguished, and EELS mapping showed clearly the nitride layer inserted in-between two layers of oxide layers, as shown in Fig. 2.29A. After electron radiation with the beam current of 10.31 nA for around 7.5 minutes, EELS mapping and line scan profile in Fig. 2.29B revealed the diffusion of N from nitride layer and segregated at the interfaces between oxide and the poly-Si layers of the control gate and floating gate. With electron radiation for 11.8 minutes, severe diffusion of both N and O occurred for which ONO structure was completely damaged. The nitride layer and two oxide layers were completely mixed with each other, as shown by the O and N EELS maps in Fig. 2.29C. The results indicated that high-dose electron beam radiation led to not only the knock-on damage, but also fast diffusion of O and N atoms. Such atomic diffusion behavior well explained the linear time-dependent thinning-down of nitride in ONO as shown in Figs. 2.25–2.27, which can be ascribed to the so-called radiation-enhanced diffusion (RED) effects, as widely reported in other materials systems [8–11]. According to the RED theory, the enhanced diffusivity of impurities or host atoms in crystalline and amorphous materials is directly related to the concentration of active point defects (vacancies and interstitials) generated by electron and ion beam radiation. As discussed above, electron beam radiation at 200 kV not only displaced Si and N atoms by knock-on effects, but also induced strong ionization effects for which the rapture of chemical bonding in-between Si and N occurred. With such both knock-on and ionization damage, lots of point defects were generated, which acted as active diffusion carriers, to promote the diffusion of Si, N, and O atoms, leading to continuous thinning-down of nitride layer in the ONO structure.

As far as the formation of pore-like defects in the middle of nitride spacer is concerned, it was associated with the mass loss induced by electron radiation. With scanning TEM (STEM) imaging at a high magnification, it was found that there was additional layer formed along the nitride spacer after electron radiation for 11.8 minutes, overlapping with the oxide layer outside of the nitride spacer, as indicated by the *red arrows* (gray in print version) in Fig. 2.30B. EELS mapping and spectroscopy analysis [in the area marked by *yellow-dotted square* (white in print version)] revealed that this newly formed layer was N-rich (Fig. 2.30C), which implied the diffusion of N atoms from the nitride spacer with electron radiation. In addition, it is interesting noted that the pore-like defect region formed in the middle of the spacer was N deficient, indicating the mass loss of N element after electron radiation for 11.8 minutes, as shown in Fig. 2.30D. Above results indicated that high-dose electron radiation at 200 kV led to not only atomic diffusion but also the mass loss of N element. Such phenomena might be attributed to the relatively large thickness of the nitride spacer. On the one hand, electron radiation–induced RED effects promoted the atomic diffusion of N atoms to both sides of the nitride spacer. On the other hand, for those N atoms in the middle of nitride spacer knocked out by electron radiation, they could not timely diffuse out due to large thickness of nitride spacer. Instead, they condensed together and diffused to the surface and finally evaporated out, leading to the N loss. Such mass

**FIGURE 2.30**

Scanning transmission electron microscopy micrographs of nitride spacer of one nonvolatile memory cell at (A) ~ 0 min beam radiation; (B) around 11.8 min beam radiation; (C); and (D) electron energy loss spectroscopy mapping in the nitride spacer region after electron radiation for 11.8 min with electron beam current of 10.31 nA at 200 kV [45].

© 2018 American Institute of Physics.

loss process is similar to halogen loss in halide materials with the formation of double halogen ion (H-center) by electron radiation [4,5,7].

With above results and discussion, it is clear that high-dose electron radiation can result in the damage to microstructure and the modification of chemical composition of silicon nitride thin films. Therefore we need to be cautious about the processes of PFA by TEM and the interpretation of the FA results related to silicon nitride. Otherwise, electron radiation-induced microstructure and phase

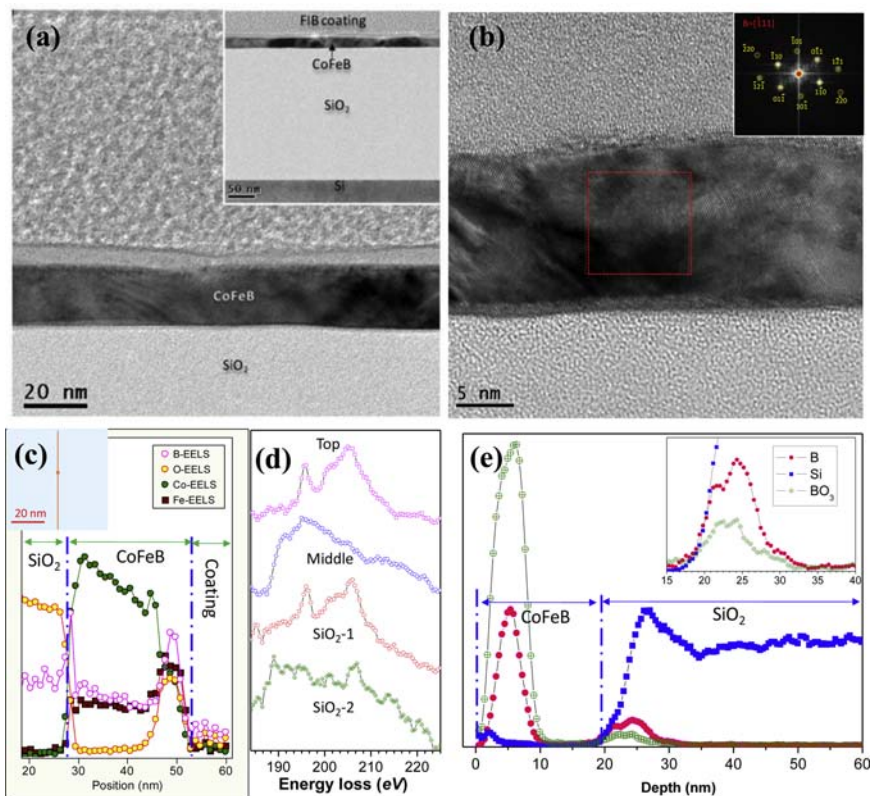
changes could give some misleading results related to some critical semiconductor structures such as ONO, as discussed above. To get authentic FA results associated with silicon nitride materials, we should take corresponding measures by using low-dose TEM technique as discussed in [Section 2.3](#). This is crucial for catching the authentic physical signatures of nitride process layers that are really correlated with the issues arising from the drift of semiconductor manufacturing processes by which we can understand the root cause of the electrical and reliability failure of the semiconductor devices.

2.5 Impact of electron beam radiation damage during TEM FA: boron diffusion and segregation induced phase and microstructure changes in CoFeB material

The CoFeB material has been receiving intensive research efforts since its first application in magnetic tunneling junction (MTJ) structures reported in 2005 [47]. As a ferromagnetic material, CoFeB is widely used as the magnetic free layer and reference layer in the MTJ heterojunctions. CoFeB/MgO-based MTJ has been implemented in commercial MRAM devices. Normally the CoFeB as-deposited in MTJ is of amorphous phase and thus suitable thermal annealing is needed in order to obtain high tunneling magnetoresistance ratio at room temperature. After thermal annealing, amorphous CoFeB will transform to polycrystalline Co_xFe_y phase with the majority of boron (B) out diffused to the neighboring process layers in MTJ stacks. Owing to the nanometer or subnanometer thickness of different process layers in MTJ stacks, TEM is essentially required for the PFA of any abnormality associated with the phase, composition and microstructure of MTJ film stacks, such as the roughness, uniformity, and crystallinity of thin films, interfacial material diffusion and segregation, and so on. Quite often it is necessary to perform high-resolution TEM (HRTEM) with spatially resolved elemental analysis by EELS and EDS. All these TEM analyses involve sample illumination by highly intensive electron beam or high-dose electron nanoprobe. Therefore under high-dose electron beam illumination, electron radiation could be a concern for the material characterization of CoFeB-based MTJ stacks. With the presence of B in amorphous CoFeB, partially crystallized Co_xFe_y , and some interfacial areas inside the MTJ stacks, electron beam radiation damage can lead to some unexpected diffusion and segregation of these B atoms, leading to some unusual changes in the phase and microstructures of MTJ film stacks [15].

The thin film stacks used for the electron radiation study was $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ (25 nm)/ SiO_2 (240 nm) deposited on Si [110] substrate by physical sputtering process. After annealing at 450°C, amorphous CoFeB was transferred to Co_3Fe phase with a thin layer of $\text{Co}_x\text{Fe}_y\text{O}_z$ on the surface which was formed due to thermal oxidation during thermal annealing, as shown in [Fig. 2.31A and B](#).

EELS and secondary ion mass spectrometry analysis revealed B segregation in both top oxide layer and the bottom $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface, as shown by two B

**FIGURE 2.31**

(A) Bright-field transmission electron microscope (TEM); (B) high-resolution TEM images; (C) scanning TEM-EELS line scan profiles; (D) boron EEL spectra at top oxide layer (top, middle, and bottom of the Co_3Fe film); and (E) secondary ion mass spectrometry depth profile of $\text{Co}_3\text{Fe}/\text{SiO}_2$ film after annealing at 450°C [15]. *EELS*, Electron energy loss spectroscopy; *FIB*, focus ion beam.

© 2017 AIP Publishing.

peaks in EELS line scan profile in Fig. 2.31C. As indicated in Fig. 2.31D, there existed two distinct B states: (1) B in oxidation state in both top oxide layer (marked as “top”) and the SiO_2 layer (marked as “ SiO_2 -1” in Fig. 2.31D) which is very close to the $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface; (2) metallic B in both Co_3Fe bulk film (marked as “middle” in Fig. 2.31D) and in bottom SiO_2 layer (marked as “ SiO_2 -2” in Fig. 2.31D) which is a bit away from the $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface.

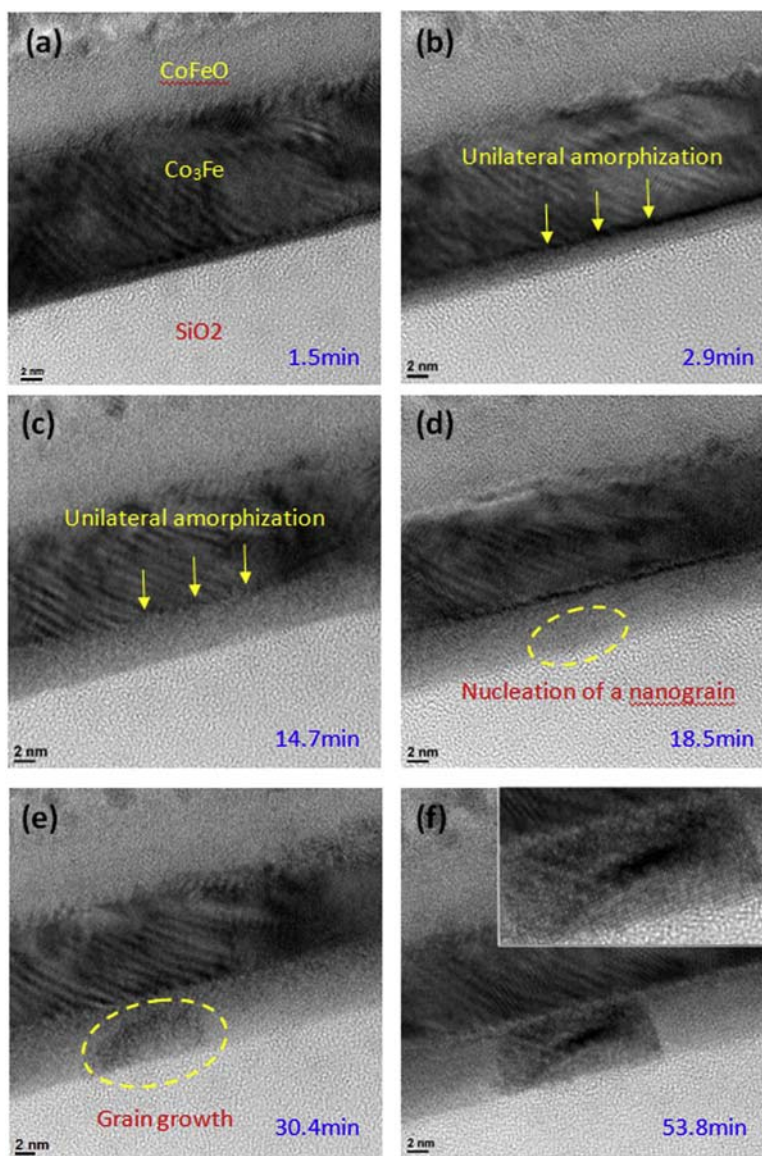
The electron beam radiation damage experiments were performed at 300 kV with the beam currents ranging from 5.88 to 17.7 nA. In situ TEM analysis under constant dose rates was carried out by recording TEM images for every 1- to 2-minute interval to examine the phase and microstructure evolution during electron

radiation. With the high-dose electron radiation, unusual phase transformation was observed in CoFeB thin film, from a polycrystalline Co_3Fe to a unilateral amorphous phase of Co_3Fe and then to nanocrystalline $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ phase. Fig. 2.32A–F shows the HRTEM images of the sample illuminated with beam current of 14.4 nA for different periods of time. Under such high-dose beam radiation, unusual unilateral amorphization of Co_3Fe film was observed along the interface of $\text{Co}_3\text{Fe}/\text{SiO}_2$. A thin amorphous layer was formed within 1.5 minutes after beam illumination, as shown in Fig. 2.32A. Further beam illumination led to the continuous thickening in amorphous layer. It is interesting to find out that prolonged beam illumination (18.5 minutes under 14.4 nA) led to the nucleation and formation of nanograins in the radiation-induced amorphous layer (Fig. 2.32D–F). Thus two different amorphization stages were identified for all the radiation experiments as shown in Fig. 2.33A, that is, fast initial unilateral amorphization stage (Stage-I) followed by a slow amorphization stage accompanying with the nucleation and growth of nanograins in the amorphous layer (Stage-II). The approximate linear time dependence of amorphous layer thickness indicates that such amorphization is possibly a diffusion-controlled process [46]. Fig. 2.33B shows the dependence of amorphization speeds of Co_3Fe film on the electron beam currents. Under all the radiation experimental conditions, the amorphization in Stage-I was always faster than that in Stage-II. This implies that the amorphization process in Stage-II might be retarded by the formation of a new crystalline phase. Moreover, with long-time electron radiation, both Co_3Fe and SiO_2 films showed apparent deformation, with hole drilling by electron beam in SiO_2 film. The continuous change in diffraction contrast and Moiré patterns in the crystalline portion of Co_3Fe films (Fig. 2.32F) indicates the microstructure changes during electron radiation, such as defect formation, materials diffusion, and microstrain, although HRTEM analysis did not reveal any phase change in the top crystalline Co_3Fe layer.

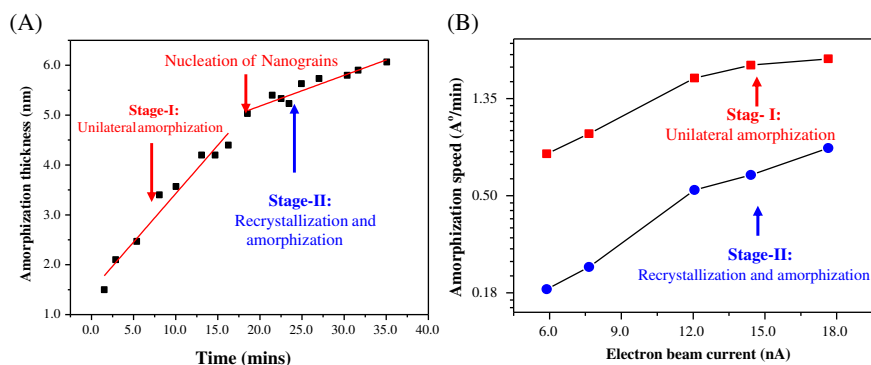
2.5.1 Stage-I: The electron radiation–induced unilateral amorphization of Co_3Fe thin film

Such unilateral amorphization behavior in Co_3Fe thin film could not be explained neither by the elastic knock-on damage nor by the inelastic ionization induced radiolysis and beam heating damage at 300 kV. Otherwise, all these kinds of radiation damage would be isotropic and globally occur across the whole Co_3Fe thin film layer rather than the unilateral amorphization behavior as observed.

Fig. 2.34A shows the EELS B maps of $\text{Co}_3\text{Fe}/\text{SiO}_2$ film after electron radiation for different periods of time. As shown in Fig. 2.34A, at the beginning of electron radiation (~ 0 minute), a thin layer with apparent B segregation was observed at the bottom of the Co_3Fe film along $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface. During the initial amorphization stage (e.g., 0–22 minutes in Stage-I and early Stage-II), the thickness of this B segregation layer continuously increased with beam

**FIGURE 2.32**

HR transmission electron microscopy micrographs of Co_3Fe film after beam illumination for different periods of time (14.4 nA, 300 kV): (A)–(C) continuous unilateral amorphization of Co_3Fe film along $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface; (D)–(F) nucleation and grain growth of nanocrystals in the amorphous layer [15].

**FIGURE 2.33**

(A) Time-dependent amorphization of Co_3Fe film under electron radiation at 14.4 nA and 300 kV. (B) The dependence of amorphization speed of Co_3Fe film on the electron beam current [15].

© 2017 AIP Publishing.

illumination, and further radiation led to the disappearance of B segregation at the bottom of Co_3Fe film in late Stage-II amorphization (e.g., 52 minutes in Fig. 2.34A). While in the top $\text{Co}_x\text{Fe}_y\text{O}_z$ layer, there was no significant change in the B segregation layer thickness even after beam illumination for 52 minutes. Most importantly, both TEM and STEM analyses indicate that the thickness of B segregation layer is same as that of amorphous layer induced by electron radiation, as shown by B maps (0–22 minutes) in Fig. 2.34A and B. The fact implies that the radiation-induced unilateral Co_3Fe amorphization was correlated with the B segregation along $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface. This is further verified by EELS line scan analysis in Fig. 2.34C, in which the B line scan profiles also clearly shows the continuous increase in the thickness of B segregation layer at the bottom of Co_3Fe film. While for the B segregation in the top $\text{Co}_x\text{Fe}_y\text{O}_z$ oxide layer, the line scan analysis shows somewhat broadened B distribution but essentially no drastic change is observed in terms of B segregation layer thickness with electron radiation up to 52 minutes. This is consistent with EELS mapping analysis, as shown in Fig. 2.34A.

Further detailed analysis revealed that B segregation at the bottom of Co_3Fe film arose from the back diffusion of metallic B atoms in the bottom SiO_2 layer [15]. Under electron beam radiation at 300 kV, these B atoms were knocked out by head-on electron collision, and diffused preferably to Co_3Fe thin film layer due to large B diffusivity in Co/Fe materials than that in SiO_2 [48–51]. The driving force behind the B diffusion was verified to be RED effects [15]. These B atoms segregated at the Co_3Fe film bottom directly resulted in large structural disordering due to the low solubility of B atoms in CoFe matrix (<1%) [52]. Therefore the unilateral amorphization of Co_3Fe film occurred along $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface, as shown in Fig. 2.32A–F.

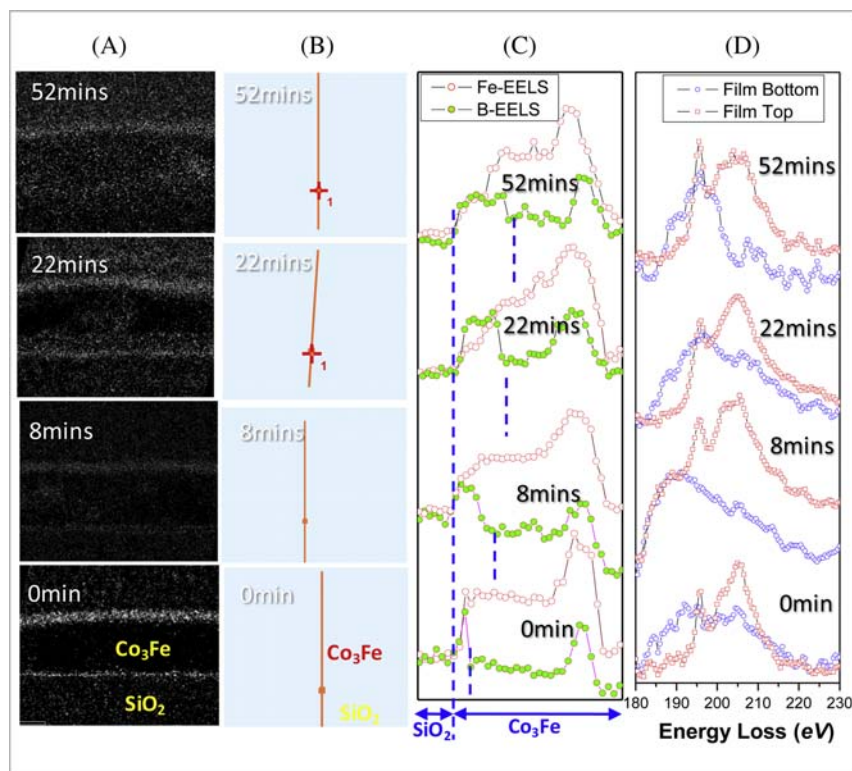


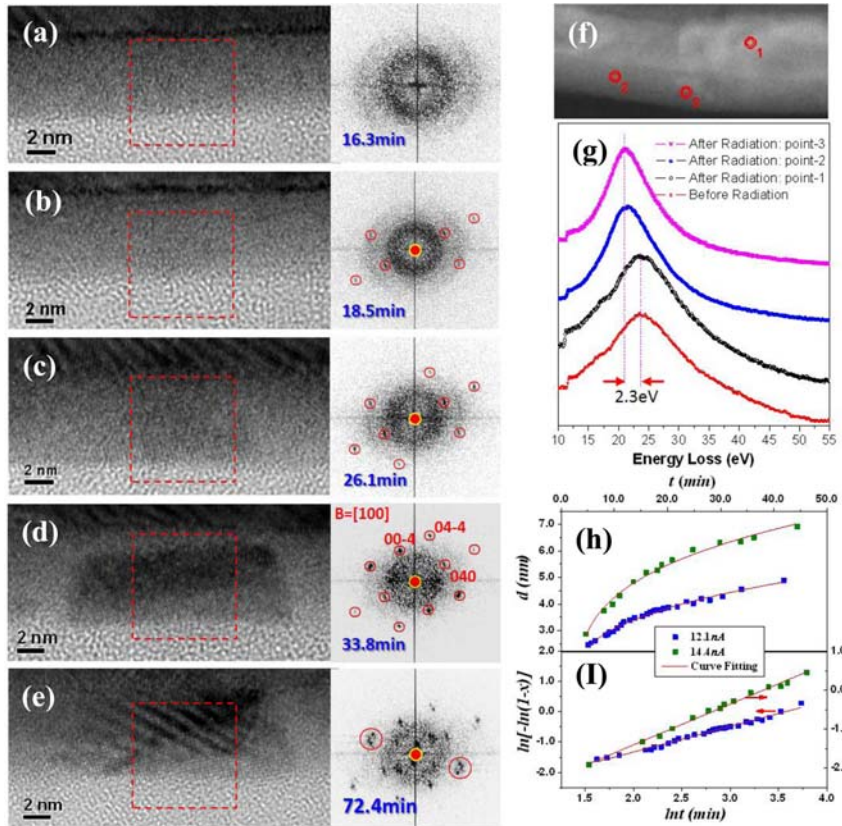
FIGURE 2.34

(A) Energy-filtered transmission electron microscope (TEM)-EELS boron maps; (B) scanning TEM Z-contrast images; (C) EELS line scan profiles of B and Fe; and (D) B EEL spectra after beam radiation for different periods of time under beam radiation at 14.4 nA and 300 kV [15].

© 2017 AIP Publishing.

2.5.2 Stage-II: The electron radiation–induced recrystallization in the amorphized Co_3Fe thin film

During Stage-II, the electron radiation–induced amorphization of Co_3Fe thin film slowed down, accompanying with the nucleation and growth of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins from the amorphous layer, as shown in Figs. 2.32D–F and 2.33. Higher electron beam current led to earlier nucleation and faster growth of nanograins. Fig. 2.35A–E shows HRTEM micrographs of the nuclei formation and grain growth in the amorphous layer of the Co_3Fe film at 14.4 nA and 300 kV. It was found that the nuclei formation was fast and started with a weak diffraction-like contrast in the amorphous matrix with somewhat short-range ordering evidenced by weak lattice fringes. The FFT pattern in Fig. 2.35B indicates the formation of

**FIGURE 2.35**

(A)–(E) High-resolution transmission electron microscopy images showing the nucleation and grain growth of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins from amorphous Co_3Fe layer with the electron beam radiation at 14.4 nA and 300 kV; (F) scanning TEM images and (G) electron energy loss spectroscopy low-loss spectra of the Co_3Fe film collected after radiation for ~ 52 min; (H) time-dependent grain growth at 14.4 nA and 300 kV; and (I) Johnson–Mehl–Avrami plots, $\ln[-\ln(1-x)]$ versus $\ln t$ at 14.4 nA and 300 kV [15].

© 2017 AIP Publishing.

two sets of crystalline planes with an angle of 45 degrees in-between them. With the continuous electron radiation up to ~ 26 minutes, one more set of crystal plane was formed, as shown in Fig. 2.35C. The streaky diffraction spots in Fig. 2.35B and C indicated a defective microstructure of the nanograin in the initial stage.

With further beam illumination to ~ 33 minutes, a well-ordered structure of the nanograin was formed, and its phase was cubic $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ with a zone axis along [100], as shown in Fig. 2.35D. The formation of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ phase was

also evidenced by the energy shift in the low-loss EEL spectra shown in Fig. 2.35F and G. The plasmon peak of the $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ phase was centered at ~ 20.9 eV, around 2.3 eV shift with respect to that of the crystalline Co_3Fe phase. The $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ plasmon energy of ~ 20.9 eV agreed with that of FeB compounds reported by Bratkovsky et al. [53]. The red shift of the Plasmon energy indicates a lower density of outer shell electrons with the formation Co–Fe–B chemical bonding compared with the metallic Co_3Fe phase.

Further electron radiation resulted in more and more nanograins formed in the amorphous matrix. Complex phase contrast such as the Moiré fringes were observed in the $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins, as is evidenced by the double diffraction in FFT pattern in Fig. 2.35E. Such Moiré fringes indicated the formation of additional nanograins overlapping with the initial nanograin. Prolonged electron radiation experiment showed that the initial amorphous layer formed in Stage-I became fully crystallized with a polycrystalline structure. With the formation of such fully polycrystalline structure, there was no further amorphization of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ and Co_3Fe phases even after electron radiation for 3 hours event with electron beam currents up to 17.7 nA in this study. The results indicated that the newly formed $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ phase was stable under electron radiation at 300 kV. It has been reported that the displacement threshold energy of Fe in Fe–B is $\sim 22 \pm 3$ eV, higher than the maximum electron energy transfer (E_{max}) of 11.8 eV for Fe at 300 kV as calculated above. Therefore no amorphization of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ phase could occur by the radiation-induced knock-on effects. While for the reason why no further amorphization of Co_3Fe film in the final Stage-II, it could be attributed to electron radiation–induced B redistribution with long-time electron radiation. As mentioned above, during Stage-I and the initial Stage-II, radiation-induced RED effects drove fast B diffusion from the bottom SiO_2 layer, leading B segregation at the bottom of Co_3Fe film and the unilateral Co_3Fe amorphization. However, with long-time electron radiation till the late Stage-II, apparent B redistribution occurred, that is, the high concentration of B originally segregated at the bottom of Co_3Fe film gradually diffused around, leading to overall relatively uniform B distribution throughout the whole film. This was evidenced by the EELS mapping and EELS line scan analysis shown in Fig. 2.34A and C, in which the high concentration of B in the segregation layer started to diffuse out after beam illumination for 52 minutes. On the other hand, the formation of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ phase in the amorphous layer may also slow down the amorphization process of Co_3Fe film, by which some B atoms were trapped with the formation of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins without further diffusion to the top crystalline Co_3Fe layer. This also helps to explain why the amorphization speed in Stage-II was slower than that in Stage-I.

To understand the kinetic behaviors of the radiation-induced nanocrystallization, we estimated that the maximum temperature induced by the highest beam current of 17.7 nA was only around 1.1°C in the CoFe film [15], which was too low to generate any thermal ordering of the amorphous phase. Therefore further investigation is needed. Fig. 2.35H shows the time-dependent grain growth under

two different electron beam currents of 7.1 and 14.4 nA. The grain size was estimated based on in situ HRTEM analysis by mapping out the 2D area occupied by the nanograins in HRTEM images. In terms of conventional thermal annealing processes, the kinetic isothermal grain growth can be generally described by the classic empirical model as below [54]:

$$d^n - d_0^n = K_T(t - t_0) \quad (2.5)$$

where d is the average grain size at time t , d_0 is the average grain size at t_0 , n is the grain growth exponent, and k_T is a kinetic constant. In case of conventional thermal annealing process, K_T can be expressed by:

$$K_T = K_0 \exp\left(\frac{E}{RT}\right) \quad (2.6)$$

where K_0 is a constant, E is the activation energy for grain growth, R is the gas constant, and T is the annealing temperature. In our radiation experiments, since there were no significant electron heating effects, K_T can be considered as a dose-rate dependent constant. By fitting the experimental data in Fig. 2.35H with Eq. (2.5), the as-fitted grain growth exponents n are around 3.37 and 3.47 for the radiation experiments at 7.1 and 14.4 nA, respectively, close to each other. While for the kinetic constants K_T , the fitted values showed a big difference, around 5.0 for the case of 7.1 nA and 25.7 for the case of 14.4 nA. The close value of the grain growth exponent n indicated that grain growth in these two radiation experiments followed a similar kinetic process, namely diffusion-controlled grain growth mechanism with n close to 3 [54]. The large difference in the kinetic constants k_T once again implies the dose-rate sensitive grain growth behaviors. With referring to Eq. (2.5), higher electron dose rate may effectively lower down the energy barrier for the nucleation and growth of the $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins during the electron radiation.

To further understand the kinetic process of the radiation-induced nanocrystallization of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ phase, further analysis was performed by using the well-known phenomenological Johnson–Mehl–Avrami (JMA) model [55–57]. Based on the JMA model, the time-dependent phase transformation or grain growth can be described by Eq. (2.7):

$$x(t) = 1 - \exp(-kt^n) \quad (2.7)$$

where x is the volume fraction of crystalline phase in amorphous matrix, t is the time, k is the kinetic constant which is related to the temperature and activation energy, and n is the Avrami exponent which is associated with the dimensionality of crystals and the mechanism of nucleation and grain growth. For the electron radiation experiments, it was assumed that $x(t)$ to be the ratio of the 2D area occupied by a nanograin at time t to that of the nanograin captured by the final in situ HRTEM analysis in the experiment. To determine Avrami exponent n , Eq. (2.7) can be transferred to

$$\ln[-\ln(1 - x(t))] = \ln k + n \ln t \quad (2.8)$$

Based on Eq. (2.8), Avrami exponent n can be determined as the slop of $\ln[-\ln(1-x(t))] \sim \ln t$ plots, which are shown in Fig. 2.35I. The obtained Avrami exponents are 0.85 and 1.05 for 7.1 nA at 14.4 nA radiation experiments, respectively. Therefore in both cases, the Avrami constant can be considered as 1. Such a low value of Avrami constant indicates that surface crystallization is the dominating kinetic process and the atomic diffusion may involve in the radiation-induced amorphous to nanocrystallization transformation process [58,59].

In terms of the above discussion, both the classic empirical model and JMA model reveal the surface crystallization and diffusion-controlled nucleation and grain growth mechanism behind the radiation-induced $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanocrystallization process. Although it is impossible to directly observe atomic diffusion of B, Fe, and Co atoms by in situ HRTEM analysis, it is interesting to find out that the nuclei of the $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins always first appeared at the interface between amorphous Co_3Fe film and SiO_2 , as is shown in Fig. 2.32. With continuous electron radiation, the nanograins gradually grew toward to the interface between crystalline Co_3Fe and amorphous CoFeB layers, as is evidenced by the HRTEM images in Fig. 2.32D–F. The preferable nucleation of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins at the interface of amorphous $\text{Co}_3\text{Fe}/\text{SiO}_2$ could be the direct evidence for the surface crystallization kinetics as revealed by JMA model, if we consider amorphous $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface as the surface of amorphous Co_3Fe film where nucleation of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins started. While for the atomic diffusion mechanism, it may be correlated with the RED effects induced by electron radiation. As mentioned earlier, with the RED effects induced by energetic electron radiation, the point defects as diffusion carrier are constantly generated if the electron dose rate is above the threshold one. On the other hand, it is well known that the grain boundaries and phase boundaries are always effective sinks for the point defects and defect clusters. During electron radiation, there are always certain amounts of point defects diffusing to the $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface and trapped there. During Stage-I amorphization process, such interface point-defect sinking effects may not effectively affect RED-assisted B diffusion with large population of B atoms knocked out from SiO_2 layer with electron radiation. While during Stage-II (amorphization and nanocrystallization), the prolonged electron radiation resulted in not only the redistribution of B atoms, but also more and more point defects trapped in $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface. With high-concentration of point defects at $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface, the atomic diffusion will be substantially enhanced near the interface, which we suspected to be the reason accounting for the preferable nucleation of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins at the interface. It has been widely reported that highly energetic electron irradiation can substantially enhance the atomic diffusivity in the amorphous state even at a low temperature, and in some cases, an increase of many orders of magnitude was observed [60]. Moreover, as pointed out by Uberuaga et al., the enhanced diffusion in polycrystalline materials is at least in part governed by higher defect concentrations at interfaces. Even if the mobility per defect could be slower, the overall diffusion constant could still be

faster [61]. With such radiation-enhanced atomic diffusivity and high concentration of radiation-induced point defect at $\text{Co}_3\text{Fe}/\text{SiO}_2$ interface, the local short-range ordering of Fe, Co, and B atoms becomes possible, favoring the nucleation and growth of $\text{Fe}_x\text{Co}_{23-x}\text{B}_6$ nanograins.

Compared with the several tens of film stacks in commercial MTJ devices, the $\text{CoFeB}/\text{SiO}_2$ thin-film stack discussed earlier is definitely much simple and thus the in situ TEM experimental study on the electron radiation effects is much more straightforward. However, the electron radiation induced complex physical and chemical processes occurred in the $\text{CoFeB}/\text{SiO}_2$ thin film stack could be the embodiments of the possible similar phase and microstructure changes in the real MTJ devices during TEM-PFA of MTJ devices. During our TEM FA of MTJ devices, it was constantly observed that high-dose electron beam led to fast degradation of the contrast between different process layers, and even changes in interfacial microstructure and the crystallinity of some process layers in the MTJ stacks. The underlying reasons may lie in the electron radiation enhanced material diffusion, namely RED effects. The main culprits are the large amount of B and its species which were out diffused from CoFeB layers after thermal annealing and segregated at the nearby interfaces. In addition, some materials used in the MTJ devices possess high diffusivity, and thus electron radiation readily induces their diffusion to the neighboring process layers. All these will greatly impact the authenticity of the TEM PFA results. Therefore to get the native physical failure signatures of CoFeB -related MTJ devices, it is necessary to take note of the electron radiation effects when performing TEM analysis. On the one hand, the analyzer should avoid the unnecessary electron radiation with a high electron dose during TEM analysis. On the other hand, when performing the analysis requiring high-dose electron beam such as elemental analysis by EDS and EELS, the whole analysis processes should be done as fast as possible to minimize the radiation-induced artificial FA signatures. Some analysis even requires multiple samples in order to obtain solid TEM FA results. Most importantly, the analyzer should be always cautious about the results interpretation, and the correlation of TEM PFA results with those of EFA is the key to understand the real mechanisms behind the MTJ device failure.

2.6 Conclusion

In this chapter, we discussed the electron beam radiation damage and its impacts on semiconductor FA by SEM, FIB, and TEM. Although the topic we discussed focused on semiconductor FA, the phenomena of electron beam radiation damage and its associated impacts may apply to the characterization and analysis of other type of electron beam-sensitive materials by electron beam-based analysis techniques. For semiconductor FA, as all we know, the task is to acquire the key

failure signatures associated with manufacturing processes and device operation. Hence, to get authentic physical signatures, understanding the beam sensitivity of the materials in the target structures or devices is essential. With understanding the nature of materials and physics behind the electron beam radiation damage, we can take corresponding measures to reduce or minimize the electron beam radiation damage—induced artificial effects. In general, the technical solutions include both voltage control and dose control, as well as cryo-SEM/FIB/TEM imaging techniques, commonly used for the imaging and analysis of biological samples, as discussed in [Sections 2.2](#) and [2.3](#). For the material characterization with intensive electron beam illumination such as TEM analysis at high magnifications, the high electron dose and highly energetic electron beam can induce the modification of microstructure, phase and composition of the materials. Depending on the physical and chemical nature of the materials and analytical conditions, such electron beam radiation damage may occur fast or advance gradually, and sometimes even almost invisible when electron dose is relatively low, just like silicon nitride and CoFeB materials as discussed in [Sections 2.4](#) and [2.5](#). To deal with such gradual radiation-induced material damage, as microscopists and material scientists, we need to have a good understanding of physical and chemical properties of materials to be analyzed, and keep aware of any appreciable microstructural changes during TEM analysis. Only in this way can the analytical results be really native and authentic.

References

- [1] G.R. Wale, Diffusion-limited annealing of radiation damage in germanium, *Phys. Rev.* 107 (1957) 471.
- [2] P.B. Price, Direct observation of ion damage in cadmium, *Phys. Rev. Lett.* 11 (1961) 615.
- [3] V.E. Cosslett, Radiation damage in the high resolution electron microscopy of biological materials: a review, *J. Microsc.* 113 (1978) 113.
- [4] L.W. Hobbs, Radiation effects in analysis by TEM, in: J.J. Hren, J.I. Goldstein, D.C. Joy (Eds.), *Introduction to Analytical Electron Microscopy*, Plenum Press, New York, 1979, p. p. 399.
- [5] R.F. Egerton, P. Li, M. Malac, Radiation damage in the TEM and SEM, *Micron* 35 (2004) 399–409.
- [6] L.E. Thomas, Light-element analysis with electrons and X-rays in a high-resolution STEM, *Ultramicroscopy* 18 (1985) 173–184.
- [7] R.F. Egerton, Control of radiation damage in the TEM, *Ultramicroscopy* 127 (2013) 100.
- [8] R.S. Averback, H. Hahn, Radiation-enhanced diffusion in amorphous Ni-Zr alloys, *Phys. Rev. B* 37 (1988) 10383.
- [9] S. Bellini, A. Montone, M. Vittori-Antisari, Radiation-enhanced diffusion in amorphous Ni-Zr studied by in situ electron irradiation in a transmission electron microscope, *Phys. Rev. B* 50 (1994) 9803.

- [10] G.J. Dienes, A.C. Damask, Radiation enhanced diffusion in solids, *J. Appl. Phys.* 29 (1958) 1713.
- [11] S. Loualich, C. Lucas, P. Baruch, J.P. Gailliard, J.C. Pfieste, Theoretic model for radiation enhanced diffusion and redistribution of impurities. Comparison with experiments, *Phys. Status Solidi A* 69 (1982) 663.
- [12] P. Lévêque, J.S. Christensen, A.Yu Kuznetsov, B.G. Svensson, A. Nylandsted Larsen, Influence of boron on radiation enhanced diffusion of antimony in delta-doped silicon, *J. Appl. Phys.* 91 (2002) 4073.
- [13] J. Shim, J.A. Rivera, Rashid Bashir, Electron beam induced local crystallization of HfO₂ nanopores for biosensing applications, *Nanoscale* 5 (2013) 10887.
- [14] A. Nino, T. Nagase, Y. Umakoshi, Electron irradiation induced crystallization and amorphization in Fe₇₇Nd_{4.5}B_{18.5} metallic glass, *Mater. Sci. and Eng. A* 449–451 (2007) 1115.
- [15] B.H. Liu, H.W. Teo, Z.H. Mo, Z.H. Mai, J. Lam, J.M. Xue, et al., In-situ TEM study of electron beam radiation damage induced boron diffusion and effects on phase and microstructure evolution in nanostructured CoFeB/SiO₂ thin film, *J. App. Phys.* 121 (2017) 01511.
- [16] L. Reimer, *Scanning Electron Microscopy: Physics of Image Formation and Microanalysis*, second ed., Springer Press, New York, 1998, p. 117.
- [17] K. Kanaya, K. Adachi, K. Yonehara, Y. Muranaka, H. Ishikawa, *Micron and Microscopia Acat* 22 (1991) 223.
- [18] S.B. Fisher, On the temperature rise in electron irradiated foils, *Radiat. Eff.* 5 (1970) 239.
- [19] I. Jenčič, M.W. Bench, I.M. Robertson, M.A. Kirk, Electron-beam-induced crystallization of isolated amorphous regions in Si, Ge, GaP, and GaAs, *J. Appl. Phys.* 78 (1995) 974.
- [20] “Executive Summary.” International Technology Roadmap for Semiconductors, published by the Semiconductor Industry Association, 2005.
- [21] “Interconnect.” In International Technology Roadmap for Semiconductors, published by the Semiconductor Industry Association, 2005.
- [22] K. Maex, M.R. Baklanov, D. Shamiryan, F. Lacopi, S.H. Brongersma, Z.S. Yanovitskaya, Low dielectric constant materials for microelectronics, *J. Appl. Phys.* 93 (2003) 8793.
- [23] R. Doering, Y. Nishi, *Handbook of semiconductor manufacturing technology*, second ed., Tylor & Francis Group, Boca Raton, London and New York, 2008, pp. 13–31.
- [24] B.D. Hatton, K. Landskron, W.J. Hunks, M.R. Bennett, D. Shukaris, D.D. Perovic, et al., Materials chemistry for low-k materials, *Materials Today* 9 (2006) 22.
- [25] D.T. Grubb, Radiation damage of organic materials in the transmission electron microscopy, *Ultramicroscopy* 12 (1984) 279.
- [26] C.M. Wang, D.R. Baer, J.E. Amonette, M.H. Engelhard, J.J. Antony, Y. Qiang, *Ultramicroscopy* 108 (2007) 43.
- [27] W.K. Chu, J.W. Mayer, M.A. Nicolet, *Backscattering Spectrometry*, Hardcourt Brace Juvanovich, Boston, Tokyo, 1982.
- [28] A. Delan, M. Rennau, S.E. Schulz, T. Gessner, Thermal conductivity of ultra-low k dielectrics, *Microelectronic Eng.* 70 (2003) 280.
- [29] R.A. Knief, *Nuclear Energy Technology*, McGraw-Hill, 1981, p. 72.
- [30] I. Müllerová, L. Frank, Scanning low-energy electron microscopy, *Adv Imag. Electr. Phys.* 128 (2003) 309.

- [31] L. Reimer, *Scanning Electron Microscopy: Physics of Image Formation and Microanalysis*, second ed., Springer Press, New York, 1998, p. 83.
- [32] B.H. Liu, Z.Q. Mo, Y.N. Hua and J. Teong, Optimization of SEM analytical conditions for low k and ULK dielectric materials. *Proceedings from the 34th International Symposium for Testing and Failure Analysis*, November 2–6, 2008, Portland, OR, p. 291.
- [33] B.E. Bammes, J. Jakana, M.F. Schmid, W. Chiu, Radiation damage effects at four specimen temperatures from 4 to 100 K, *J. Struct. Biol.* 169 (2010) 331.
- [34] J.Z. Chen, C. Sachse, C. Xu, T. Mielke, C.M.T. Sphahn, N. Grigorieff, A dose-rate effect in single-particle electron microscopy, *J. Struct. Biol.* 161 (2008) 92.
- [35] B. Deng, C.M. O'Connor, D.H. Kedes, Z.H. Zhou, Cryo-electron tomography of Kaposi's sarcoma-associated herpesvirus capsids reveals dynamic scaffolding structures essential to capsid assembly and maturation, *J. Struct. Biol.* 161 (2008) 419.
- [36] S.E. Thompson, M. Armstrong, C. Auth, S. Cea, R. Chau, G. Glass, et al., A logic nanotechnology featuring strained-silicon, *IEEE Elect. Dev. Lett.* 25 (2004) 191.
- [37] S.E. Thompson, M. Armstrong, C. Auth, M. Alavi, M. Buehler, R. Chau, et al., A 90-nm logic technology featuring strained-silicon, *IEEE Trans. ED* 51 (2004) 1790.
- [38] M.C. Lee, H.Y. Wong, Charge loss mechanisms of nitride-based charge trap flash memory devices, *IEEE Trans. Elec. Dev.* 60 (2013) 3256.
- [39] H. Tanaka, H. Uchida, T. Ajioka, N. Hirashita, The degradation of TDDB characteristics of $\text{SiO}_2/\text{Si}_3\text{N}_4/\text{SiO}_2$ stacked films caused by surface roughness of Si_3N_4 films, *IEEE Trans. Elec. Dev.* 40 (1993) 2231.
- [40] Y. Yang, A. Purwarb, M.H. White, Reliability considerations in scaled SONOS non-volatile memory devices, *Solid-State Electronics* 43 (1999) 2025.
- [41] L. Grifoni, P. Massimi, F. Fama, Si_3N_4 thickness and its oxidation time influence on O/N/O DRAM cell performance, *J. Non-cryst Solids* 216 (1997) 202.
- [42] M.Y. Wu, D. Krapf, M. Zandbergen, H. Zandbergen, P.E. Batson, Formation of nanopores in a SiN/SiO_2 membrane with an electron beam, *Appl. Phys. Lett.* 87 (2005) 113106.
- [43] Y. Kuznetsova, M. Zamoryanskaya, Unstable luminescence of nitrides under electron-beam irradiation, *Jpn. J. Appl. Phys.* 52 (2013) 08JJ06.
- [44] F. Fransen, R. Vanden Berghe, R. Vlaeminck, M. Hinoul, J. Remmerie, H.E. Maes, Electron and ion beam degradation effects in AES analysis of silicon nitride thin films, *Surf. Int. Anal.* 7 (1985) 79.
- [45] B.H. Liu, Z.L. Dong, Y.N. Hua, C. Fu, X.M. Li, P.K. Tan, et al., Electron beam radiation damage induced degradation of silicon nitride and its impact to semiconductor failure analysis by TEM, *AIP Adv.* 8 (2018) 115327.
- [46] U. Gosele, K.N. Tu, Growth kinetics of planar binary diffusion couples: thin-film case versus bulk cases, *J. Appl. Phys.* 53 (1982) 3252.
- [47] D.D. Djayaprawira, K. Tsunekawa, M. Nagai, H. Maehara, S. Yamagata, N. Watanabe, et al., 230% room-temperature magnetoresistance in $\text{CoFeB}/\text{MgO}/\text{CoFeB}$ magnetic tunnel junctions, *Appl. Phys. Lett.* 86 (2005) 092502.
- [48] K.A. Ellis, R.A. Buhrman, Boron diffusion in silicon oxides and oxynitrides, *J. Electrochem. Soc.* 145 (1998) 2068.
- [49] T. Aoyama, K. Suzuki, H. Tashiro, Y. Toda, T. Yamazaki, K. Takasaki, et al., Effect of fluorine on boron diffusion in thin silicon dioxides and oxynitride, *J. Appl. Phys.* 77 (1995) 417.

- [50] T. Aoyama, K. Suzuki, H. Tashiro, Y. Toda, T. Yamazaki, K. Takasaki, et al., Boron diffusion through pure silicon oxide and oxynitride used for metal-oxide-semiconductor devices, *J. Electrochem. Soc.* 12 (1993) 3624.
- [51] R.B. Mclellan, C. Ko, The diffusion of boron in f.c.c iron, *J. Phys. Chem. Sol.* 54 (1993) 465.
- [52] Y. Lu, B. Lépine, G. Jézéquel, S. Ababou, M. Alnot, J. Lambert, et al., Electronic structure of nanopolycrystalline pulsed laser deposited LaB₆ films and single crystals: the boron perspective, *J. Appl. Phys.* 108 (2010) 043703.
- [53] A.M. Bratkovsky, N. Rashev, A.V. Smirnov, G. Wendin, Universality in electronic structure and EELS spectra of Fe-B and Ni-B crystalline and amorphous systems, *Europhys. Lett.* 26 (1994) 43.
- [54] L. Lu, N.R. Tao, L.B. Wang, N.Z. Ding, K. Lu, Grain growth and strain release in nanocrystalline copper, *J. Appl. Phys.* 89 (2001) 6408.
- [55] W.A. Johnson, R.F. Mehl, *Trans. Am. Inst. Min., Metall. Pet. Eng.* 135 (1939) 416.
- [56] M. Avrami, M. Granulation, Granulation, phase change, and microstructure kinetics of phase change. III, *J. Chem. Phys.* 9 (1941) 177.
- [57] H. Yinnon, D.R. Uhlmann, Applications of thermoanalytical techniques to the study of crystallization kinetics in glass-forming liquids, part I: Theory, *J. Non-Cryst. Solids* 54 (1983) 253.
- [58] M. Çelikkilek, A.E. Ersundu, N. Solak, S. Aydin, Crystallization kinetics of the tungsten—tellurite glasses, *J. Non-Cryst. Solids.* 357 (2011) 88.
- [59] J.Z. Jiang, Y.X. Zhuang, H. Rasmussen, Formation of quasicrystals and amorphous-to-quasicrystalline phase transformation kinetics in Zr₆₅Al_{7.5}Ni₁₀Cu_{7.5}Ag₁₀ metallic glass under pressure, *Phys. Rev. B* 64 (2001) 094208.
- [60] Z.Q. Bai, L. Shen, Q.Y. Wu, M.G. Zeng, J.S. Wang, G.H. Han, et al., Boron diffusion induced symmetry reduction and scattering in CoFeB/MgO/CoFeB magnetic tunnel junctions, *Phys. Rev. B* 87 (2013) 014114.
- [61] B.P. Uberuaga, L.J. Vernon, E. Martinez, A.F. Voter, *Scientific Report* 5 (2015) 9095.

This page intentionally left blank

Failure of intermetallic solder ball due to stress shielding and amplification effects

E.P. Ooi¹, R. Daud¹, N.A.M. Amin¹, M.S. Abdul Majid¹, M. Afendi¹, A. Mohamad¹
and A.K. Ariffin²

¹Fracture and Damage Mechanic Research Group, School of Mechatronic Engineering,
Universiti Malaysia Perlis, Arau, Malaysia

²Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, Bangi,
Malaysia

3.1 Introduction

One of the major factors causing microelectronic products failure is solder joint fracture due to thermomechanical stress. Recently, many of the intermetallic compounds (IMCs) have been found to grow on the interface between the pad and the solder material [1]. The brittle property of IMCs causes the strong stress concentration effect during the mechanical impacts [1–13] and thermal cycles [14,15]. The cracks were found to initiate and propagate near the IMC layers. Thus the presence of IMC has greatly influenced the solder joints reliability and many studies [1–4,6,7,11–15] were based on it. There are few other factors that influence the fracture toughness of solder joint, such as solder joint thickness and crack location related to an interface of the solder joint. Alam et al. [2] have showed that the stress intensity factors (SIFs) (e.g., K_I and K_{II}) increase with the thickness of solder alloys at the loading rate of 0.8 MPa/s for the crack located at the middle of IMC layer. Alam et al. [2] also highlighted that the nearer the crack location to the interface, the higher will be the SIF values (both K_I and K_{II}). K_{II} increases to a very high value that could lead to a very unstable crack due to its higher creep resistance. Thus the simulation result proves that the crack near the interface always has more tendencies to propagate.

Nadimpalli and Spelt[3] highlighted the effect of geometry on the fracture behavior of solder joints by changing the thickness of lead-free solder joints, there was no effect on the initiation strain energy release rate, G_{ci} , if the solder length is short.

Li et al. [16] found that a smaller grain size is beneficial to the generation of voids at the Cu_3Sn/Cu interface, because there are much more diffusion paths to

facilitate the effective vacancies through grain boundary to coalesce to form voids. Therefore more voids will be formed as the solder joint is being heated by electrical concentric stress during the operation of electronic devices. Liang et al. [17] developed a diffuse interface model of solder joint to simulate the effect of an electric field on the morphological evolution migration behavior of the microvoid in the solder which causes migration of the microvoid in the Sn/Cu solder interface. He also found that the migration velocity of the microvoid increases directly with the voltage but inversely proportional to the size of the microvoid. The studies also noticed that a small microvoid can combine with an adjacent big microvoid, and merge to form a larger microvoid, which may lead to an open-circuit failure near the solder/Cu interface. The coupled effect of surface diffusion and electric field revealed that the coalescence of microvoids driven only by surface energy occurs when the microvoids contact with one another.

Most of the simulation models and calculations were used based on a single crack [2] and less interest were put on multiple crack behaviors. Therefore more focus is required on the interaction between multiple cracks in solder ball which was lately found on the morphology of the IMC grain formed by the electric stress and reflow heat effect [1,16–21].

This study aims to investigate the effect of the interaction between multiple edge cracks in solder joint to measure the shielding and amplification effects based on the stress singularity approach. A finite element (FE) model of two parallel edge cracks subjected to shear loading was developed. The effect of crack length between two parallel edge cracks on the SIF was evaluated. The interaction between the two colinear cracks was also evaluated. In addition, the effect of inner crack length and vertical distance were highlighted.

3.2 Methodology

3.2.1 Finite element modeling

3.2.1.1 Finite element modeling based on Brown & Srawley analytical model

Following the same optimization method as Srawley analytical mathematical equation, the optimum dimension for a numerical model is obtained from the modeling process based on Brown & Srawley analytical equation.

According to Brown & Srawley analytical equation, C , the SIF value is calculated as:

$$K_{BS} = C\sigma\sqrt{\pi a} \quad (3.3)$$

$$C = 1.12 - 0.23\left(\frac{a}{w}\right) + 10.6\left(\frac{a}{w}\right)^2 - 21.7\left(\frac{a}{w}\right)^3 + 30.4\left(\frac{a}{w}\right)^4 \quad (3.4)$$

where K_{BS} is the SIF for Brown & Srawley model, C is the single edge correction factor (constant), σ is the loading applied, a is the crack length, and w is the width of solder.

An FE test model with geometry height 3.0 mm and width 2.0 mm, and IMC thickness of 0.010 mm is set up, as shown in Fig. 3.1. A crack with a length of 0.2–1.8 mm is introduced to study the SIF at the crack tip where a tensile stress σ of 6 MPa is applied on both upper and bottom surfaces.

3.2.1.2 Finite element solder joint modeling

In order to simulate and characterize the typical solder joint, one solder joint FE model which was validated with Brown & Srawley analytical equation has been developed. The FE analysis was conducted using ANSYS Release13. The FE model involved three types of materials—copper, lead-free tin–silver–copper (SnAgCu), and IMC. The meshing scheme was set to isoperimetric quadrilateral elements (PLANE 183) with Barsoum singularity element around the crack tip. Table 3.1 shows the material properties of solder joints. Copper and IMC layer are isotropic linear elastic materials, while solder matter is a viscoplastic material. This analysis is based on static loading without applying loading rate and thermal effect. A crack is introduced at the left middle edge of the IMC layer in order to measure the SIF at its crack tip.

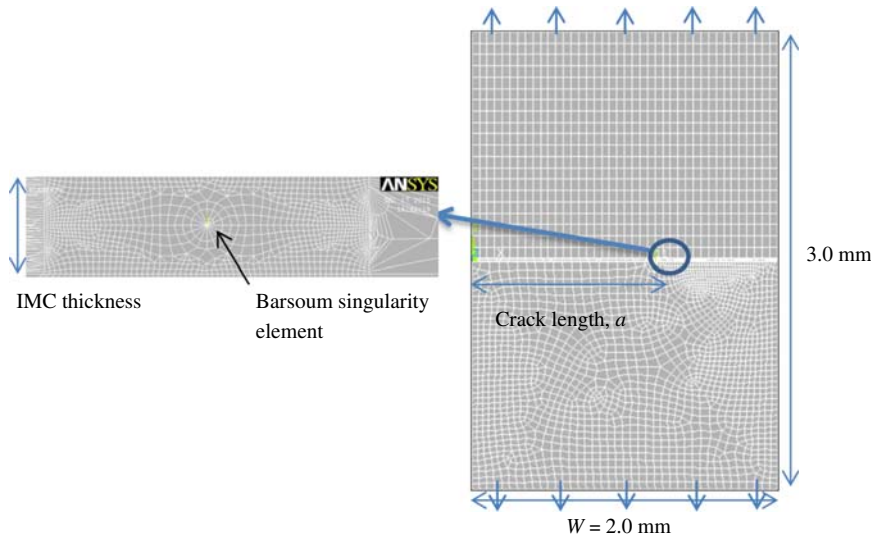


FIGURE 3.1

Finite element model on Brown & Srawley analytical equation. *IMC*, Intermetallic compound.

Table 3.1 Material properties for the solder joint modeling.

Materials	Elastic modulus, E (GPa)	Poisson ratio, ν
Intermetallic compound (IMC)	110	0.30
Lead-free solder tin–silver–copper (SnAgCu)	43	0.30
Leaded solder Sn37Pb Eutectic (SnPb)	35	0.36
Copper (Cu)	129	0.34

According to Alam et al. [2], the critical value for fracture toughness (K_{IC}) of Cu_6Sn_5 was in the range of $1.4\text{--}3 \text{ MPa}\sqrt{\text{m}}$. For this study, $K_{IC} = 1.4 \text{ MPa}\sqrt{\text{m}}$ is selected which would propagate crack when the K_I is more than K_{IC} .

3.2.1.3 Multiple cracks analysis on solder joint behavior—parallel edge cracks

The first study investigates the effect of vertical distance between the two parallel cracks edge on crack tip fracture SIF. Two parallel (stack) cracks are introduced to solder joint in the solder bulk area. About 80 MPa shear stresses are applied on the model to study the effect of distance between two parallel cracks on fracture parameter (SIF) as it would produce SIF around K_{IC} of the IMC. The geometry detail is shown in Fig. 3.2. The model height is 3.0 mm, while its copper thickness and solder thickness are set at 1.35 and 0.288 mm, respectively. Its width is 2.0 mm and its IMC thickness is 0.006 mm. Vertical distance between A1 and copper is set at 0.010 mm, while distance between A1 and A2 is manipulated from 0.004 to 0.250 mm. The edge crack length A1 is set at 0.012 mm, while crack length A2 is set at 0.0012 and 0.060 mm so that the simulation is repeated for difference ratio of A2 to A1 crack length with values 0.1, 0.5, and 1.0.

The experiment is also simulated with a single crack A1 alone and also single crack A2 only to compare with the parallel cracks. The setup is illustrated in Fig. 3.2.

3.2.1.4 Multiple cracks analysis on solder joint behavior—coplanar cracks

Two coplanar cracks interacting with each other for manipulated variables are described in the following steps:

1. The first study on coplanar cracks behavior is the relationship between SIF and the horizontal length (x) between the two opposites coplanar crack tips A and B. About 120 MPa shear stress is applied to top copper edge and fixed at the bottom copper edge on the opposite side, as shown in Fig. 3.3. Shear stress of 120 MPa produces SIF at the crack tip around the K_{IC} value of the IMC. The optimized geometry of the model is described below. The model

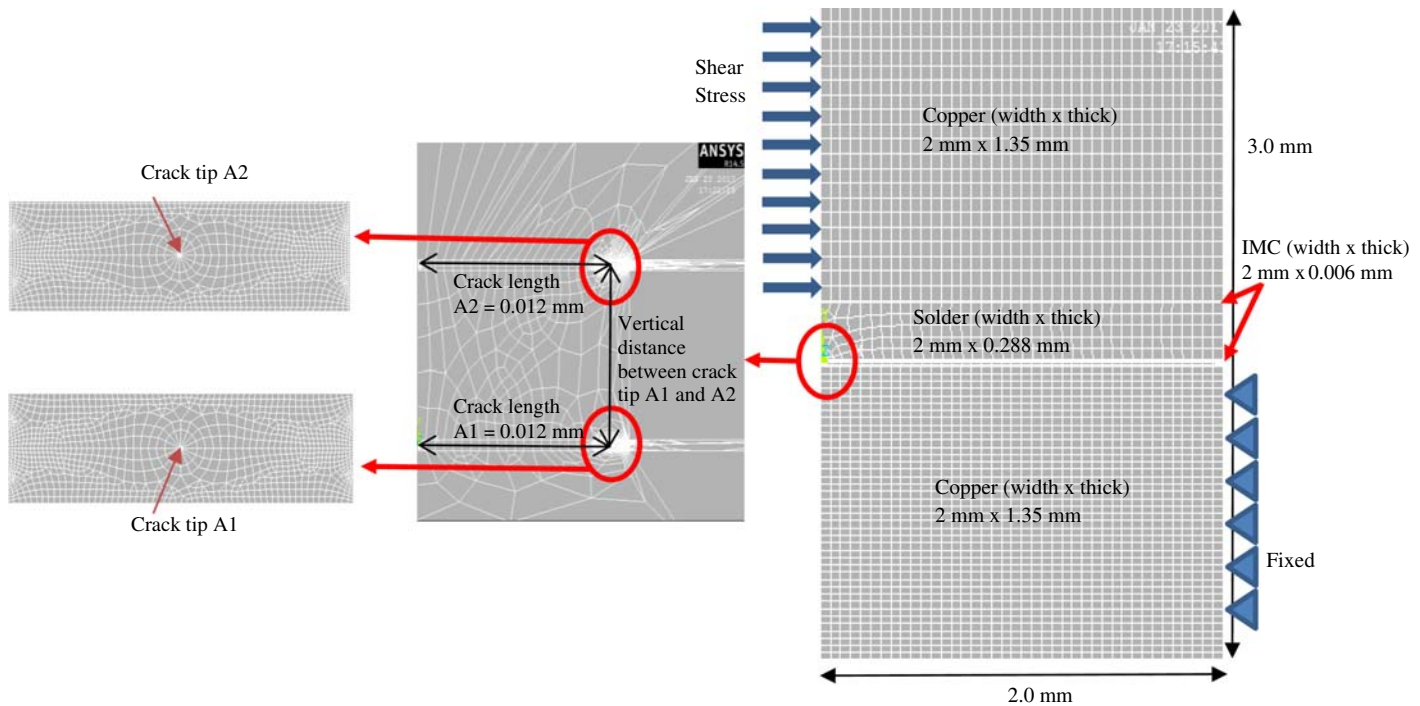
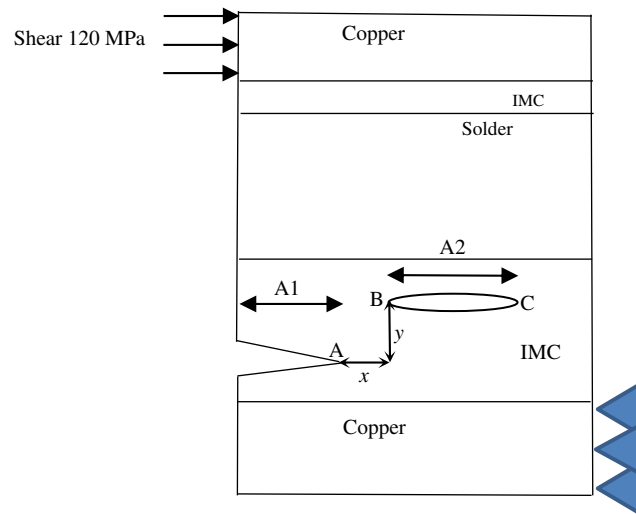


FIGURE 3.2

Finite element model validated with Brown & Srawley analytical equation for two parallel crack length studies. *IMC*, Intermetallic compound.

**FIGURE 3.3**

Finite element model for two coplanar crack tips studies. *IMC*, Intermetallic compound.

height is 3.0 mm, while its copper thickness and solder thickness is set at 1.35 and 0.3 mm, respectively. Its width is 2.0 mm and the edge crack length A1 is set at 0.012 mm, while crack length A2 is set at 0.025 mm. The IMC thickness is a total of 0.010 mm plus the vertical distance between A and B crack tips. The vertical distance between A1 and copper is set at 0.005 mm, while the vertical distance (y) between A and B is 0.0026 mm. The horizontal distance between A and B (x) is manipulated from 0.0035 to 0.005, 0.008, 0.010, 0.015, 0.020, 0.050, 0.080, 0.100, 0.200, 0.250, and 0.400 mm. The experiment is simulated with single crack A only FE model, as well as the single crack B/C only. The experiment is repeated with a vertical distance (y) between A and B for 0.005 and 0.020 mm.

2. The second study on coplanar crack's behavior is the relationship between SIF of the two closed coplanar crack tips A, B, C, and the vertical distance (y) between them. About 120 MPa shear stress is applied to the top copper edge and fixed at the bottom copper edge on the opposite side, as shown in Fig. 3.3. The optimized geometry of the model is list below. The model height is 3.0 mm, while its copper thickness and solder thickness is set at 1.3374 and 0.3 mm, respectively. Its width is 2.0 mm and the edge crack length A1 is set at 0.012 mm, while crack length A2 is set at 0.025 mm. The IMC thickness is 0.0126 mm. The vertical distance between A1 and copper is set at 0.005 mm while the horizontal distance (x) between A and B is 0.0035 mm. The vertical distance (y) between A and B is manipulated from 0.0026 to 0.005, 0.008, 0.010, 0.015, 0.020, 0.030, 0.050, 0.060, 0.0750, and 0.100 mm. The experiment is simulated with single crack A only FE model, as well as single crack B/C only.

3.3 Results and discussion

3.3.1 The effect of distance (B) between two parallel edge cracks

The result of simulation between two edge cracks of the crack length $A2/A1$ ratio of 0.1 in a solder joint FE model is shown in Fig. 3.4, where B is the vertical distance between the two parallel crack tips.

The graphs in Fig. 3.4 show the relationship between SIF of crack points A2 and A1 when the A2 crack length is very small. It is obvious that only the SIF (stress) at crack tip A2 shows shielding effect while longer crack length A1 does not have any changes in this interaction. The shielding effective start take place when the distance between both crack smaller than 0.1 mm and the effect become larger for a distance less than 0.05 mm. The closer the distance, the greater the effect. It drops from 0.28 to 0.012 MPa $\sqrt{m}$ at a 4 μm gap.

When the crack length A2 grow to half the size of A1, as shown in Fig. 3.5, the shielding effect at A2 becomes greater while the effect start to appear at A1 when their distance is less than 40 μm . The stress at A2 reduces about 75% from 0.64 to 0.167 MPa $\sqrt{m}$ when their vertical crack distance is 4 μm between each other.

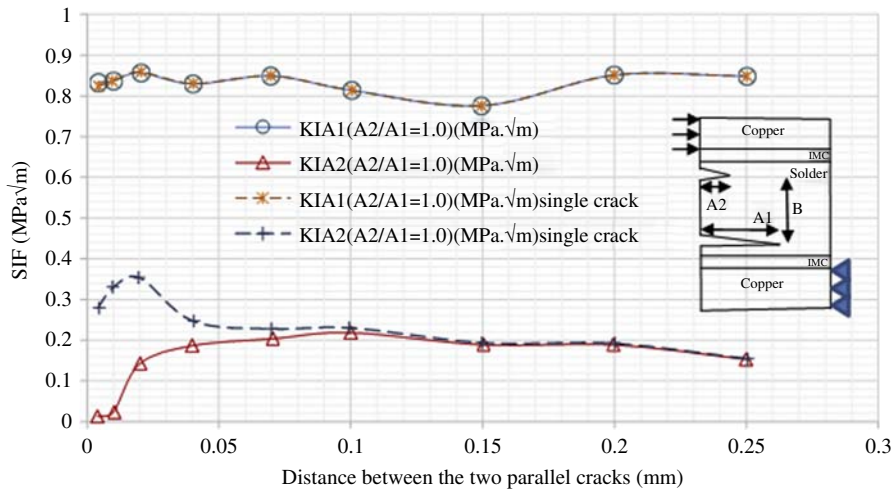
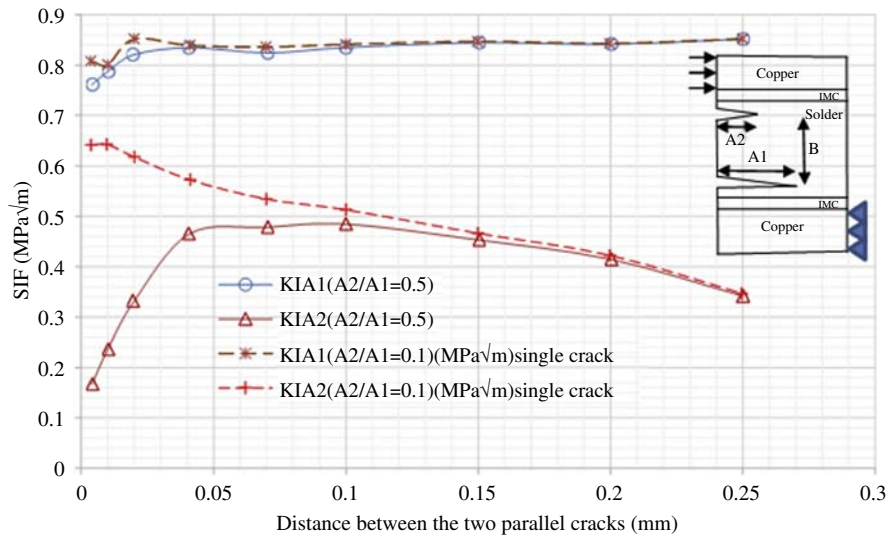


FIGURE 3.4

Stress intensity factor (SIF) at A1 and A2 crack tips against distance between the two parallel cracks with crack length ratio of $A2:A1 = 0.1$.

**FIGURE 3.5**

Stress intensity factor (SIF) at A1 and A2 crack tips against distance between the two parallel cracks with crack length ratio of $A2:A1 = 0.5$.

For SIF mode 1 stress with A2 crack length to A1 crack length ratio of 0.1 and 0.5, the greater the length of A2 crack, the shielding effect decrease while the one with smaller crack length, the more protecting effect it has gained.

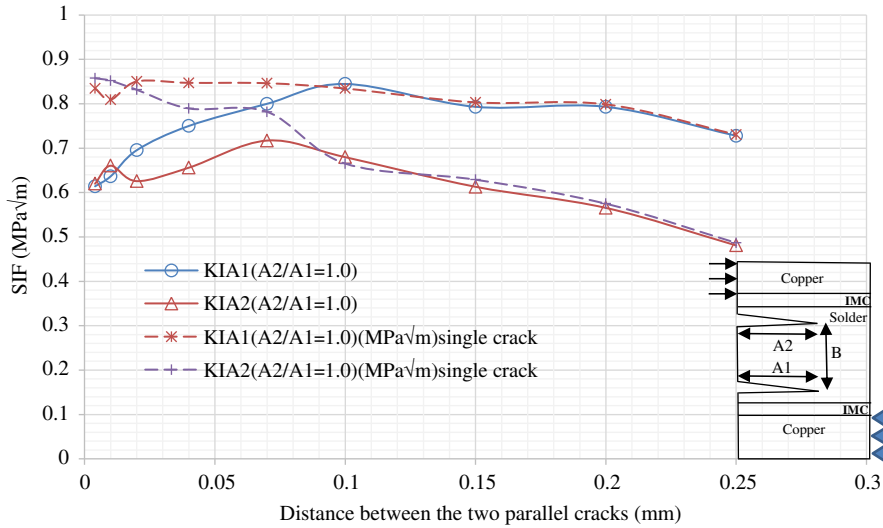
As for Fig. 3.6, when both crack lengths are of the same size of 0.012 mm, comparing the single crack and two cracks from the graphs plotted, and the shielding effect are almost the same for both tips. The SIF at crack tip A1 and A2 reduces when both crack tips are close together (less than 90 μm). The shielding disappears when both crack tips are more than 90 μm away from each other. On the other hand, without the presence of A1 crack, SIF at crack tip A2 decreases with the distance from the IMC layer. The stress at both crack tip A1 and A2 reduces from 0.86 and 0.83 to 0.62 $\text{MPa}\sqrt{\text{m}}$ when both are very near together at 4 μm . The shielding effect decreases with distance between both crack tips.

In summary, the shielding effect of macrocracks and microcracks becomes less significant when the distance between them becomes further. The microcrack tip has more shielding effect.

3.3.2 Multiple crack analysis—coplanar cracks

3.3.2.1 The effect of horizontal (x) distance between the coplanar crack tips

From the graph in Figs. 3.7–3.9, it is clearly seen that when the two-coplanar crack tips are nearer to each other (crack tips A and B) with the distance less than

**FIGURE 3.6**

Stress intensity factor (SIF) at A1 and A2 crack tips against distance between the two parallel cracks with crack length ratio of $A2:A1 = 1.0$.

0.040 mm, their SIF value increase exponentially and show amplification effect. This contributes to the propagation on both crack tips and makes them merge together. As a result, a longer crack length is produced and causes greater stress (SIF value) to further propagate the cracks.

For more detail in edge crack, graph in Fig. 3.7 shows that the edge crack which produce crack tip A, the SIF value increase exponentially from $1.0 \text{ MPa}\sqrt{\text{m}}$ at a horizontal distance (x) 0.040 mm between both the crack tip to $1.93 \text{ MPa}\sqrt{\text{m}}$ when the horizontal distance (x) become very closed at 0.0026 mm. Its SIF remains around $1.018\text{--}1.026 \text{ MPa}\sqrt{\text{m}}$ if there is no coplanar crack or inner crack near it for the distance less than 0.004 mm. The coplanar inner crack gives a very minor effect to the edge crack tip for a distance more than 0.040 mm and no effect at all for the distance of 0.200 mm onward.

As for crack tip B of inner crack, at 0.040 mm distance between the two coplanar crack tips, the SIF value is $0.41 \text{ MPa}\sqrt{\text{m}}$. It increases drastically to $2.0 \text{ MPa}\sqrt{\text{m}}$ when the distance becomes nearer to each other at 0.0035 mm. This amplification effect can be seen in Fig. 3.7 when compare the graph of KI at crack tip B with the dotted line of KI at B (single crack) which is only inner crack and no edge crack.

For crack tip C which is 0.025 mm further from crack tip B, the effect is less. It shows a significant effect when it is very close to the edge crack tip, which is less than 0.010 mm between the crack tips A and B.

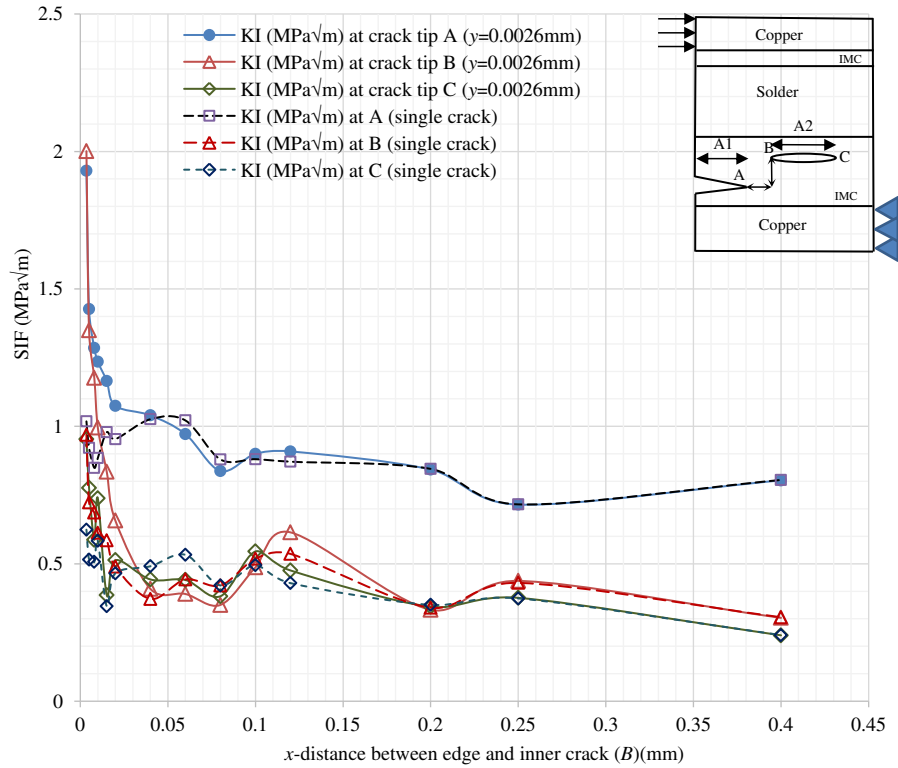


FIGURE 3.7

Stress intensity factor (SIF) against x -distance between two coplanar crack tips for vertical distance (y) at 0.0026 mm.

As for Figs. 3.8 and 3.9, it can be seen that when the vertical distance (y) between crack tips A and B is further apart, the amplification effect become less.

3.3.2.2 The effect of horizontal (y) distance between the coplanar crack tips

When analyzing the graph in Fig. 3.10 on the effect of y -distance between the two coplanar crack tips, it shows that the amplification effect only start to take place when the y -distance is smaller than 0.050 mm. For crack tips A and B, the amplification takes effect from the distances 0.050 and 0.04 mm, respectively. For crack tip C, the effect is less but it affect only from 0.050 mm onward.

As an overall result for the coplanar cracks study, the amplification effect of coplanar crack tip only take place when the x -distance and y -distance between the two near crack tips are less than 0.050 mm and increase exponentially with the decrease in the distance.

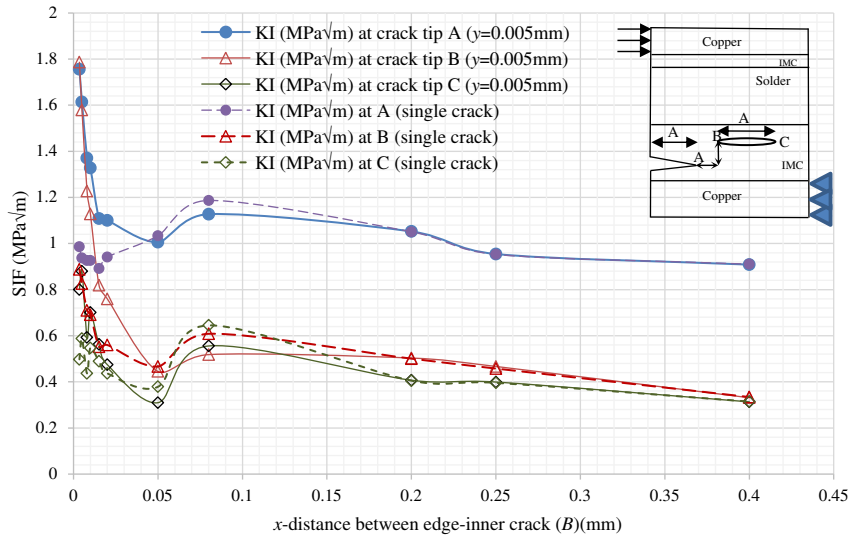


FIGURE 3.8

Stress intensity factor (SIF) against x -distance between two coplanar crack tips for vertical distance (y) at 0.005 mm.

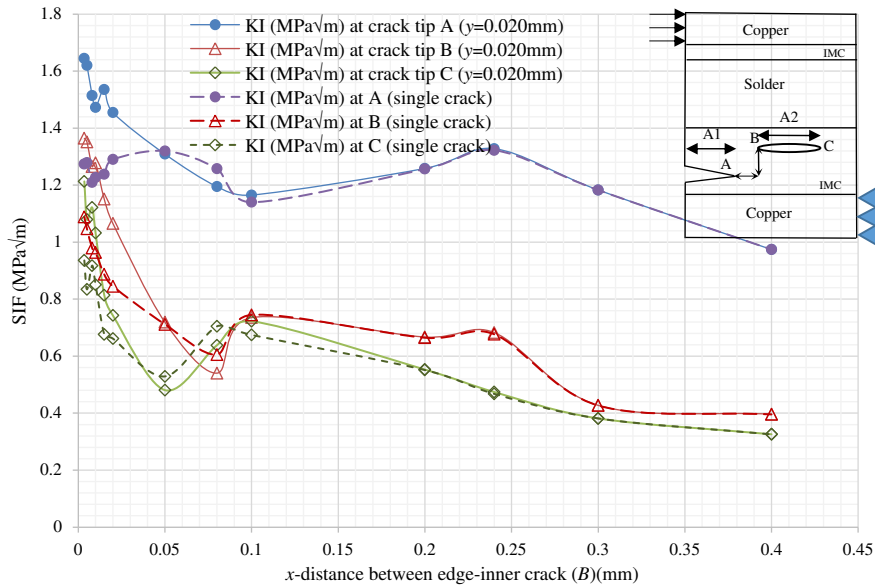
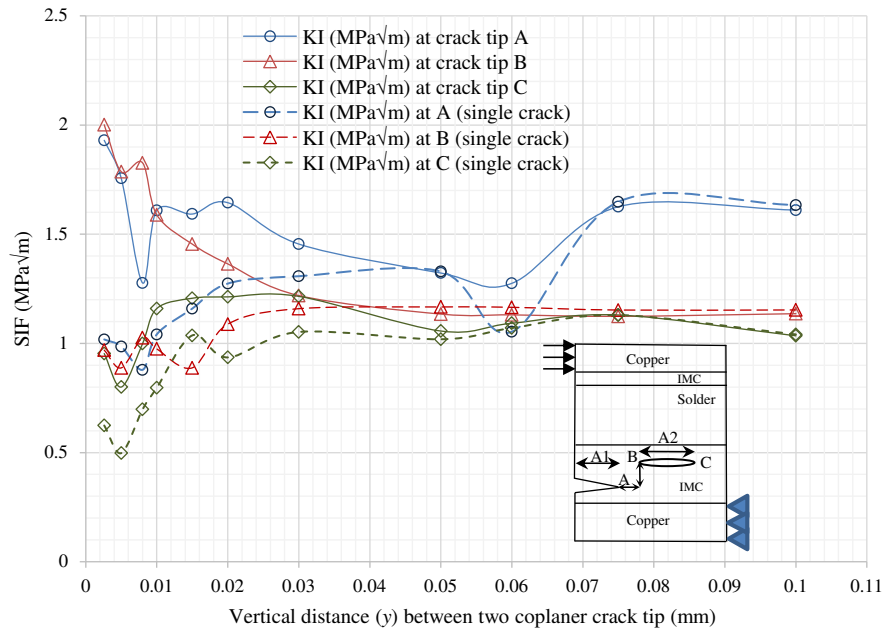


FIGURE 3.9

Stress intensity factor (SIF) against x -distance between two coplanar crack tips for vertical distance (y) at 0.020 mm.

**FIGURE 3.10**

Stress intensity factor (SIF) versus vertical distance (y) between two coplanar crack tips for x -distance 0.0035 mm.

In summary, the results from numerical models based on single crack and multiple cracks approach have given a promising output to the solder joint fracture analysis study. The single crack approach has confirmed the solder joint fracture behavior found in many studies. Finally, the multiple crack interaction has provided a further detailed description of the failure mechanism found by many researchers in their investigations in IMC layer.

3.4 Conclusion

This simulation study has showed the characterization of the solder joint based on the multiple crack approach.

1. The ratio of crack length between the two parallel edge cracks giving significant remarks to the interaction.
 - a. The one that having longer crack length will bear more stress than the one having the shorter crack length, whereas the shorter crack length crack tip will receive protection from the longer crack tip. When related to the single crack behavior on crack length, it is tally that the longer crack

- length receives greater stress. Nevertheless, the presence of one nearer parallel crack will result in a shielding effect.
- b. Multiple cracks interaction between two parallel cracks shows that the fracture stress reduces when they are approaching each other and provide shielding effect.
 2. The simulation of horizontal and vertical distance between the two coplanar cracks has showed that the amplification effect of coplanar crack tips only take place when the x -distance and y -distance between the two near crack tips are very close together (less than 0.050 mm) and increase greatly with the decrease of the distance. The SIF becomes double when both tips are very close together [horizontal (x) distance $\leq 3.5 \mu\text{m}$ and vertical (y) distance $\leq 2.6 \mu\text{m}$].

In conclusion, the results from numerical models based on single crack and multiple cracks approach have given a promising output to the solder joint fracture analysis study. It has been found in many studies that the single crack approach has confirmed the solder joint fracture behavior. Finally, the multiple crack interaction has provided a further detailed description on failure mechanism found by many researchers in their investigation in the IMC layer. More works are recommended in the multiple cracks behavior.

Acknowledgment

The authors would like to acknowledge the support from Fundamental Research Grant Scheme (FRGS) under a grant number of FRGS/2/2013/TK01/UNIMAP/02/1 from the Ministry of Higher Education Malaysia.

References

- [1] Y. Tian, C. Hang, C. Wang, S. Yang, P. Lin, Effects of bump size on deformation and fracture behavior of Sn3.0Ag0.5Cu/Cu solder joints during shear testing, *Mater. Sci. Eng. A* 529 (2011) 468–478.
- [2] M.O. Alam, H. Lu, Chris Bailey, Y.C. Chan, Fracture mechanics analysis of solder joint intermetallic compounds in shear test, *Comp. Mater. Sci.* 45 (2009) 576–583.
- [3] S.P.V. Nadimpalli, J.K. Spelt, Effect of geometry on the fracture behavior of lead-free solder joints, *Eng. Fract. Mech.* 78 (2011) 1169–1181.
- [4] Y. Yao, L.M. Keer, M.E. Fine, Modeling the failure of intermetallic/solder interfaces, *Intermetallics* 18 (2010) 1603–1611.
- [5] X. Chen, Y.C. Lin, X. Liu, G.-Q. Lu, Fracture mechanics analysis of the effect of substrate flexibility on solder joint reliability, *Eng. Fract. Mech.* 72 (2005) 2628–2646.

- [6] D.-S. Liu, C.-Y. Kuo, C.-L. Hsu, G.-S. Shen, Y.-R. Chen, K.-C. Lo, Failure mode analysis of lead-free solder joints under high speed impact testing, *Mater. Sci. Eng. A* 494 (2008) 196–202.
- [7] Y.-L. Shen, K. Aluru, Numerical study of ductile failure morphology in solder joints under fast loading conditions, *Microelectron. Reliab.* 50 (2010) 2059–2070.
- [8] K. Aluru, F.-L. Wen, Y.-L. Shen, Direct simulation of fatigue failure in solder joints during cyclic shear, *Mater. Des.* 32 (2011) 1940–1947.
- [9] J.H. Lee, H.-Y. Jeong, Fatigue life prediction of solder joints with consideration of frequency, temperature and cracking energy density, *Int. J. Fatigue* 61 (2014) 264–270.
- [10] S.P.V. Nadimpalli, J.K. Spelt, Mixed-mode fracture load prediction in lead-free solder joints, *Eng. Fract. Mech.* 78 (2011) 317–333.
- [11] S.P.V. Nadimpalli, J.K. Spelt, Fracture load prediction of lead-free solder joints, *Eng. Fract. Mech.* 77 (2010) 3446–3461.
- [12] Q.K. Zhang, Z.F. Zhang, Thermal fatigue behaviors of Sn–4Ag/Cu solder joints at low strain amplitude, *Mater. Sci. Eng. A* 580 (2013) 374–384.
- [13] K.S. Siow, M. Manoharan, Mixed mode fracture toughness of lead–tin and tin–silver solder joints with nickel-plated substrate, *Mater. Sci. Eng. A* 404 (2005) 244–250.
- [14] Y.-M. Jen, Y.-C. Chiou, C.-L. Yu, Fracture mechanics study on the intermetallic compound cracks for the solder joints of electronic packages, *Eng. Fail. Anal.* 18 (2011) 797–810.
- [15] E.H. Amalu, N.N. Ekere, High temperature reliability of lead-free solder joints in a flip chip assembly, *J. Mater. Process. Tech.* 212 (2012) 471–483.
- [16] H.L. Li, R. An, C.Q. Wang, Y.H. Tian, Z. Jiang, Effect of Cu grain size on the voiding propensity at the interface of SnAgCu/Cu solder joints, *Mater. Lett.* 144 (2015) 97–99.
- [17] S.B. Liang, C.B. Ke, W.J. Ma, M.B. Zhou, X.P. Zhang, Numerical simulations of migration and coalescence behavior of microvoids driven by diffusion and electric field in solder interconnects, *Microelectron. Reliab.* 71 (2017) 77–81.
- [18] Y.H. Tian, B.L. Liu, R. Zhang, J.K. Qin, Electromigration failure of SnAgCu lead-free BGA package assembled with SnPb solder paste, in: 2013 14th International Conference on Electronic Packaging Technology, 2013, pp. 892–895.
- [19] Y.H. Tian, R. Zhang, C.J. Hang, L. Niu, C.Q. Wang, Relationship between morphologies and orientations of Cu₆Sn₅ grains in Sn 3.0 Ag 0.5 Cu solder joints on different Cu pads, *Mater. Characterization* 88 (2014) 58–68.
- [20] C.J. Hang, Y.H. Tian, R. Zhang, D.S. Yang, Phase transformation and grain orientation of Cu–Sn intermetallic compounds during low temperature bonding process, *J. Mater. Sci. Mater. Electron.* (2014) 1–9.
- [21] B.L. Liu, Y.H. Tian, S. Wang, R. Zhang, X. Zhao, C.L. Dong, et al., Rapid formation of Cu–Sn intermetallic compounds by strong electric current, in: 2014 15th International Conference on Electronic Packaging Technology, 2014, pp. 489–492.

Further reading

R. Daud, A.K. Ariffin, S. Abdullah, Validation of crack interaction limit model for parallel edge cracks using two-dimensional finite element analysis, *Int. J. Automotive Mech. Eng. (IJAME)* 7 (2013) 993–1004.

- R. Daud, K.A. Ariffin, S. Abdullah, M.S. Abdul Majid, M.A. Rojan, Mathematical model of elastic crack unification interaction and two-dimensional finite element analyses based on Griffith energy release rate, *Adv. Mater. Res.* 795 (2013) 587–590.
- M. Kachanov, Interaction of a crack with certain microcrack arrays, *Eng. Fract. Mech.* 25 (5/6) (1986) 625–636.
- M. Kachanov, Elastic solids with many cracks: a simple method of analysis, *Int. J. Sol. Struct.* 23 (1) (1987) 23–43.
- M. Kachanov, A simple analysis of intersecting cracks and cracks intersecting a hole, *Int. J. Fract.* 40 (1989) R61–R65.
- M.M. Kachanov, Mechanics of crack-microcrack interactions, *Mech. Mater.* 10 (1990) 59–71.
- M. Kachanov, On the problems of crack interactions and crack coalescence, *Int. J. Fract.* 120 (2003) 537–543.
- E.P. Ooi, R. Daud, N.A.M. Amin, F. Mat, M.H. Sulaiman, M.S. Abdul Majid, et al., Fracture Behaviour of Intermetallic Compound (IMC) of Solder Joints Based on Finite Elements' Simulation Result. *Lecture Notes in Mechanical Engineering* (ISSN: 2195-4356), *Advances in Joining Technology*, 2017, pp. 49–57.

This page intentionally left blank

Assessment of failure of consumer electronics due to indoor corrosion in subtropical climates

Armando Ortiz, Víctor Hugo Jacobo and Rafael Schouwenaars

Departamento de materiales y manufactura, Facultad de ingeniería, Edificio O, Universidad Nacional Autónoma de México, Coyoacán, Ciudad de México, México

4.1 Introduction

Indoor corrosion is generally considered as a specific case of atmospheric corrosion and has been studied for many decades [1]. Most of the studies have focused on the effect of controlled atmospheres in moderate climates with atmospheric pollution within certain health-related limits imposed by authorities. This corresponds to the general situation in Europe, the United States, and Canada. These regions have also been historically the main markets for consumer electronics. In general, the studies conclude that indoor corrosion is a mild form of normal atmospheric corrosion [2–8]. The main explanation for this is that the indoor atmosphere is much more controlled compared with outdoor, limiting temperature effects, and variations of humidity. This difference has been found to be negligible in the case of silver and extreme in the case of steel [2,3,9]. It was reminded by Rice et al. [2] that the corrosion kinetics and mechanisms are not necessarily the same between the outdoors and indoors.

More recently, several studies have described the corrosion conditions in the Caribbean zone [9–12]. It was indicated that corrosion mechanisms and velocities are different in this region and that the differences between indoor and outdoor corrosion are much smaller. Dew is the predominant factor replaces rain plays a major role in determining time of wetness. It was found that the ISO9223 [13] classification of atmospheric corrosivity did not apply to coastal subtropical climates, and the generation of class VI corrosivity was suggested. Studies on materials under indoor conditions were tested at the seafront, corrosion in tropical climates was even more extreme due to the influence of saline water spray carried by the wind [14–16]. Also in Baja California, which has a desert climate, very specific climate conditions could be found to accelerate corrosion in an extreme manner [17]. Such extreme conditions should, however, not be used to draw conclusions on tropical indoor corrosion and were not included in the present work.

Conditions on the east coast of Mexico are found to be very similar [10,18] to that of the ones determined in Cuba. Studies in New Zealand [19], Argentina [20,21], and Colombia [22,23] also indicate a strong effect of temperature and humidity, while studies in the Canary Islands [24] and Baja California [25–27], with coastal conditions but arid climate discover strong but not extreme corrosion conditions. These studies have clearly shown that even if common features exist in both indoor and outdoor atmospheric corrosion, a good knowledge of the local environmental conditions is needed to explain the both phenomena and atmospheric pollution, which is different at each site, will affect the specific corrosion mechanisms and products.

Most of the cited references conducted research on standard coupons exposed in racks for outdoor conditions. The tests for indoor conditions are more varied. An easy and direct comparison is obtained by enclosing the specimens in some ventilated container at the same site as the outdoor racks [8,9]. These studies conclude that dew is at least as aggressive as rain. Other studies measure standard coupons inside the installations of interest [2,6]. Some researchers have reported on the damage observed in real electronic components after damage or malfunction was detected in the systems [28,29]. Advanced analysis of corrosion products generally involves refined instrumental methods. Most of these methods are not really quantitative, with the exception of the gravimetric method, which is not very specific in relation to the specific products formed on the corroded surfaces. The results of these methods are easy to interpret when the corrosion mechanisms of the corresponding metals are well known, as is the case for steel [24,30].

A complicating factor in the study of atmospheric corrosion is the huge variety of corrosion products that can be formed on a single pure metal species under different conditions. Another important problem is the determination of the pollutants involved at any test site and their mutual interactions. Besides the principal pollutants SO_2 and Cl^- , other pollutants such as oxides of nitrogen (NO_x), O_3 , H_2S (or reduced S in general), CO_2 , and NH_3 were also mentioned. Monitoring all these species on site in general would mean heavy investment in instruments. This has led some researchers to develop laboratory tests simulating the effects of indoor corrosion [3,5,6,31–34]. These tests give excellent results with respect to the interactions of a limited number of gases but do not take into account that particulate matter forms a major source of pollutants rather than gases [6,29]. Airborne salinity also plays an important role, even at relatively large distances from the coast [19,24].

The complexity of atmospheric and indoor corrosion makes a fast prediction of the expected damage difficult in regions that have not previously been studied. Nevertheless, such fast assessment was needed for the producer of telephone sets for domestic use in Mexico. It had been found that an abnormal number of sets failed during the warranty period. In spite of the increasing use of wireless communication, each landline still is installed with a fixed telephone, so the high failure frequency involved a considerable financial risk.

The time available to respond to this problem excluded the execution of standard field tests, which may require several years, or the design of an accelerated laboratory test, which would require detailed knowledge of the local climatic and atmospheric conditions. Instead, failed telephone sets from three zones in Mexico were collected and analyzed. This led to limited sets of samples that had been exposed to uncontrolled but realistic indoor conditions. The advantages and limitations of this approach will be discussed. It will be indicated how the method allowed reaching some preliminary conclusions on corrosivity of atmospheres in Mexico and the main pollutants involved in the indoor corrosion of electronic materials under nonstandard conditions. This will permit formulating some simple rules that can help designing corrosion-resistant systems for tropical and subtropical zones.

4.2 Methods

Three telephones were investigated from each of three different regions in the Republic of Mexico. The first zone was Mexico City, situated at an altitude of 2200 m above sea level, about 400 km of both the Pacific and the Atlantic oceans. The city was once considered the most polluted on earth, but now has ceded this honor to the Asian megacities. The climate is moderate with night humidity raising to high levels only half of the year. The second zone where telephones were collected was Cocoyoc, a small rural town in the sugarcane region of the state of Morelos. It is situated at a height of 1500 m above sea level and separated from the metropolitan area of Mexico by mountains ranging between 3000 and 3900 m. The climate is similar, except for the temperature, which is about 8°C higher due to the difference in altitude. Humidity is somewhat higher as well. No atmospheric pollution data are available for this region, but it may be assumed that the mountains separating it from Mexico City form a reasonably efficient barrier against pollution. The third zone, selected due to known problems in telephone sets, was the city of Tampico on the Mexican Gulf coast. This place is characterized by a hot and humid climate (maximum temperature around 40°C). At night, the condensation of humidity is significant all year round. Tampico is also the center of Mexico's oil industry for the northern half of the Gulf Coast, resulting in high (if unknown) amounts of sulfur oxides. It is subject to a strong coastal influence with periodic strong winds both in "winter" and in the fall hurricane season.

All telephones included in the study were the same model, but some were of older generations' model. Even if they are nominally equal, small changes in design and component selection were found, most noticeably in the Tampico zone, as these specimens were returned for malfunctions after 12, 14, and 15 months. The other sets were all between 6 and 9 years old. All telephones were retrieved from normal middle-class houses, except for one from Cocoyoc that had

stood in a restaurant for 9 years and one from Mexico City that had functioned for 6 years in a nonconditioned office space. The study followed the general scheme of a failure analysis. First, the history of the equipment was tracked, as well as the possible and all essential functions were tested. The systems were then opened, and the connections, the printed circuit boards (PCBs), and the handsets were inspected visually. All parts were photographed in detail. They were then exhaustively researched by stereomicroscopy at magnifications of $12\text{--}32\times$. Next, the samples were taken apart and investigated in a Philips XL20 scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS) for chemical analysis.

A word of caution must be added concerning the SEM for the analysis of corrosion products. SEM has the advantage that small amounts of salts and oxides can easily be found using the backscattered electron detector, as they appear dark due the lower atomic weight of O, S, N, and Cl compared with the pure metal. Complemented with EDS, the pollutants that cause corrosion are easily identified. This analysis is not quantitative for O and N. In X-ray detectors with a beryllium window, the nitrogen radiation is almost completely absorbed. Oxygen and nitrogen radiation is strongly adsorbed by the matrix. Neither ZAF (ZAF, in EDS analysis represent a composition correction factor by the mean atomic number of the sample (Z- atomic number correction or generation factor), the absorption of generated X rays as they exit the sample (A- absorption correction), and the generation of secondary fluorescent X rays (F-fluorescent correction). ZAF is a usual correction algorithms used en analysis by EDS) nor $\phi\rho Z$ —correction algorithms account for these effects in light elements [35]. Corrosion products are often heterogeneous or porous and this compromises quantitative analysis too. As a consequence, the results presented in this chapter are qualitative and permitting to distinguish the importance of each of the pollutants in the process but without the ambition of determining the exact composition of any of the corrosion products observed.

To facilitate the interpretation of the data and to allow the comparison of corrosion phenomena in different samples, damage charts were designed for the main components of the telephones. These charts consist of a schematic drawing of the parts, indicating the location and type of all representative electronic components present on it. Five degrees of severity of atmospheric influences are then defined (Table 4.1) and mapped as five gray levels on the chart. The damage charts are linked to a damage database containing detailed photographs, SEM images, and EDS analysis of the corresponding phenomena.

4.3 Damage analysis

The main component of a telephone is the PCB, on which integrated circuits (ICs), resistors, and capacitors are soldered. All these components consist of a

Table 4.1 Damage levels as observed in the stereomicroscope at $32\times$.

1	Very light deposit of electrolytic solutions or salts without signs of corrosion.
2	Notable deposit of electrolytic solution or salts without signs of corrosion.
3	Initiating general corrosion or localized spots of corrosion products.
4	Clear and advanced general corrosion or generalized pitting with abundant corrosion products.
5	Advanced general corrosion, pitting or stress corrosion cracking threatening or affecting component integrity.

Levels of atmospheric influence on the components of electronic equipment as defined for the present study. Levels 1 and 2 do not represent significant corrosion, but are useful to predict the future risk for corrosion in systems that have not failed yet. Levels 3–5 indicate different degrees of corrosion, level 5 indicating imminent failure.

polymeric package containing the component and the connecting wires, which are tin-plated without exception. Connectors and switches that must be accessible to the outside occupy a small portion of the PCB. In these components, a wide variety of materials are found, from tin-plated connecting wires for soldering to gold- and nickel-plated contact wires. Silver and brass are common, and in one occasion bronze was found in switches. Exposed copper was not observed in any of the systems. Housings for switches and contacts mostly consist of polymers or chromated steel. Brass and silver are used in buzzers and can also be found in a variety of components in the handset. In the newest models, the PCB also contains a flat battery that provides a fixed potential to the digital memory. This was the case for the Tampico sets only. One particular part in the telephone design studied is the presence of a galvanized steel sheet at the bottom. This is an easy and very cheap solution to avoid electronic noise and interference by other electrical applications by acting as a Faraday cage. This sheet is not connected to the PCB or to the earth.

The tin plating and soldering metal were corrosion resistant in all the analyzed parts. Tin is known to passivate easily [36]. Chlorine is the only common pollutant that provokes corrosion in this material, but even then the corrosion products only form a thin film on the surface. This film limits the solderability of individual components but is unimportant once the parts are mounted. A fairly large amount of resistors, capacitors, and ICs were revised, but no significant corrosion could be detected. These components will not be discussed any further.

Damage charts are useful if corrosion affects the entire system. If only a limited number of components are affected, the damage chart gives additional information. An example of their use is shown for the three Tampico samples (Fig. 4.1). The charts show the lower side of the PCB, which contains the majority of components. Corresponding charts for the galvanized sheet and the upside of the PCB are not presented but show a high degree of coincidence with respect to the location and intensity of the corrosion phenomena. The three samples were returned to the telephone company due to their failure, but sets 1 and 2 showed

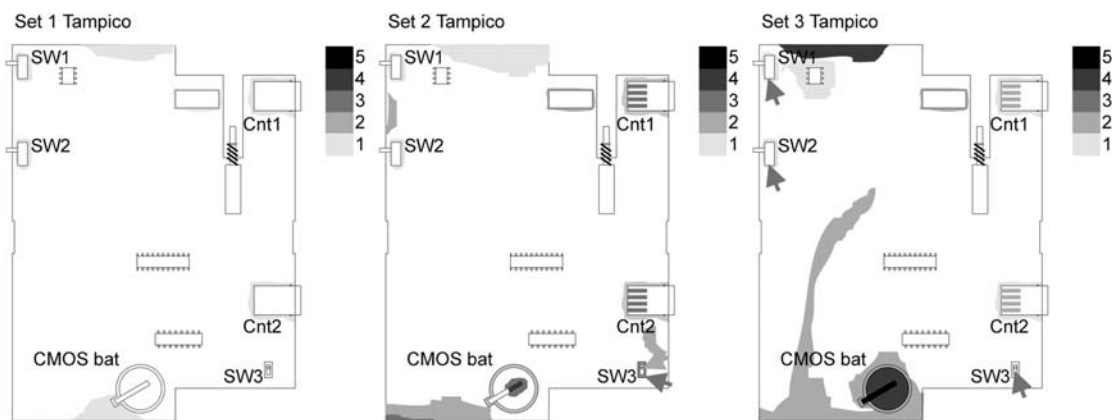


FIGURE 4.1

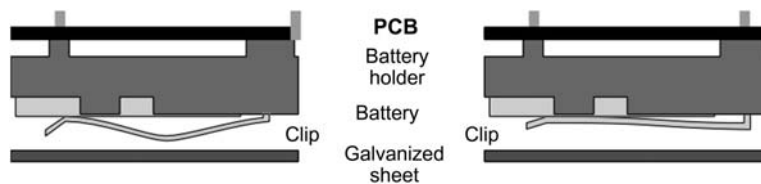
Damage charts for the three telephones from Tampico. The damage scale is indicated in [Table 4.1](#). Components referred to in the text are SW1, SW2, and SW3 (switches), CMOS battery and CNT1 and CNT2 (contacts consisting of nickel-plated copper wires with a partial gold overcoating in a polyvinyl chloride casing).

no defects when tested in the laboratory. Set 1 did not show signs of corrosion; set 2 showed some localized effects and fairly strong corrosion on the battery and its clip, as well as some pits perforating the plating of the galvanized sheet. Set 3 showed advanced corrosion of the galvanized sheet, the battery, and of the battery clip. Abundant deposits of iron oxide on large portions of the PCB and the galvanized sheet originally led to the hypothesis that this was not the result of atmospheric corrosion but it was because of that liquid poured over the telephone. The comparison of the three charts shows this was not the case. Salt deposits are found on all three PCBs at exactly the same location. In sets 2 and 3, these start to affect the substrate of the PCB. Salts were also found around the switches and the contacts. Deposits are present on the contact wires (CNT1 and CNT2) and incipient corrosion of these components is seen in set 2. Limited corrosion was found at SW3 for set 2 and is observed on all switches in set 3. The clip of the battery is strongly affected in set 2 and was broken in set 3. The galvanized sheet underneath the clip showed pitting due to the perforation of its chrome-plated zinc coating in set 2 and was heavily corroded in set 3. The carbon steel underneath the coating was the source of the iron oxides.

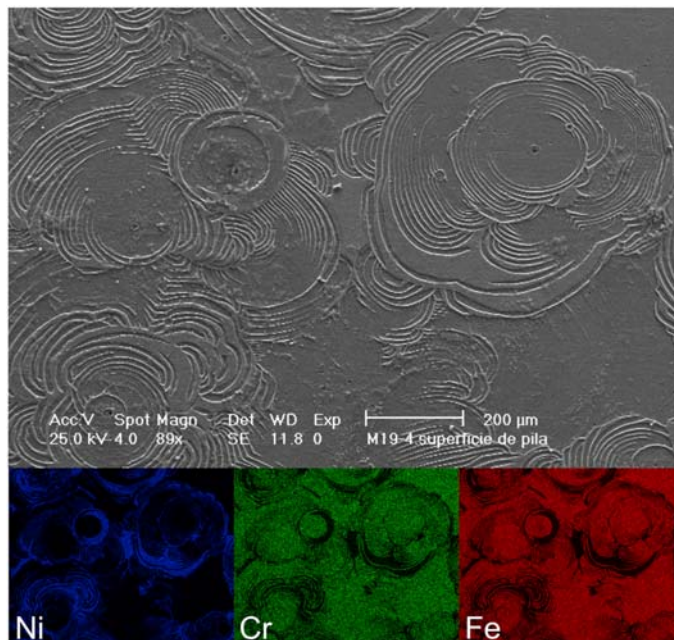
The coincidence between the three damage charts demonstrates that the same process is observed to varying degrees in the three cases. The tropical climate causes abundant condensation on certain portions of the PCB. Combined with atmospheric pollutants and dissolved metal ions, an electrolyte is formed that can cause failure due to leak currents or short circuits, even if corrosion cannot yet be detected (set 1). Incipient corrosion per se does not mean failure, as was shown in set 2, which worked fine once transported to the dryer climate of Mexico City. This shows a first problem for consumer electronics in humid tropical climates. The condensation of water during the nighttime cooling cycle is sufficient to cause failure. The contamination of the atmosphere is important, as it enhances the conductivity of the electrolyte.

The different corrosion phenomena observed were the result of small but important design details. The most prominent damage was observed in the clip of the battery and the underlying galvanized sheet. The geometry of the affected zone is sketched in [Fig. 4.2](#). The battery is made of stainless steel. The clip is carbon steel coated with tin. The housing is black polyvinyl chloride (PVC). In the original design of the clip, it was bent downward. Just underneath this point, pitting was observed with strong undercoat corrosion in the galvanized sheet for set 2. Zinc is a sacrificial coating with respect to steel. Tin, on the other hand is nobler than steel, zinc, and chromium. Corrosion starts when drops form on the metal surface of the battery and clip. These drip onto the galvanized sheet. When the electrolyte layer on the sheet becomes sufficiently thick and drops keep accumulating, contact is made between the clip and the sheet. Galvanic corrosion quickly perforates the chromium and zinc coating and the undercoat corrosion of the steel initiates.

In sets 2 and 3, incipient pitting corrosion of the stainless steel casing of the battery was observed. This is illustrated in [Fig. 4.3](#). A pattern of concentric rings

**FIGURE 4.2**

Original design of the battery holder with a bent clip (left). The curvature of the clip provides a dripping point and is too close to the galvanized sheet, causing galvanic corrosion between the chromium overcoating of the sheet and the tin coating of the clip. The corrected design (right) eliminates the dripping point and increases the distance between both metals.

**FIGURE 4.3**

Corrosion on the stainless steel surface of the battery and the corresponding energy dispersive X-ray spectrometer maps for Ni, Cr, and Fe. The center of the circles is formed by pits generated by chlorine salts. The circles are formed by nickel contaminated by Cl and minor quantities of S.

is formed around a central pit. Whether the location of this pit is determined by some microstructural feature or by the deposition of an airborne dust particle cannot be discerned from the observations. Chemical mapping shows that the rings consist mainly of Nickel, which is the noblest of the three metals making up the

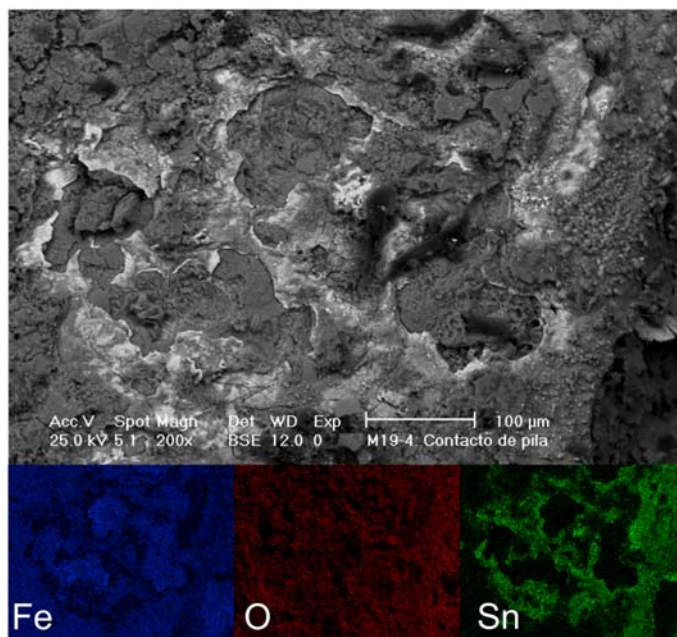


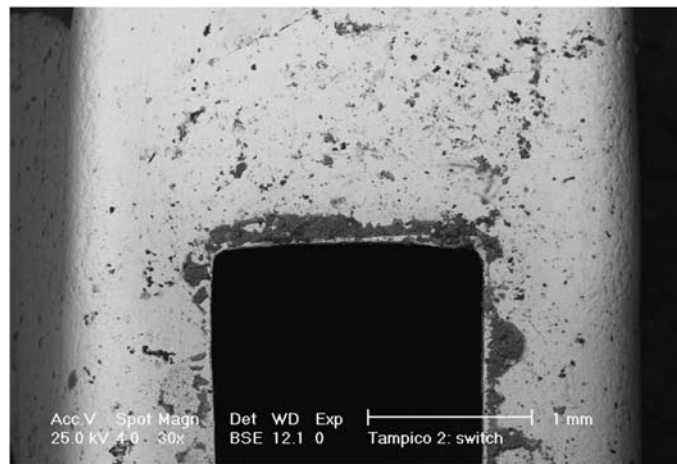
FIGURE 4.4

Corrosion of the tin-plated clip which retains the battery. Composition maps for Fe, O, and Sn are also shown. In order for this process to occur, the Sn-coating of the clip must either be damaged or depassivated.

stainless steel. The ring pattern is explained as the effect of condensed humidity, which shrinks and grows due to periodic variation of the atmospheric humidity.

In set 3, also the clip was corroded to failure and the tin coating had disintegrated, indicating the presence of substances that are capable of depassivating tin (Fig. 4.4). Considering that the main contaminant found was S, this points toward a medium (condensed water) that was considerably acidified. The problem presented by the battery assembly was improved by a small change in design, eliminating the dripping point and increasing the distance between the clip and the sheet (Fig. 4.2). It must be noted that the presence of a battery exposed to atmospheric influences always forms a corrosion risk and that hermetic packaging of the part must be considered preventing the observed problems completely.

Similarly, the switches combine a galvanic cell with a point where water accumulates (Fig. 4.5). Here, a small gap exists between the housing, made from chromium-plated steel, and the handle of the switch. Electrolytes accumulate easily in the punched hole of the housing, activating the chromium–steel cell and causing superficial damage to the housing but not to the interior parts of the switch. The design solution is easy, as there is no real need to use chromated parts. Polymers can easily substitute them. It is noteworthy that inside the

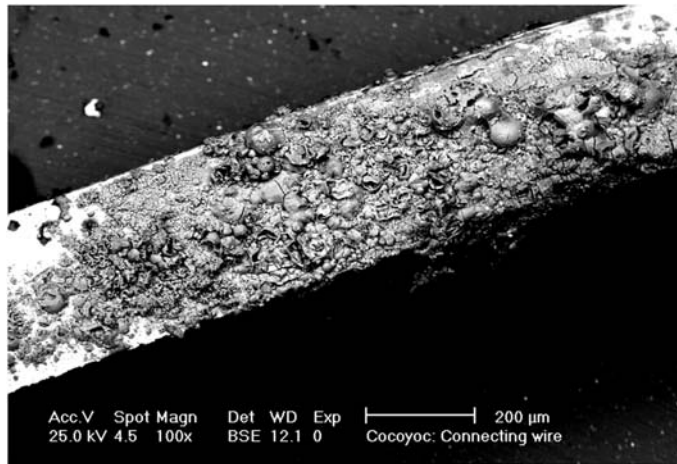
**FIGURE 4.5**

Corrosion of the housing of a switch. The phenomenon initiates when electrolyte accumulates in the punched hole of the chromated steel strip, activating the galvanic cell between the metals.

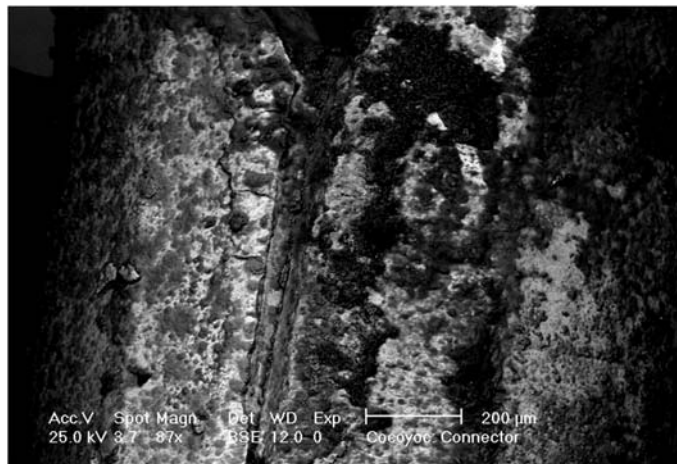
housings, corrosion is much less pronounced than on parts directly exposed to the atmosphere. This was found to be generally valid, showing that even inside the telephone, pollution gradients exist and that packaging can prevent corrosion of sensitive parts, even if it is not hermetic.

A final and important example of galvanic corrosion is the one that is caused when gold plating is used. Gold, being the most noble metal, will form strong galvanic cells in contact with any other material. It has been seen that undercoating corrosion is a problem with gold on Ni and Cu substrates, as electroplated gold tends to be porous [6]. Through-the-coating corrosion was found in a partially gold-coated contact wire with nickel underplating on copper exposed in the Cocoyoc zone (Fig. 4.6). The corrosion products were formed by chlorine compounds of Ni and Cu with minor additions of sulfur. Lateral transport of ions through a superficial electrolyte layer seemed to generate the damage on a contact shoe made of brass that was strongly corroded near its contact with a gold-coated wire (Fig. 4.7). Considerable amounts of copper- and zinc-based corrosion products were deposited on the wire. They contained chlorine and minor amounts of sulfur, together with significant oxygen, which was not quantified by the applied methods. This confirms the view that using gold promotes corrosion in electronic equipment rather than preventing it [20].

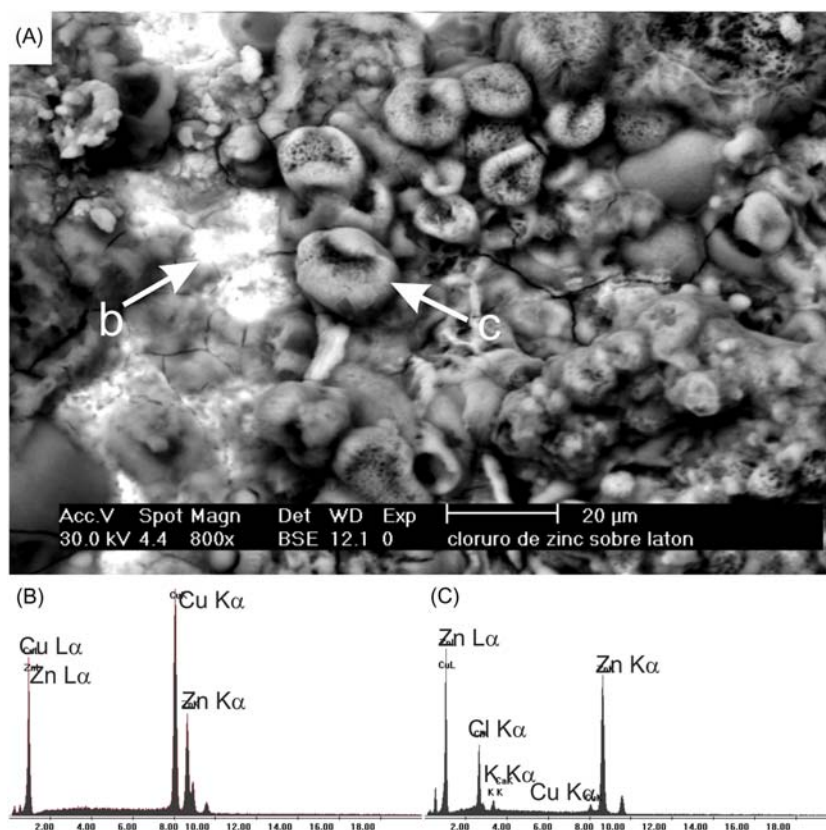
The role of airborne particulate contaminants is illustrated by the corrosion of the buzzer of a telephone retrieved from the Cocoyoc zone (Fig. 4.8). This component contains a circular brass membrane, which is exposed to the environment. Zinc is selectively dissolved from the alloy and deposited as chlorides with nodular shape. Remarkable is the presence of K in the deposits.

**FIGURE 4.6**

Corrosion products on a Ni-coated copper wire partially overplated with gold in the contact zones. The deposits are formed by nickel and copper compounds containing significant amounts of Cl and traces of S.

**FIGURE 4.7**

Brass contact shoe clipped onto a gold-coated wire. The entire surface is affected by corrosion, as is shown by the dark zones on the image. Corrosion products contain chlorine and limited amounts of sulfur.

**FIGURE 4.8**

Dezinification of brass by a chlorine-rich condensate (A). The clear parts of the image correspond to the Cu-rich phase (energy dispersive X-ray spectrometer (EDS) spectrum in B) and the darker nodules are highly enriched in Zn (EDS-spectrum in C). K is present from airborne particulate contaminants.

In a final note referring to the damage analysis, no significant corrosion products were detected in the three telephones studied in Mexico City. This does not mean corrosion is nonexistent, but it was not observed in the stereomicroscope or found to be insignificant. It must also be pointed out that desktop telephones are relatively simple electronic devices and it cannot be excluded that more refined systems may be affected to some degree even in Mexico City.

4.4 Discussion

The experimental technique followed only allows analyzing the relative importance of sulfur and chlorine in the corrosion products. The effect of ozone and

nitrogen oxides (NO_x) cannot be assessed. The latter have been discussed in various studies and their role is all but clear [8,9,29,31,33,36,37]. This lack of conclusion is probably due to the fact that their chemistry is far more complex than is generally assumed in corrosion studies. Volatile organic components (VOCs) play an important role in this chemistry. The removal of ozone and NO_x from the atmosphere can happen in the form of nitrous acid in NO_x -controlled chemistry and nighttime processes [38]. If VOCs control the chemistry, ozone and NO_x are removed through the formation of hydrogen peroxide, which is readily absorbed in the aqueous phase and catalyzes the oxidation of SO_3^{2-} to SO_4^{2-} . Daytime processes are intensified by sunlight, so these processes are expected to be more important in subtropical zones than in moderate climates. For the case of Tampico, where VOCs predominate, one might suppose that the combination of night- and daytime processes can cause the especially corrosive mix of sulfuric and nitric acid in aqueous solution during the evening condensation phase. The situation for Mexico City is less clear though strong acidification of condensate layers is highly probable through any of the processes described in [38]. However, the absence of strong corrosion in Mexico City indicates that one of the necessary conditions is not fulfilled, which is probably the formation of the condensation layer on the metallic surfaces.

The relative influence of chlorine and sulfur as corrosive agents is illustrated in Fig. 4.9. A summary is made of all measurements of corrosion products classified by alloy. The low values for S and Cl correspond to light corrosion spots, as a large portion of the EDS-signal comes from the substrate in this case. The high values correspond to fully developed salt or oxide deposits. It is readily seen that silver and nickel are mainly affected by sulfur, though strong chlorine presence is seen in some silver-based corrosion products. Brass is mostly affected by chlorine. The pits formed in the center of the circular corrosion pattern of the batteries from Tampico (Fig. 4.3) showed high chlorine contents. It must be said, however, that a clearly determined trend is not observed in the chart. This is because in very few cases, a single alloy corroded on its own. In almost all cases, macroscale galvanic cells were present and then, only the presence of a conductive electrolyte is required and the composition of the contaminating ions is of secondary importance.

The results for Mexico City are somewhat surprising. It was relatively difficult to find evidence of corrosion in most parts of the telephones. Silver forms an exception, as it forms a sulfur-based corrosion layer similar to the one observed in other zones. Pollution in Mexico City is severe beyond doubt. However, the main problems are O_3 , NO_x , VOCs, and particulate matter. All these are known to accelerate corrosion in presence of other pollutants. SO_2 is present, while it may be assumed that Cl plays a minor role. In the industrialized coastal zone of Tampico, the same pollutants are found, though SO_2 levels are probably higher. Also here, Cl is less important, indicating that the coastal influence is less important than the petroleum industry for provoking corrosion. The main difference between Tampico and Mexico City is humidity. In Tampico, nighttime condensation is strong enough to cause failure in electronic systems even without

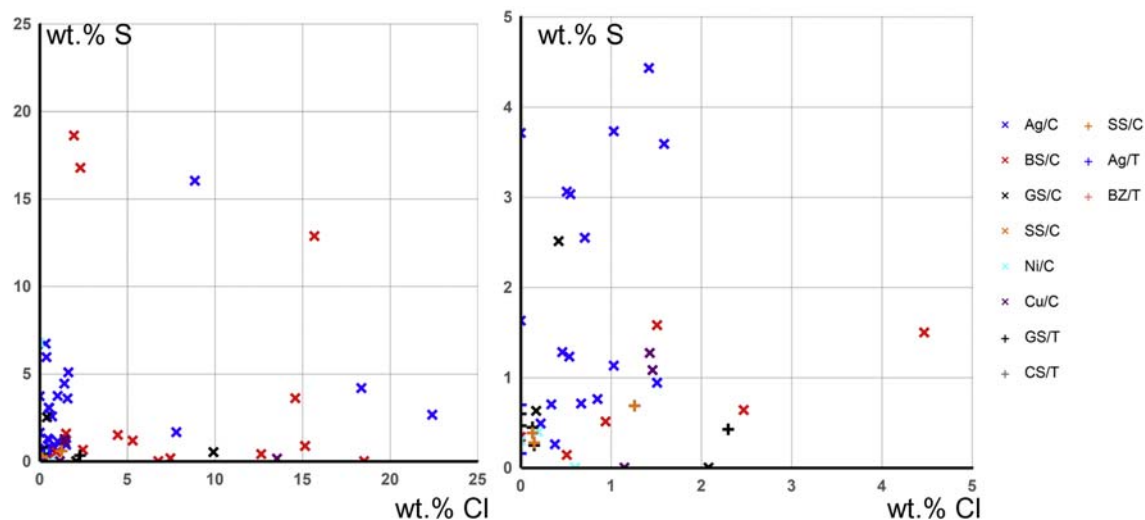


FIGURE 4.9

Amount of Cl and S detected in the corrosion products found on different alloys in the telephones investigated. The remainder of the products is made up of metal ions and combinations of oxygen, hydroxyl ions, and nitrates, all of which cannot be quantified by energy dispersive X-ray spectrometer (the chart on the left is a magnification of the right). The symbol $\times$ refers to Cocoyoc, + to Tampico. *Ag*, Silver; *BS*, brass; *BZ*, bronze; *CS*, carbon steel; *Cu*, copper; *GS*, galvanized steel; *Ni*, nickel; *SS*, stainless steel.

corrosion. Such phenomenon is nonexistent in Mexico City. Removal of NO_x and O_3 in the form of HNO_3 and H_2O_2 needs the presence of an aqueous phase [30], so their influence in the Tampico region will be stronger. The accumulation of SO_2 will follow the same logic, with an exception for silver, which is known to corrode in dry atmospheres [2,3].

Cocoyoc ranks in corrosivity between Mexico and Tampico, and so does the relative humidity. Nevertheless, the presence of relatively high amounts of sulfur may seem surprising for a rural zone, while the high levels of chlorine seem abnormal at 350 km from the coast. It was found that the chlorine was accompanied by potassium, not sodium, and soot was abundant on all exposed metal surfaces. This points to the agricultural practices in the sugarcane industry as the source of corrosive agents, as the fields are burned before harvesting to remove the leaves from the canes to facilitate the labor. Another contaminant that was present in higher amounts than in the other two zones were particles, resulting in the presence of small amounts of Al, Si, and Ca in the corrosion products. These elements have been found consistently in any of the corrosion studies executed by the authors in central Mexico and have their origin in the local geology, where almost all soils and rocks are created through volcanic processes, although volcanism itself has not been found to be directly involved in the corrosion phenomena.

Corrosion in Cocoyoc generally concentrated around spots containing particles or crystallized salts. This is an indication of intermittent, limited condensation on the surfaces, where pollutants accumulate during the wet phase and crystallized when drying. The presence of hygroscopic salts or dust particles that serve as inoculants causes condensation to occur at the same spot during every wet–dry cycle.

The former indicates that the connotation that rural zones are unpolluted cannot be accepted without critical examination. In fact, it was found that rural zones in Mexico are completely different from those in Sweden or from what is found at the Canary Islands or in New Zealand [8,19,24]. Similarly, indoor corrosion in subtropical coastal zones is far more severe than what has been predicted in other studies, even in areas which are not directly exposed to the salty sea breeze. In the present study, S is equally important as Cl for the Tampico zone, pointing to the impact of industry (mainly petroleum in this case) as an influencing factor.

To account for the severity of the environment in tropical and subtropical regions, it has been proposed [9–11] to add a class VI corrosivity to the existing ISO9223 norm. The authors do not believe this is justified at this moment. The present study indicates that the conditions to which equipment may be exposed are highly variable and dependent on very specific local circumstances. Little similarities exist to the conditions in the fully industrialized countries with a moderate climate, for which ISO9223 has been designed.

Nevertheless, further study on the topic is fully justified by the fact that more than half of the world population lives in tropical and subtropical coastal regions. Pollution in these zones is generally much higher than in the strongly regulated industrial zones of the classical industrialized countries. Most of these regions show rapid economic expansion, leading to increasing demand for consumer

electronics. The results of the present survey are limited, and it is clear that the variability in atmospheric, economic, and industrial conditions makes an extrapolation difficult. However, it is likely that conditions as the ones encountered in Tampico can serve as an indication for what can happen in other hot and humid coastal regions. The Cocoyoc case represents an example of conditions in places where agricultural practices are significantly different from those in moderate climate zones. Although the present work focuses on a fairly low-technology application, the importance of indoor corrosion is likely to increase in the future, as illustrated by recent studies on corrosion in control rooms and data centers [39,40].

The method used in the present study does not provide information on the exact nature of the mechanisms involved in subtropical indoor corrosion. One of the main disadvantages is the lack of quantitative data with respect to the role of oxygen and nitrogen in the products. A rough assessment of corrosion velocity can be made only based on the microscopic observation of the amount of damage; the original weight of the components was unknown. However, the method is fast, as it was not necessary to wait for the results of the exposure tests. Rapid assessment was a key condition of the present study. It also allowed detecting problems related to design, which would have been impossible on standard specimens. The present approach is interesting as it relates more closely to the specific needs of the electronics industry. Existing laboratory tests on standard coupons are likely to detect only a small fraction of the possible corrosion problems.

Recommendations to avoid failure of consumer electronics in severe climates can be formulated based on the observations in this work. It was shown that almost all cases of severe corrosion can be linked to the presence of galvanic cells. This can be avoided by giving due consideration to materials selection, which was reflected by the fact that two components with the same function in different telephones could be made from different materials. It is evident that the electronic engineers who designed the part were only considering the price and not the material. Use of nonfunctional plating or additions to components must be avoided. This was illustrated by the presence of a chrome plating which caused corrosion problems while it does not comply with any functional need and enhances costs. A general rule that can be derived from these observations is that the amount of different materials used in the design should be kept as low as possible and coatings should be avoided unless absolutely necessary.

Gold and tin plating were clearly associated with corrosion problems as well. Although gold and tin are corrosion resistant by themselves, they can provoke strong corrosion to less-noble materials around them, either by direct physical contact or by the formation of liquid bridges formed by condensation. Also, the geometry the components is important, as water condensates preferentially on large metal surfaces and accumulates in small gaps between components. Dripping points, consisting of downward-bend metal surfaces, must be avoided.

4.5 Conclusion

The experimental approach followed in the present study is rather unusual, but allowed for a fast assessment of the main factors involved in the corrosion of consumer electronics in subtropical climates. No standard tests exist for the moment that would have been able to reach the same results within the strict timeframe available for the analysis. The method can be considered as an interesting complement in more extensive studies into the same problem.

It was found that indoor corrosion in consumer electronics in subtropical climates can be far more severe than in moderate climate zones. Higher pollution levels may play a role, but it was found that humidity and the resulting amounts of condensation during nighttime cooling are the main factors in the process. This effect is stronger in zones subject to higher daytime temperatures. Failure in electronic systems can occur before corrosion causes significant damage.

Under these conditions, design details that are not important in moderate climates become vital factors. Galvanic cells must be avoided as much as possible, which means that materials choice should focus on minimizing the number of different alloys used, which may include their substitution by polymers where possible. Gold provokes galvanic corrosion due to its high electrochemical potential. Dripping points on large metal surfaces cause the accumulation of corrosive electrolytes and must be eliminated. The use of coatings of any type induces a galvanic cell and should be critically evaluated. Organic coatings may be preferred instead of classical electroplating.

Acknowledgments

The authors would like to thank E. Ramos, I. Cueva, and G. Álvarez for their technical support. The project was supported by DGAPA grant PAPIIT IN117412.

References

- [1] C. Leygraf, I.O. Wallinder, J. Tidblad, T. Graedel, *Atmospheric Corrosion*, John Wiley & Sons, 2016.
- [2] D.W. Rice, R.J. Cappel, W. Kinsolving, J.J. Laskowski, *Indoor corrosion in metals*, *J. Electrochem. Soc.* 127 (1980) 891–901.
- [3] D.W. Rice, P. Peterson, E.B. Rigby, P.B.P. Phipps, R.J. Cappel, R. Tremoureux, *Atmospheric corrosion of copper and silver*, *J. Electrochem. Soc.* 128 (2) (1981) 275–284.
- [4] D.W. Rice, P.B.P. Phipps, R. Tremoureux, *Atmospheric corrosion of Nickel*, *J. Electrochem. Soc.* 127 (1980) 536–568.
- [5] W.H. Abbot, *Corrosion of electrical contacts: review of flowing mixed gas test developments*, *Br. Corros. J.* 24 (1989) 153–160.

- [6] S. Zakipour, C. Leygraf, Evaluation of laboratory tests to simulate indoor corrosion of electrical contacts, *J. Electrochem. Soc.* 133 (1986) 21–29.
- [7] J.D. Sinclair, L.A. Psota-Kelty, C.J. Weschler, H.C. Shields, Deposition of airborne sulphate, nitrate and chloride salts as it relates to corrosion of electronics, *J. Electrochem. Soc.* 137 (1990) 1200–1206.
- [8] J. Tidblad, C. Leygraf, Acid deposition effects on materials: evaluation of nickel and copper, *J. Electrochem. Soc.* 138 (1991) 3592–3598.
- [9] A.R. Mendoza, F. Corvo, Outdoor and indoor corrosion of carbon steel, *Corros. Sci.* 41 (1999) 75–86.
- [10] A.R. Mendoza, F. Corvo, Outdoor and indoor corrosion atmospheric corrosion of non-ferrous materials, *Corr. Sci.* 42 (2000) 1123–1147.
- [11] F. Corvo, C. Haces, N. Betancourt, L. Maldonado, L. Véleva, M. Echeverría, et al., Atmospheric corrosivity in the Caribbean area, *Corr. Sci.* 39 (1997) 823–833.
- [12] Y. Martín-Regueira, O. Ledea, F. Corvo, C. Lariot, Indoor atmospheric corrosion of copper and steel under heat trap conditions in Cuban tropical climate, *Corr. Eng. Sci. Tech.* 46 (2011) 624–633.
- [13] ISO 9223, Corrosion of Metals and Alloys-Corrosivity of Atmospheres. Classification, 1992.
- [14] F. Corvo, N. Betancourt, A. Mendoza, The influence of airborne salinity on the atmospheric corrosion of steel, *Corr. Sci.* 73 (1995) 1889–1901.
- [15] F. Corvo, A.D. Torrens, N. Betancourt, J. Perez, E. Gonzalez, Indoor atmospheric corrosion in Cuba. A report about indoor localized corrosion, *Corr. Sci.* 49 (2007) 418–435.
- [16] F. Corvo, T. Perez, L.R. Dzib, Y. Martin, A. Castañeda, E. Gonzalez, et al., Outdoor–indoor corrosion of metals in tropical coastal atmospheres, *Corros. Sci.* 50 (2008) 220–230.
- [17] G. Lopez, B. Valdez, M. Schorr, Spectroscopy analysis of corrosion in the electronic industry influenced by Santa Ana winds in marine environments of Mexico, *Indoor and Outdoor Air Pollution*, InTech, 2011.
- [18] R. Ramanauskas, L. Muleshkova, L. Maldonado, P. Dobrovolskis, Characterization of the corrosion behaviour of Zn and Zn alloy electrodeposits: atmospheric and accelerated tests, *Corros. Sci.* 40 (1998) 401–410.
- [19] R.J. Cordner, Atmospheric corrosion survey of New Zealand, *Br. Corros. J.* 25 (1990) 115–118.
- [20] J.R. Vilche, F.E. Varela, G. Acuna, E.N. Codaro, B.M. Rosales, A. Fernandez, et al., A survey of Argentinean atmospheric corrosion: I—aluminium and zinc samples, *Corros. Sci.* 37 (1995) 941–961.
- [21] J.R. Vilche, F.E. Varela, E.N. Codaro, B.M. Rosales, G. Moriena, A. Fernández, A survey of Argentinean atmospheric corrosion: II—copper samples, *Corros. Sci.* 39 (1997) 655–679.
- [22] H. Gil, J.A. Calderón, C.P. Buitrago, A. Echavarría, F. Echeverría, Indoor atmospheric corrosion of electronic materials in tropical-mountain environments, *Corros. Sci.* 52 (2010) 327–337.
- [23] H. Gil, C.P. Buitrago, A. Echavarría, Characterization of atmospheric corrosion products formed on silver in tropical-mountain environments, *J. Solid State Electrochem.* 19 (2015) 1817–1825.
- [24] J.J. Santana Rodríguez, F.J. Santana Hernández, J.E. González González, XRD and SEM studies of the layer of corrosion products for carbon steel in various different

- environments in the Province of Las Palmas (The Canary Islands, Spain), *Corros. Sci.* 44 (2002) 2425–2438.
- [25] B.G. Lopez, S.B. Valdez, K.R. Zlatev, P.J. Flores, B.M. Carrillo, W.M. Schorr, Corrosion of metals at indoor conditions in the electronics manufacturing industry, *Anti-Corros. Methods and Mater.* 54 (2007) 354–359.
- [26] G. Lopez, H. Tiznado, G. Soto Herrera, W. De la Cruz, B. Valdez, M. Schorr, et al., Use of AES in corrosion of copper connectors of electronic devices and equipments in arid and marine environments, *Anti-Corros. Methods and Mater.* 58 (2011) 331–336.
- [27] F. Moreno Ramirez, S. Velarde Cordova, H.A. Pelaez Molina, H.J. Rodriguez Centeno, S.L. Toledo Perea, K.C. Arredondo Soto, ABC analysis of industrial process in the electronic industry of Mexicali, *Sci. Fed. Mater. Res. Lett.* 2 (3) (2018).
- [28] J.D. Sinclair, Corrosion of electronics: the role of ionic substances, *J. Electrochem. Soc.* 135 (1988) 89C–95C.
- [29] V.K. Gouda, J.A. Carew, W.T. Riad, Investigation of computer hardware failure due to corrosion, *Br. Corros. J.* 24 (1989) 192–194.
- [30] A.M.G. Pacheco, M.G.I.B. Teixeira, M.G.S. Ferreira, Initial stages of chloride induced atmospheric corrosion of iron: an infrared spectroscopy study, *Br. Corros. J.* 25 (1990) 57–59.
- [31] H. Strandberg, L.G. Johansson, Role of O_3 in the atmospheric corrosion of copper in the presence of SO_2 , *J. Electrochem. Soc.* 144 (1997) 2334–2342.
- [32] S.P. Sharma, Atmospheric corrosion of silver, copper and nickel - environmental test, *J. Electrochem. Soc.* 125 (1978) 2005–2011.
- [33] S. Oesch, M. Faller, Environmental effects on materials: the effect of the air pollutants SO_2 , NO_2 , NO and O_3 on the corrosion of copper, zinc and aluminium. A short literature survey and results of laboratory exposures, *Corros. Sci.* 39 (1997) 1505–1530.
- [34] H. Conseil, V. Chakravarthy Gudla, M. Stendahl Jellesen, R. Ambat, Humidity build-up in a typical electronic enclosure exposed to cycling conditions and effect on corrosion reliability, *IEEE Transact. Compon., Packag. Manuf. Tech.* 6 (2016) 1379–1388.
- [35] J.I. Goldstein, D.E. Newbury, P. Echlin, D.C. Joy, A.D. Romig, C.E. Lyman, et al., *Scanning Electron Microscopy and X-ray Microanalysis*, second ed., Plenum Press, New York, 1992.
- [36] M. Jordan, Electrodeposition of tin-lead alloys, in: M. Schlesinger, M. Paunovic (Eds.), *Modern Electroplating*, John Wiley & Sons, New York, 2000.
- [37] C. Arroyave, M. Morcillo, Effect of nitrogen oxides in atmospheric corrosion of metals, *Corros. Sci.* 37 (1995) 293–305.
- [38] M.E. Jenkin, K.C. Clemitshaw, Ozone and other secondary photochemical pollutants: chemical processes governing their formation in the planetary boundary layer, *Atmos. Environ.* 34 (2000) 2499–2527.
- [39] X. Liu, Y. Hu, F. Wang, Y. Pang, H. Liu, A real-time evaluation method on Internet Data Centre's Equipment Corrosion by wireless sensor networks, in: 2016 International Symposium on Computer, Consumer and Control (IS3C), IEEE, 2016, pp. 622–627.
- [40] C. Muller, What's corroding your control room? *Chem. Eng.* 124 (2017) 35–43.

This page intentionally left blank

Pb-free solder— microstructural, material reliability, and failure relationships

5

Guang Ren¹, Maurice N. Collins¹, Jeff Punch¹, Eric Dalton¹ and Richard Coyle²

¹*Stokes Labs, Bernal Institute, University of Limerick, Limerick, Ireland*

²*Nokia Bell Labs, Murray Hill, NJ, United States*

5.1 Introduction

Electronic products such as smart phones, wearable devices, and various portable gadgets are getting smaller with increasing functionality, enabled by the development of miniaturized, high density integrated components. Solder joints play a key role in these products as interconnect materials between components and printed circuit boards (PCBs), serving both an electrical and mechanical function. The lifetime of commonly used electronic products greatly depends on the reliability of these solder joint materials, operating under various loading conditions such as drop impact, vibration, thermal cycling, and high electrical currents.

5.1.1 Development of Pb-free solder alloys

In 2006 the European Union introduced a restriction on hazardous substances (RoHS) directive that effectively phased out the utilization of SnPb solder in the electronics industry. Since the legislative driven Pb-free initiative, the establishment of near-eutectic Sn–Ag–Cu (SAC) alloys has marked the beginning of Pb-free solder alloy development in the electronic packaging industry [1–5]. Second-generation alloys with lower Ag content were introduced to address shortcomings such as poor mechanical shock performance and high cost [2,6,7]. In response to complex new manufacturing and high reliability requirements, such as automotive, telecommunication, mission-critical aeronautics, military, and medical applications, where high operating temperatures, rapid thermal and power cycles, long dwell in combination with vibration, and mechanical shock are common, new third generation Pb-free alloys have been developed [8]. Table 5.1 lists the trade name and nominal compositions of some alloys that will be discussed in this chapter.

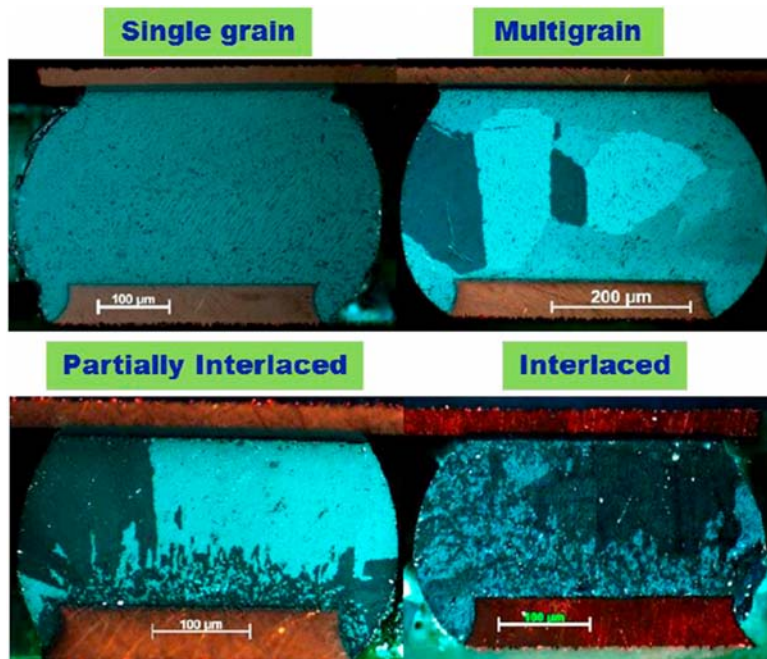
Table 5.1 Trade names of alloys and their nominal compositions in wt.% [9].

Solder alloy composition	Trade name
Sn–37Pb	SnPb
Sn–0.7Cu–0.05Ni + Ge	Sn100C
Sn–0.3Ag–0.7Cu	SAC0307
Sn–0.3Ag–0.7Cu + 0.09Bi	SACX
Sn–1.0Ag–0.5Cu	SAC105
Sn–1.0Ag–0.5Cu + 0.03Mn	SAC105 + Mn
Sn–1.0Ag–0.5Cu + 0.05Ni	SAC105 + Ni
Sn–1.0Ag–0.7Cu	SAC107
Sn–1.7Ag–0.7Cu + 0.4Sb	SACi
Sn–2.0Ag–0.5Cu	SAC205
Sn–2.0Ag–0.5Cu + 0.05Ni	SAC205 + Ni
Sn–2.5Ag–0.8Cu–0.5Sb	Castin
Sn–3.0Ag–0.5Cu	SAC305
Sn–3.8Ag–0.7Cu	SAC387
Sn–3.8Ag3–0.7Cu0–3.0Bi–1.4Sb–0.15Ni	Innolot
Sn–3.9Ag–0.6Cu	SAC396
Sn–4.0Ag–0.5Cu	SAC405

5.1.2 Failure and microstructure

The combination of thermal fatigue and solder joint creep is considered a major source of failure of surface-mount components [10]. Accelerated temperature cycling (ATC) is a widely used reliability evaluation technique that simultaneously produces static and strain-induced microstructural evolution in solders, thereby accelerating fatigue and creep damage mechanisms. The number of cycles to reach 63.2% failure (N63.2) extracted from Weibull plots is usually accepted as the indicator of characteristic lifetime. High homologous temperatures associated with Pb-free solders contribute further to creep with associated recrystallization processes further complicating the failure mechanisms. It is well understood that the mechanical performance of an alloy is determined by its microstructure; therefore its evolution during ATC is critical. Studies have confirmed that the creep and fatigue behavior of solder alloys is significantly influenced by microstructure, substrate surface finish, mechanical loading, and thermal cycling profile [11–13]. These studies have been used to predict solder performance in a range of likely deployment conditions.

As a major component in every SAC solder alloy, Sn, which solidifies as a β -Sn phase, has a tetragonal unit cell and displays large anisotropies [9]. The orientations of Sn grains are critical to estimate the thermomechanical behavior of SAC solder joints. Polarized light microscopy (PLM) is an useful technique to identify Sn grain morphology after reflow and thermal cycling. As shown in Fig. 5.1, different crystal orientations result in different colors due to the

**FIGURE 5.1**

Various Sn grain morphologies [14].

birefringence of Sn. A more powerful technique, electron backscattered diffraction (EBSD), can tell the orientation of each Sn grain.

As an example, Fig. 5.2 shows the microstructures of SAC–Mn solder joints from different phases under ATC, indicating the microstructural development. When the solder joint is cycled to its 20% characteristic life, the microstructural change is not significant. Upon reaching 50% characteristic life, precipitates in the upper area adjacent to the component clearly coarsens while a tiny crack appears in the same region. At this stage, new grains with high angle grain boundaries are formed due to recrystallization, enhanced by the thermal strain, as shown in Fig. 5.3. Subsequently, the fatigue crack propagates along the continuous network of high angle grain boundaries, through the recrystallized area, leading to the eventual failure, as illustrated in Fig. 5.4.

5.1.3 An overview of the chapter

An improved understanding of failure mechanisms provides insights into how to enhance the fatigue performance of Pb-free solder joints, in order to better survive extremes of temperature and strains, thereby allowing deployment in harsher environments. This chapter will specifically focus on Pb-free solder alloys for electronic interconnections, describing their microstructural evolution,

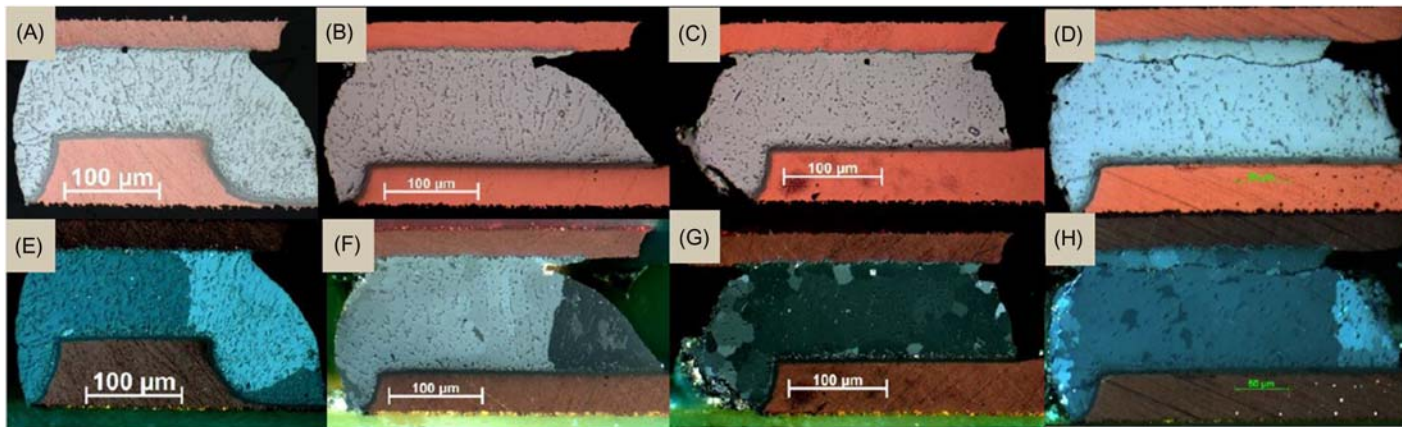


FIGURE 5.2

Bright-field and PLM images of SAC–Mn solder joints from left to right: (A, E) as reflowed, (B, F) cycled for 20% of characteristic life, (C, G) cycled for 50% of characteristic life and (D, H) failed [14].

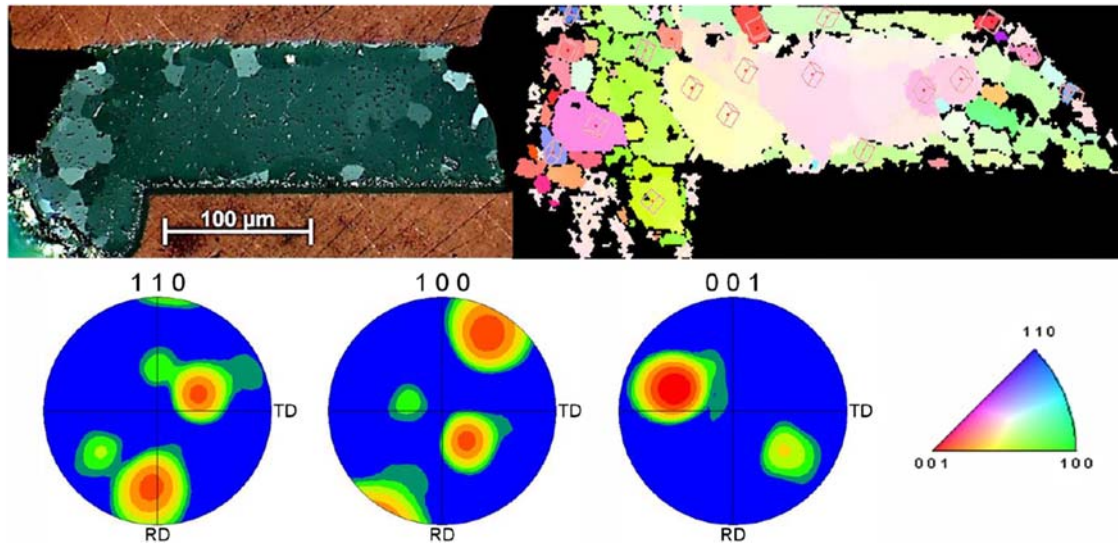
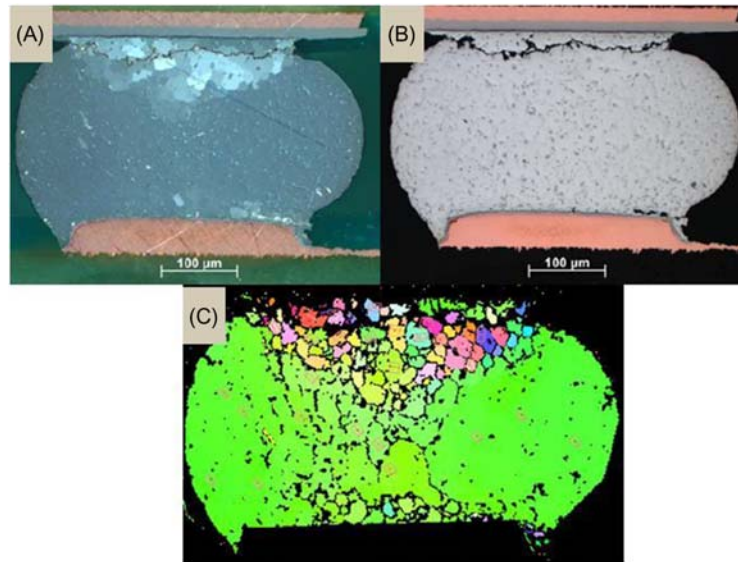


FIGURE 5.3

PLM image and EBSD map of SAC-Mn solder joint after 50% characteristic life [14].

**FIGURE 5.4**

Microstructure of solder joint failed after ATC: (A) PLM, (B) bright field, (C) EBSD map [15].

recrystallization and failure mechanisms through crack growth and initiation analysis. Case studies on thermal fatigue and isothermal mechanical performance of various Pb-free solders will be reported in order to outline the evolution of these materials:

- Case study I deals with Pb-doped solder alloys which are a result of processing mixed assemblies of Pb and Pb-free materials as there has been a Pb-free exemption from legislation for some high reliability applications.
- Case study II details the reliability and failure mechanisms associated with the first- and second-generation totally Pb-free interconnect materials based on the SAC alloy. Special emphasis has been dedicated to Ag content and the influence of thermal cycling.
- Case study III outlines the higher reliability applications and the incorporation of micro-alloy additions to achieve these reliability standards.
- Case study IV looks to the future and the development of low temperature Sn–Zn-based solder alloys for temperature sensitive applications. The realization of these alloys has been in conjunction with solder flux developments that have allowed surface-mount processing of these materials for the first time. Here, we present preliminary reliability data for these emerging solder alloys.

5.2 Case study I—Pb-doped solder alloys

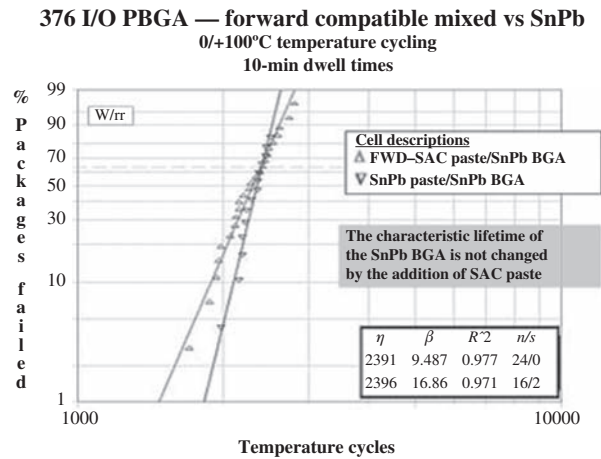
For high reliability fields, such as telecommunication, military, aeronautics, and medical, exemptions were given to producers allowing the Pb-free transition to proceed at a slower pace. Before full-scale Pb-free implementation, SnPb manufacturing processes were allowed to continue in tandem with the development of high reliability Pb-free alternative approaches. However, the supply chain is dominated by the high volume consumer electronic market, which has completed the transition to Pb-free solder assembly. As a result, the adoption of Pb-free components and materials has become increasingly necessary for high reliability equipment producers, due to the limited availability of SnPb ball grid array (BGA) components on the market. Using Pb-free SAC components with SnPb paste, backward compatible soldering offers an alternative to complete a swift conversion to Pb-free manufacturing. On the other hand, using SnPb components with Pb-free solder pastes, forward compatibility soldering provides a workaround for high reliability users to overcome the challenges faced in supply chains.

Substantial work has been carried out by electronic manufacturing industries, along with academia, some employing ATC, to assess the effect of Pb on the reliability of aforementioned mixed solder assemblies [16–29]. Despite a large number of experimental inconsistencies, it can be concluded that acceptable solder joint quality can be achieved with mixed alloy assemblies. Depending on the package and ATC profiles, the mixed solder fatigue life can be better, equal, or sometimes worse than that of SnPb or SAC.

When SnPb BGAs are used with SAC305 paste, it is named as “forward compatible mixing,” which will be introduced in [Section 5.2.1](#). On the other hand, [Section 5.2.2](#), will present “backward compatible mixing,” referring to the combination of SAC305 BGAs and SnPb paste, which is a more common practice due to the supply chain issue as discussed previously.

5.2.1 Forward compatible mixing

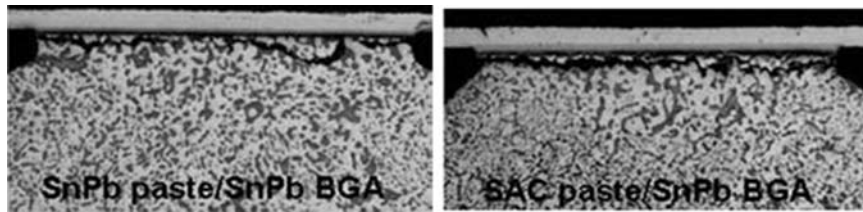
A study by Coyle et al. investigated the thermal fatigue reliability of SAC305 paste/SnPb BGA forward compatible assemblies, using SAC paste/SAC BGA assemblies and SnPb paste/SnPb BGA assemblies as controls, with a 0/100°C ATC profile [30]. The Weibull plot in [Fig. 5.5](#) shows that the characteristic lifetime of SAC305 paste/SnPb BGA forward compatible mixed assembly is practically identical to that of SnPb. This is due to the metallurgical composition of the forward compatible assembly being equivalent to that of SnPb with only a trace amount of Ag addition. However, higher Ag content can moderately improve the thermal fatigue behavior, as in for example Sn36Pb2Ag solder [31]. The forward compatible mixed cell has a lower slope than the SnPb cell, an indication of greater sample-to-sample variation which may be attributed to inconsistencies in mixing that results in local deviations from the eutectic composition.



Assembly	Characteristic life η	Slope β	Coefficient of determination R^2
FWD — SAC paste/SnPb BGA	2391	9.487	0.977
SnPb paste/SnPb BGA	2396	16.86	0.971

FIGURE 5.5

A Weibull plot of a forward compatible mixed assembly and an SnPb assembly [30].

**FIGURE 5.6**

Optical microscope (OM) images of failed SnPb assembly (left) and forward compatible mixed assembly (right) [30].

Fig. 5.6 shows the microstructures of a failed forward compatible mixed assembly and a failed SnPb assembly, which both exhibit typical SnPb fatigue failure modes, with grain coarsening induced by the combination of strain and thermal exposure during temperature cycling. The similarity of the failure modes is consistent with the practically identical characteristic lives.

5.2.2 Backward compatible mixing

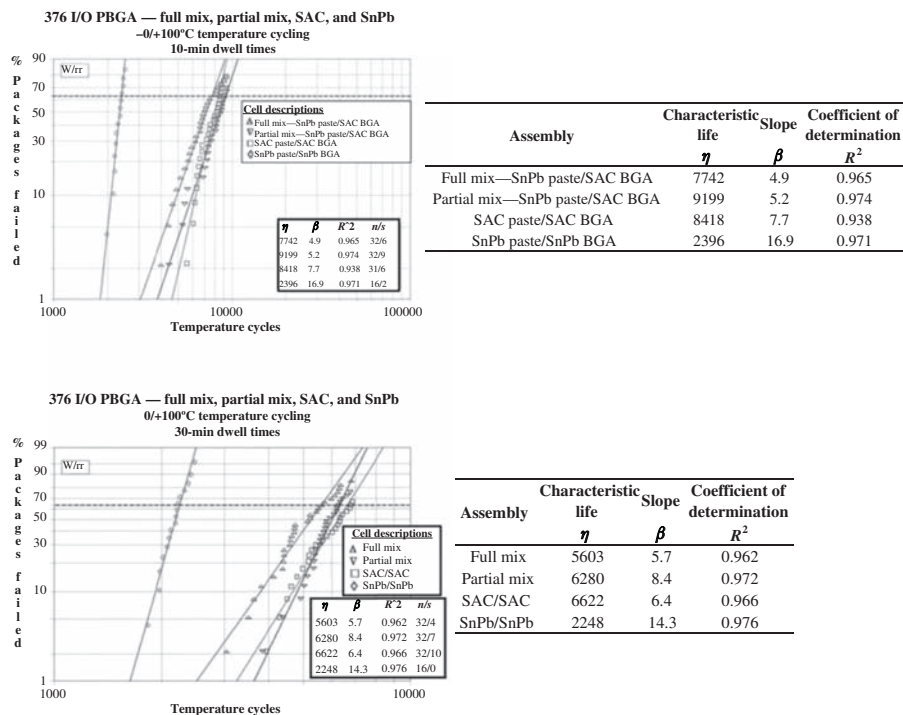
The study also investigated the thermal fatigue reliability of an SnPb paste/SAC305 BGA backward compatible assembly, with both fully mixed and partially mixed assemblies, using multiple dwell times [30].

5.2.2.1 Full mix and partial mix

Fig. 5.7 shows the Weibull plot of mixed, SAC, and SnPb solder joint data at 10- and 30-minute dwell times during 0/100°C ATC (10-minute ramp between temperature extremes). Overall, the reliability of these mixed solder joints outperformed that of SnPb joints by a factor of three. The characteristic lifetime of fully mixed solder assemblies is only 10%–15% lower than the pure SAC assembly, which indicates that Pb contamination does not significantly deteriorate the thermal fatigue reliability of SAC solder joints. Meanwhile, the reliability of partially mixed solder assemblies is comparable to SAC assemblies during 10-minute dwells, but is 10% higher during 30-minute dwell times.

It was hypothesized that by forming an Sn–Pb–Ag eutectic or an intermetallic compound (IMC), Pb redistribution and segregation at interfaces could accelerate thermal fatigue and therefore decrease the reliability of mixed assemblies [16,17,32,33]. However, without direct evidence of induced fatigue cracking, Pb distribution results in defects that lead to mixed mode or nonfatigue failures, but this is not deemed to represent a long-term reliability risk. Snugovsky et al. suggest that it is the interaction of Pb morphology and solder microstructure which controls the reliability [34].

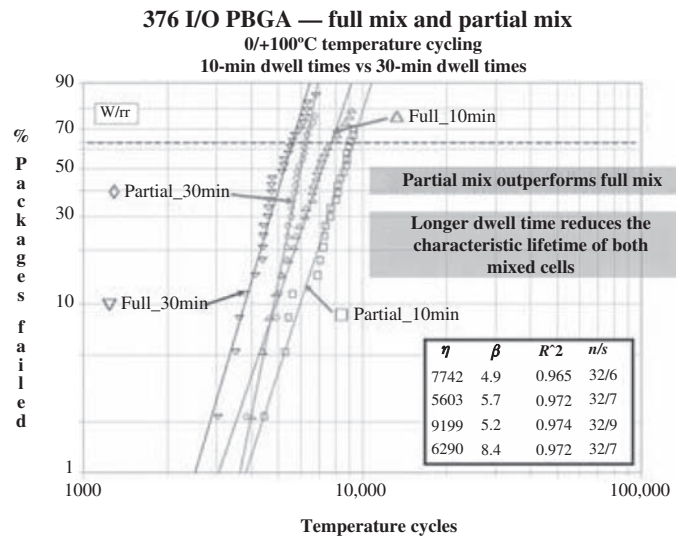
As full mixing cannot always be achievable in product manufacturing, it is essential to understand any possible reliability limitations of partial mixing. It had

**FIGURE 5.7**

Weibull plots of fully mixed, partially mixed, SAC, and SnPb assemblies at 10-min dwell (upper) and 30-min dwell (lower) [30].

been assumed that a fully mixed system is required for optimum reliability, however, as shown in Fig. 5.8, this is not the case and partially mixed assemblies in fact display better reliability than fully mixed assemblies for both 10- and 30-minute dwells during ATC. Since satisfactory solder joint quality can be attained with partial mixing assembly, the processing window for mixed alloy assembly is less constrained during manufacturing. Similar to their Pb-free counterparts, the characteristic lifetime of both mixed assemblies reduced with longer dwell time. To be specific, the reliability of mixed alloy assemblies was overestimated by 25% under ATC with the shorter, 10-minute dwell time.

In a fully mixed assembly, the Pb introduced by solder paste distributes randomly but not homogeneously throughout the solder joint during reflow, extensively from PCB pad to BGA pad, as shown in Fig. 5.9. On the other hand, in a partially mixed assembly, the Pb only distributes in the lower region adjacent to PCB pad, ~60%–90% of the solder joint. The upper region of the partially mixed assembly does not melt during the surface-mount assembly process and shows an unmixed microstructure similar to that of a conventional SAC305 solder joint, as shown in Fig. 5.10.



Assembly	Characteristic life η	Slope β	Coefficient of determination R^2
Full_10min	7742	4.9	0.965
Full_30min	5603	5.7	0.972
Partial_10min	9199	5.2	0.974
Partial_30min	6290	8.4	0.972

FIGURE 5.8

A Weibull plot of fully mixed and partially mixed assembly at 10- and 30-min dwell times [30].

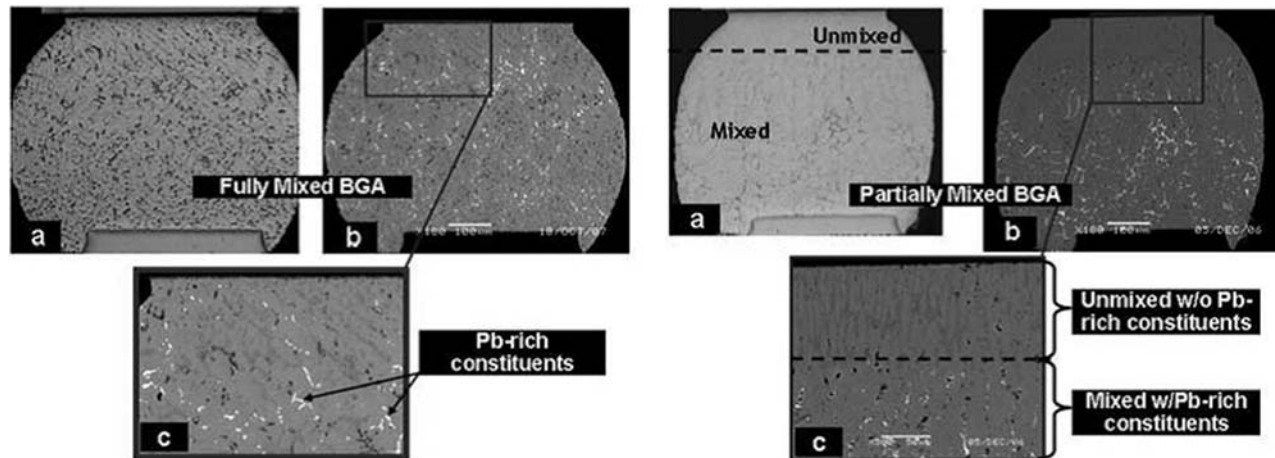


FIGURE 5.9

Optical microscope (OM) images of fully mixed solder joints (left) and partially mixed solder joints (right) [30].

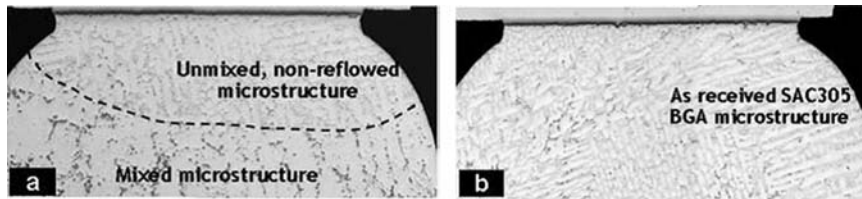


FIGURE 5.10

OM images of partially mixed solder joint (left) and SAC305 solder joint (right) [30].

5.2.2.2 Effect of accelerated temperature cycling profile

Regardless of solder alloy composition, surface-mount assembly processing conditions or ATC profiles, the first failure in BGA components almost always occurs in the outer most row of the center ball array close to the die edge, due to the higher strains caused by coefficient of thermal expansion (CTE) mismatches. After 0/100°C ATC with either 10-minute dwell or 30-minute dwell times, as shown in Fig. 5.11, all failed solder joints display very similar fracture features, consisting of crack branching, recrystallization, and cavitation [30], which is in agreement with the report by Dunford et al. [35].

More details can be observed in Fig. 5.12: located at Sn dendrite boundaries and throughout the solder ball, Pb particles have not interacted with the crack propagation path, since the fatigue fracture mode of the fully mixed solder joint has not obviously altered from that of a typical SAC assembly [30]. Combining these observations with the Weibull data, a conclusion can be drawn that Pb has minimal, if any, influence on the thermal fatigue reliability of the mixed solder joint assembly.

With higher magnification images shown in Fig. 5.13, bright white Pb particles can be identified in the upper nonreflowed region, indicating that Pb has migrated during temperature cycling, from the lower mixed region of the solder joint into the upper unmixed region adjacent to the interface. However, the Pb particles are much smaller compared to those in the lower reflowed region or in the fully mixed assembly. In a 30-minute dwell time ATC, a larger number of fine Pb particles appear in the upper nonreflowed region, due to increased cumulative time for Pb diffusion at peak temperature.

At the peak temperature of ATC, the Ag_3Sn IMC particles coarsen and diminish the creep resistance of the SAC solder joint, leading to a reduced fatigue reliability. Reliability of a SAC assembly is therefore dwell time dependent as longer dwell times can boost IMC coarsening, resulting in a reduced thermal fatigue characteristic lifetime. With more Pb particles existing in the critical region, however, the characteristic lifetimes of partially mixed and SAC assemblies are similar for longer dwells. It can therefore be assumed that the fatigue reliability is determined more by SAC microstructure evolution rather than the presence of Pb.

As shown in Fig. 5.14, the 10-minute dwell coarsens the Ag_3Sn IMC particles in all three solder joints, but the partially mixed assembly displays slightly larger Sn grains with a much wider network of Ag_3Sn IMC particles, a microstructure found to improve thermal fatigue resistance [18,36,37]. The integrity of the

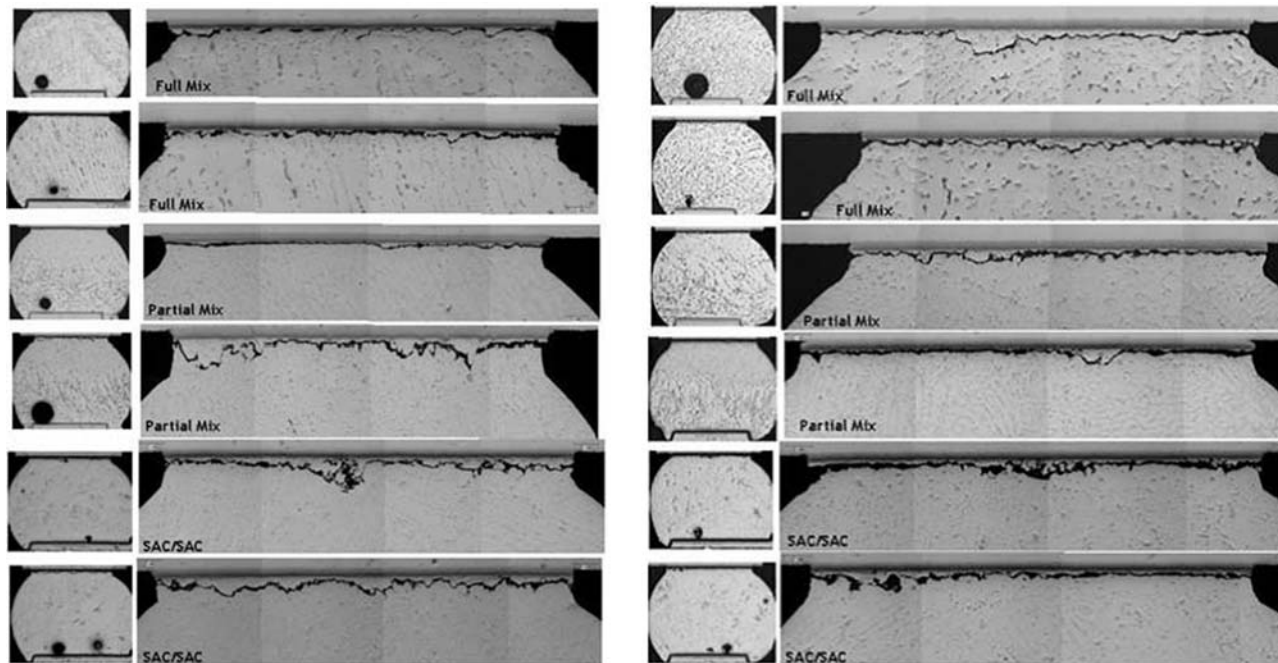
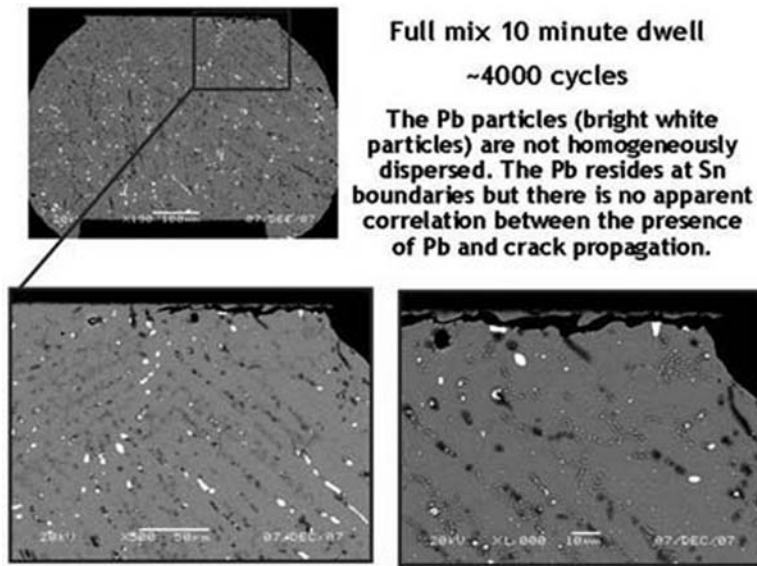


FIGURE 5.11

Thermal fatigue failure of fully mixed, partially mixed, and SAC solder joints at 0/100°C ATC with 10-min dwell (left) and 30-min dwell (right) [30].

**FIGURE 5.12**

Distribution of Pb particles in a fully mixed solder joint after 0/100°C ATC with 10-min dwell [30].

Ag_3Sn network in the partially mixed assembly has been retained to a large extent, which could be the reason why it achieves an even higher characteristic lifetime than the SAC assembly.

Significant coarsening is observed for 30-minute dwell times, and this diminishes the Ag_3Sn network of the SAC solder joint while almost eliminating it from the fully mixed one. However, the interdendritic Ag_3Sn network of partially mixed solder joint remains more intact than that of the SAC joint. Regardless of the Pb, the microstructure of the partially mixed assembly is quite similar to that of SAC, explaining why they display similar characteristic lives.

On the other hand, for the fully mixed solder joint, a dramatic Ag_3Sn coarsening and Sn grain growth is observed for both 10- or 30-minute dwells, and therefore, the lifetime consistently ranks the lowest among the three assemblies. It can be concluded that rather than Pb distribution from the mixed assembly processing, the thermal fatigue reliability is more dependent on SAC microstructure evolution, which is strongly influenced by the ATC profile.

5.2.2.3 Effect of Ag content

Collins et al. investigated the thermal fatigue performance of various SAC-based solder alloys with lead terminations using ATC and 2512 chip resistors, and found that with a relatively higher Ag content, SAC305, and SAC405 exhibit higher characteristic lifetimes than SAC105 and SAC205, as shown in Fig. 5.15. It was concluded that increased Ag content contributed to a refined grain structure which

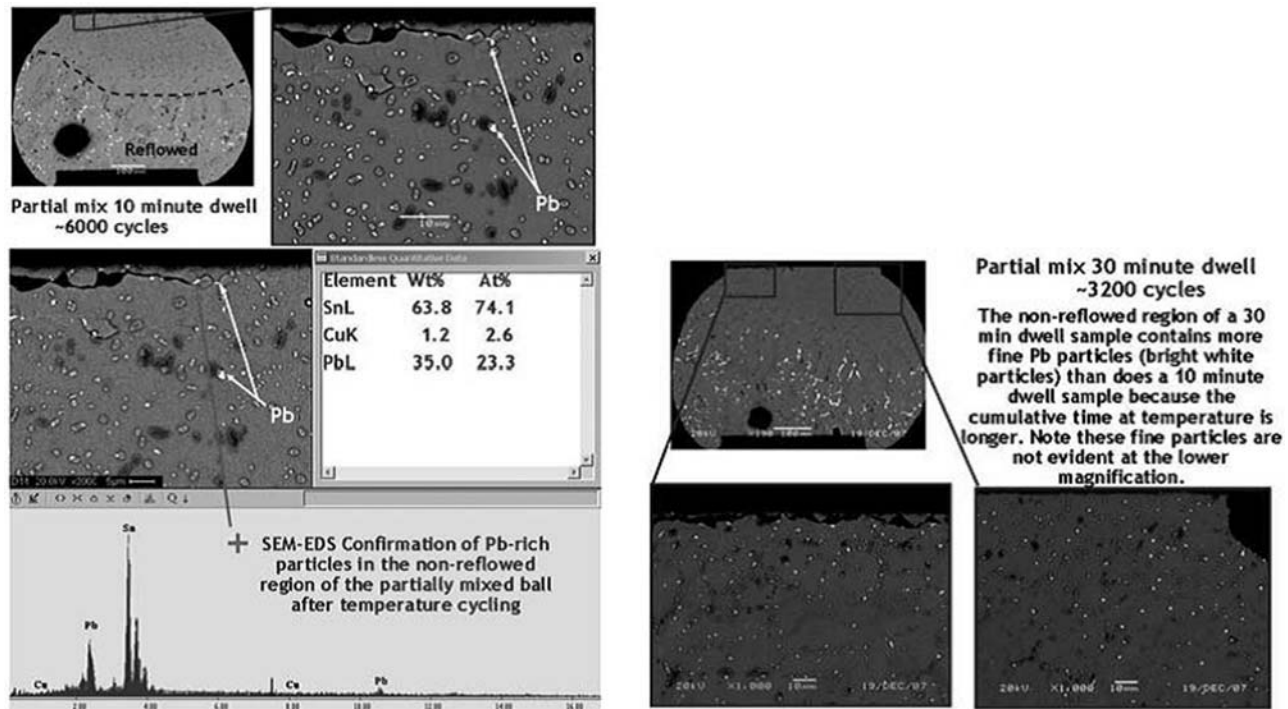


FIGURE 5.13

Scanning electron microscope (SEM) images of partially mixed solder alloys failed after ATC with 10-min dwell (left) and 30-min dwell (right) [30].

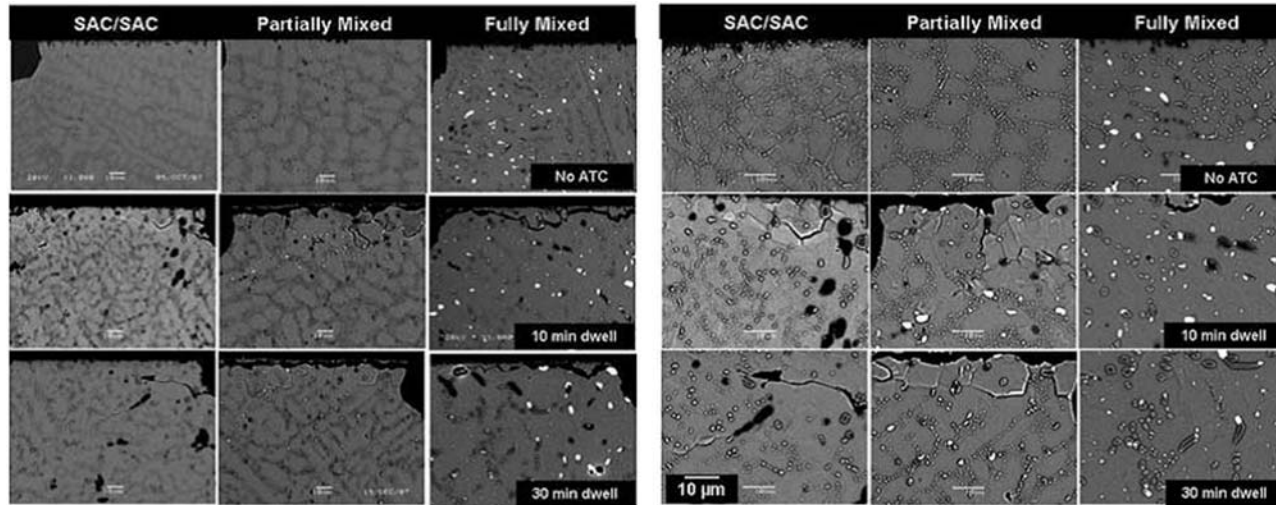


FIGURE 5.14

SEM images with lower magnification (left) and higher magnification (right) showing the microstructure evolution of SAC and mixed solder joints under ATC with different dwell times [30].

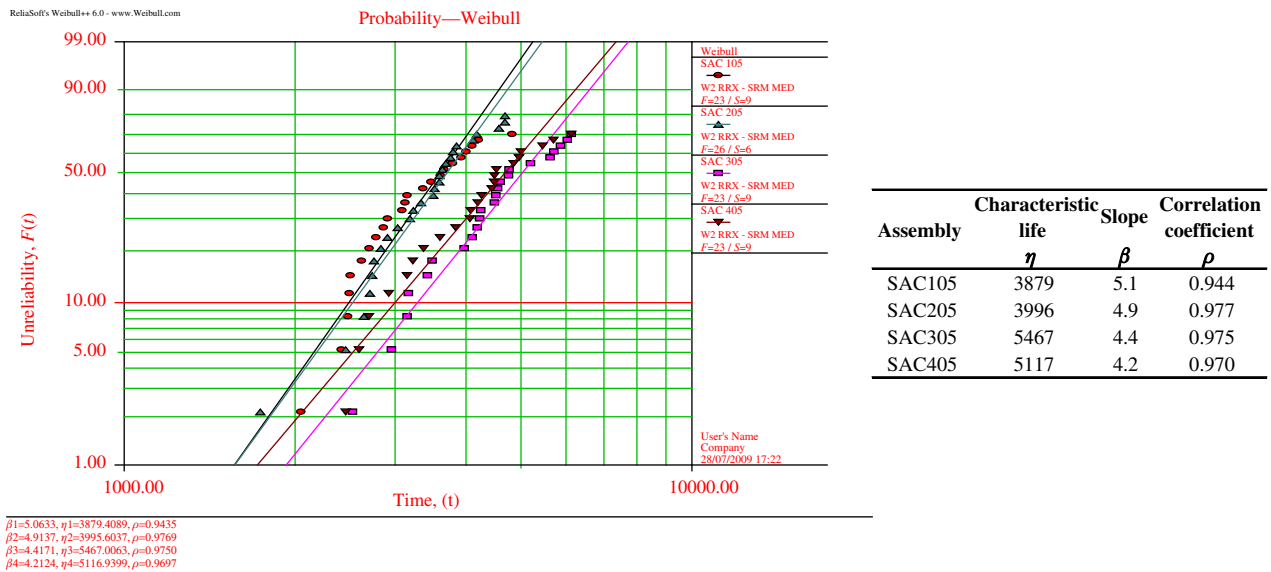


FIGURE 5.15

A Weibull plot comparing reliability of SAC105, SAC205, SAC305, and SAC405 assemblies.

Data from the same study as Adapted from M.N. Collins, J. Punch, R. Coyle, M. Reid, R. Popowich, P. Read, et al., Thermal fatigue and failure analysis of SnAgCu solder alloys with minor Pb additions, IEEE Transact. Components Packaging Manufact. Technol. 1 (2011) 1594–1600 [38].

coarsened at a slower rate under the stresses imposed by the thermal cycling, in conjunction with a finer distribution of Ag_3Sn IMC that seems have had a dispersion strengthening effect [38].

As shown in Fig. 5.16, all four SAC alloys showed similar microstructures that consist of a primary Sn matrix embedded within a combination of darker

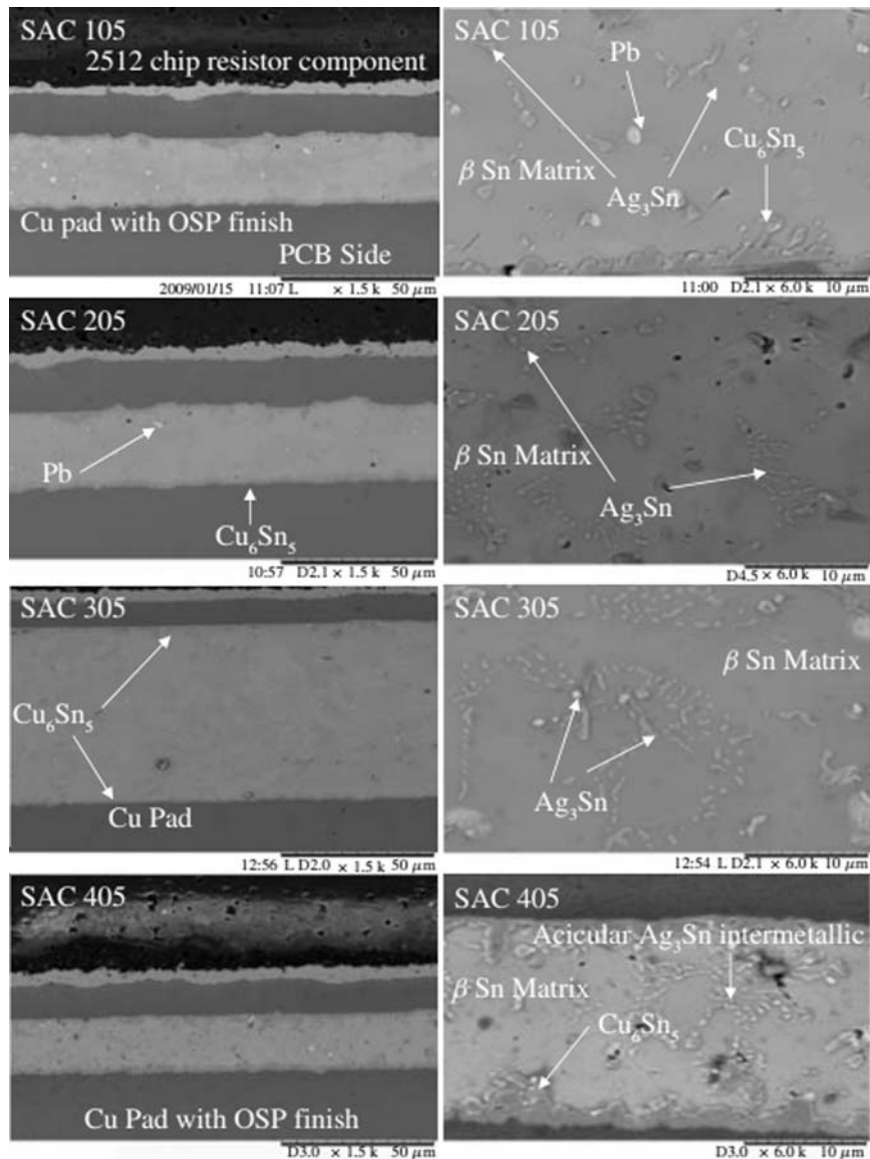


FIGURE 5.16

SEM images of as received microstructures of SAC105, SAC205, SAC305, and SAC405 [38].

Cu-rich Cu_6Sn_5 and brighter flake-like Ag-rich $\epsilon\text{-Ag}_3\text{Sn}$ IMCs. However, the volume fraction of the primary Sn phase in SAC305 and SAC405 is lower than that in SAC105 and SAC205. The higher Ag content in SAC405 facilitates the primary solidification of more Ag_3Sn IMC, leading to the formation of a typical hypereutectic microstructure containing dispersed fine needle-like IMCs within an Sn matrix. Larger $\epsilon\text{-Ag}_3\text{Sn}$ IMC plates formed in the SAC405 solder alloy were found to effectively act as blocks for fatigue crack propagation, thus offering a strengthening mechanism.

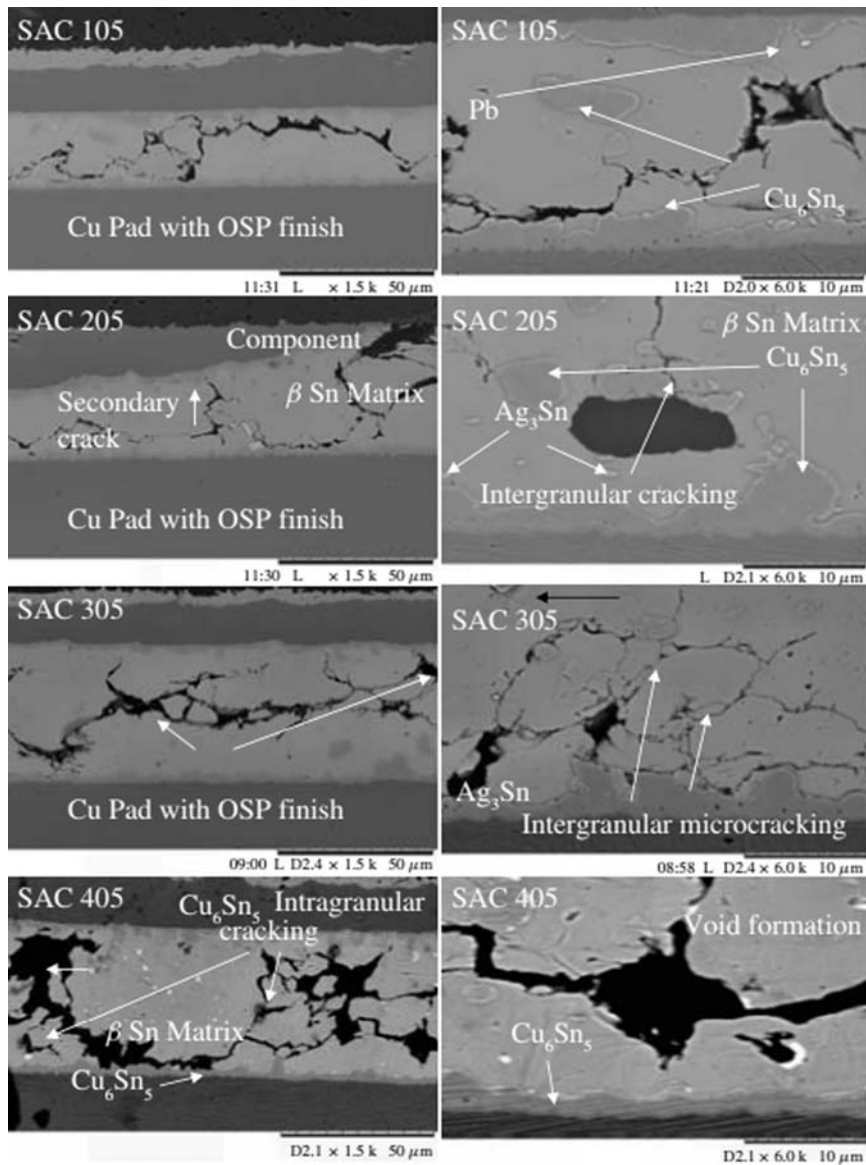
For samples containing Pb terminations, it can be clearly seen that the Pb has diffused from the terminations into all four solder alloy microstructures, displayed as dispersed bright particles, evenly distributed throughout the solder joint, accumulating predominantly at grain boundaries. The authors suggest that creep strain originating from grain-boundary sliding can be affected by Pb precipitates at the grain boundaries, and strain that originates from dislocation movement within the grains can be also be influenced by Pb through a solid solution strengthening effect. Both of these processes contribute to a more creep resistant alloy. Furthermore, during the thermal cycle, the Pb phase can dissolve at higher temperature and subsequently precipitate at lower temperatures, nucleating at dislocations and thereby restricting their movement. Nonetheless, the easy recrystallization of primary Pb may have a negative influence on the reliability of the solder joint.

Fig. 5.17 shows the microstructures of solder joints after 1500 thermal cycles. A fine crack with multiple fronts has propagated through the joint, with portions located in the bulk solder and other portions propagated into the intermetallic layer (IML). Overall, the wide, single path style cracks were observed near the component side, whereas the shattered appearance was likely to occur near the Cu pad of the PCB side. Vertical cracking was observed in failures of all four SAC-based Pb-free solder joints. Cracks appear to propagate alongside the outline of Cu_6Sn_5 phases near the PCB pad, as shown in the microstructure of the SAC105 solder joint. In the crack regions of SAC305 and SAC405 solder joints, several small voids can be observed, presumably due to IMC formation and internal stresses which subsequently deform the joint. These small voids were most often observed at or near the thick IML. Intragranular cracking can also be observed in the SAC305 and SAC405 solder joints.

Fracture surface microstructures of the SAC105 and SAC205 both at the component and PCB interfaces are shown in Fig. 5.18. Fracture surface morphology observed is quite similar, showing a mixed intergranular and transgranular fracture modes, although the previous SEM images indicate a dominant intergranular fracture mode. Striations associated with fatigue failures can also be observed.

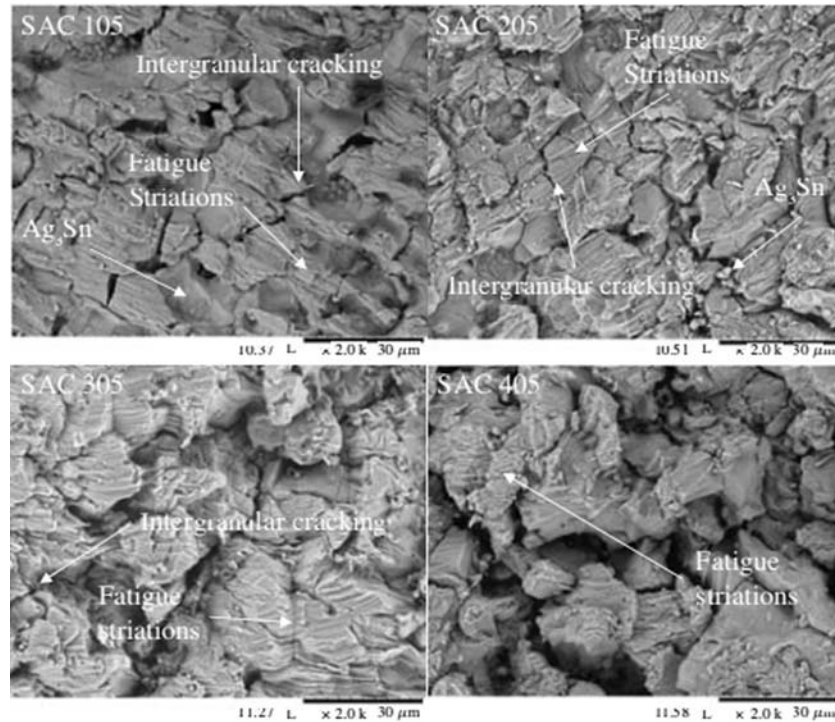
5.2.3 Lessons learnt from case study I

The study of Coyle et al. above shows that the thermal fatigue performance of forward compatible mixed assemblies are similar to that of SnPb/SnPb. However, all SAC-based backward compatible mixings outperform their SnPb counterparts and

**FIGURE 5.17**

SEM images of post-ATC microstructures of SAC105, SAC205, SAC305, and SAC405 [38].

can provide acceptable reliability, even with partially mixing. No evidence has been found that the presence of Pb degrades the reliability of solder joint. On the other hand, the study of Collins et al. shows that Ag content resulted in increased reliability due to greater submicron sized Ag₃Sn formation. Besides solder alloy and

**FIGURE 5.18**

SEM images of post ATC fracture surfaces for SAC alloys [38].

BGA assembly combinations, ATC profiles (including temperature range and dwell time) also has a significant impact on solder joint thermal fatigue performance, and should be taken into consideration when it comes to reliability evaluation.

5.3 Case study II—First- and second-generation Sn–Ag–Cu solder alloys

As already discussed, thermal fatigue is a major failure mechanism for solder joints in surface-mount technology, and it is caused by the CTE mismatch between the component and the PCB [10]. This is of critical importance in many high reliability applications. Back to the time when RoHS was initially enacted, near-eutectic SAC with high Ag content was widely considered as the solder alloy to meet high reliability requirements. For example, Eutectic SAC387 was identified as the best choice by the European Consortium Brite-Euram in the European Union, and SAC396 was recommended by the National Electronics Manufacturing Initiative in the United States [4,39]. However, based on a study

with ATC and thermal shock testing of three SAC alloys with >3 wt.% Ag, the IPC Solder Product Value Council (SPVC), an industry working group consisting mainly of solder suppliers, decided to adopt the mildly hypoeutectic SAC305 as Pb-free replacement in the first phase of implementation [40]. SAC305 was also endorsed by the Japanese Electronics Industry Association (JEITA) [4,39]. Although the study satisfied the ATC requirements for consumer applications, for many high-reliability end users, it was not comprehensive or rigorous enough to justify the adoption of SAC305 in their applications.

Before long, several drawbacks of the near-eutectic SAC were revealed by mainstream volume manufacturing and field experiences in consumer markets, notably poor mechanical shock performance, along with higher cost than the SnPb alloy being replaced, and process shortcomings like rapid dissolution of Cu substrates, shrinkage cracks in fillets due to noneutectic solidification and high dross rates in wave soldering [9]. Over the following years, however, a number of Pb-free solder alloys emerged and significant effort has been invested by both academia and industry to study the reliability of these near-eutectic SAC alloys. It has been concluded that there is no “drop-in” substitute for SnPb solder and that alloy choice is application specific.

The poor performance of near-eutectic SAC305 solder alloys under drop impact loading is associated with its higher strength, modulus, and work hardening, which is attributed to the strengthening effect of Ag. Therefore low Ag alloys have been developed and implemented, to improve the drop/impact behavior of solder joints, under dynamic loading conditions. The content of Ag in the alloy was reduced to around 1 wt.% or less, while maintaining 0.5 wt.% Cu. However, these low Ag alloys do not display sufficient fatigue resistance to survive the temperature cycling required for high reliability applications, such as desktops and servers. Nevertheless, mechanical properties and reliability of SAC solders can be enhanced by means of micro-alloying minor dopants to Sn-based alloys.

The latest news around RoHS exemption renewal for high reliability applications is scheduled to be announced in late 2017. Uncertainty about whether the exemptions will be renewed has provided extra emphasis for high reliability electronic equipment manufacturers to convert to Pb-free solders.

Coyle et al. conducted a detailed study on the thermal fatigue performance of second-generation Pb-free solder alloys, including SnPb, Sn100C, SAC0307, SACX, SAC105, SAC105 + Mn, SAC105 + Ni, SAC107, SACi, SAC205, SAC205 + Ni, SAC305, and SAC405. ATC was imposed in accordance with the IPC-9701A industry test guideline. Assembled circuit boards were thermally cycled with a ramp rate of $10^{\circ}\text{C}/\text{min}$ between temperature extremes consisting $-40/125^{\circ}\text{C}$, $-15/125^{\circ}\text{C}$, $25/125^{\circ}\text{C}$, $-40/100^{\circ}\text{C}$, and $0/100^{\circ}\text{C}$. The hot and cold dwell times were either 10 or 60 minutes [9]. The following subsections will address the influences of the BGA component, Ag content, dwell time, and ATC profile on the number of cycles to failure of studied assemblies. The effect of microstructural evolution on failure mechanism during thermal cycling process will also be discussed.

5.3.1 Effect of ball grid array component

Two BGA components were used in this study, Amkor's ChipArray Ball Grid Array (192CABGA) and ChipArray Thin Core Ball Grid Array (84CTBGA), as shown in Fig. 5.19, with the former being much larger. The CTE of the PCB used was 13 ppm/°C, while the composite CTE of 192CABGA (7.0/7.6 ppm/°C along X/Y axis) was on average 20% lower than that of 84CTBGA (8.5/9.6 ppm/°C along X/Y axis), resulting in a greater CTE mismatch between 192CABGA and the PCB.

The characteristic life of 84CTBGA substantially exceeds (30%–100%) that of 192CABGA. On one hand, the larger CTE mismatch between 192CABGA and PCB leads to a higher strain that the solder joints have to endure during ATC process. On the other hand, because of its larger die size, the distance from the center point of 192CABGA to its outer rows is greater, which drives failure sooner as a result of higher strains. For 0/100°C ATC, the characteristic lifetime of SAC405 was 20% higher than that of SAC105 for 192CABGA, while the difference increased to 40% for 84CTBGA. Similar trends were observed for other alloys in this study when used to compare these components, the higher strain level in 192CABGA resulted in general lower characteristic lifetimes, and was therefore less sensitive to Ag content [9].

5.3.2 Effect of Ag content

With more Ag content in the solder alloy adopted, a higher characteristic lifetime was achieved in ATC, presumably due to the dislocation pinning effect of Ag₃Sn IMC particles. However, in a more aggressive thermal profile, the –40/125°C cycle, the strengthening effect of Ag content on characteristic lifetime was dramatically decreased with either the 192CABGA or 84CTBGA packages [9], probably due to the coarsening of Ag₃Sn precipitates and the interparticle spacing

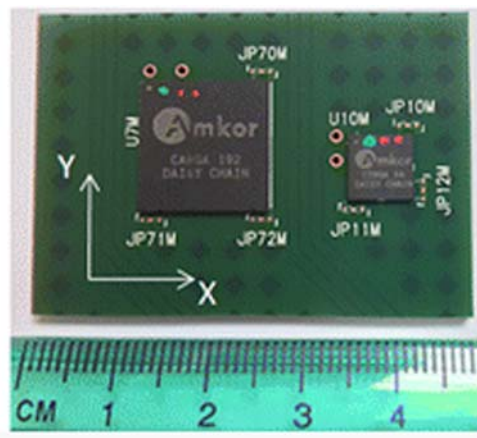


FIGURE 5.19

Components of 192CABGA (left) and 84CTBGA (right) [9].

increasing as a result of Ostwald ripening. There is a possibility that the combination of the highest strain (ΔT) and the highest peak temperature, 125°C, resulted in accelerated Ag_3Sn particle coarsening which greatly diminished their dislocation pinning effect on thermal fatigue life. Similar results can also be found in the report from Lee and Ma [41], who observed that the performance of SAC305 and SAC105 under high-strain test configurations showed minimal difference.

5.3.3 Effect of dwell time

With prolonged ATC dwell time, more creep occurs and this generates more damage that drives time dependent recrystallization. Ag_3Sn precipitates coarsen, grain boundary sliding occurs along with grain rotation which initiates and propagates cracks. All the aforementioned processes generate or accelerate failure and, as a result, characteristic lifetime is reduced. For example, reliability was significantly reduced by 25%–40% for almost all Sn-based Pb-free alloys when the ATC dwell time was extended from 10 to 60 minutes, while keeping the upper/lower temperatures at 0/100°C [9].

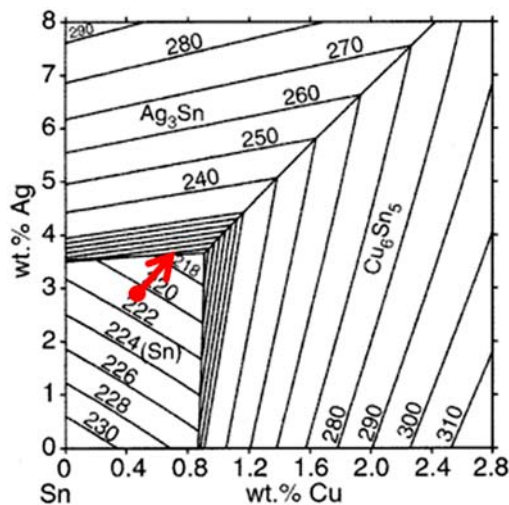
Under either 0/100°C or –40/100 100°C ATC, second-generation Pb-free solder alloys exhibited remarkably improved characteristic lifetimes compared to the eutectic SnPb solder alloy, which was used as a benchmark, even with the extended 60-minute ATC dwell time [9]. First generation Pb-free alloys with higher Ag content were even more reliable.

5.3.4 Effect of accelerated temperature cycling profile

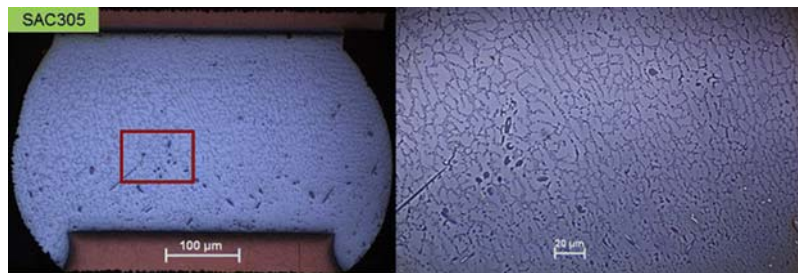
Either a higher peak temperature with the same ΔT , or a higher ΔT with same peak temperature, leads to the reduction of characteristic lifetime. Generally, there is a 20%–50% reduction in lifetime under more aggressive thermal cycle test conditions [9]. Predictive models show that the shear strain generated in the Pb-free solders is proportional to the CTE mismatch and ΔT [42]. Therefore larger ΔT generates greater strain which drives Ag_3Sn coarsening and recrystallization, which occur at peak temperature with the longer dwell providing more time for recovery processes. As the reliability reduction is not quantitatively consistent from alloy to alloy, it is difficult to determine if ΔT (strain) or dwell time is the dominant factor involved.

5.3.5 Microstructural evolution and failure mechanisms

As indicated in Fig. 5.20, solidification of SAC305 initiates at the *red mark* (gray in print version), the composition of remaining liquid moves down the temperature gradient in the direction of the arrow, freezing out of primary dendrites until reaching the pseudo-binary Sn– Cu_6Sn_5 eutectic. As the solidification of this eutectic advances, the composition moves toward the ternary eutectic Sn– Ag_3Sn – Cu_6Sn_5 , where the remaining liquid solidifies. Taking a typical SAC

**FIGURE 5.20**

The liquidus projection of the tin-rich corner of Sn–Ag–Cu equilibrium phase diagram with equilibrium freezing trajectories of SAC305 [43].

**FIGURE 5.21**

As received SAC305 solder ball with 192CABGA package [9].

alloy as an example, the microstructure of SAC305 is dominated by primary Sn dendrites, with fine Ag_3Sn IMC particles dispersed on the Sn boundaries to outline the shape of the dendrite arms, with larger Cu_6Sn_5 IMC particles randomly distributed in the interdendritic regions, as illustrated in Fig. 5.21.

Presumably, under equilibrium conditions, the microstructures of other SAC-based alloys would be similar, only differing in the volume fraction of the Ag_3Sn IMC phases in the interdendritic regions, which should be proportional to the content of Ag in the solder alloy. However, even with the SAC305 near-eutectic composition, during practical reflow soldering, it is usually the nonequilibrium conditions that determine the solidification sequence, which subsequently dominates the distribution of the Ag_3Sn IMC phase. The degree of undercooling can affect the morphology of the Sn grains as well as the size and distribution of

precipitates. As a large amount of undercooling cannot be fulfilled to nucleate the β -Sn, the solder cools well below the equilibrium liquidus temperature before nucleation actually occurs. Few nuclei can be formed due to the difficulty of nucleation, resulting in limited Sn grains [9].

BGA solder joints, typically form either a single Sn grain, or the so-called beach ball structure (Fig. 5.22A–C), in which Sn grains arranged radially around a point showing clearly defined boundaries. However, in land grid array (LGA) solder joints with relatively smaller volume, alternatively, an interlaced twinning microstructure usually takes shape (Fig. 5.22F). It has been presumed that higher percentages of interlaced joints may cause higher characteristic lifetimes in ATC, as indicated in Fig. 5.23.

Fig. 5.24 shows the cross-sectional PLM image of the SAC405 solder joint, the microstructure of which consists of a fine-grain interlaced twinned morphology, as well as the aforementioned beach ball morphology, which is made up of several large Sn grains with varying crystallographic orientations. Generally, the beach-ball structure is found in samples solidified at higher temperatures, whereas the interlaced twinning morphology has been observed more commonly in solder alloys with higher Ag content and in smaller scale solder joints [44,46–49]. Also, micro-alloying elements like Ni could possibly promote the formation of an interlaced structure [50].

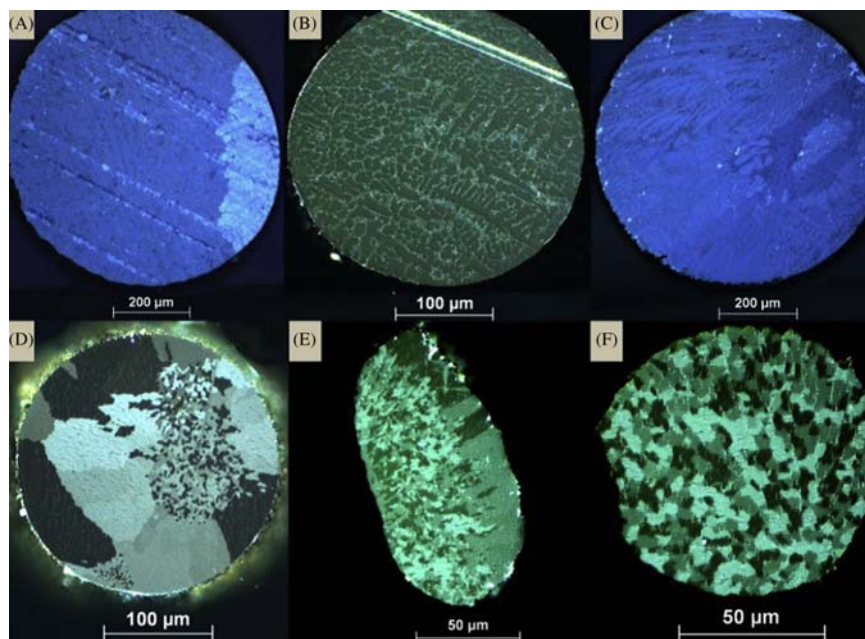
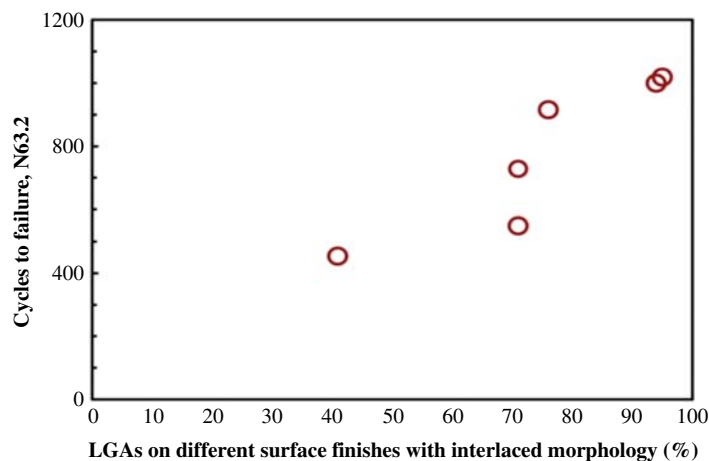
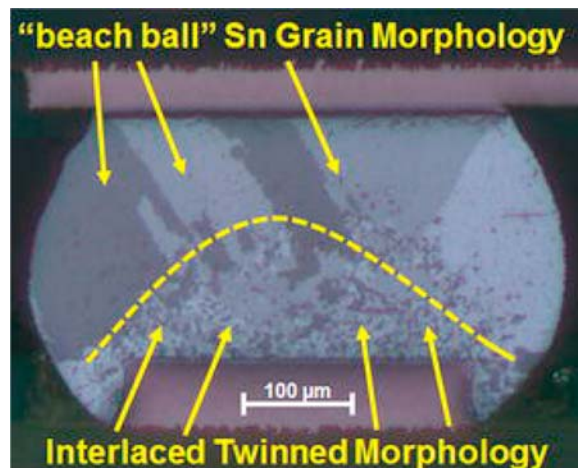


FIGURE 5.22

PLM images showing the microstructure of SAC305 solder joints from various of sizes and solidification temperatures: (A) 750 μm , 200°C; (B) 300 μm , 195°C; (C) 750 μm , 181°C; (D) 300 μm , 172°C; (E) 100 μm , 165°C; and (F) 125 μm , 138°C [44].

**FIGURE 5.23**

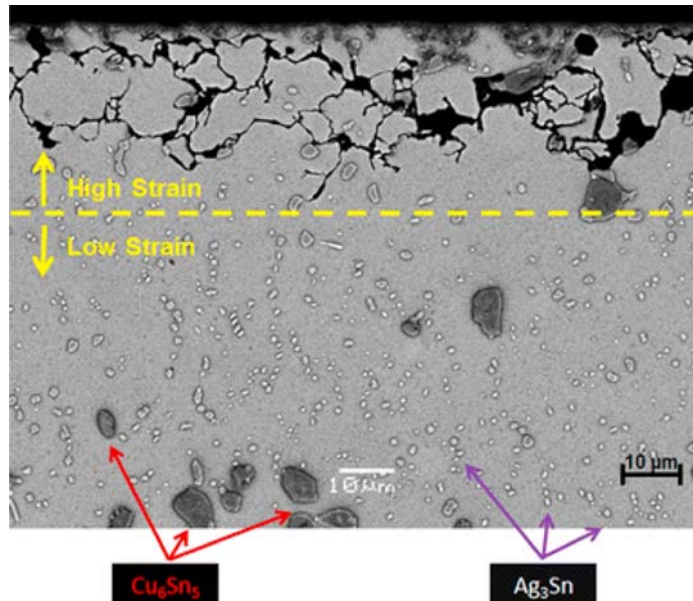
Relationship between the characteristic lifetimes of LGA solder joints and percentage of interlaced joints [45].

**FIGURE 5.24**

PLM image of a SAC405 ball [9].

Other reports also found that solder joints with this particular fine-grain interlaced twinned morphology exhibit higher thermal fatigue resistance [51,52].

With the similar mechanisms of crack nucleation and propagation, Pb-free SAC-based solder alloys usually show typical thermal fatigue failure under various ATC tests, independent of dwell time. As illustrated in Fig. 5.25, in the upper region adjacent to the IML, the relatively higher strain accelerates the coarsening of Ag_3Sn IMC particles and the recrystallization of Sn grains, followed by sliding

**FIGURE 5.25**

Microstructure of SAC305/192CABGA solder joint failed after 0/100°C, 10-min dwell ATC [9].

and rotation of the small grains, and a fatigue crack then initiates and propagates along the network of grain boundaries through the recrystallized area until failure. As an example, in the lower region of the bulk solder, due to lower strain, finer IMC particles are dispersed more densely and are clearly observed, indicating a moderate coarsening as shown below in Fig. 5.25.

5.3.6 Lessons learnt from case study II

A significant finding from the study of Coyle et al. is that the beneficial effect of Ag on ATC reliability diminishes as the severity of the thermal cycling profile increases, defined by greater ΔT , higher peak temperature, and longer dwell times. The results also indicate that all the Pb-free solders show superior reliability than the SnPb alloy they have replaced. Besides OM and SEM, advanced analytical methods such as PLM and EBSD can provide a better understanding of the effect of microstructure and its evolution on thermal fatigue performance.

5.4 Case study III—High-performance solders (third generation)

For end users with a requirement for high reliability, thermal fatigue resistance of Pb-free solders is an attractive topic. Solder joint fatigue is recognized as one of

the primary failure mechanisms of electronic assemblies. Due to differing CTE, there will be thermal expansion mismatch between components and their circuit board, resulting in thermomechanical fatigue of solder joints during service.

As elucidated earlier, although Ag is very effective to enhance the reliability (evaluated by characteristic lifetime under ATC) of solder joints under mild ATC conditions, for example, 0/100°C 10-minute dwell, the strengthening effect of Ag that benefits the reliability lessens drastically with increased severity of the ATC profile, such as greater ΔT , higher peak temperature, and longer dwell times. It is necessary to find an alternative or additional strengthening mechanism that is less vulnerable under more severe conditions, which degrade the effectiveness of strengthening mechanism from Ag. Therefore third-generation Pb-free alloys have been studied and developed; a wide range of elements have been used as micro-alloys, such as Ni, Al, Mn, Ti, Zn, Bi, as well as some rare-earth elements, to study their effects on solder alloy reliability.

5.4.1 Effect of micro-alloying on Sn—Ag—Cu

In a review study, Shnawah et al. reported that the low Ag-content SAC alloys with different minor alloying elements such as Mn, Ce, Bi, Ni, and Ti display good performance in terms of thermal cycling conditions [53]. For example, IMC coarsening suppression phenomenon was observed in SAC105 solder alloy with Mn or Ce additives. In addition, grain coarsening was also suppressed through pinning grain boundary by refined IMC particles. As a result, a fine IMC structure and a stable grain structure jointly lead to an improved thermal cycling reliability.

Ali et al. reported that by adding 0.05 wt.% Fe and 1 wt.% Bi to Sn—1Ag—0.5Cu (SAC105), the impact of absorbed energy of the solder bulk is increased by about 20%, which is corroborated by the corresponding modulus. It was found that the sizes of β -Sn grains and IMCs (Ag_3Sn and Cu_6Sn_5) were reduced. Fe and Bi additions to SAC105 solders improved mechanical performance and were more stable than SAC105 under high thermal aging conditions, due to the microstructure refinement and solid solution hardening mechanisms [54].

Fleshman et al. found that doping small amount of Ni (0.1 wt.%) to Sn—3.0Ag—0.5Cu can alter the grain structure of a solder ball and improve its shear strength. Nickel can serve as heterogeneous nucleation sites for IMCs and induce finer IMCs precipitates, which can then pin the grain boundaries of β -Sn and suppress the growth of grains, resulting in a refined microstructure. Meanwhile, Ni-doped SAC305 displays multiple grains with a partially interlaced structure, differing from SAC305 which shows mainly single grain and only occasionally multiple grains. These refined grains, altered orientation and enhanced dispersion of fine IMC precipitates all contribute to enhanced mechanical performance of Ni-doped SAC305 [55].

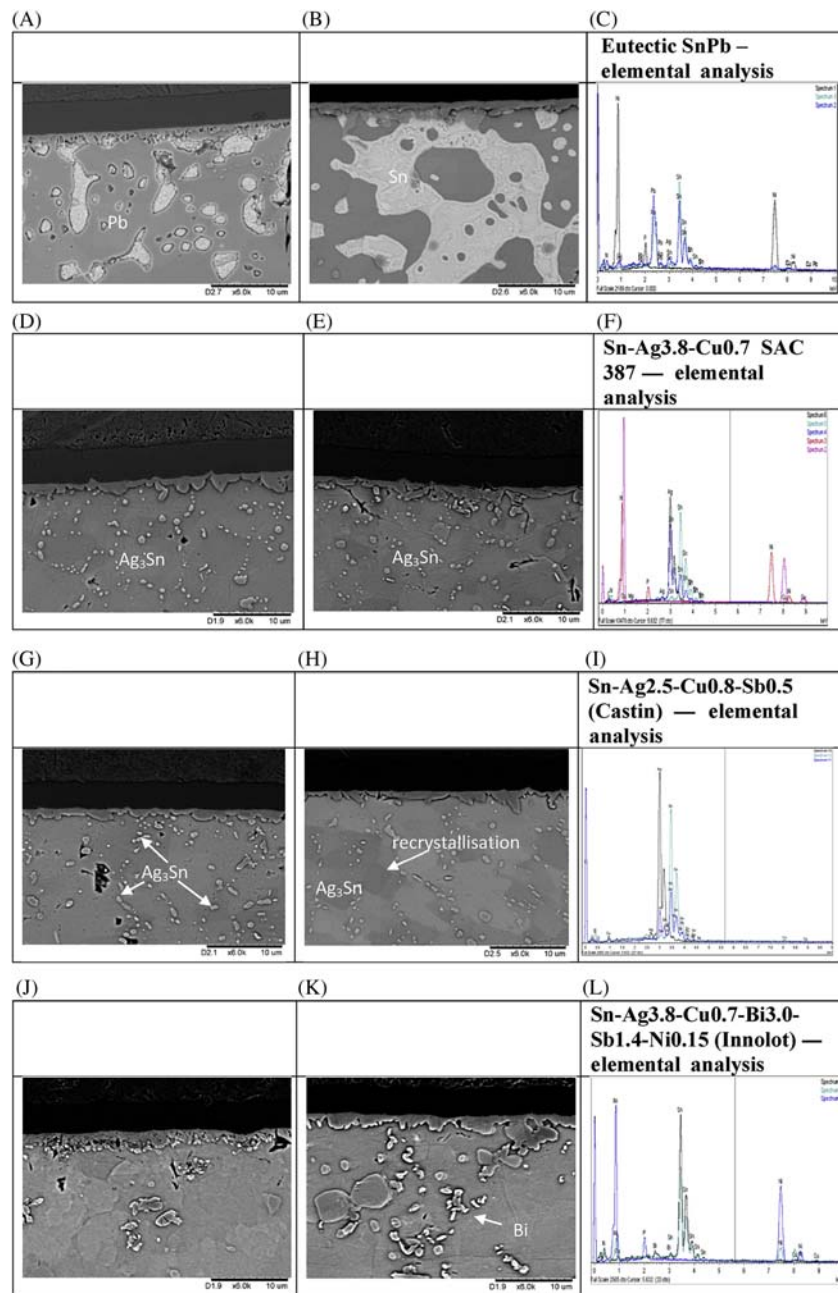
5.4.2 Two commercialized alloys

Collins et al. evaluated the thermomechanical fatigue resistance of a range of commercially available Pb-free solder alloys that are known to exhibit fatigue

resistance: (1) SAC387 as a conventional Pb-free alloy, (2) a Sb-doped SAC alloy (Castin), (3) a Bi-bearing alloy (Innolot), and (4) eutectic SnPb that was used as reference material. The thermal cycle consisted of 0/100°C and –40/125°C with a ramp rate of 4°C/min on heating and cooling. The dwell times were 15 minutes at the upper and lower limits. These thermal cycle conditions are suitable to accelerate failures in long-life equipment for telecommunications, defense, or aerospace applications [8,56]. Due to its relatively high silver content, SAC387 is known to possess good fatigue resistance [57,58], whereas the Castin alloy is known to perform well under fatigue [1] and the Innolot alloy has been designed as a fatigue-resistant solder alloy [59]. These alloys have primarily focused on improving toughness of the interfacial IML, and have been targeted for deployment in harsh operating environments such as automotive, industrial, military, and aerospace applications. Micro-alloying can provide solid solution strengthening to compensate for the loss of strength once the Ag_3Sn particles coarsen.

The initial microstructures of Castin, Innolot, and SAC387 are highly dependent on preconditioning (dry heat at 125°C for 10 days) and alloy composition. As shown in Fig. 5.26, with the electroless nickel immersion gold (ENIG) finish on copper pads as a substrate, the dark Ni layer can be clearly observed just above the Cu_6Sn_5 intermetallic, where cracks are most likely to initiate in the solder joint. Due to its tetragonal crystal structure, an extensive degree of undercooling is required in order to induce nucleation and the eutectic reaction of the Sn dendrites, and eventually form the dendritic primary Sn phase structure. Eutectic SnPb shows the microstructure of alternating layers of Pb-rich α and Sn-rich β phases due to a diffusion controlled distribution. For SAC387-based alloys (SAC387, Castin, and Innolot), a combination of darker Cu-rich Cu_6Sn_5 and brighter flake-like Ag-rich ϵ - Ag_3Sn IMCs are embedded in the Sn-rich matrix. For SAC387, preconditioned alloys show a significantly coarser Ag_3Sn particle network compared to as received ones. In Castin, Sb dopant predominantly dissolves in the β -Sn matrix to form a solid solution, whereas the white Bi phase is clearly identified in Innolot.

The reliability of the solder alloys is ranked as follows $\text{Innolot} > \text{Castin} > \text{SAC387} \gg \text{SnPb}$, as shown in Table 5.2, in terms of characteristic life data. Compared with traditional SnPb, the higher characteristic lifetime of SAC387 solder could be attributed to its relatively high silver content. Based on SAC387, the higher characteristic lifetime of Castin is due to solid solution strengthening by micro-alloying Sb, which also resulted in an initial finer grain structure that coarsened at a slower rate under the stresses imposed by the thermal cycling. Possessing the solid solution strengthening and microstructure refinement from micro-alloying Sb, the reliability of Innolot was further improved by micro-alloying Bi, which introduced additional solid solution strengthening as well as an extra IMC that could offer a precipitation hardening mechanism. On the other hand, as mainly concentrated in the Cu_6Sn_5 phase which mostly was located at the interface between the solder and the copper substrate, the Ni dopant in Innolot is expected to have a toughening effect on the IMC thereby inhibiting crack formation and growth.

**FIGURE 5.26**

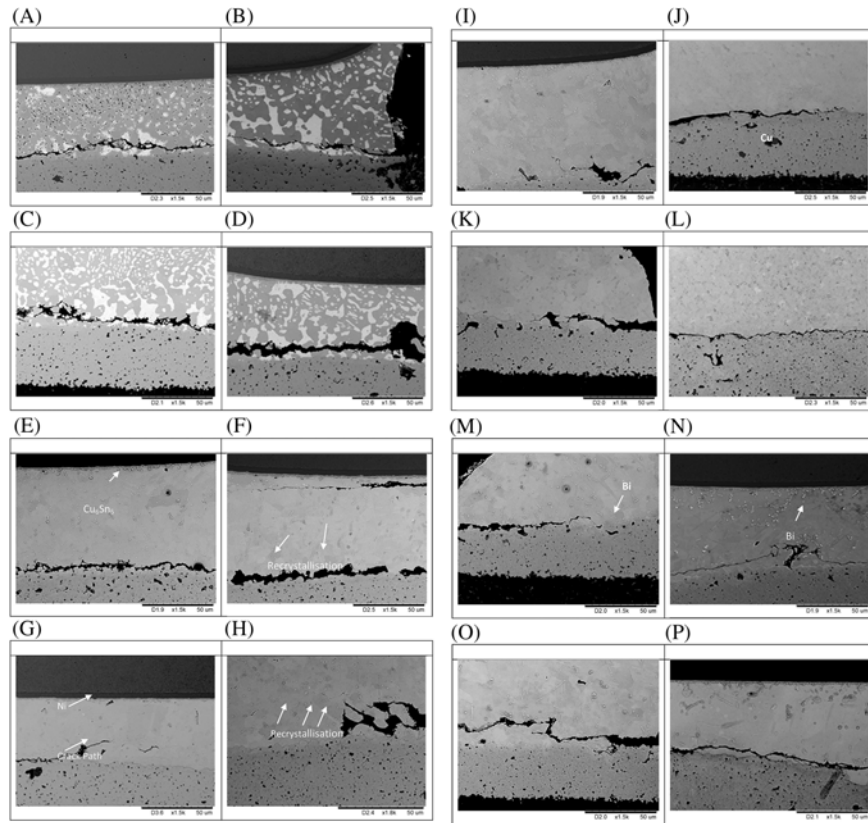
As received microstructures of (A) SnPb, (D) SAC387, (G) Castin, and (J) Innolot; preconditioned microstructures of (B) SnPb, (E) SAC387, (H) Castin, and (K) Innolot; EDS of (C) SnPb, (F) SAC387, (I) Castin, and (L) Innolot [8].

Table 5.2 Summarized ATC reliability data [8].

Alloy	Preconditioning	Thermal cycle (°C)	Characteristic life η	Slope β
SnPb	No	0/100	148.8	8.23
SAC387	No	0/100	756.1	8.55
Innolot	No	0/100	1341	3.37
Castin	No	0/100	609.3	6.99
SnPb	No	−40/125	74.6	5.43
SAC387	No	−40/125	212.9	18.4
Innolot	No	−40/125	320.5	6.51
Castin	No	−40/125	205.9	8.46
SnPb	Yes	0/100	215.6	8.06
SAC387	Yes	0/100	688.6	12.2
Innolot	Yes	0/100	1215	4.19
Castin	Yes	0/100	911.7	7.58
SnPb	Yes	−40/125	136.7	6.81
SAC387	Yes	−40/125	185.9	9.09
Innolot	Yes	−40/125	258.9	8.25
Castin	Yes	−40/125	211.9	6.81

Characteristic lifetime of all alloys, without exception, was remarkably reduced by $\sim 50\%$ – 80% under a $-40/125^\circ\text{C}$ thermal cycle compared to a $0/100^\circ\text{C}$ thermal cycle. The lower characteristic lifetimes can be ascribed as before to the larger strain of the cycle with greater temperature difference (ΔT , 165°C vs 85°C) and hence longer time is required for recovery processes. The higher peak temperature in the more aggressive cycle will also result in enhanced Ag_3Sn coarsening and recrystallization, driven by the strain damage associated with the larger ΔT as discussed previously. Therefore rather than thermal exposure alone, it is the synergy of strain induced by temperature cycling coupled with the thermal exposure at the high end of the cycle that dominantly drives the IMC coarsening. Preconditioning generally resulted in lower characteristic lifetimes for the Pb-free alloys. However, for SAC387-based alloys, although isothermal preconditioning had an obvious impact on initial microstructure, it has a negligible effect on the characteristic lifetime, and neither does it alter the failure mode nor the final microstructure. Similar results have also been found for SAC405 alloys by Manock et al. [60]. By micro-alloying Bi and Sb, the microstructure coarsening and grain growth in Castin and Innolot solder alloys may also be inhibited during thermal cycling, through the solid solution strengthening mechanism. Similar to Ag–Sn IMC precipitates, Bi could migrate from the bulk Sn to the grain boundaries and accumulate there during thermal cycling, to provide a strengthening mechanism which can hinder dislocation motion.

As the failures represented in Fig. 5.27 show, cracking is observed both in the bulk solder and at the interface with the board for all alloys. Slight contrast

**FIGURE 5.27**

Microstructures of as received post ATC 1 (A) SnPb, (E) SAC387, (I) Castin, and (M) Innolot; preconditioned post ATC 1 (B) SnPb, (F) SAC387, (J) Castin, and (N) Innolot; as received post-ATC 2 (C) SnPb, (G) SAC387, (K) Castin, and (O) Innolot; preconditioned post-ATC 2 (D) SnPb, (H) SAC387, (L) Castin, and (P) Innolot [8].

differences in the Sn phase were assumed to be subgrains, which may form during thermal cycling of solder alloys, due to recrystallization or recovery during creep at locations where high strain conditions occur that provide nucleation sites. As the thermal cycling proceeds, a number of micro-cracks appear to connect voids that form preferentially at triple junction points induced by recrystallization and grain boundary motion. Secondary cracking is seen in all the failures, while Cu_6Sn_5 and Ag_3Sn IMCs are commonly observed throughout the bulk solder in all the SAC387-based solder joints. Recently, utilizing microstructural analysis and crack imaging software, Zhong et al. reported that Innolot exhibited better crack resistance during early cycles ($-40/125^\circ\text{C}$ cycle) as energy was consumed during recrystallization processes. However, the larger amount of grain

boundaries from the early recrystallization could act as crack propagation paths, leading to significant acceleration of its failure in later cycles, when compared to SAC387. It is concluded that recrystallization speed should be controlled to enhance reliability and that Innolot is more suited to short term reliability products [61]. Contradictory reliability findings from Collins et al. showed that while Innolot may fail at early cycles, the overall long term reliability is better than SAC387 statistically. When compared to the other alloys, the Castin and Innolot exhibit the longest fatigue lives during the more aggressive temperature cycles ($-40/125^{\circ}\text{C}$ cycle). The increased reliability is due to the inhibition of microstructural coarsening by enhanced Ag_3Sn precipitate formation, which is a result of Bi and Sb micro-alloying.

5.4.3 Lessons learnt from case study III

All the third generation Pb-free solders discussed above are more reliable in ATC than the SnPb alloy they have replaced. Alloys are being designed to improve toughness of IMLs and to provide solid solution strengthening to compensate for the loss of strength once the Ag_3Sn particles coarsen. While solid solution strengthening by additions of Sb is effective (Castin), it is not sufficient, and other strengthening options through the introduction of additional precipitates and solid solution strengthening using Bi (Innolot) is most desirable. Presumably, Ni also has a toughening effect on the IMCs thereby inhibiting crack formation and growth. The strain induced by temperature cycling coupled with the thermal exposure at the high end of the cycle (not temperature exposure alone) was the dominant factor driving the particle coarsening.

5.5 Case study IV—Low-temperature solders

Currently, SAC305 alloy is widely used by the industry as the most prevalent replacement for Pb-free solder due to its thermal reliability [11,38]. However, it exhibits a much higher melting temperature (217°C) compared with traditional SnPb (183°C) [62], and this has become its drawback with regard to wider application in the electronics industry. Recently, driven by specialist applications such as temperature-sensitive components and optoelectronics as well as environmental awareness, there has been a trend toward Sn–Zn-based solder alloys due to their lower melting points (198.5°C), good mechanical performance at room temperature, and low cost [63]. Villain et al. found that the solder joints of eutectic Sn–9Zn showed comparable shear force to eutectic SnPb and higher creep resistance than Sn–40Pb under the same test conditions [64]. Zhang et al. reported that Sn–9Zn shows a higher fatigue life than SnPb with solder joints in chip scale packages in a finite element analysis modeling study [65]. The LEADFREE project reported that a large fraction of the Sn–9Zn solder joints had failed after 400

cycles whereas Sn–36Pb–2Ag had the first failure at 1000 cycles under the same ATC conditions [66]. In the following subsections, the effects of substrate and micro-alloying on reliability of Sn–Zn-based solders will be reviewed respectively.

5.5.1 Effect of substrate

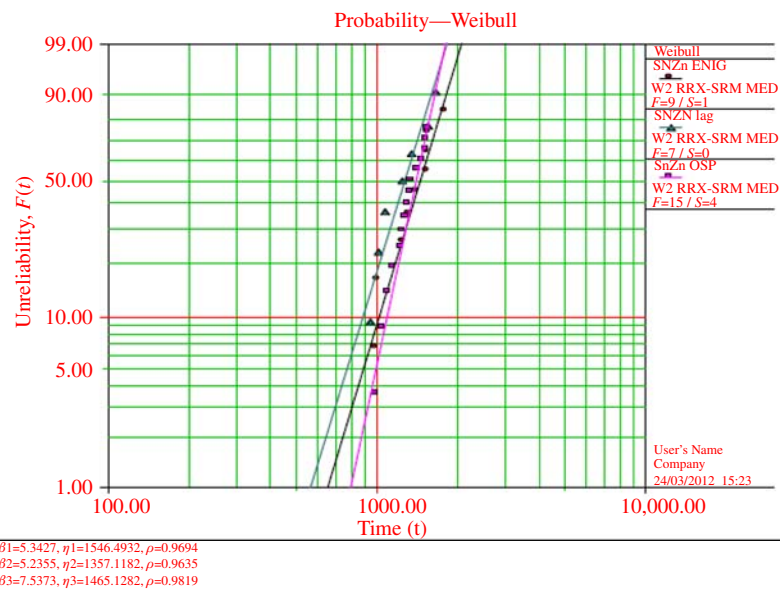
The inferior reliability of Sn–9Zn is attributed to partially its faster interfacial reaction and partially to the insufficient soldering due to its poor wettability. Due to the high activity of Zn, Sn–Zn-based solder alloys generally show poor wettability and short paste shelf life. However, these disadvantages can be overcome by developments in flux technology [62,67]. A specifically formulated Rosin-based flux with high activator level to overcome wettability and oxidation problems was developed to produce the robust Sn–9Zn solder joint, by Collins et al. [68]. After 2500 thermal cycles, the characteristic lifetime of Sn–9Zn soldered 2512 chip resistors on the different finishes can be ranked as follows: ENIG > organic solderability preservative (OSP) > immersion silver (IAG), as shown in Fig. 5.28. The results show that Sn–9Zn could potentially be a viable alternative to SAC305 for high reliability components [68].

As shown in Fig. 5.29, as received solder joint microstructures with three different finishes look morphologically similar, with Zn phase (black) existing as needle-like primary Zn and platelet or fibrous Zn precipitates dispersed in the eutectic phase. The solder joint with ENIG finish shows remarkably finer grain structure, however, which may coarsen at a slower rate under the stresses introduced by thermal cycling, leading to an increased characteristic lifetime. For IAG and OSP, γ -Cu₅Zn₈ is the primary IML, between which and Cu a thin β -CuZn layer is also formed. On the other hand, Ni₅Zn₂₁ is the only IML formed along the interface for ENIG. During thermal cycling, cracks form adjacent to the IML and propagate along the coarsened brittle IMC.

In contrast to the fast Sn–9Zn/Cu interfacial reaction which can significantly degrade the joint strength [69], Yoon et al. found that the ENIG layer effectively protected the Sn–9Zn/ENIG interface by forming an AuZn₃ IMC layer which can act as a diffusion barrier that suppresses the undesirable interfacial reaction, resulting in superior joint reliability [70]. However, in the case of the Au/Ni electroplated Cu substrate, the AuZn₃ IMC layer became detached from the interface during reflow, presumably due to the CTE mismatch and weak adhesion between the AuZn₃ IMC layer and Ni layer caused by the depletion of the Au layer [71,72].

5.5.2 Effect of micro-alloying

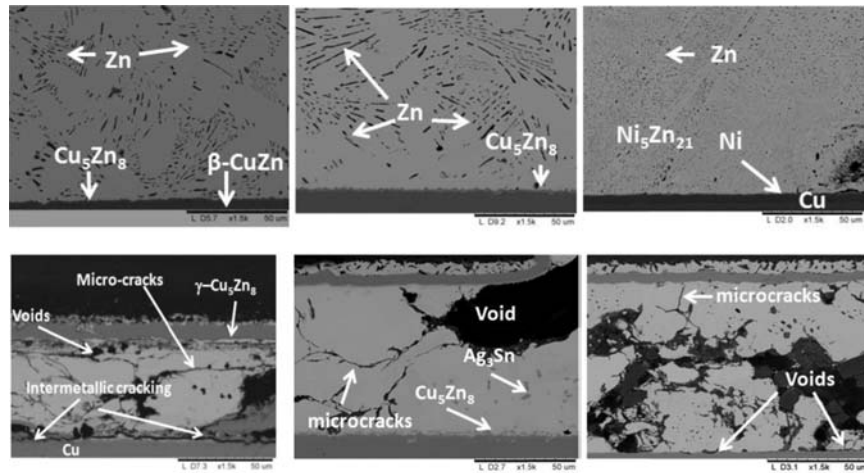
It has been reported that micro-alloying has a critical influence on mechanical properties, as well as microstructural evolution and reliability of Sn–Zn-based solder alloys [73–76]. El-Daly et al. reported that alloying of Sb can effectively increase mechanical strength and creep resistance of Sn–Zn-based solder alloys



Assembly	Characteristic life η	Slope β	Correlation coefficient ρ
SnZn ENIG	1546	5.3	0.969
SnZn IAg	1357	5.2	0.964
SnZn OSP	1465	7.5	0.982

FIGURE 5.28

Weibull plot of Sn—9Zn with ENIG, IAg, and OSP surface finish [68].

**FIGURE 5.29**

As received solder joint (upper) and failed solder joint (lower) of Sn–9Zn with OSP (left), IAg (middle), and ENIG (right) [68].

[77,78]. In a micro-alloying study, Shen and Chan found that ZrO_2 nanoparticles embedded in the solder matrix of Sn–9Zn can obstruct the motion of dislocations by pinning and also pin grain boundaries, therefore, significantly enhancing the shear strength of the solder joint with an electrodeposited Ni/Au BGA substrate. Due to their strong adsorption effect, ZrO_2 nanoparticles can also absorb Zn atoms on their surfaces, impeding Zn from gathering and therefore restraining the growth of Ni–Zn IMC layer, therefore improving the reliability of solder joints [79].

The addition of Bi into Sn–Zn binary alloys can improve mechanical performance and corrosion resistance [80–82]. Due to the great potential of Sn–Zn–Bi solder systems to replace SnPb as a low-temperature Pb-free solder, many studies, both industrial [2,83,84] and academic [73,74,85], have been carried out to study the influence of micro-alloying on Sn–Zn–Bi-based solder alloys. It is found that micro-alloying In, Nd, Ni, or Sb can enhance mechanical performance of Sn–8Zn–3Bi-based alloys through microstructure refinement and solid solution strengthening [76,86–89], micro-alloying Ge or Cr can improve the antioxidation resistance [90,91], whereas micro-alloying Ag or Cu can effectively suppress the formation and growth of IMCs [92–94]. In addition, alloying 0.3 wt.% Ag into Sn–8Zn–3Bi can regulate the spalling of the Au–Zn IMC layer as well as limit the formation of Ni–Zn compound which can deteriorate the mechanical strength of the joint [92]. In a comprehensive study on interfacial IMCs and reliability, Sharif and Chen concluded that Sn–8Zn–3Bi solder and ENIG is a good combination for BGA soldering technology [95].

5.5.3 Lessons learnt from case study IV

The study of Collins et al. shows that drawbacks of low temperature Sn–Zn-based solders like poor wettability and short paste shelf life can be prevented by the development of tailor-made flux technology. In addition, other studies show that micro-alloying can enhance the reliability of Sn–Zn-based solder assembly. With more commercial potential in the application space of consumer and “throw away” electronics, due to lower raw material cost and processing temperatures, the novel micro-alloyed Sn–Zn-based solders could potentially lead to a viable alternative to conventional SAC solder solders for niche applications.

5.6 Conclusion

The evolution of Pb-free solders has been reviewed in this chapter. Substantial research has been carried in this field and it has provided valuable guidance for applications in the electronics industry and for understanding the ATC performance of Pb-free solders. With an increasingly compositional complexity, SAC-based alloys have made it possible for most consumer electronics, and much of the electronics for which the EU RoHS Directive had granted exemption, to transition to Pb-free solder assembly without encountering significant reliability issues. In addition, emerging low temperature Sn–Zn-based solders have their own place in specific applications.

Finally, failure mechanisms of solder joints are largely dominated by micro-structural evolution during thermal cycling, which is influenced by alloy composition, component type, as well as ATC profile including temperature range and dwell time. Therefore it would be prudent for future reliability studies to understand their undercooling behavior during reflow, Sn grain orientation, morphology, and recrystallization behavior during thermal processing.

References

- [1] E. Bradley, C.A. Handwerker, J. Bath, R.D. Parker, R.W. Gedney, *Lead-Free Electronics: iNEMI Projects Lead to Successful Manufacturing*, John Wiley & Sons, 2007.
- [2] G. Henshall, R. Healey, R.S. Pandher, K. Sweatman, K. Howell, R. Coyle, et al., iNEMI Pb-free alloy alternatives project report: state of the industry, in: *Proceedings SMTA International*, 2008.
- [3] D. Shangguan, *Lead-Free Solder Interconnect Reliability*, ASM International, 2005.
- [4] H. Ma, J.C. Suhling, A review of mechanical properties of lead-free solders for electronic packaging, *J. Mater. Sci.* 44 (Mar 2009) 1141–1158.
- [5] K. Suganuma, Advances in lead-free electronics soldering, *Curr. Opin. Solid State Mater. Sci.* 5 (2001) 55–64.

- [6] M. Reid, J. Punch, M. Collins, C. Ryan, Effect of Ag content on the microstructure of Sn-Ag-Cu based solder alloys, *Solder. Surface Mount Technol.* 20 (2008) 3–8.
- [7] F.J. Cheng, F. Gao, J.Y. Zhang, W.S. Jin, X. Xiao, Tensile properties and wettability of SAC0307 and SAC105 low Ag lead-free solder alloys, *J. Mater. Sci.* 46 (2011) 3424–3429.
- [8] M.N. Collins, E. Dalton, J. Punch, Microstructural influences on thermomechanical fatigue behaviour of third generation high Ag content Pb-Free solder alloys, *J. Alloys Compd.* 688 (2016) 164–170.
- [9] R.J. Coyle, K. Sweatman, B. Arfaei, Thermal fatigue evaluation of Pb-free solder joints: results, lessons learned, and future trends, *JOM* 67 (2015) 2394–2415.
- [10] W. Engelmaier, Surface mount solder joint long-term reliability: design, testing, prediction, *Solder. Surface Mount Technol.* 1 (1989) 14–22.
- [11] M.N. Collins, J. Punch, R. Coyle, Surface finish effect on reliability of SAC 305 soldered chip resistors, *Solder. Surface Mount Technol.* 24 (2012) 240–248.
- [12] R. Coyle, J. Osenbach, M.N. Collins, H. McCormick, P. Read, D. Fleming, et al., Phenomenological study of the effect of microstructural evolution on the thermal fatigue resistance of Pb-free solder joints, in: *IEEE Transactions on Components Packaging and Manufacturing Technology*, vol. 1, 2011, pp. 1583–1593.
- [13] R. Coyle, M. Reid, C. Ryan, R. Popowich, P. Read, D. Fleming, et al., The influence of the Pb-free solder alloy composition and processing parameters on thermal fatigue performance of a ceramic chip resistor, in: *2009 IEEE 59th Electronic Components and Technology Conference*, vols. 1–4, 2009, pp. 423–430.
- [14] B. Arfaei, F. Mutuku, R. Coyle, E. Cotts, and J. Wilcox, Failure mechanism and microstructural evolution of Pb-free solder alloys in thermal cycling tests: effect of solder composition and Sn grain morphology, in: *Electronic Components and Technology Conference (ECTC)*, 2015 IEEE 65th, 2015, pp. 118–126.
- [15] B. Arfaei, M. Anselm, S. Joshi, S. M-Shirazi, P. Borgesen, E. Cotts, et al., Effect of Sn grain orientation on failure mechanism and reliability of lead-free solder joints in thermal cycling tests, in: *Proceedings of the SMTA International*, 2013, pp. 539–550.
- [16] K. Seelig and D. Suraski, A study of lead-contamination in lead-free electronics assembly and its impact on reliability, in: *Proceedings of SMTA*, 2002.
- [17] J. Oliver, O. Rod, M. Nylén, C. Markou, Fatigue properties of Sn13. 5Ag10. 7Cu solder joints and effects of Pb-contamination, *J. Surface Mount Technol.* 15 (2002) 23–29.
- [18] S. Choi, T. Bieler, K. Subramanian, J. Lucas, Effects of Pb contamination on the eutectic Sn-Ag solder joint, *Solder. Surface Mount Technol.* 13 (2001) 26–29.
- [19] P. Vianco, J. Rejent, I. Artaki, U. Ray, D. Finley, and A. Jackson, Compatibility of lead-free solders with lead containing surface finishes as a reliability issue in electronic assemblies, in: *Electronic Components and Technology Conference*, 1996. *Proceedings*, 46th, 1996, pp. 1172–1183.
- [20] Q. Zhu, M. Sheng, L. Luo, The effect of Pb contamination on the microstructure and mechanical properties of SnAg/Cu and SnSb/Cu solder joints in SMT, *Solder. Surface Mount Technol.* 12 (2000) 19–24.
- [21] F. Hua, R. Aspandiar, T. Rothman, C. Anderson, G. Clemons, M. Klier, Solder joint reliability of Sn-Ag-Cu BGA components attached with eutectic Pb-Sn solder paste, *J. Surface Mount Technol.* 16 (2003) 34–42.

- [22] C.K. Chung, R. Aspandiar, K.F. Leong, C.S. Tay, The interactions of lead (Pb) in lead free solder (Sn/Ag/Cu) system, in: Electronic Components and Technology Conference, 2002. Proceedings. 52nd, 2002, pp. 168–175.
- [23] P. Snugovsky, A. Zbrzezny, M. Kelly, M. Romansky, Theory and practice of lead-free BGA assembly using Sn-Pb solder, in: Proceedings CMAP (Centre for Microelectronics Assembly and Packaging in Canada) International Conference on Lead-free Soldering, 2005.
- [24] J. Bath, S. Sethuraman, X. Zhou, D. Willie, K. Hyland, K. Newman, et al., Reliability evaluation of lead-free SnAgCu PBGA676 components using tin-lead and lead-free SnAgCu solder paste, in: Proceedings of SMTAI, pp. 891–901, 2005.
- [25] J. Nguyen, D. Geiger, D.D. Rooney, D.D. Shangguan, Backward compatibility study of lead-free area array packages with tin-lead soldering process, in: Proceedings of the IPC SMTA Council APEX Conference, 2006, pp. S34–03–1-S34–03-9.
- [26] J. Nguyen, D. Geiger, Reliability study of lead-free area array packages with tin-lead soldering processes, in: Proceedings of SMTAI, pp. 433–438, 2006.
- [27] R. Kinyanjui, Q. Chu, P. Snugovsky, R. Coyle, Solder joint reliability of Pb-free Sn-Ag-Cu ball grid array (BGA) components in Sn-Pb assembly process, in: Proceedings of APEX 2008, pp. S15–02, 2008.
- [28] H. McCormick, P. Snugovsky, Z. Bagheri, S. Bagheri, C. Hamilton, G. Riccitelli, et al., Mixing metallurgy: reliability of SAC balled area array packages assembled using SnPb solder, *SMTA News J. Surface Mount Technol.* 20 (2007) 11.
- [29] B. Nandagopal, M. Zequn, T. Sue, Microstructure and thermal fatigue life of BGAs with eutectic Sn-Ag-Cu balls assembled at 210 C with eutectic Sn-Pb solder paste, in: Proceedings of the 56th Electronic Components and Technology Conference, 2006, pp. 875–883.
- [30] R. Coyle, P. Read, S. Kummerl, D. Fleming, R. Popowich, I. Chatterji, A comprehensive analysis of the thermal fatigue reliability of SnPb and Pb free plastic ball grid arrays (PBGA) using backward and forward compatible assembly processes, *J. Surface Mount Technol.* 21 (2008) 33–47.
- [31] R.J. Coyle, D.H. Poppo, A. Mawer, D.P. Cullen, G.M. Wenger, P.P. Solan, The effect of modifications to the nickel/gold surface finish on assembly quality and attachment reliability of a plastic ball grid array (peer review version), *IEEE Transact. Components Packaging Technol.* 26 (2003) 724–732.
- [32] A.R. Zbrzezny, P. Snugovsky, T. Lindsay, R. Lau, Reliability investigation of mixed BGA assemblies, *IEEE Transact. Electron. Packaging Manufact.* 29 (2006) 211–216.
- [33] D. Hillman, M. Wells, K. Cho, R. Collins, The Impact of Reflowing a Pb-free solder alloy using a tin/lead solder alloy reflow profile on solder joint integrity, in: Proceedings CMAP (Centre for Microelectronics Assembly and Packaging in Canada) International Conference on Lead-free Soldering, 2005.
- [34] P. Snugovsky, H. McCormick, S. Bagheri, Z. Bagheri, C. Hamilton, M. Romansky, Microstructure, defects, and reliability of mixed Pb-free/Sn-Pb assemblies, *J. Electron. Mater.* 38 (2009) 292–302.
- [35] S. Dunford, S. Canumalla, P. Viswanadharn, Intermetallic morphology and damage evolution under thermomechanical fatigue of lead (Pb)-free solder interconnections, in: Electronic Components and Technology Conference, 2004. Proceedings. 54th, 2004, pp. 726–736.

- [36] S.K. Kang, P. Lauro, D.-Y. Shih, D.W. Henderson, T. Gosselin, J. Bartelo, et al., Evaluation of thermal fatigue life and failure mechanisms of Sn-Ag-Cu solder joints with reduced Ag contents, in: *Electronic Components and Technology Conference*, 2004. Proceedings. 54th, 2004, pp. 661–667.
- [37] J. Liang, N. Dariavach, D. Shangguan, Solidification condition effects on microstructures and creep resistance of Sn-3.8 Ag-0.7 Cu lead-free solder, *Metallurg. Mater. Transact. A* 38 (2007) 1530–1538.
- [38] M.N. Collins, J. Punch, R. Coyle, M. Reid, R. Popowich, P. Read, et al., Thermal fatigue and failure analysis of SnAgCu solder alloys with minor Pb additions, *IEEE Transact. Components Packaging Manufact. Technol.* 1 (2011) 1594–1600.
- [39] W.P. Liu, N.C. Lee, The effects of additives to SnAgCu alloys on microstructure and drop impact reliability of solder joints, *JOM* 59 (2007) 26–31.
- [40] L.F.T.S. IPC, Solder Products Value Council, Round Robin Testing and Analysis of Lead Free Solder Pastes with Alloys of Tin, Silver and Copper: Final Report, 2005.
- [41] T.-K. Lee, H. Ma, Aging impact on the accelerated thermal cycling performance of lead-free BGA solder joints in various stress conditions, in: *Electronic Components and Technology Conference (ECTC)*, 2012 IEEE 62nd, 2012, pp. 477–482.
- [42] J.-P. Clech, G. Henshall, J. Miremadi, Closed-form, strain-energy based acceleration factors for thermal cycling of lead-free assemblies, in: *SMTA International Conference Proceedings*, 2009.
- [43] K.-W. Moon, W.J. Boettinger, U.R. Kattner, F.S. Biancaniello, C. Handwerker, Experimental and thermodynamic assessment of Sn-Ag-Cu solder alloys, *J. Electron. Mater.* 29 (2000) 1122–1136.
- [44] B. Arfaei, N. Kim, E. Cotts, Dependence of Sn grain morphology of Sn-Ag-Cu solder on solidification temperature, *J. Electron. Mater.* 41 (2012) 362–374.
- [45] B. Arfaei, Mtuku, F., Anselm, M., Effect of PCB surface finish on Sn grain morphology and thermal fatigue performance of SnPb and lead free solder joints, in: *SMTA International*, 2014.
- [46] B. Arfaei, M. Benedict, E. Cotts, Nucleation rates of Sn in undercooled Sn-Ag-Cu flip-chip solder joints, *J. Appl. Phys.* 114 (2013) 173506.
- [47] L. Lehman, Y. Xing, T. Bieler, E. Cotts, Cyclic twin nucleation in tin-based solder alloys, *Acta Mater.* 58 (2010) 3546–3556.
- [48] R. Kinyanjui, L. Lehman, L. Zavalij, E. Cotts, Effect of sample size on the solidification temperature and microstructure of SnAgCu near eutectic alloys, *J. Mater. Res.* 20 (2005) 2914–2918.
- [49] J. Wilcox, R. Coyle, L. Lehman, J. Smetana, Effect of isothermal preconditioning on thermal fatigue life and microstructure of a SAC305 BGA, in: *SMTA International Conference*, Chicago, IL, 2014, pp. 122–133.
- [50] R. Coyle, R. Parker, B. Arfaei, F. Mutuku, K. Sweatman, K. Howell, et al., The effect of nickel microalloying on thermal fatigue reliability and microstructure of SAC105 and SAC205 solders, in: *Electronic Components and Technology Conference (ECTC)*, 2014 IEEE 64th, 2014, pp. 425–440.
- [51] B. Arfaei, L. Wentlent, S. Joshi, M. Anselm, P. Borgeses, Controlling the superior reliability of lead free assemblies with short standoff height through design and materials selection, in: *Proceedings of IMECE*, 2012.
- [52] B. Arfaei, L. Wentlent, S. Joshi, A. Alazzam, T. Tashtoush, M. Halaweh, et al., Improving the thermomechanical behavior of lead free solder joints by controlling

- the microstructure, in: *Thermal and Thermomechanical Phenomena in Electronic Systems (ITherm)*, 2012 13th IEEE Intersociety Conference on, 2012, pp. 392–398.
- [53] D.A.A. Shnawah, M.F.B. Sabri, I.A. Badruddin, S. Said, A review on effect of minor alloying elements on thermal cycling and drop impact reliability of low-Ag Sn-Ag-Cu solder joints, *Microelectron. Int.* 29 (2012) 47–57.
 - [54] B. Ali, M. Sabri, S.M. Said, N.L. Sukiman, I. Jauhari, N. Soin, High impact reliability and high temperature performance of Fe and Bi added Sn-1Ag-0.5Cu solder alloys, *J. Mater. Sci. Mater. Electron.* 28 (2017) 7277–7285.
 - [55] C. Fleshman, W.Y. Chen, T.T. Chou, J.H. Huang, J.G. Duh, The variation of grain structure and the enhancement of shear strength in SAC305-0.1Ni/OSP Cu solder joint, *Mater. Chem. Phys.* 189 (2017) 76–79.
 - [56] E. Dalton, G. Ren, J. Punch, M.N. Collins, Accelerated temperature cycling induced strain and failure behaviour for BGA assemblies of third generation high Ag content Pb-free solder alloys, *Mater. Design* 154 (2018) 184–191.
 - [57] J. Putaala, O. Salmela, O. Nousiainen, T. Kangasvieri, J. Vahakangas, A. Uusimäki, et al., Lifetime prediction and design aspects of reliable lead-free non-collapsible BGA joints in LTCC packages for RF/microwave telecommunication applications, *Solder. Surface Mount Technol.* 26 (2014) 117–128.
 - [58] C. Williams, K.E. Tan, J.H.L. Pang, Thermal cycling fatigue analysis of Sac387 solder joints, in: *2010 12th IEEE Intersociety Conference on Thermal and Thermomechanical Phenomena in Electronic Systems*, 2010.
 - [59] R. Dudek, M. Hildebrandt, R. Doering, S. Rzepka, H. Trageser, R. Kohl, et al., Solder fatigue acceleration prediction and testing results for different thermal test- and field cycling environments, in: *Electronics System-Integration Technology Conference (ESTC)*, 2014, 2014, pp. 1–8.
 - [60] J. Manock, R. Coyle, B. Vaccaro, H. McCormick, R. Popowich, D. Fleming, et al., Effect of temperature cycling parameters on the solder joint reliability of a Pb-free PBGA package, *SMTA J.* 21 (2008) 36–45.
 - [61] Y. Zhong, W. Liu, C.Q. Wang, X.J. Zhao, J.F.J.M. Caers, The influence of strengthening and recrystallization to the cracking behavior of Ni, Sb, Bi alloyed SnAgCu solder during thermal cycling, *Mater. Sci. Eng. A Struct. Mater. Properties Microstruct. Process.* 652 (2016) 264–270.
 - [62] G.J. Jackson, I.J. Wilding, R. Boyle, M.N. Collins, E. Dalton, J. Punch, et al., SnZn solder alternative for low-cost Pb-free surface mount assemblies, in: *SMTA International*, 2012.
 - [63] C.M. Chen, Y.M. Hung, C.P. Lin, W.C. Su, Effect of temperature on microstructural changes of the Sn-9 wt.% Zn lead-free solder stripe under current stressing, *Mater. Chem. Phys.* 115 (2009) 367–370.
 - [64] J. Villain, W. Jillick, E. Schmitt, T. Qasim, Properties and reliability of SnZn-based lead-free solder alloys, in: *Asian Green Electronics, 2004. AGECE. Proceedings of 2004 International IEEE Conference on the*, 2004, pp. 38–41.
 - [65] L. Zhang, L. Sun, Y.H. Guo, C.W. He, Reliability of lead-free solder joints in CSP device under thermal cycling, *J. Mater. Sci. Mater. Electron.* 25 (2014) 1209–1213.
 - [66] G. Grossmann, G. Nicoletti, U. Soler, Results of comparative reliability tests on lead-free solder alloys, in: *52nd Electronic Components & Technology Conference, 2002 Proceedings*, 2002, pp. 1232–1237.

- [67] S. Nakagawa, M. Collins, Soldering flux, solder paste and method for manufacturing soldering flux, Ed: Google Patents, 2016.
- [68] M.N. Collins, G.J. Jackson, E. Dalton, H. Steen, P. Liu, M. Holloway, et al., Accelerated temperature cycling and microstructural analysis of SnZn solder in surface mount assemblies, in: ICSR (Soldering and Reliability), 2012.
- [69] J.-W. Yoon, S.-B. Jung, Reliability studies of Sn–9Zn/Cu solder joints with aging treatment, *J. Alloys Compd.* 407 (2006) 141–149.
- [70] J.-W. Yoon, H.-S. Chun, S.-B. Jung, Interfacial reaction and mechanical characterization of eutectic Sn–Zn/ENIG solder joints during reflow and aging, *Mater. Transact.* 46 (2005) 2386–2393.
- [71] J.-W. Yoon, S.-B. Jung, Solder joint reliability evaluation of Sn–Zn/Au/Ni/Cu ball-grid-array package during aging, *Mater. Sci. Eng. A* 452 (2007) 46–54.
- [72] J.W. Yoon, S.B. Jung, Interfacial reactions and shear strength on Cu and electrolytic Au/Ni metallization with Sn–Zn solder, *J. Mater. Res.* 21 (2006) 1590–1599.
- [73] G. Ren, I.J. Wilding, M.N. Collins, Alloying influences on low melt temperature SnZn and SnBi solder alloys for electronic interconnections, *J. Alloys Compd.* 665 (2016) 251–260.
- [74] L. Zhang, K.N. Tu, Structure and properties of lead-free solders bearing micro and nano particles, *Mater. Sci. Eng. R-Reports* 82 (2014) 1–32.
- [75] S. Liu, S.B. Xue, P. Xue, D.X. Luo, Present status of Sn–Zn lead-free solders bearing alloying elements, *J. Mater. Sci. Mater. Electron.* 26 (2015) 4389–4411.
- [76] G. Ren, M.N. Collins, The effects of antimony additions on microstructures, thermal and mechanical properties of Sn–8Zn–3Bi alloys, *Mater. Design* 119 (2017) 133–140.
- [77] A.A. El-Daly, A.E. Hammad, G.S. Al-Ganainy, A.A. Ibrahiem, Design of lead-free candidate alloys for low-temperature soldering applications based on the hypoeutectic Sn–6.5Zn alloy, *Mater. Design* 56 (2014) 594–603.
- [78] A.A. El-Daly, A.E. Hammad, G.A. Al-Ganainy, A.A. Ibrahiem, Enhancing mechanical response of hypoeutectic Sn–6.5Zn solder alloy using Ni and Sb additions, *Mater. Design* 52 (2013) 966–973.
- [79] J. Shen, Y.C. Chan, Effects of ZrO₂ nanoparticles on the mechanical properties of Sn–Zn solder joints on Au/Ni/Cu pads, *J. Alloys Compd.* 477 (2009) 552–559.
- [80] K. Bukat, Z. Moser, W. Gasior, J. Sitek, M. Koscielski, J. Pstrus, Trends in wettability studies of Pb-free solders. Basic and application. Part II. Relation between surface tension, interfacial tension and wettability of lead-free Sn–Zn and Sn–Zn–Bi–Sb alloys, *Arch. Metall. Mater.* 53 (2008) 1065–1074.
- [81] K. Suganuma, K.S. Kim, Sn–Zn low temperature solder, *J. Mater. Sci. Mater. Electron.* 18 (2007) 121–127.
- [82] J. Zhou, Y.S. Sun, F. Xue, Properties of low melting point Sn–Zn–Bi solders, *J. Alloys Compd.* 397 (2005) 260–264.
- [83] N.C. Lee, Getting ready for lead-free solders, *Solder. Surface Mount Technol.* 9 (1997) 65–69.
- [84] G. Henshall, R. Healey, R.S. Pandher, K. Sweatman, K. Howell, R. Coyle, et al., Addressing opportunities and risks of pb-free solder alloy alternatives, in: Microelectronics and Packaging Conference, 2009. EMPC 2009. European, 2009, pp. 1–11.
- [85] H.R. Kotadia, P.D. Howes, S.H. Mannan, A review: on the development of low melting temperature Pb-free solders, *Microelectron. Reliab.* 54 (2014) 1253–1273.

- [86] M.M. Billah, K.M. Shorowordi, A. Sharif, Effect of micron size Ni particle addition in Sn-8Zn-3Bi lead-free solder alloy on the microstructure, thermal and mechanical properties, *J. Alloys Compd.* 585 (2014) 32–39.
- [87] J.C. Liu, G. Zhang, Z.H. Wang, J.S. Ma, K. Suganuma, Thermal property, wettability and interfacial characterization of novel Sn-Zn-Bi-In alloys as low-temperature lead-free solders, *Mater. Design* 84 (2015) 331–339.
- [88] J. Zhou, D. Huang, Y.L. Fang, F. Xue, Investigation on properties of Sn-8Zn-3Bi lead-free solder by Nd addition, *J. Alloys Compd.* 480 (2009) 903–907.
- [89] G. Ren, M.N. Collins, On the mechanism of Sn tunnelling induced intermetallic formation between Sn-8Zn-3Bi solder alloys and Cu substrates, *J. Alloys Compd.* (2019).
- [90] S.H. Wang, T.S. Chin, C.F. Yang, S.W. Chen, C.T. Chuang, Pb-free solder-alloy based on Sn-Zn-Bi with the addition of germanium, *J. Alloys Compd.* 497 (2010) 428–431.
- [91] N. Huang, A.M. Hu, M. Li, D.L. Mao, Influence of Cr alloying on the oxidation resistance of Sn-8Zn-3Bi solders, *J. Mater. Sci. Mater. Electron.* 24 (2013) 2812–2817.
- [92] A. Sharif, Y. Chan, Retardation of spalling by the addition of Ag in Sn–Zn–Bi solder with the Au/Ni metallization, *Mater. Sci. Eng. A* 445 (2007) 686–690.
- [93] L.J. Liu, W. Zhou, B.L. Li, P. Wu, Interfacial reactions between Sn-8Zn-3Bi-xAg lead-free solders and Cu substrate, *J. Alloys Compd.* 482 (2009) 90–98.
- [94] L. Liu, P. Wu, W. Zhou, Effects of Cu on the interfacial reactions between Sn-8Zn-3Bi-xCu solders and Cu substrate, *Microelectron. Reliab.* 54 (2014) 259–264.
- [95] A. Sharif, Y. Chan, Effect of substrate metallization on interfacial reactions and reliability of Sn–Zn–Bi solder joints, *Microelectron. Eng.* 84 (2007) 328–335.

This page intentionally left blank

The role of contamination in the failure of electronics—case studies

W. John Wolfgang, Joseph Colangelo and Jason Wheeler

Raytheon Company, McKinney Failure Analysis Laboratory, McKinney, TX, United States

6.1 Introduction

Contamination, for the purposes of this work, is defined as the presence of an unexpected material at electrically sensitive areas of an electrical circuit. The result of such contamination can range from no effect at all to degradation of system performance to catastrophic system failures. While the presence of contamination having a negative effect on any type of system's performance is intuitive, the modes of deposition and types thereof, in particular with regards to electronics, can be less than obvious. For example, many types of contamination that can lead to system failures derive from gaseous species via “outgassing” from a variety of sources. These gaseous materials can lead to conditions such as silver tarnish or create new seemingly difficult to rationalize contaminants such as frictional polymers or insulating decomposition products at electrical contact surfaces [1,2]. It is also common that contaminants are native to systems, deriving from the construction materials of the systems themselves, as opposed to foreign sources. No matter the source, the effects of contamination may be transitory and the responsible contaminants difficult or impossible to identify with only indirect evidence that contamination contributed to a systems failure. Case in point—some metals common to electronics (e.g., pure tin) can form “whiskers” which can either directly grow to form a short between energetic contacts or break off of the growth site and become lodged between other electrically sensitive areas of a circuit [3]. The short which results from this contamination can vaporize the small whisker making identification of this mechanism difficult.

The nature of the contaminants that may affect electrical circuits range from insulative commonly leading to failures associated with opens and/or current leakage, to conductive which may lead to shorts and catastrophic systems failures. It is also possible that over time a contaminant may start as one of these classes but, because of environmental conditions and other factors, may convert to another. An example of such an occurrence is the presence of electrolytic contamination (e.g., salts) on the surface of a circuit (or more generally, between conductors)

that, under dry conditions, creates no detectable effects but, when exposed to a high-humidity environment, can lead to resistive shorting [4].

The purpose of this contribution is to highlight examples in which contamination is the primary contributor to the failure of electronics. Selected examples were chosen to highlight the fact that, in many cases, identification of contamination as a root cause mechanism, as well as various sources of contaminants, can be less than obvious.

6.2 Case studies

6.2.1 Example 1—Contamination as a primary cause of motor failures

6.2.1.1 Introduction

Contamination at high-energy electrical contacts, for examples at motor brush/commutator interfaces, is typified by the formation of insulative reaction products and/or decomposition products of the source contaminants. These reaction and decomposition products, which are distinct from the initial contaminants which led to their formation, lead to failures which range from unacceptably high ohmic contact to opens [5]. The following case study is an example in which such mechanisms were found to be the root cause of a system failure.

In this example, a multispectral viewing and imaging systems were noted to fail upon switching between the various viewing modes. These failures were noted well short of the expected system lifetime, and, in some cases, occurred after only about 10% of operational life. Switching between the various viewing modes is accomplished by the action of a mechanical system driven by an electric motor. The system performance varied significantly across the sample set evaluated; the performance generally ranged from normal operation to “sluggish” switching time, and, in some cases, failed to switch altogether.

Initial investigations were carried out at the systems level in hopes of isolating wherein the switching drive train the failures were occurring. For example, an inspection of the complete mechanical system did not reveal any anomalies—associated gears and drive shafts exhibited no damage or abnormalities. Because of these observations, it was decided that the failures were most likely associated with improper motor operation. As a result, motors from several systems which were noted to exhibit sporadic switching operations were removed for analyses. It was noted that the replacement of the motor with a “known good” sample restored system functionality. In addition to these, example motors from stock in the “as received” condition were also inspected for comparative purposes.

6.2.1.2 Motor operation investigation

After removal, the failing motors were mounted to appropriate fixturing and connected to power, 27 V under no load, for characterization (similar characterization

was also performed of the new from stock motors for comparison). This first level investigation was performed to determine how the motors operated in isolation. This investigation yielded some interesting observations. For example:

1. It was found that motors which did not operate at all could be induced to operate upon gently turning the motor shaft or upon gently tapping on the motor housing.
2. Upon supplying power, some of the motors were found to operate initially very slowly coming to speed only after a few initial rotations.
3. In many cases the motors exhibited sporadic motion with apparent stalls and starts before coming up to speed.
4. The control stock motors performed as expected with no anomalous behavior observed.

These initial motor operational characteristics were judged to be consistent with the overall systems level failures. The data also suggest that contributions from the systems, be they materials related such as contamination or purely functional related to how the motors are operated in the systems in question, could contribute to the failures as the motors from stock operated as expected. With this in mind the systems level failure analysis was isolated to investigations of the motors.

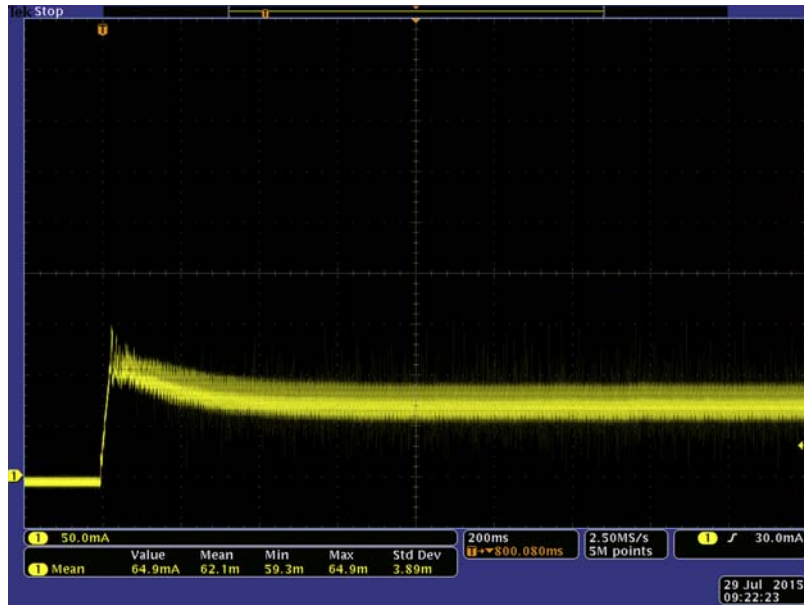
6.2.1.3 Electrical characterization

The motor assemblies in questions are comprised of a planetary gearbox mounted to a permanent magnet DC brushed motor. There are numerous failure mechanisms associated with DC brushed motors, some of which could result in similar failure modes [6,7]. Often, these failure mechanisms produce distinctive electrical characteristics, which could be used to direct the analytical approach and complement various other analytical techniques in identifying a potential root cause. As such, electrical characterization of the various motors during operation was carried out.

6.2.1.4 Control stock motor

During failure analysis in general, and when possible, it is desirable to obtain known control examples of whatever may be under investigations. These “as received,” “from stock,” “known good,” etc., examples are often useful control samples to establish baseline operating characteristics and any other parameters of interest for comparison to the failures under investigation. Such samples may also be used to determine possible flaws or design deficiencies that may contribute to the failures under investigation. It is also common to obtain examples of parts built from different lots to help understand if lot to lot variations contribute to the observed failures.

In this case, stock motors representing the “as received” condition were provided to obtain baseline “time zero” operational characteristics as well as allowing for a thorough investigation of the motor construction.

**FIGURE 6.1**

Oscilloscope capture displaying the current waveform through a stock motor versus time.

In this particular example, a stock motor (new from stock in the “as received” condition) and a failing motor were operated at nominal voltage with no load on the gearbox. The current was plotted with an oscilloscope using a high bandwidth current probe. Fig. 6.1 displays the current through a stock motor for a duration of approximately 2 seconds after power application; the RMS current was determined to be consistent with the vendor’s specifications. Fig. 6.2 displays the commutation current through the same motor over a period of approximately two revolutions. The commutation spikes displayed in the oscilloscope trace can be used to identify points at which the brushes transition from one commutation segment to another. Therefore, for example, a brushed DC motor with 5 commutator segments and 2 brushes, as was the case with the motors under investigation and reported here, display 10 commutation spikes over the course of one revolution. This can also provide useful information, such as calculating the RPM of the motor using an oscilloscope. Also noteworthy of this stock motor the instantaneous motor current is always nonzero, an indicator that the motor is generating continuous torque output.

6.2.1.5 Failed motor

After electrical characterization was performed on control motors as discussed in the previous section, electrical characterization of failed motor examples was performed in a similar fashion. These investigations revealed some interesting



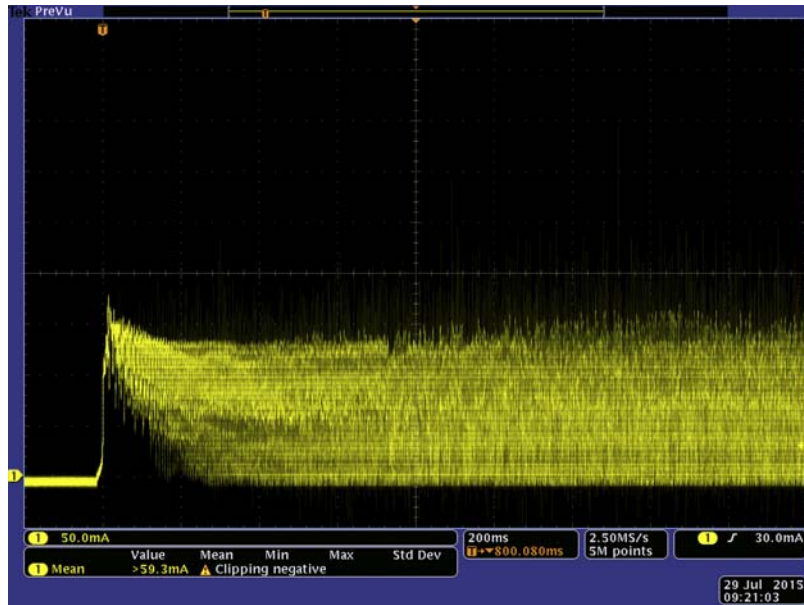
FIGURE 6.2

Oscilloscope capture displaying the commutation current through a stock motor over a period of approximately two revolutions. Commutation spikes are highlighted by the red arrows.

operational characteristics of the failing motors; in particular, some failing motors displayed evidence of varying shaft speed. In this section the results for one of these failing motors are discussed.

After power cycling numerous times, it was noted that, on occasion, the motor would not drive; however, gentle tapping on the side of the motor could restore operation (although the restored shaft speed was still varied). Fig. 6.3 displays the current through the failing motor for a duration of approximately 2 seconds after power application. The RMS current was measured, and was lower than that of the stock motor. Additionally, the waveform characteristics were significantly different than that of a stock motor, and produced instantaneous current values that dropped to zero.

Fig. 6.4 displays the commutation current through a failed motor over a period of approximately two revolutions. As was the case with the control motor, the commutation spikes provide useful information about the relative location for each brush/commutator combination. Regions A, B, and C in Fig. 6.4 represent specific brush/commutator locations where the instantaneous motor current is abnormally low. The periodic nature of these observations are indicative of abnormal contact between the brush and commutator interfaces in a localized fashion—this is as

**FIGURE 6.3**

Oscilloscope capture displaying the current waveform through a failing motor versus time.

opposed to such phenomena occurring in a random fashion that is also commonly observed during motor failure analyses using these methods. It is important to note that the failing motor's torque output in this example is minimal when the brush/commutator mating occurs in these regions, as current and torque are directly proportional. However, it is determined that the inertia of the gearbox carried the motor past these highly resistive regions enabling degraded motor operation consistent with reported field failures. Another important outcome of these findings is that if during power cycling the commutators stop at one of these high-resistance regions, the motors will not operate upon activation. However, if the regions are moved, for example, by applying mechanical input such as tapping or rotating the motors a few degrees, the motors start in a sluggish manner with decreased performance. These observations are also consistent with reported field failures and strongly suggest that a mechanism such as the localized presence of resistive contamination at the commutator/brush interfaces is the mechanism responsible for the motor failures.

6.2.1.6 Chemical characterizations

The electrical performance discussed in the previous section showed in a dramatic fashion that insulating contaminants at the brush/commutator interfaces most likely accounted for the system failures. Supporting this supposition is the fact

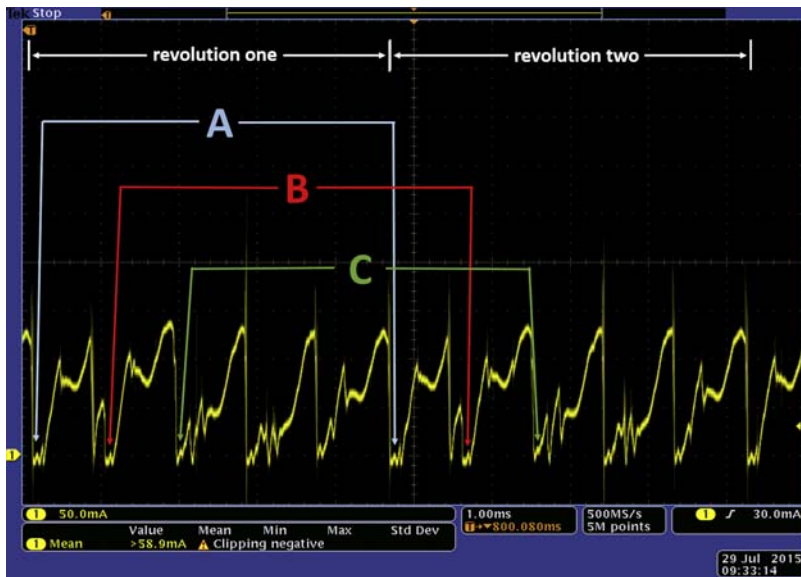


FIGURE 6.4

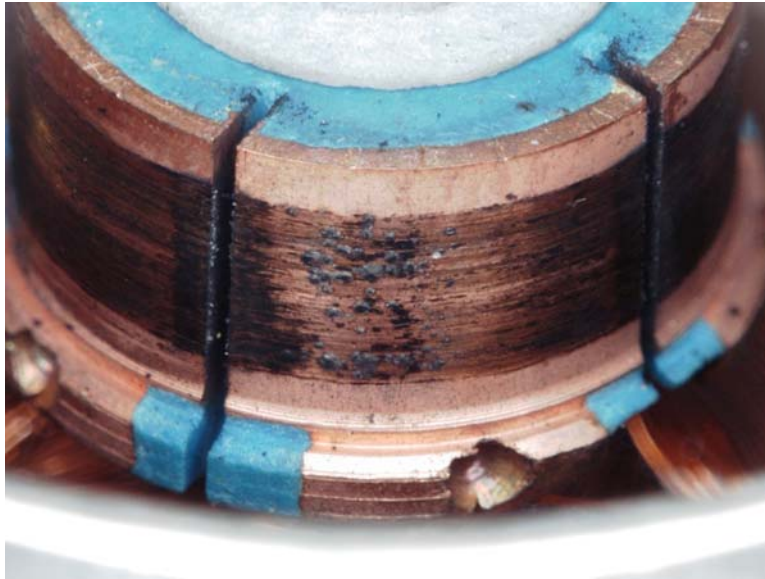
Oscilloscope capture displaying the commutation current through the failing motor over a period of approximately two revolutions. A, B, and C represent distinct regions on the commutator segments where the instantaneous motor current drops to zero.

that the step-by-step deconstruction analyses (not presented here) of the failing motors did not indicate any other possible contributors.

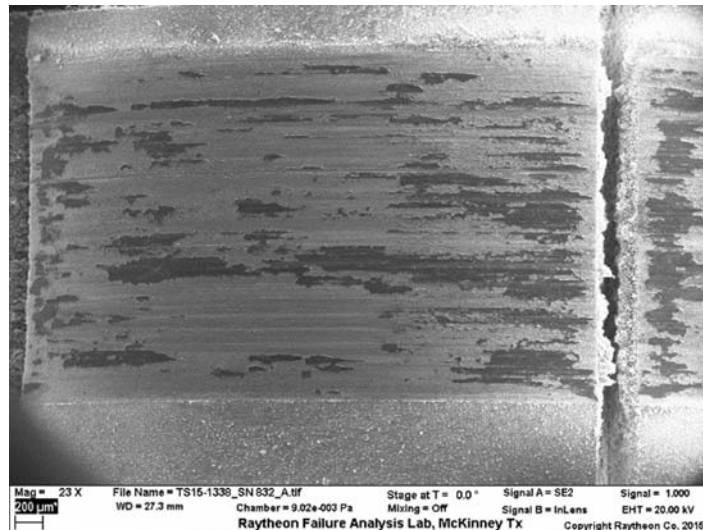
For example, the internal motor bearings were properly lubricated and found to rotate smoothly with no indications of any anomalies. However, during this deconstruction analysis and as shown in Figs. 6.5 and 6.6, unusual levels of surface residues on the commutators were judged to be present. This further indicated that contamination at the brush/commutator interfaces were responsible for the systems' failures. As a result, chemical analyses of these interfaces were carried out to test the contamination hypothesis as well as help to identify possible contamination sources [8].

Over the course of the chemical analysis portion of the investigations, a number of motors were investigated with an emphasis on the brushes and commutators, producing, in many cases, complex data. However, the findings were all consistent with the following failure mechanism:

1. Chemical species were liberated from a variety of sources, not the least of which were silicone grommets, depositing materials onto the commutator/brush surfaces as well as being absorbed into the carbon of the brush construction (carbon acts as a chemical trap, e.g., its use in water filtration).
2. The deposited and absorbed materials were converted into other more tenacious chemical forms via the action of the energy from motor arcing, heat

**FIGURE 6.5**

Commutators with surface deposits. Dark spots were noted on the commutator segments in two specific locations, separated by approximately 180 degrees. The dark streaks appear to be particulate deposits from normal brush wear.

**FIGURE 6.6**

Scanning electron microscope image showing large deposits (dark features) on a commutator surface.

from operation, and direct frictional energy at the contact surfaces from the rotation of the motor. For example, the formation of silica upon the decomposition of source silicones or the polymerization of source organic materials forming a resistive surface “varnish” at the contact interfaces [9].

3. These deposited materials formed indirectly from distinct source materials released from the motor construction materials as well as system construction materials and were removed at a rate much slower than their rate of formation (motor brush commutator pairs are “self-cleaning” via normal operational wear and at times even have added “wipers” to help remove surface contaminants).

The following chemical analysis data are representative of some of the findings gained during the investigation.

The commutator surface deposits displayed in scanning electron microscope (SEM) Figs. 6.7 and 6.8 were typical of many of the commutator surface residues. While the filmlike residues displayed in Fig. 6.8 are expected to some degree even on the commutator surfaces of properly operating motors, the large deposits displayed in Figs. 6.5 and 6.7 are not. In fact these large contamination features were some of the most pronounced ever observed by the lab from a number of similar motor failure analyses.

These findings were consistently observed toward the ends of the commutators indicating that such regions were responsible for the periodic current drops displayed in Fig. 6.4. To further characterize the sources of the contaminants, elemental

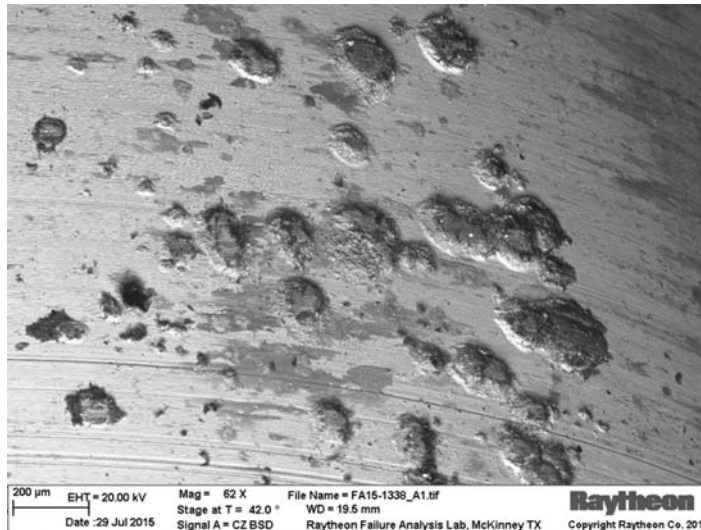
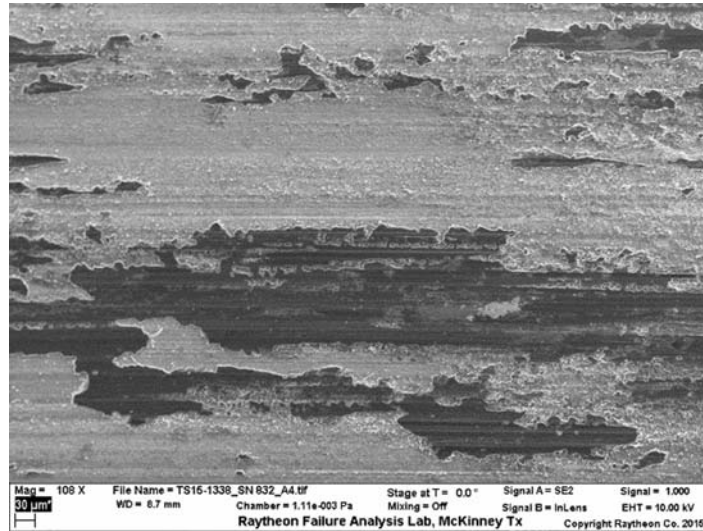


FIGURE 6.7

Large deposits on commutator surface.

**FIGURE 6.8**

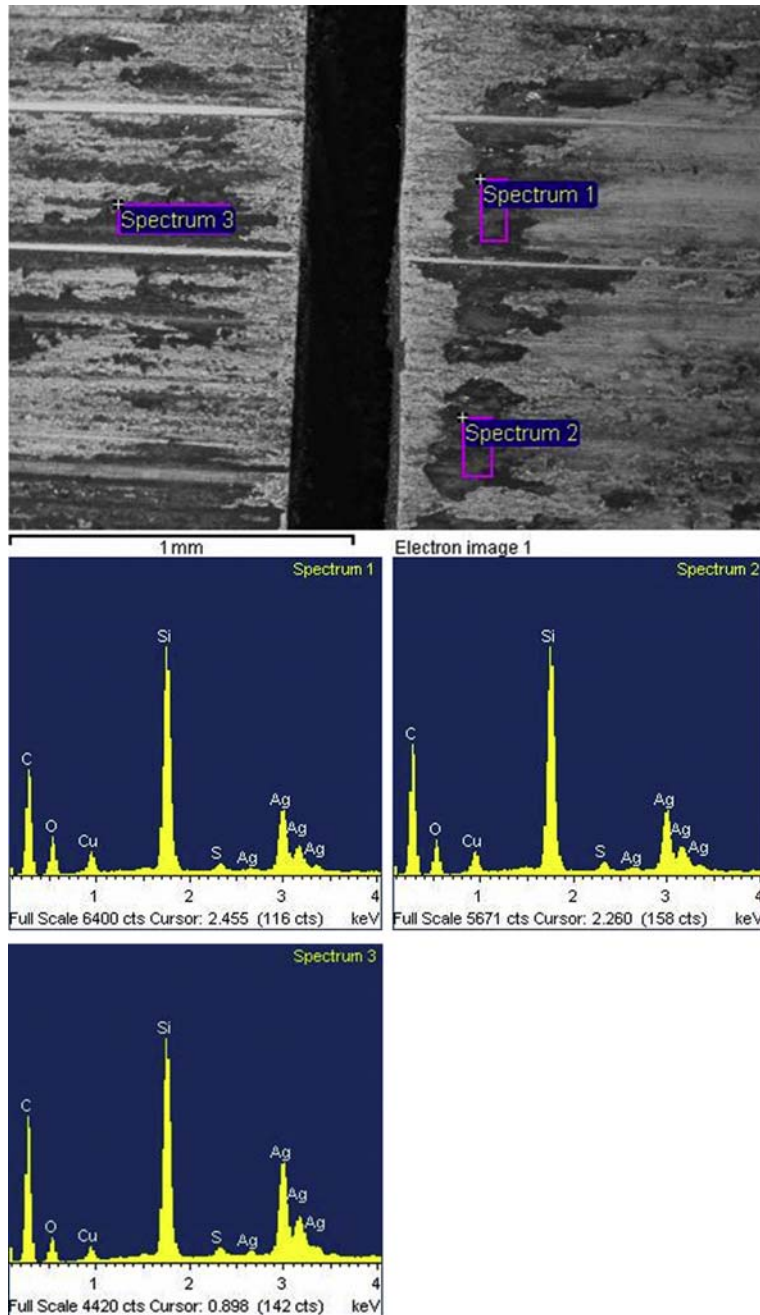
Filmlike deposits on commutator surfaces.

spectra were also obtained of the residues using energy dispersive spectroscopy (EDS) while imaging in the SEM.

The elemental spectra displayed in Fig. 6.9 collected from commutator surfaces from a failed motor are distinguished by the intense return for silicon (Si). Also indicated as possible contamination contributors are the findings of carbon (C), oxygen (O), and sulfur (S). The findings of Si in combination with C and O indicate that silicones as well as silica formed upon the decomposition thereof likely account for much of the electrically insulating surface contaminates (these also account for the observed system failures) [10].

It is worth noting in passing that gas chromatography/mass spectrometry (GC/MS) analyses of brushes indicated that the brushes from failing motors released sulfur-containing gaseous molecules that can help explain the presence of high sulfur deposits on some commutator surfaces (both the example shown here as well as others in which the level of sulfur in deposits was more than $10\times$ the presented example). These data show that it is likely that a variety of materials are present on the brush and commutator surfaces. It is important for the analyst to realize that some contamination is expected at commutator and/or brush surfaces of common motor designs and in some cases is even beneficial to motor operation [11].

Fourier-transform infrared (FTIR) spectroscopy methods were also employed to further characterize the surface contaminates (in most cases microscope reflectance methods were employed). The following spectrum was commonly

**FIGURE 6.9**

Elemental spectra obtained from commutator edges of a failed motor.

associated with areas such as those shown in Fig. 6.9 in which the elemental analysis data suggested that silicones and silica could be present in these areas.

The infrared spectrum displayed in Fig. 6.10 indicates that silica and silicones are present on the commutator surface. The band at about 1100 cm^{-1} , in particular, is associated with Si—O—Si bonding of these materials. Review of the motor construction indicated that silicone grommets were used to electrically isolate conductors from the motor housing and, as such, were deemed as the likely source of the silicone outgassed materials. These grommets were found to liberate relatively high levels of gaseous silicones using GC/MS methods (data not presented here). Additionally, review of outgassing data published by NASA of the silicone grommets used for these motor construction indicated that the materials release a relatively high level of materials via outgassing mechanisms [12].

A number of other chemical analyses were performed that showed the presence of organic films as well as other contaminants in/on the brushes and on the commutator surfaces during these analyses that were distinct from silicone-derived materials (data not shown). Likely source materials of these residues were also identified.

Consultation with the supplier revealed that similar failures had never been reported for the motors in question; the implication being that there were no inherent design problems associated with these motors. Supporting this supposition was the fact that Raytheon had successfully employed this same motor in separate applications. This anecdotal information suggested that the motor, while perfectly appropriate for common applications, was likely incompatible for use

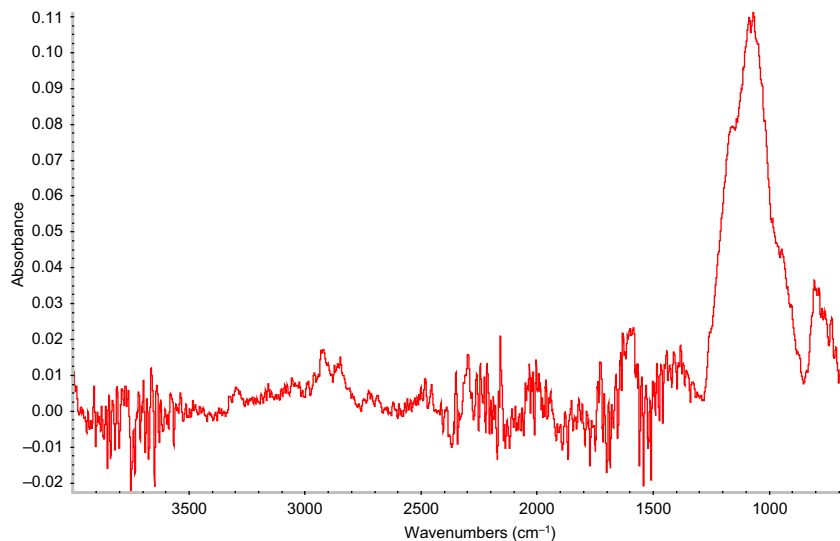


FIGURE 6.10

Infrared spectrum collected from an area such as displayed in Fig. 6.9.

with these imaging systems. A review of the operating conditions, in conjunction with controlled experiments, ultimately showed this to be the case.

For example, as employed with the failing systems, the motors were activated in a very sporadic fashion when compared to other applications in which similar failures had not been reported. Specifically, when switching between the various system viewing modes, the motors were determined to operate for only a few seconds upon actuation. This is in contrast to the other examples in which the motors were used in a more continuous fashion with applied power for much longer intervals (minutes to hours).

These findings suggested two possible paths forward—select a different motor more suited to the application or alter the motor construction such that the specific contamination mechanism (deposit of contaminants followed by decomposition thereof) is eliminated to an acceptable level. In this case the latter path was chosen via the use of alternate nonsilicone grommets. This materials change was shown to eliminate the failures completely.

In passing it is worth noting that such a mechanism of contamination outgassing and deposition at motor contact surfaces leading to the formation of insulating residues had been observed several times by Raytheon. For example, in one case the effects of silicone materials outgassing in the immediate vicinity of motors were shown in a dramatic fashion. Specifically, when these motors were continually operated over an 8-hour period, they gradually slowed to a complete stop. However, upon eliminating the ingress of volatile silicones via sealing the motors from the environment, the test of a control motor was stopped after several months of continual operation. In this case, the sporadic on-then-off operational condition was not necessary to affect motor failures. In another example, a nonsilicone contamination source was shown to lead to this same failure mechanism. In that case consultation with the supplier revealed that brushes were temporarily affixed to tape adhesive to allow for the facile application of springs. The contaminants thusly deposited were shown to lead to surface contact resistance via the formation of brush/commutator surface contamination.

Finally, it must be pointed out that that motor contact surfaces are normally expected to display some level of surface contamination. For example, carbon brushes are particularly susceptible to contamination as the carbon serves as a trap for gaseous materials [13]. In most cases such contamination is not problematic as the self-cleaning action of motors typically keep this failure mechanism under control; however, under specific conditions, for example, as shown here, surface contamination can lead to motor failures.

6.2.2 Example 2—Electrolyte contamination

Electrolytes are materials that, when dissolved in water, increase the conductivity of the resulting solutions beyond that of water alone. Salts such as sodium chloride and potassium carbonate are examples of strong electrolytes as they are highly soluble in water forming hydrated ions in solution. On the other hand,

organic acids, for example, those found in many soldering fluxes, are examples of weak electrolytes. This results from the fact that when these materials are dissolved in water only a small fraction of the resultant species hydrated ions (the acids are present primarily in the unionized form). Because of these properties, it is common that these materials are collectively referred to as “ionic” contamination. Common failure modes associated with such contamination include electrical leakage and shorting as well as corrosion of metals. Because even small amounts of such contamination can result in catastrophic failures of electronics, several industry standards exist with regard to analytical methods for the detection of such materials as well as providing guidance with regard to acceptable levels [14]. It should be pointed out that the level of water necessary for the formation of conductive solutions with regard to electronics is rarely beyond the levels native to materials exposed to ambient conditions—this does not require visible condensed liquid solutions [15].

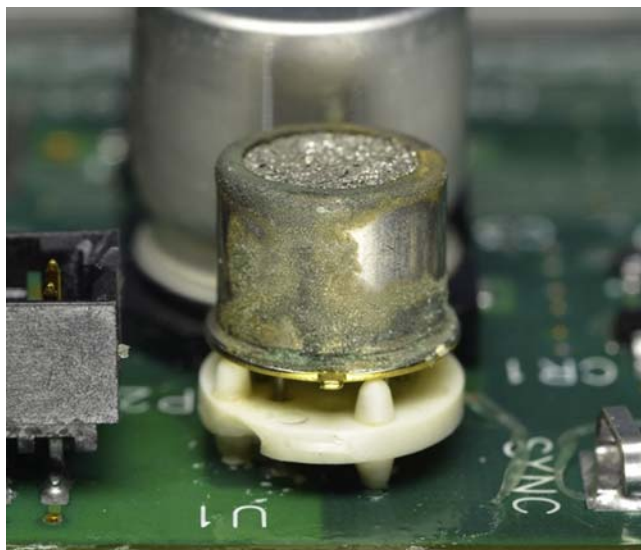
Presented here are examples in which such contamination have led to the failures of electronics. The examples selected were chosen to highlight that, at times, the failures associated with such materials are not immediately obvious.

6.2.2.1 A leaking capacitor

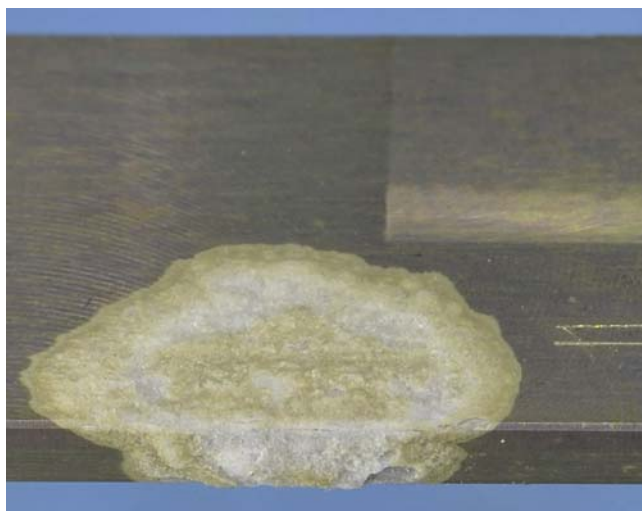
Liquid electrolytes are commonly employed for the construction of capacitors and as such it is crucial that these liquids be sealed within capacitors and not allowed to leak onto sensitive surfaces [16]. While in many cases the effects of leakage of capacitor electrolyte onto sensitive surfaces are subtle and difficult to identify during failure analysis, the example presented here, of a failure resulting from such liquid leakage, was manifest in dramatic fashion.

In this example, the lab was presented with a system failure associated with the malfunction of a humidity detector component. Upon disassembly of the system, the humidity detector was noted to be discolored with apparent residue contamination as shown in Fig. 6.11. Interestingly, and initially assumed to be distinct from this component level failure, residues were also noted on an aluminum bracket of the larger system construction, Fig. 6.12, as well as associated with a connector, Fig. 6.13.

Among initial hypotheses, based solely on the appearance of the residues, was that a possible electrical overstress event may have occurred leading to an arcing event accounting for the appearance of the connector. It was also thought that such an event may have affected the humidity detector. However, the only reported systems failure was the humidity detector malfunction with the remainder of the system operationally unaffected, seemingly discounting this initial hypothesis. Another more plausible explanation was that the residue formation at these three distinct areas of the overall system resulted from independent events. For example, the aluminum frame was in a distinct location with regard to the connector and humidity detector and is, of course, not part of the circuitry. With this in mind, analyses of the residues for chemical composition were carried out.

**FIGURE 6.11**

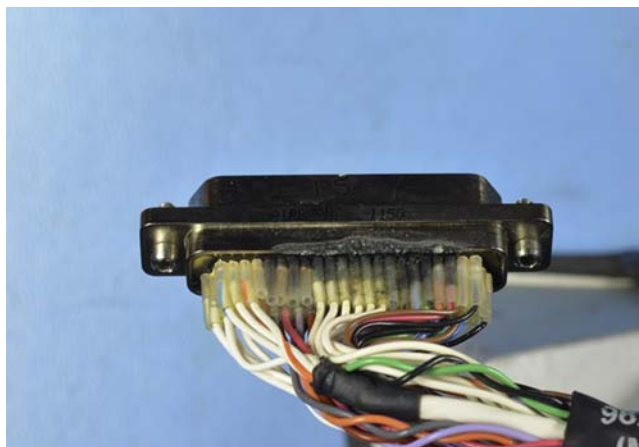
Humidity detector with residues on the surface.

**FIGURE 6.12**

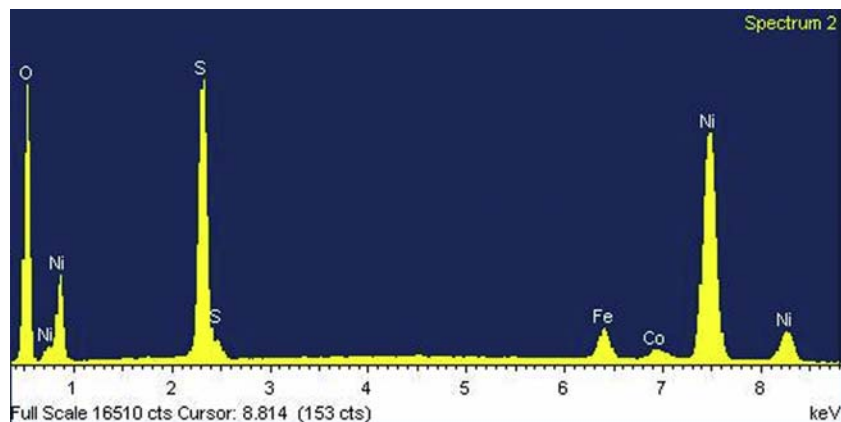
Residues on a connector frame.

Elemental analyses were carried out using SEM/EDS methods while infrared spectra were collected using FTIR methods.

The elemental spectra displayed in [Figs. 6.14–6.16](#) have relatively intense peaks indicating that sulfur (S) and oxygen (O) are present in all the residues.

**FIGURE 6.13**

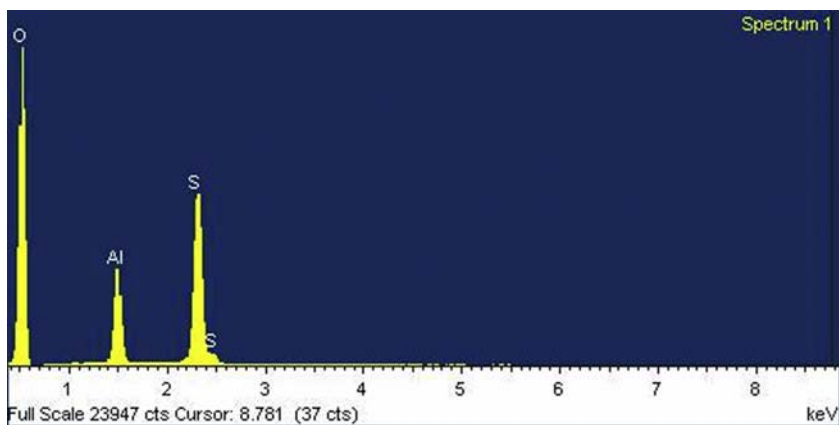
Residue on an aluminum frame.

**FIGURE 6.14**

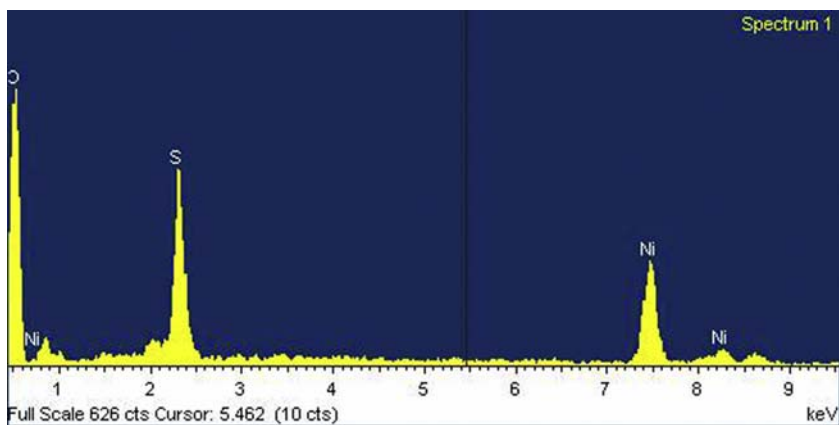
Elemental spectrum collected of humidity detector residue.

In addition to these elemental spectral similarities, the infrared spectra displayed in Fig. 6.17 also share many commonalities. Finally, as shown Fig. 6.18, the data-base matching result of the residues are composed of the sulfate salts of the base metals on which they reside (The base metals in question were determined to be a nickel plating for the connector frame, aluminum from the housing frame, and an Inconel alloy for the humidity detector. These compositions were verified from the parts specification data sheets.).

This chemical characterization data indicating that the residues are all composed of the sulfate salts of the base metals on which residues were initially hard

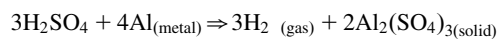
**FIGURE 6.15**

Elemental spectrum collected of aluminum bracket residue.

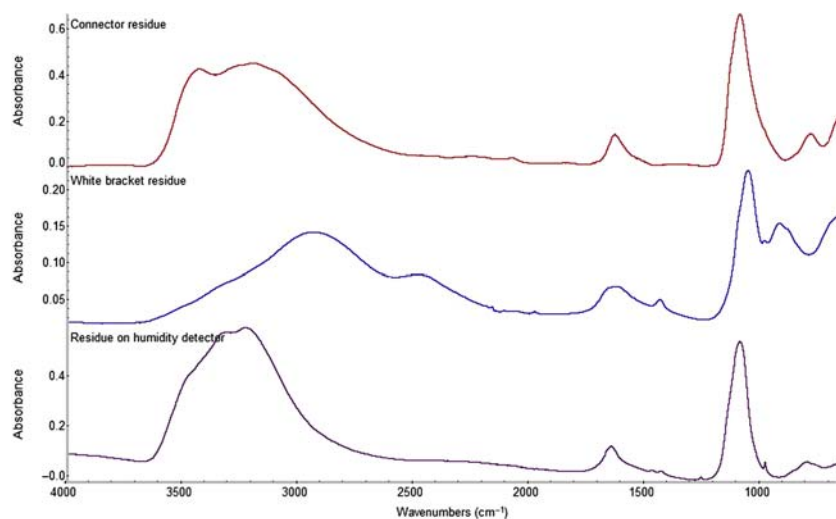
**FIGURE 6.16**

Elemental spectrum collected from the connector frame.

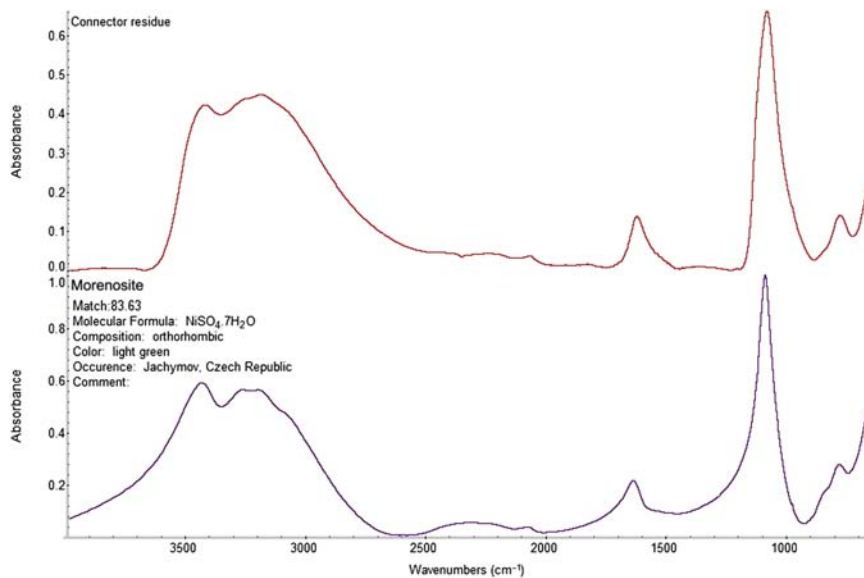
to rationalize. For example, their formation was judged to have most likely occurred via the reaction of sulfuric acid solutions with the base metals on which they reside. This reaction is displayed for the aluminum frame in the following chemical equation.



The findings, suggesting that a sulfuric acid solution had dropped onto the surfaces, were initially very confounding. Discussion with the customer revealed that such solutions, for example, those which may have been used for aluminum

**FIGURE 6.17**

Infrared spectra of the various residues (as labeled).

**FIGURE 6.18**

Infrared spectral database matching result of connector residue spectrum (top spectrum is of the residue, bottom spectrum is the database match).

conversion coat touch up, had not been applied at any point in the construction of the system. Furthermore, the distribution of these residues, at first, seemed unrelated as the areas in question were not in immediate proximity to each other. Further investigation into the composition of the various components of the system was thusly undertaken.

The only possible explanation for a source of sulfuric acid which remained, upon review of all processing the system had undergone, was that either a sulfuric acid solution had been used in proximity to the system by some unreported mechanism or that a source existed which was native to the system construction. The later explanation seemed most likely, and it was theorized that a wet-tantalum capacitor had been used within the system. Review of the various specifications for the components in the system as installed by Raytheon indicated that no such capacitors had been used during the construction of circuitry built by Raytheon. However, it was found that the small circuit card on which the humidity detector resided represented a commercial off-the-shelf part. As such, a careful examination of this circuit card was undertaken. The results were revealing.

Fig. 6.19 shows the presence of liquid droplets near one end of a capacitor affixed to the humidity detector Circuit Card Assembly (CCA). It should be pointed out that this area of the CCA was not initially visible as it was buried deep within the system and considerable disassembly was necessary for access.

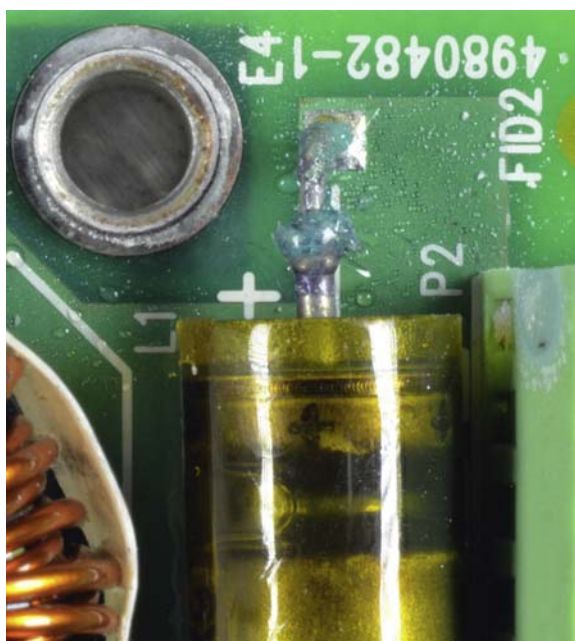


FIGURE 6.19

Capacitor on the commercial off-the-shelf humidity detector circuit card.

Chemical analysis of the droplets using FTIR methods revealed that they were in fact composed of highly concentrated sulfuric acid. In addition to these findings, a simple demonstration showed when liquids were allowed to drop off the detector CCA, they landed on the connector and aluminum housing frame in a fashion consistent with the observed residues. This finding allowed for rationalization of formation of these residues at seemingly distinct areas.

In addition to these findings, the markings on the capacitor revealed that it was, in fact, a sulfuric acid electrolyte wet-tantalum capacitor. Additionally, as shown in Fig. 6.20, upon removal and disassembly, the inner chamber of the capacitor was found to be dry. Finally, upon analysis of a cross section of the capacitor end near the droplets found on the humid detector CCA, shown in Fig. 6.21, the leakage path was discovered. These findings allowed for unambiguous assignment of the source of the sulfuric acid that led to the residue formations.

It is worth pointing out that the potential damage that may have resulted was surprisingly limited in this case. For example, the acid droplets which landed on the connector could have caused significantly more damage via formation of conductive pathways between individual pins. The fact that the damage was limited to the formation of corrosion products of the outer connector frame was fortunate. Additionally, if the droplets had deposited at other more electrically sensitive areas, it is likely that a more catastrophic systems level failure would have occurred.

Remediation included moving the card in the systems such that if a similar incident were to occur the leaking acid would be isolated to an electrically less sensitive area. While the card to which this capacitor is mounted fails when such



FIGURE 6.20

The inner electrolyte chamber was found to be dry.

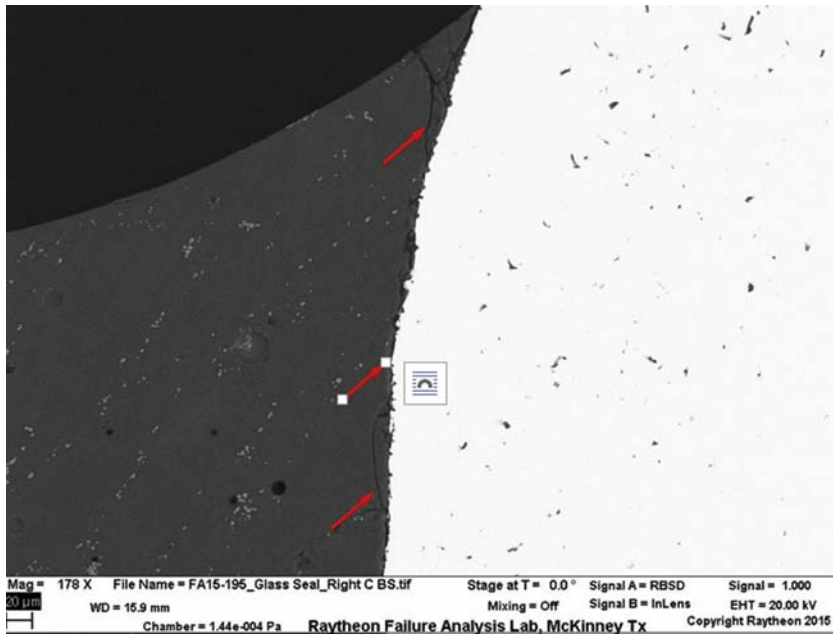


FIGURE 6.21

Scanning electron microscope image showing a crack along with the metal/glass seal at the leaking end of the capacitor.

leakage occurs, it will not cause catastrophic system failure (loss of a system sensor only). However, when mounted in the system as is, catastrophic system failure is a distinct possibility as noted.

6.2.2.2 Other types of electrolyte contamination damage

The example presented in the previous section is not the most common with regard to electrolyte contamination and electronics failures (sulfuric acid solutions are not typically encountered during such investigations). It was chosen to highlight the fact that during failure analyses, sources of contamination as well as the results thereof are not always obvious.

Some of the more common sources of electrolyte contamination include entrapment of cleaner solutions, handling, process chemicals, and solder fluxes [17,18]. While such sources of electrolyte contamination rarely contain materials as aggressive as sulfuric acid as presented in the previous example, these more common sources in many cases also contain acidic compounds such as activators found in fluxes as well as alkaline materials found in board cleaners. Such aggressive compounds can also directly lead to corrosion of metals as well as create leakage paths [19].

Fig. 6.22 displays corrosion at a copper mag wire with a polymer coating soldered to a lead. Residual water-soluble flux which was not rinsed off after application was shown to have led to the formation of the corrosion products. The wire in this area was completely consumed, causing an open circuit.

It is worth noting that the presence of residual water-soluble fluxes, in particular, on electronics often leads to greatly accelerated corrosion as well as shorting. This is in contrast to the presence of residuals of benign fluxes such as no-clean varieties (no-clean fluxes are designed to remain on components and boards, hence the designation “no-clean”). Additionally, while in many cases water-soluble fluxes may be advertised as being pH neutral resulting from careful buffering of the fluxes, these still, in fact, contain aggressive chemicals including strong electrolytes. The aggressive nature of most of these fluxes is reflected by the requirement of removal in a relatively short time after application per the guidelines of their use.

Another common outcome of the presence of electrolyte contamination is the electromigration of metals [20]. It is an unfortunate coincidence that metals common to electronics (e.g., tin/lead solders, copper, and silver) are those that are most likely to form the dendritic growths associated with electromigration [21]. An example in which cleaner entrapment in a connector that led to electromigration has been presented in a previous handbook in this series [22].

Fig. 6.23 shows an example in which flux residue was found to have accelerated electromigration of metals associated with a connector. The pins labeled 44 and 45 have plated copper formations on the gold coating of the pins. The dull appearance of these pins results from the presence of copper and its oxides on the outermost gold pin plating layer. These materials identification as well as



FIGURE 6.22

Corrosion of copper wire resulting from the presence of residual flux.

**FIGURE 6.23**

Electromigration of metals resulting from flux contamination.

verification of the presence of the gold under the deposits was verified using SEM/EDS methods. Additional lead rich electromigration products are represented by the gray residues at the base of several of the pins. Additional corrosion products, whose growth was accelerated by the presence of electrolyte contamination and applied power, are the greenish tinted residues just above the labels 25 and 26 in the figure.

In one example presented to the lab, there was disagreement between the customer and lab personnel with regard to the formation of dendritic growths via the electromigration mechanism. The customer held that electromigration was not possible as deionized (DI) water had been used in a humidity chamber after which the growths were noted. This, of course, was an improper argument as even trace levels of electrolyte is sufficient in many cases to accelerate electromigration of metals. The lab created a real-time movie to demonstrate electromigration of metals on components of the same dimension of those on which the formations were observed. This demonstration is included in the electronic version of this publication [23] and shows in rather dramatic fashion that electromigration can occur even when DI water is applied and can occur very rapidly.

6.3 Discussion

Contamination of electronic circuits leading to failures is both intuitive and common knowledge with examples encountered in everyday life. The examples given

here were chosen to highlight the fact that sources of contamination and the effects thereof are often not straightforward. It was also the goal of the authors to demonstrate the logical progression of the failure investigations presented as well as providing the rationale for the application of common analytical methods employed during such analyses.

With regard to the highlighted modes of contamination via outgassing of materials with concurrent deposition at electrically sensitive areas is, in the authors' opinion, an extremely important root cause of electronics failures. This is particularly important as it is almost certainly extremely underreported in industry as it generally requires sophisticated analytical methodologies and complex testing and experimentation for root cause determination. On the other hand, in space systems design, this is a very well-known mechanism and is factored into all stages of development [12].

Avoidance of such failure modes starts with the understanding that such mechanisms are more common than typically anticipated. During development stages of product design, it is essential that potential environments to which the products may be exposed be taken into account beyond the obvious. For example, the authors have investigated examples in which the localized environments to which table-top electronics used in the restaurant industry have directly led to failures resulting from accelerated oxidation of silver coated contacts (silver sulfide formation) resulting from high concentrations of sulfur-containing gases from steaming foods.

Electrolyte contamination is extremely well known and a very large driver of failures during all stages of an electronic product's life. During the assembly process, chemicals such as active soldering fluxes are well-known sources of such contamination. While in-whole the industry has improved processes and materials to minimize electrolyte contamination during electronics production via a multitude of approaches (e.g., the emergence of near electrolyte free "no-clean" soldering fluxes, improved and automated cleaning processes, development of standardized, and quick testing procedures of electrolyte contamination) the importance of such contamination is reflected in the sheer number of investigations regarding such contamination [24].

Perhaps the most important way to avoid such contamination is process control. For example, the use of active soldering fluxes (in particular water-soluble products) offers many advantages including simple cleaning after soldering using water alone. However, the advantages offered by such products commonly result from the presence of potentially deleterious electrolytes. Such cleaning of these fluxes is typically specified within 8–24 hours of application and deviations from such strict process controls can lead to unexpected failures (although it should be noted that solutions can wick into small crevices and "standard" rinse and drying processes may not adequately remove it).

The examples given herein and the logical progression of the failure analyses reported were selected to highlight the often elusive nature of the role contamination plays in electronic device failures.

References

- [1] W. Wolfgong, K. Wiggins, The silicone conundrum, *J. Failure Anal. Prev.* 10 (4) (2010) 264–269.
- [2] M. Antler, Field studies of contact materials: contact resistance behavior of some base and noble metals, *IEEE Trans. Compon., Hybrids, Manuf. Technol.* CHMT-5 (1982) 301.
- [3] W.J. Wolfgong, R. Ogden, B. Waller, Surface oxidation as a tin whisker growth mechanism, *Circuits Assembly* 16 (12) (2005) 24.
- [4] V. Khanna, Humidity and contamination effects on electronics, *Extreme-Temperature and Harsh-Environment Electronics: Physics, Technology and Applications*, IOP Publishing, Bristol, 2017, pp. 15-1–15-11.
- [5] W. Rieder, G. Witter, Arc Interactions with contaminants, *Electrical Contact: Principles and Applications*, Marcel Dekker, Inc., New York, NY, 1999, pp. 809–837.
- [6] Z. Tong, Z. Dong, M. Li, A. New, Entropy Bi-cepstrum based-method for DC motor brush abnormality recognition, *IEEE. Sens. J.* 17 (3) (2017) 745–754.
- [7] R. El-Mahayni, K. Al-Qahtani, A. Al-Gheeth, Large synchronous motor failure investigation: measurements, analysis, and lessons learned, *IEEE Trans. Ind. Appl.* 52 (6) (2016) 5318–5326.
- [8] A review of a number of chemical analysis methods employed during electronics failure analysis are available in a previous handbook of this series – W.J. Wolfgong, *Chemical analysis techniques for failure analysis Part 1, common instrumental methods*, *Handbook of Materials Failure Analysis: with Case Studies from the Aerospace and Automotive Industries*, Chapter 14, Elsevier, Oxford, 2016pp. 279–307.
- [9] B. Reagor, L. Seibles, Structural analysis of deuterated and nondeuterated frictional polymers using Fourier transform infrared spectroscopy and pyrolysis gas chromatography/mass spectroscopy, *IEEE Trans. Compon, Hybrids, Manuf. Technol.* 1 (1) (1981) 102–108.
- [10] A. Okada, M. Toda, Influence of silicone contamination on brush-commutator contacts in small-size DC motors, *IEEE Trans. Compon., Hybrids, Manuf. Technol.* 5 (2) (1982) 281–286.
- [11] G. Cornelissen, M. Elmquist, I. Groth, O. Gustafsson, Effect of sorbate planarity on environmental black carbon sorption, *Environ. Sci. Technol.* 38 (13) (2004) 3574–3580.
- [12] Outgassing Data for Selecting Spacecraft Materials Online. Available for free at: <<https://outgassing.nasa.gov/>> (site last checked 12.05.18).
- [13] W. Kalb, Maintenance of good brush performance, *Electr. Eng.* 64 (12) (1945) 996–997.
- [14] IPC TM-650 Test Methods Manual.
- [15] P. Isaacs, J. Porto, D. Braun, T. Munson, Comparison of ionic contamination test methods to determine their ability to reliably predict performance risks, in: 2017 Pan Pacific Microelectronics Symposium (Pan Pacific), 2017, pp. 1–7.
- [16] B. Gosse, J.P. Gosse, M. Sauviat, Interactions between solid and the ions of liquid in impregnated paper, *IEEE Trans. Electr. Insul.* EI-15 (2) (1980) 104–111.
- [17] W.J. Wolfgong, Cleaning COTS Parts in Military Applications, *Circuits Assembly*, October 2009.
- [18] X. He, L. Zhou, J. Shen, A study for a typical leakage failure of PCBA with no-cleaning process, in: 17th International Conference on Electronic Packaging Technology (ICEPT), 2016, pp. 53–56.

- [19] S. Kolesar, Principles of corrosion, in: 12th International Reliability Physics Symposium, 1974, pp. 155–167.
- [20] Y. Chen, M. Hsiao, Investigation of electrochemical migration on a fine pitch BGA package, in: 19th IEEE International Symposium on Physical and Failure Analysis of Integrated Circuits, 2012, pp. 1–4.
- [21] S. Krumbein, Metallic electromigration phenomena, *IEEE Trans. Compon., Hybrids, Manuf. Technol.* 11 (1) (1988) 5–15.
- [22] W.J. Wolfgong, Chemical analysis techniques for failure analysis Part 2, examples from the lab, *Handbook of Materials Failure Analysis: With Case Studies from the Aerospace and Automotive Industries*, Chapter 15, Elsevier, Oxford, 2016, pp. 331–336.
- [23] In this example the customer was insistent that because DI water had been used in a humidity chamber no electromigration could occur from exposure to that water (not to mention there is always some contamination transferred from the part itself as well as the humidity chamber). The movie was created, which can be found at <https://doi.org/10.1016/B978-0-08-101937-5.00006-3>, to demonstrate this phenomena by submerging a capacitor in DI water at the conditions specified in the movie.
- [24] A simple web search using terms such as “electrolyte contamination of electronics” results in page after page of results.

Analytical solutions for electronic assemblies subjected to shock and vibration loadings

Mohammad A. Gharaibeh

Department of Mechanical Engineering, The Hashemite University, Zarqa, Jordan

7.1 Introduction

In service life, electronic products are prone to mechanical shock and vibration loadings and that leads to catastrophic solder interconnects failures. For this reason, the fatigue life assessment of electronic assemblies under shock and vibrations has become a major concern in industry. Researchers have discovered, developed, and conducted several experimental studies, finite element simulations, and analytical solutions to study and evaluate the fatigue, that is, reliability, performance of electronics due to shock/impact and vibration loading conditions [1–14].

In general, for an electronic assembly under bending, studies have shown that the solder failures are mainly due to the flexural differences between the integrated circuit (IC) component and the printed circuit board (PCB) [15–17]. Wong et al. [18,19] used the two elastically coupled beams problem approach to assess solder axial stresses due to mechanical shock and vibration. The same problem of coupled beams was employed to compute solder stresses due to symmetrical static loadings [20] and concentrated forces [21,22] in the cases of full and partial elastic couplings [23]. Perkins et al. [24] proposed the beam-spring-mass model to characterize the dynamic properties and solder deformations of a ceramic column grid array package subjected to harmonic vibration. Recently, Gharaibeh et al. [25] presented the elastically coupled two-parallel-plate system to derive the equation of motion of a vibrating assembly.

This chapter adopts the analytical solution presented by the author [25]. This solution is employed here to solve for the free and forced problems of an electronic package under shock and harmonic vibrations. The details of this analytical solution development and validation with experiments and finite element simulations are provided in this chapter. Finally, the effect of the package geometric and material configurations on the fatigue performance due to shock/impact loadings are also presented.

7.2 Test assembly details

As shown in Fig. 7.1, a squared PCB of 76.2 mm length and 1 mm thickness, with a centrally placed $17 \times 17 \times 1 \text{ mm}^3$ Amkor CABGA 256 dummy component having 280 μm height and 540 μm width eutectic 63Sn37Pb BGA solder joints, is considered in this work. The solders are evenly separated by a 1 mm pitch.

7.3 Experimental modal analysis

The natural frequencies and mode shapes of the present test vehicle are measured using hammer testing experiment, that is, modal analysis. The setup of this experiment is depicted in Fig. 7.2. In the setup, the test piece is attached to an Aluminum fixture at the four screw holes. An integrated circuit piezoelectric impact hammer was used to gently hit the test sample at certain grid locations. A lightweight accelerometer was placed at a fixed point and used to measure the response acceleration after each hammer impact. The National Instruments data acquisition system model 4413 (NIDAQ-4413) was used to acquire the acceleration-to-force transfer function for each measurement point. Finally, STAR Modal Version 7.0 [26] was adopted to produce the modal data of the test piece.

7.4 Finite element modeling

ANSYS Release 17.0 [27] was used for constructing the FE model of the assembly. In this model, only 3-D hexahedron elements, specified as SOLID185 in ANSYS package, were chosen to generate the mesh through the strategic

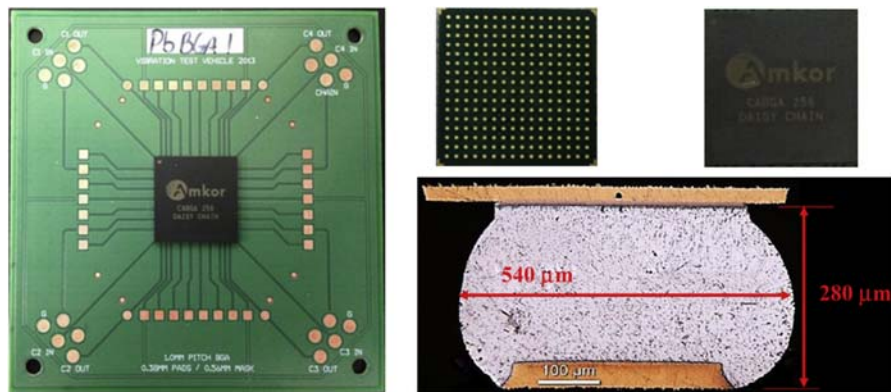
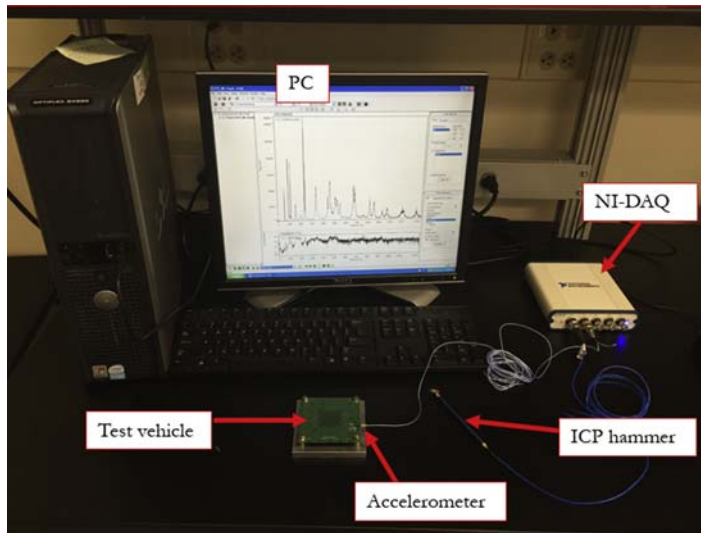
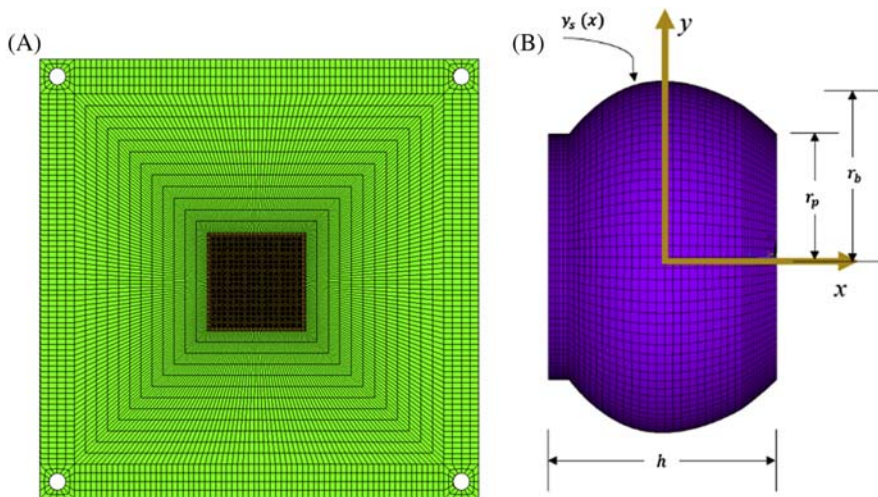


FIGURE 7.1

Test vehicle details.

**FIGURE 7.2**

Modal analysis experiment. *ICP*, Integrated circuit piezoelectric.

**FIGURE 7.3**

(A) Finite element model and (B) solder joint mesh and geometry.

selection of the isoparametric mapping concept. This element type was attributed to the PCB, component, solder joints as well as copper pads. The FE model of the current assembly is depicted in [Fig. 7.3](#).

Table 7.1 Linear elastic material properties used in FEA model.

	PCB	Component	Solder joints 63Sn37Pb
Young's modulus (GPa)	32.0	27.0	34.0
Poisson's ratio	0.14	0.25	0.40
Density (kg/m ³)	3000	1100	8410

FEA, *Finite element analysis*; PCB, *printed circuit board*.

The assembly material properties used in the finite element analysis (FEA) model are listed in Table 7.1. All the materials are assumed to be linear elastic isotropic. The boundary conditions were imposed by restraining the PCB at the top and bottom surfaces of the PCB at the screw holes' locations in all directions, that is, all degrees of freedom were set to 0. The constrained regions on the PCB are equal to those fixed by the screws, standoffs, and washers of the modal analysis experiment.

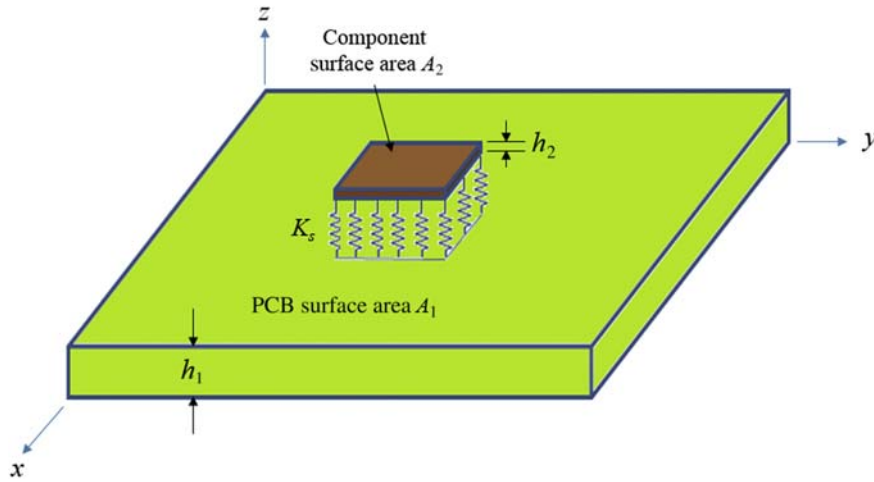
Three primary FEA analyses were conducted: modal, harmonic, and transient analyses. In modal analysis, the structure natural frequencies and mode shapes were computed. Large mass method was used to simulate base vibration. In this method, the model is attached to a very large mass ($\mathcal{M}$), then a force $F = \mathcal{M}a$ (where a is the required acceleration input level) was applied on the large mass in the transverse direction. Additionally, a mode superposition transient analysis using enforced motion method, available in ANSYS, was adopted to simulate the test board response due to base excitation shock loading.

7.5 Analytical solution details

7.5.1 Free vibration

Generally, the electronic assembly can be faithfully described by a two elastically coupled plates system [25] as depicted in Fig. 7.4, with the geometric and material parameters listed in Table 7.2. This analytical model suggested that the PCB is a bottom elastic plate; the component is a rigid top plate and both are connected by linear axial springs. Springs here, represent the solder interconnects. In that model and according to Ritz method, the PCB and component displacement solutions, $v(x, y, t)$ and $u(x, y, t)$, respectively, were written in series form of admissible functions composed of multiplication of the PCB mode shapes $V_i(x, y)$ or component mode shapes $U_i(x, y)$ by the time-dependent generalized coordinates $z_i(t)$ as:

$$\begin{aligned} v(x, y, t) &= \sum_i^N V_i(x, y) z_i(t) \\ u(x, y, t) &= \sum_i^N U_i(x, y) z_i(t) \end{aligned} \quad (7.1)$$

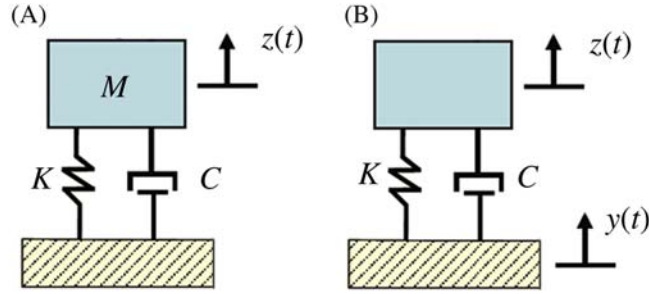
**FIGURE 7.4**

Two elastically coupled plates problem description.

Table 7.2 Geometric and material parameters of the two elastically coupled plates problem.

	PCB (bottom plate)	Component (top plate)
Deflection	$v(x, y, t)$	$u(x, y, t)$
Young's modulus	E_1	E_2
Poisson's ratio	ν_1	ν_2
Density	ρ_1	ρ_2
Damping coefficient	C_1	C_2
Plate flexural rigidity $D = \frac{Eh^3}{12(1-\nu^2)}$	D_1	D_2
Surface area	A_1	A_2
Thickness	h_1	h_2
Length and width	l_1, w_1	l_2, w_2
Number of springs (solders)	$N_s = 256$	
Spring constant	$K_s = \frac{2\pi r_p E_s}{h_s} \left[\frac{\tanh^{-1} \left(\sqrt{\frac{r_b - r_p}{r_b}} \right)}{\sqrt{(r_b - r_p)r_b}} + \frac{1}{r_p} \right]^{-1}$, see Fig. 7.3B	
Solder pad radius, solder ball radius, and standoff height	r_p, r_b , and h_s	
Solder modulus of elasticity	E_s	
Spring (solder) position in x and y	x_s, y_s	

PCB, Printed circuit board.

**FIGURE 7.5**

SDOF system: (A) free and (B) with base excitation.

This solution states that for this continuous system, the vibration problem can be reduced to the simple single-degree-of-freedom (SDOF) system, shown in Fig. 7.5A, with the governing equation:

$$M\ddot{z} + C\dot{z} + Kz = 0 \quad (7.2)$$

where M , C , and K are the mass, damping, and stiffness coefficients, respectively, and expressed as:

$$M = \iint_{A_1} \rho_1 h_1 V_i V_j dx dy + \iint_{A_2} \rho_2 h_2 U_i U_j dx dy \quad (7.3)$$

$$C = \iint_{A_1} C_1 V_i V_j dx dy + \iint_{A_2} C_2 U_i U_j dx dy \quad (7.4)$$

$$\begin{aligned} K = & \iint_{A_1} D_1 \{ (V_{ixx} V_{jxx} + V_{ixx} V_{jyy} + V_{iyy} V_{jxx} + V_{iyy} V_{jyy}) \\ & - (1 - \nu_1)(V_{ixx} V_{jyy} + V_{iyy} V_{jxx} - 2V_{ixy} V_{jxy}) \} dx dy \\ & + \iint_{A_2} D_2 (U_{ixx} U_{jxx} + U_{ixx} U_{jyy} + U_{iyy} U_{jxx} + U_{iyy} U_{jyy}) \\ & - (1 - \nu_2)(U_{ixx} U_{jyy} + U_{iyy} U_{jxx} - 2U_{ixy} U_{jxy}) \} dx dy \\ & + \sum_{s=1}^{N_s} K_s (U_i U_j - U_i V_j - V_i U_j + V_i V_j) \end{aligned} \quad (7.5)$$

While the solution only considers first mode, thus $i = j = 1$.

Therefore the first natural frequency, in rad/s, is:

$$\omega_o = \sqrt{\frac{K}{M}} \quad (7.6)$$

Considering first mode analysis, thus $N = 1$. Therefore the first mode shape of the PCB is:

$$V(x, y) = \sum_{n=1}^{N_t} c_{(2n-1)1} \sin\left(\frac{(2n-1)\pi x}{l_1}\right) + c_{(2n-1)2} \sin\left(\frac{(2n-1)\pi y}{w_1}\right) + c_{(2n-1)3} \sin\left(\frac{(2n-1)\pi x}{l_1}\right) \sin\left(\frac{(2n-1)\pi y}{w_1}\right) \quad (7.7)$$

where the coefficients matrix, $c_{(2n-1)m}$, is:

$$c_{(2n-1)m} = \begin{bmatrix} 0.5566 & 0.5566 & -0.0691 \\ -0.0199 & -0.0199 & -0.0141 \\ -0.0153 & -0.0153 & -0.0002 \\ -0.0054 & -0.0054 & 0.0001 \\ 0.0042 & 0.0042 & 0.0004 \\ -0.0032 & -0.0032 & 0.0002 \\ -0.0020 & -0.0020 & 0.0001 \end{bmatrix} \quad (7.8)$$

Also, the component (rigid top plate) mode shape is:

$$U(x, y) = 1 \quad (7.9)$$

It is important to mention that the subscripts in of the PCB and component mode shapes, considering one mode analysis, were dropped for simplicity. For further information on the procedure and the validation of the mode shape equations above, the reader is strongly encouraged to refer to reference [25].

7.5.2 Forced vibration: harmonic loading

After solving for the free vibration properties, that is, first natural frequency and mode shapes, the analytical solution could be taken now to the next level by solving for the forced vibration problem. This section discusses the details of the harmonic vibration problem.

Considering that the electronic system is subjected base excitation, Fig. 7.6A, and for harmonic base input $y(t) = Y e^{i\omega t}$, where Y is the input level, ω is the driving frequency and i is the complex number. Considering the SDOF system, Fig. 7.5B, the governing equation of this configuration is expressed as:

$$M\ddot{z} + C\dot{z} + Kz = C\dot{y} + Ky \quad (7.10)$$

As the input is harmonic, the displacement response solution is also harmonic as $z(t) = Z e^{i\omega t}$. Therefore the displacement amplitude Z is:

$$Z = \frac{Y(\omega_o^2 + 2i\zeta\omega\omega_o)}{\omega^2 - \omega_o^2 + 2i\zeta\omega\omega_o} \quad (7.11)$$

where $C/M = 2\zeta\omega_o$, and is ζ the damping ratio.

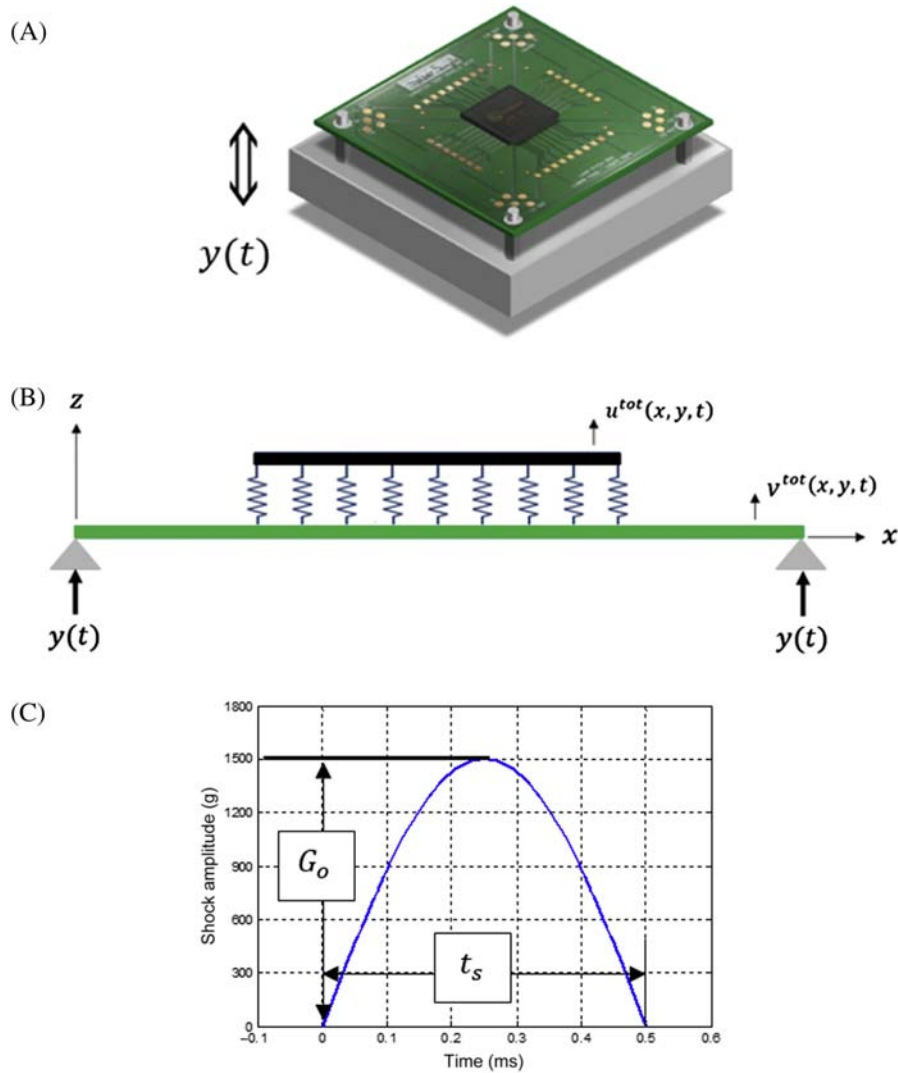


FIGURE 7.6

(A) Electronic package under base excitation, (b) two parallel plates under base excitation schematic (side view), and (C) half-sine wave shock pulse (1500 g input level and 0.5-ms duration).

By combining the above equation with Eq. (7.1) and by considering one mode analysis ($N = 1$), the PCB and component displacement responses are:

$$v(x, y, t) = V(x, y) \left(\frac{Y(\omega_o^2 + 2i\zeta\omega\omega_o)}{\omega^2 - \omega_o^2 + 2i\zeta\omega\omega_o} \right) e^{i\omega t} \quad (7.12)$$

$$u(x, y, t) = U(x, y) \left(\frac{Y(\omega_o^2 + 2i\zeta\omega\omega_o)}{\omega^2 - \omega_o^2 + 2i\zeta\omega\omega_o} \right) e^{i\omega t} \quad (7.13)$$

Or if considering acceleration base input $\ddot{y}(t) = -\omega^2 Y e^{i\omega t} = \ddot{Y} e^{i\omega t}$, where $\ddot{Y} = -\omega^2 Y$ is the acceleration input:

$$v(x, y, t) = V(x, y) \left(\frac{\ddot{Y}}{-\omega^2} \right) \left(\frac{\omega_o^2 + 2i\zeta\omega\omega_o}{\omega^2 - \omega_o^2 + 2i\zeta\omega\omega_o} \right) e^{i\omega t} \quad (7.14)$$

$$u(x, y, t) = U(x, y) \left(\frac{\ddot{Y}}{-\omega^2} \right) \left(\frac{\omega_o^2 + 2i\zeta\omega\omega_o}{\omega^2 - \omega_o^2 + 2i\zeta\omega\omega_o} \right) e^{i\omega t} \quad (7.15)$$

Now the solder axial deflection is simply:

$$\Delta(x, y, \omega) = [U(x, y) - V(x, y)] \left(\frac{\ddot{Y}}{-\omega^2} \right) \left(\frac{\omega_o^2 + 2i\zeta\omega\omega_o}{\omega^2 - \omega_o^2 + 2i\zeta\omega\omega_o} \right) \quad (7.16)$$

Considering the solder deflection at resonance ($\omega = \omega_o$), thus:

$$\Delta_r(x, y, \omega_o) = [U(x, y) - V(x, y)] \left(\frac{-\ddot{Y}(1 + 2i\zeta)}{2i\zeta\omega_o^2} \right) \quad (7.17)$$

At this stage of the analysis, the analytical solution was used to mathematically solve for the free vibration characteristics of the electronic assembly as well as for the harmonic base excitation problem. As will be shown next, this solution is expanded to solve for transient response of the electronic structure due to impact or shock loading.

7.5.3 Forced vibration: shock loading

Generally, if the electronic package was subjected to base excitation $y(t)$ through the mounting fixture, as depicted in Fig. 7.6A, then the PCB and component deflections (relative motions) of the two plates system Fig. 7.6B can be written as:

$$\begin{aligned} v(x, y, t) &= v^{tot}(x, y, t) - y(t) \\ u(x, y, t) &= u^{tot}(x, y, t) - y(t) \end{aligned} \quad (7.18)$$

where $v^{tot}(x, y, t)$ and $u^{tot}(x, y, t)$ are the PCB and component total (or absolute) motions, respectively. As proved by Gharaibeh, if this continuous system is subjected to base excitation, the vibration problem can be trustfully reduced to the problem of the simple SDOF system under base excitation $y(t)$. Considering the relative displacement $z(t)$, the governing equation of this system can be expressed as:

$$M\ddot{z}(t) + C\dot{z}(t) + Kz(t) = -M\ddot{y}(t) \quad (7.19)$$

where M , C , and K are the mass, damping, and stiffness coefficients, respectively, and given by Eqs. (7.24)–(7.27).

By dividing both sides of Eq. (7.19) by M , the equation of motion of the SDOF system becomes:

$$\ddot{z}(t) + 2\zeta\omega_o\dot{z}(t) + \omega_o^2z(t) = -\ddot{y}(t) \quad (7.20)$$

where $\zeta = C/2M\omega_o$ is the damping ratio.

Eq. (7.20) represents the equation of motion of a drop test where $\ddot{y}(t)$ is the input shock acceleration profile. Considering the half-sine wave input $\ddot{y}(t) = G_o\sin(\pi t/t_s)$ where G_o is the excitation amplitude (level) and t_s is the pulse duration, as in Fig. 7.6C. Therefore:

$$\ddot{z}(t) + 2\zeta\omega_o\dot{z}(t) + \omega_o^2z(t) = \begin{cases} -G_o\sin\left(\frac{\pi t}{t_s}\right) & 0 \leq t \leq t_s \\ 0 & t > t_s \end{cases} \quad (7.21)$$

For an underdamped system ($\zeta < 1$) and if the system is initially at rest, the solution of the ordinary differential equation (ODE) of Eq. (7.21) can be expressed as a linear combination of the homogeneous solution $z_h(t)$ and the particular solution $z_p(t)$ for $0 \leq t \leq t_s$, as:

$$z(t) = z_h(t) + z_p(t) \quad (7.22)$$

where the homogeneous solution is the solution of the homogeneous part of the ODE of Eq. (7.21):

$$\ddot{z}(t) + 2\zeta\omega_o\dot{z}(t) + \omega_o^2z(t) = 0 \quad (7.23)$$

Using the ODE solutions basics, thus:

$$z_h(t) = e^{-\zeta\omega_o t}(\alpha_1\cos(\omega_d t) + \beta_1\sin(\omega_d t)) \quad (7.24)$$

where $\omega_d = \omega_o\sqrt{1-\zeta^2}$ is the damped natural frequency of the system; α_1 and β_1 are constant coefficient to be determined from the initial conditions ($z(0) = \dot{z}(0) = 0$), remembering that the system is initially at rest.

Additionally, the particular solution can be obtained using the method of undetermined coefficients. Thus:

$$z_p(t) = \frac{G_o}{\alpha^2 + \beta^2} \left(\alpha \cos\left(\frac{\pi t}{t_s}\right) - \beta \sin\left(\frac{\pi t}{t_s}\right) \right) \quad (7.25)$$

where:

$$\begin{aligned} \alpha &= 2\zeta\omega_o \frac{\pi}{t_s} \\ \beta &= \omega_o^2 - \left(\frac{\pi}{t_s}\right)^2 \end{aligned} \quad (7.26)$$

Now, by combining the homogeneous solution of Eq. (7.24) and the particular solution of Eqs. (7.25)–(7.26) and applying the initial conditions, the α_1 and β_1 constants can be written as:

$$\begin{aligned}\alpha_1 &= -G_o \frac{\alpha}{\alpha^2 + \beta^2} \\ \beta_1 &= \frac{1}{\omega_d} \frac{G_o}{\alpha^2 + \beta^2} \left(\beta \frac{\pi}{t_s} - \zeta \omega_o \alpha \right)\end{aligned}\quad (7.27)$$

Considering the system after the shock ends ($t > t_s$), the equation of motion becomes:

$$\ddot{z}(t) + 2\zeta\omega_o\dot{z}(t) + \omega_o^2 z(t) = 0 \quad (7.28)$$

The solution of the above homogeneous ODE can be generally expressed as:

$$z(t) = e^{-\zeta\omega_o t} (\alpha_2 \cos(\omega_d t) + \beta_2 \sin(\omega_d t)) \quad (7.29)$$

where α_2 and β_2 are constant coefficients to be determined from $z(t = t_s)$ and $\dot{z}(t = t_s)$ from the solution of Eqs. (7.24)–(7.27). Thus the constants are:

$$\begin{aligned}\alpha_2 &= \frac{1}{e^{-\zeta\omega_o t_s}} \left(z(t_s) \cos(\omega_d t_s) - \frac{1}{\omega_d} \dot{z}(t_s) \sin(\omega_d t_s) \right) \\ \beta_2 &= \frac{1}{e^{-\zeta\omega_o t_s}} \left(z(t_s) \sin(\omega_d t_s) + \frac{1}{\omega_d} \dot{z}(t_s) \cos(\omega_d t_s) \right)\end{aligned}\quad (7.30)$$

Keeping in mind that $z(t_s)$ and $\dot{z}(t_s)$ are to be calculated from the solution of $0 \leq t \leq t_s$.

Now, by combining the $z(t)$ solutions with Eq. (7.1), and by considering one mode analysis ($N = 1$), the PCB and component time-dependent displacements are:

$$\begin{aligned}v(x, y, t) &= V(x, y)z(t) \\ u(x, y, t) &= U(x, y)z(t)\end{aligned}\quad (7.31)$$

At this point of the derivation process, the solder joint axial deflection, which is generally the difference between the component and PCB deflections, can be obtained as:

$$\Delta(x, y, t) = [U(x, y) - V(x, y)]z(t) \quad (7.32)$$

In summary, the above equation can be used to calculate the axial deflections of any solder interconnect available in the electronic assembly as a function of time.

7.6 Results and discussions

7.6.1 Free vibration: natural frequencies and mode shapes

The results of the current analytical solution were compared with FEA in terms of the first natural frequency and mode shape. For mode shape correlation, the modal assurance criterion (MAC) number between analytical and FEA mode shapes was calculated as:

$$[\text{MAC}(\mathbf{V}_j, \hat{\mathbf{V}}_k)] = \frac{|\mathbf{V}_j^T \hat{\mathbf{V}}_k|^2}{\mathbf{V}_j^T \mathbf{V}_j \hat{\mathbf{V}}_k^T \hat{\mathbf{V}}_k} \quad (7.33)$$

where $\mathbf{V}_j$ is the analytical mode shape vector from $j = 1$ to $j = \text{no. of mode shapes}$ and $\hat{\mathbf{V}}_k$ is the finite element mode shape vector from $k = 1$ to $k = \text{no. of mode shapes}$.

The natural frequency and mode shape correlation results are listed in Table 7.3 and mode shapes are plotted in Fig. 7.7. The correlation results showed good agreement between the analytical, FEA, and experimental solutions in terms of natural frequencies and mode shapes (MAC) for the first vibratory mode.

Additionally, it is important to mention that the modal damping ratio of the first mode is obtained as 0.55% as extracted from the modal analysis experiment. This value was used in further analysis as will be shown in subsequent sections of this chapter.

7.6.2 Forced vibration: harmonic loading

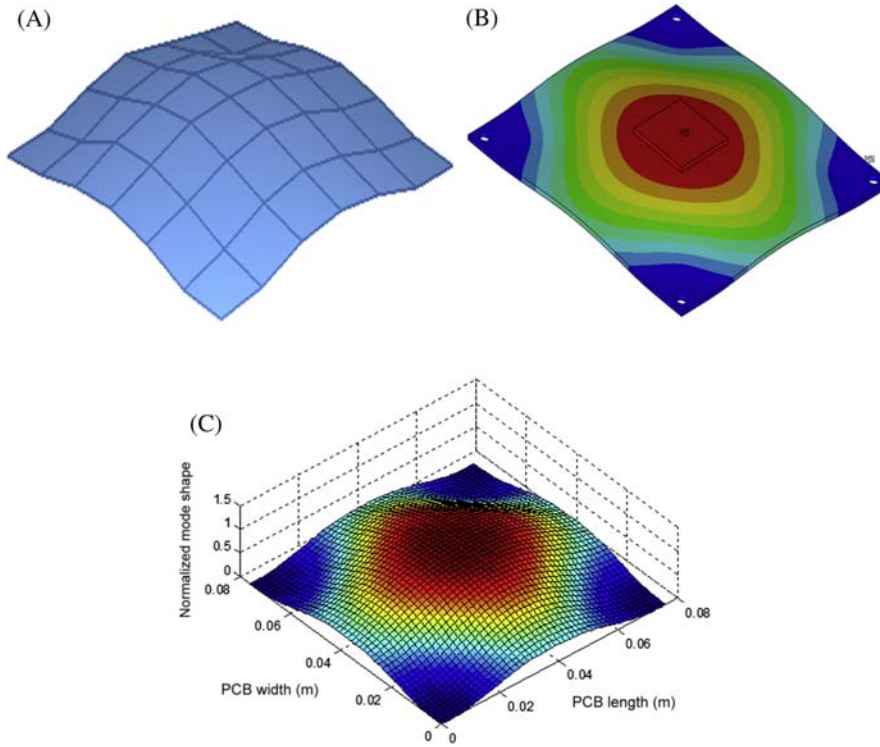
7.6.2.1 Corner solder joint deflection validation

As the outermost solder is the most damaged solder in the assembly, the analysis in this work is limited for this critical interconnect. The results of Eqs. (7.16) and (7.17) were compared with FEA models data for the outermost solder joint at 1 g base excitation and damping ratio of 0.55%. In FEA, the solder deflection was calculated by subtracting PCB nodal response from the component nodal response. The results of this comparison are shown in Fig. 7.8. Also, the solder resonance deflections are listed in Table 7.4.

Table 7.3 Free vibration correlation results: first natural frequency and mode shape.

Natural frequency (Hz)			MAC number	
Analytical	FEA	Experiment	Analytical/FEA	Analytical/experiment
579	576	566	0.99	0.97

FEA, Finite element analysis.

**FIGURE 7.7**

First mode shapes of the test vehicle from (A) experiment, (B) finite element analysis model, and (C) analytical solution.

The comparison results showed that the analytical solution precisely estimates the maximum deflection of the outermost joint, that is, at resonance, with only 10% approximate error.

In summary, the present analytical solution is faithfully able to simulate the assembly dynamic characteristics and accurately enough estimates solder deflections due to harmonic base vibration.

7.6.2.2 Critical solder joint stress analysis

The so validated analytical solution was used to study PCB and solder joint geometric and material configuration effect on solder stresses due to harmonic vibration. For this study, one parameter is changed at a time with respect to all other parameters. Also, the solder stresses for each case were computed at resonance frequency for a 2 g harmonic base vibration input with 0.55% damping ratio. The solder axial stresses (σ_s) were analytically calculated through the simple Hooke's law equation ($\sigma_s = E_s \epsilon_s$), where solder axial strain (ϵ_s) was obtained from the

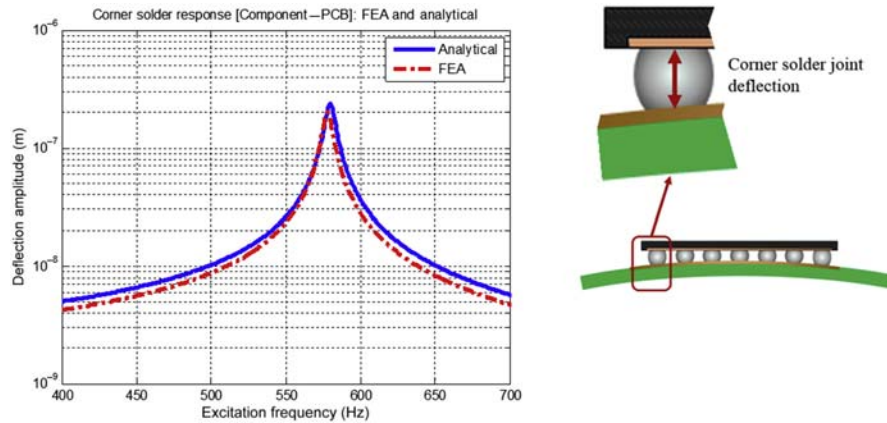


FIGURE 7.8 Outermost solder joint axial deflection response. *FEA*, Finite element analysis; *PCB*, printed circuit board.

Table 7.4 Outermost solder joint deflections at resonant frequency due to harmonic base vibration.

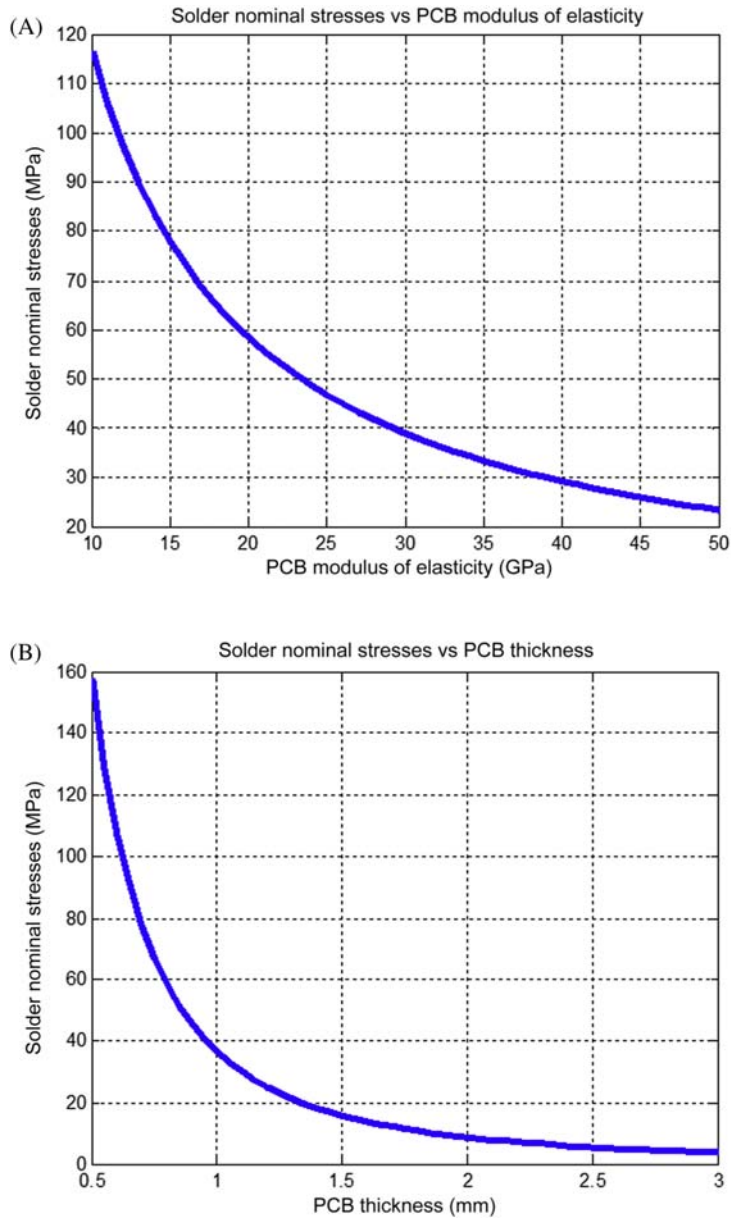
	Analytical	FEA	%Error
Solder axial deflection (μm)	0.221	0.201	9.9

FEA, Finite element analysis.

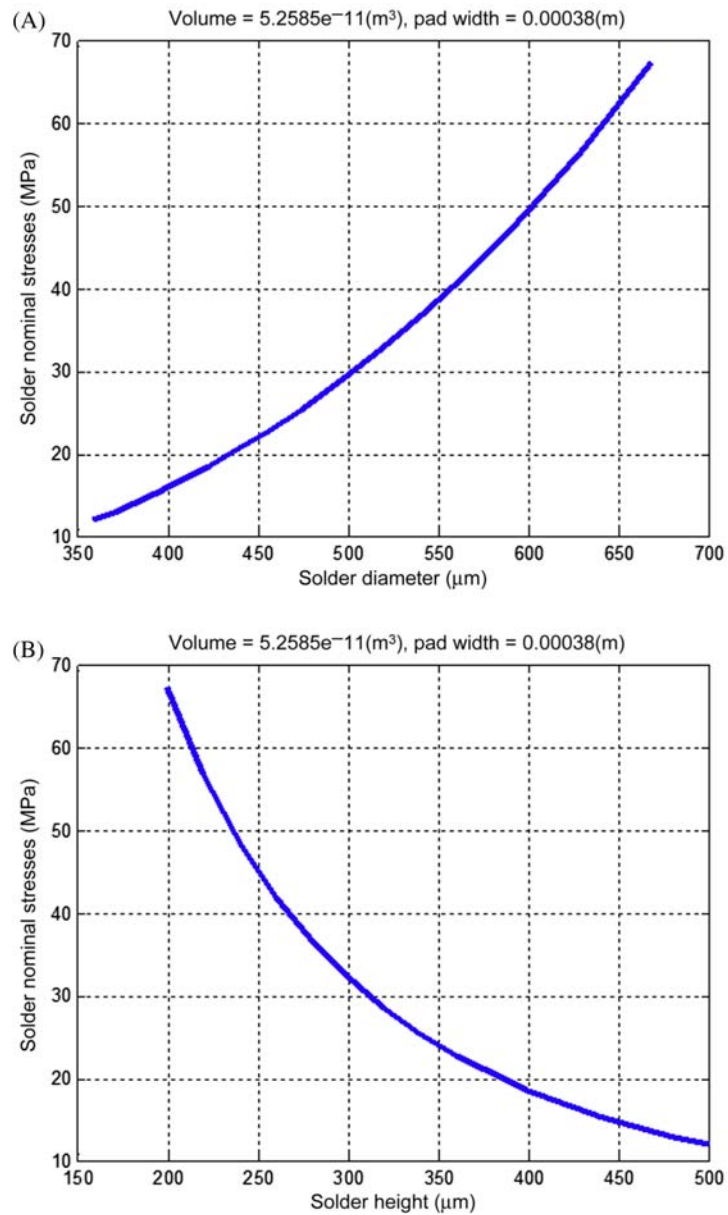
definition that the solder strain is the ratio between the solder change in length, i.e. deflection, and the solder original standoff height.

7.6.2.2.1 Printed circuit board stiffness: thickness and modulus of elasticity

The first parameter studied was the stiffness of the PCB. Specifically, PCB elastic modulus and thickness. First, a wide range of board modulus of elasticity was considered. As shown in Fig. 7.9A, the stiffer PCB can significantly reduce solder axial stresses. The second factor is PCB thickness. The results depicted in Fig. 7.9B showed that doubling the board thickness from 1 to 2 mm can reflect in more than four times solder stress reduction. To this instance, the stiffer PCB structure will cause less board bending and hence lower solder deformations and stresses. Therefore stiffer board can significantly improve electronic assembly fatigue performance due to harmonic vibration loading.

**FIGURE 7.9**

Effects of PCB stiffness: (A) elastic modulus and (B) thickness on solder stresses. *PCB*, Printed circuit board.

**FIGURE 7.10**

Effect of solder ball (A) diameter and (B) height on solder stresses.

7.6.2.2.2 Solder joint geometry: standoff height and diameter

It is strongly believed that solder joint shape has a significant effect on solder stresses. In this study, the solder diameter and height were changed while keeping solder volume as well as pad size constant. Then, solder joint nominal stresses versus solder diameter and height were plotted in Fig. 7.10A and B, respectively. From the figure, it appears to be that the fatter joint (joint with larger diameter) will have higher stresses. In contradiction, the taller joint can have lower stresses. In conclusion, a solder joint with smaller diameter and larger standoff height will exhibit lower stresses and hence better fatigue life under harmonic vibration loading.

7.6.3 Forced vibration: impact loading

7.6.3.1 Corner solder joint deflection

In this correlation step, Eq. (7.32) was used to obtain the time-history axial deflection of the outermost solder interconnect due to JEDEC B-condition of JESD B111 standard half-sine wave acceleration of 1500 g input level and 0.5-ms pulse duration using $\zeta = 0.55\%$. This damping ratio value was extracted from the modal analysis test. The analytically derived solder response was compared with that of FEA simulations due to the same lading damping conditions, as presented in Fig. 7.11. This comparison showed that the solder axial deflections of the analytical solution are in well agreement with FEA solder deflections. Additionally, the relative approximate error between two solutions of the maximum solder deflection is about 16%, as listed in Table 7.5.

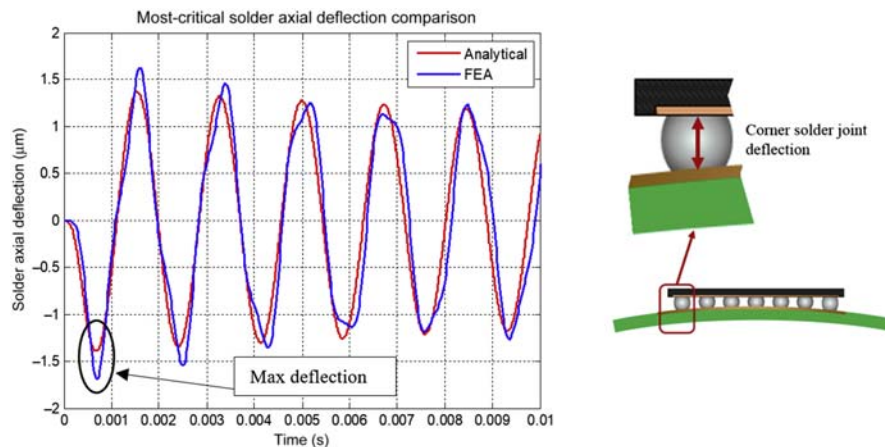


FIGURE 7.11

Axial deflection of the outermost corner solder joint due to shock loading. *FEA*, Finite element analysis.

Table 7.5 Maximum axial deflections of the outermost solder interconnect due to shock loading.

	Analytical	FEA	%Error
Solder axial deflection (μm)	– 1.393	– 1.686	15.52

FEA, *Finite element analysis*.

7.6.3.2 Critical solder joint stress analysis

The presently correlated analytical solution was further used to test the various geometric and material properties of the PCB and solder interconnect effect on the electronic system fatigue behavior in shock loading environments. This was achieved based on the evaluation of outermost corner solder axial stress value. Again, the solder axial stresses (σ_s) were analytically calculated through the simple Hooke's law equation ($\sigma_s = E_s \epsilon_s$), where solder axial strain (ϵ_s) was obtained from the definition that the solder strain is the ratio between the solder change in length, that is, deflection, and the solder original standoff height. Unless otherwise stated, only absolute values of the outermost solder maximum axial deflections, strains, and stresses were in considered in the present analysis.

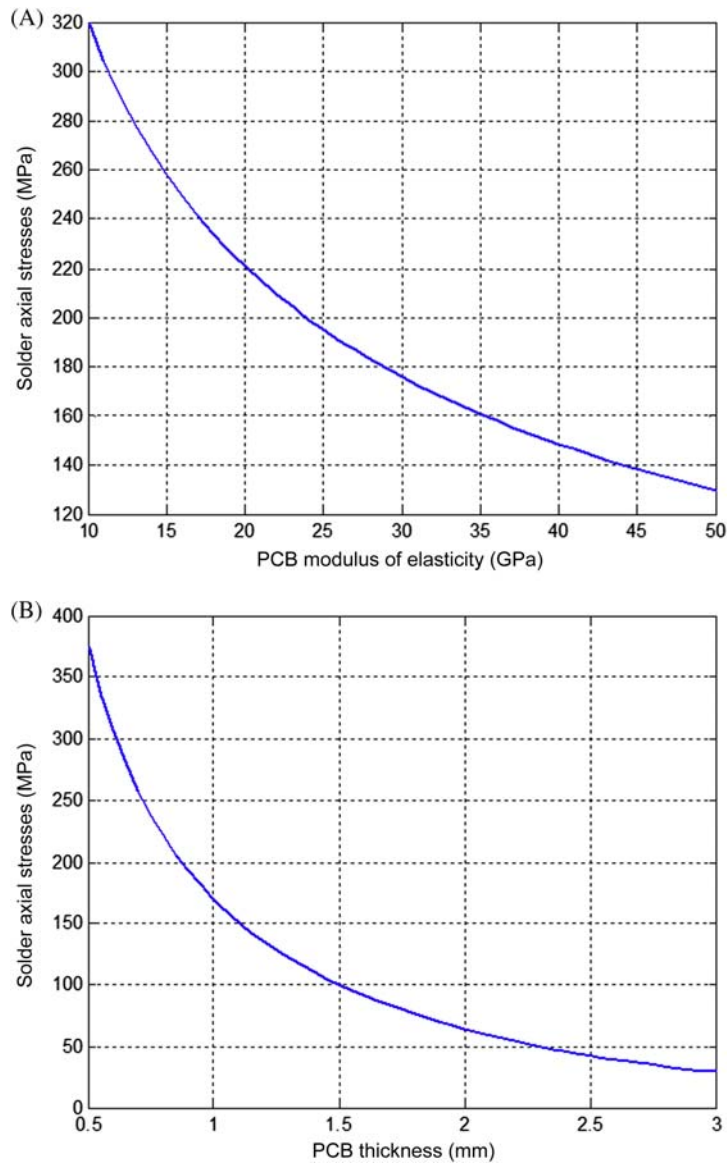
In this investigation, one geometric or material parameter was changed while all other parameters remain unchanged. The loading condition considered here is a half-sine wave impact profile of 1500 g amplitude and 0.5-ms duration (JEDEC B-condition) and 0.55% damping ratio value, unless otherwise stated.

7.6.3.2.1 Printed circuit board stiffness: thickness and modulus of elasticity

In an electronic device, the stiffness of the PCB plays an important role in the fatigue life, that is, the reliability of the product. In this paper, the PCB stiffness effect was tested based on the thickness and the modulus of elasticity. The solder axial stresses were analytically computed for a wide range of thickness and modulus, as depicted in Fig. 7.12. The results showed that the stiffer PCB, that is, thicker and of higher elastic modulus value, could significantly reduce solder stresses, and therefore enhance the fatigue performance of electronics in mechanical shock environments.

7.6.3.2.2 Solder joint geometry: standoff height and diameter

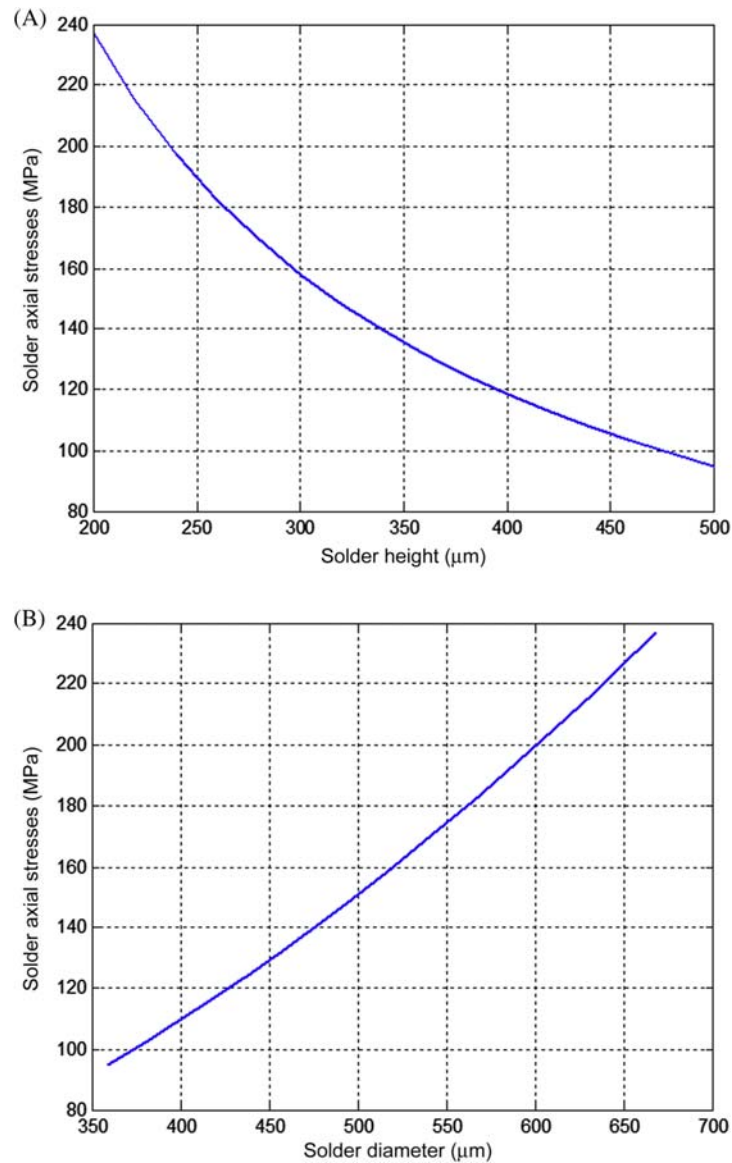
Researchers always believed that the solder geometry could highly affect the solder reliability behavior. Therefore the present analysis used the so-developed analytical solution to evaluate the solder geometry, that is, standoff height and diameter, effect on the axial stresses. The results are in Fig. 7.13. In this investigation, the solder height and diameter were varied while the solder volume kept unchanged. The results say that the thinner and taller solders might possess lower stresses, and hence, better reliability and fatigue life in impact loadings.

**FIGURE 7.12**

PCB stiffness effect on solder axial stresses: (A) elastic modulus and (B) thickness.

7.6.3.3 Solder stress response spectrum

In shock analysis, it is often convenient to characterize and plot the maximum absolute value of the system's time response versus the undamped natural

**FIGURE 7.13**

Solder joint geometry effect on solder axial stresses: (A) height and (B) diameter.

frequency (or the undamped natural period) of the system. This characterization is called shock response spectrum (SRS). In this paper, the SRS of the most-critical solder interconnect axial stress is obtained. To achieve this, for an SDOF system,

we substitute $\omega_o = 2\pi/\tau_o$ and $\omega_d = (2\pi/\tau_o)\sqrt{1-\zeta^2}$, where τ_o is the undamped natural period of the SDOF system, in Eqs. (7.24)–(7.27) and Eqs. (7.30)–(7.31). Therefore for $0 \leq t \leq t_s$, the homogenous and particular solutions, respectively, are

$$z_h(t) = e^{-\zeta\omega_o t} \left(\alpha_1 \cos\left(2\pi\sqrt{1-\zeta^2}\frac{t}{\tau_o}\right) + \beta_1 \sin\left(2\pi\sqrt{1-\zeta^2}\frac{t}{\tau_o}\right) \right) \quad (7.34)$$

and

$$z_p(t) = \frac{G_o}{\alpha^2 + \beta^2} \left(\alpha \cos\left(\frac{\pi t}{t_s}\right) - \beta \sin\left(\frac{\pi t}{t_s}\right) \right) \quad (7.35)$$

where

$$\begin{aligned} \alpha_1 &= -G_o \frac{\alpha}{\alpha^2 + \beta^2} \\ \beta_1 &= \frac{\tau_o}{2\pi\sqrt{1-\zeta^2}} \frac{G_o}{\alpha^2 + \beta^2} \left(\beta \frac{\pi}{t_s} - \frac{2\pi}{\tau_o} \zeta \alpha \right) \end{aligned} \quad (7.36)$$

and

$$\begin{aligned} \alpha &= \frac{(2\pi)^2 \zeta}{\tau_o t_s} \\ \beta &= \left(\frac{2\pi}{\tau_o} \right)^2 - \left(\frac{\pi}{t_s} \right)^2 \end{aligned} \quad (7.37)$$

Also, after the shock ends ($t > t_s$), the solution becomes:

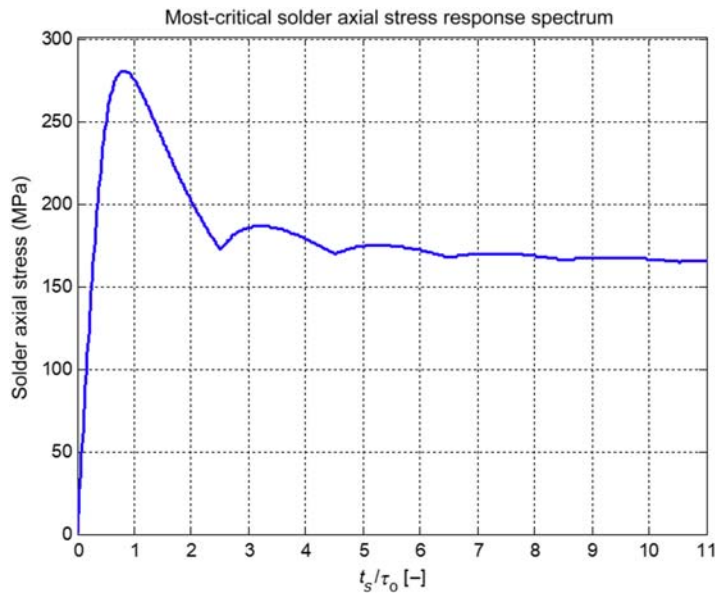
$$z(t) = e^{-\frac{\zeta 2\pi t}{\tau_o}} \left(\alpha_2 \cos\left(2\pi\sqrt{1-\zeta^2}\frac{t}{\tau_o}\right) + \beta_2 \sin\left(2\pi\sqrt{1-\zeta^2}\frac{t}{\tau_o}\right) \right) \quad (7.38)$$

where:

$$\begin{aligned} \alpha_2 &= \frac{1}{e^{-\frac{\zeta 2\pi t_s}{\tau_o}}} \left(z(t_s) \cos\left(2\pi\sqrt{1-\zeta^2}\frac{t_s}{\tau_o}\right) - \frac{\tau_o}{2\pi\sqrt{1-\zeta^2}} \dot{z}(t_s) \sin\left(2\pi\sqrt{1-\zeta^2}\frac{t_s}{\tau_o}\right) \right) \\ \beta_2 &= \frac{1}{e^{-\frac{\zeta 2\pi t_s}{\tau_o}}} \left(z(t_s) \sin\left(2\pi\sqrt{1-\zeta^2}\frac{t_s}{\tau_o}\right) + \frac{\tau_o}{2\pi\sqrt{1-\zeta^2}} \dot{z}(t_s) \cos\left(2\pi\sqrt{1-\zeta^2}\frac{t_s}{\tau_o}\right) \right) \end{aligned} \quad (7.39)$$

Again, $z(t_s)$ and $\dot{z}(t_s)$ are to be determined from the solution for $0 \leq t \leq t_s$ of Eqs. (7.34)–(7.37).

Thus by combining the above equations with Eqs. (7.31) and (7.32) as well as Hooke's law discussed previously, the solder joint stress can be determined accordingly. As now the solder ball axial stresses are expressed in terms of t_s/τ_o

**FIGURE 7.14**

Most-critical solder joint axial stress response spectrum.

(shock duration to the undamped natural period ratio), the maximum absolute stress value of the most-critical solder interconnect ($\sigma_{s,max}$) can be calculated for several t_s/τ_o ratios as shown in Fig. 7.14. From this figure, it can be observed that the maximum value of $\sigma_{s,max} \cong 280$ MPa occurred at $t_s/\tau_o \cong 0.75$. It is highly preferable to perform the drop test at such condition to ensure maximum damage case scenario, if possible. Another observation can be made here is that it is very important, in a drop reliability test, to precisely control the shock duration throughout the consecutive drops in the test. Otherwise, erroneous fatigue (or reliability) data would be generated.

7.7 Conclusion

This chapter presented an analytical solution for the problem of electronic assemblies subjected to shock and vibration loadings. A previously developed solution was adopted. The results of this analytical solution were in great match with experiments and finite element simulations in terms of free and forced vibration properties. Using this approach, a thorough investigation of the fatigue performance of electronic assemblies under shock and vibration loadings was introduced. This reliability assessment study showed that stiffer PCB structures as well as taller and thinner ball grid array solders could reasonably improve the

reliability behavior, that is, fatigue life, of electronic devices subjected to such mechanical loadings. Such findings are considered to be very useful in the design of electronic products in mechanical shock and vibration environments.

Nomenclature

A_1, A_2	surface area of PCB, component
C_1, C_2	damping coefficient of PCB, component
D_1, D_2	flexural rigidity of PCB, component $D = \frac{Eh^3}{12(1-\nu^2)}$
E_1, E_2, E_s	elastic modulus of PCB, component and solder joint
G_o	shock amplitude
h_1, h_2	thickness of PCB, component
K_s	solder axial stiffness
N	number of mode shapes used in Ritz solutions
N_t	number of terms used in PCB first mode shape
t_s	shock duration
M, K, C	system mass, stiffness, and damping coefficient
r_p, r_b, h	pad width, solder radius, and solder height
l, w	PCB length and width
$u(x, y, t), v(x, y, t)$	deflection of component, PCB
$u^{tot}(x, y, t), v^{tot}(x, y, t)$	total deflection of component, PCB
$U(x, y), V(x, y)$	mode shape of component, PCB
$y(t), \dot{y}(t), \ddot{y}(t)$	base displacement, velocity and acceleration
$y_s(x)$	solder joint profile function
$z_h(t), z_p(t)$	homogenous and particular solutions of the equation of motion
V_{xx}, V_{yy}, V_{xy}	partial derivatives: $\frac{\partial^2 v}{\partial x^2}, \frac{\partial^2 v}{\partial y^2}, \frac{\partial^2 v}{\partial x \partial y}$
U_{xx}, U_{yy}, U_{xy}	partial derivatives: $\frac{\partial^2 u}{\partial x^2}, \frac{\partial^2 u}{\partial y^2}, \frac{\partial^2 u}{\partial x \partial y}$
ν_1, ν_2	Poisson's ratio of PCB, component
ρ_1, ρ_2	density of PCB, component
Δ, Δ_r	most-corner solder joint deflection response, resonant
$z_i(t)$	time-dependent function
ζ	first mode damping ratio
τ_o	undamped natural period time
σ_s, ϵ_s	outermost solder axial stress, strain
$\omega, \omega_o, \omega_d$	angular driving frequency, system angular first natural frequency, system angular first damped natural frequency

References

- [1] E. Suhir, R. Burke, Dynamic response of a rectangular plate to a shock load, with application to portable electronic products, IEEE Trans. Compon, Packag., Manuf. Technol.: Part B 17 (3) (1994) 449–460.
- [2] J. Xi, X. Zhai, J. Wang, D. Yang, M. Ru, F. Xiao, et al., Reliability assessment of wafer level packages with novel FeNi under bump metallization, J. Electron. Packag. 137 (3) (2015) 031016.

- [3] S. Kallolimath, J.J. Zhou, Optimal shock pulse in a drop test simulation of standardized board for uniform shock response, *J. Electron. Packag.* 138 (4) (2016) 041001.
- [4] S. Park, C. Shah, J. Kwak, C. Jang, J. Pitarresi, T. Park, S. Jang, Transient dynamic simulation and full-field test validation for a slim-PCB of mobile phone under drop/impact, in: *Electronic Components and Technology Conference, 2007. ECTC'07. Proceedings. 57th, IEEE, May 2007*, pp. 914–923.
- [5] J. Pitarresi, B. Roggeman, S. Chaparala, P. Geng, Mechanical shock testing and modeling of PC motherboards, in: *54th Electronic Components and Technology Conference, 2004*.
- [6] M. Gharaibeh, Q. Su, J. Pitarresi, M. Anselm, Board level drop test: comparison of two ANSYS modeling approaches and correlation with testing, in: *Univ. Instrum. Adv. Res. Electron. Assemblies (AREA) Consortium, Conklin, NY, USA, 2013*.
- [7] M. Gharaibeh, J. Pitarresi, M. Anselm, Strain correlation: finite element modeling and experimental data, in: *Univ. Instrum. Adv. Res. Electron. Assemblies (AREA) Consortium, Conklin, NY, USA, 2013*.
- [8] M. Gharaibeh, J. Pitarresi, M. Anselm, Modeling and characterization for vibration, in: *Univ. Instrum. Adv. Res. Electron. Assemblies (AREA) Consortium, Conklin, NY, USA, 2013*.
- [9] Q. Su, J. Pitarresi, M. Gharaibeh, A. Stewart, G. Joshi, M. Anselm, Accelerated vibration reliability testing of electronic assemblies using sine dwell with resonance tracking, in: *Electronic Components and Technology Conference (ECTC), 2014 IEEE 64th, IEEE, May 2014*, pp. 119–125.
- [10] S. Joshi, B. Arfaei, A. Singh, M. Gharaibeh, M. Obaidat, A. Alazzam, et al., LGAs vs. BGAs—lower profile and better reliability?, in: *Proc. SMTAI, October 2012*, pp. 47–57.
- [11] M.A. Gharaibeh, Reliability analysis of vibrating electronic assemblies using analytical solutions and response surface methodology, *Microelectron. Reliab.* 84 (2018) 238–247.
- [12] M.A. Gharaibeh, Reliability assessment of electronic assemblies under vibration by statistical factorial analysis approach, *Soldering Surf. Mount Technol.* 30 (3) (2018) 171–181.
- [13] M.A. Gharaibeh, Finite element model updating of board-level electronic packages by factorial analysis and modal measurements, *Microelectron. Int.* 35 (2) (2018) 74–84.
- [14] M.H. Obaidat, O.T. Al Meanazel, M.A. Gharaibeh, H.A. Almomani, Pad cratering: reliability of assembly level and joint level, *Jordan J. Mech. Ind. Eng.* 10 (4) (2016) 271–277.
- [15] E.H. Wong, S.K.W. Seah, V.P.W. Shim, A review of board level solder joints for mobile applications, *Microelectron. Reliab.* 48 (11) (2008) 1747–1758.
- [16] E.H. Wong, K.M. Lim, N. Lee, S. Seah, C. Hoe, J. Wang, Drop impact test-mechanics & physics of failure, in: *4th Electronics Packaging Technology Conference, 2002*.
- [17] E.H. Wong, C.K. Wong, Approximate solutions for the stresses in the solder joints of a printed circuit board subjected to mechanical bending, *Int. J. Mech. Sci.* 51 (2) (2009) 152–158.
- [18] E.H. Wong, Y.W. Mai, S.K. Seah, K.M. Lim, T.B. Lim, Analytical solutions for interconnect stress in board level drop impact, in: *Advanced Packaging, IEEE Transactions on, 2007*.

- [19] E.H. Wong, Y.W. Mai, S.K. Seah, K.M. Lim, T.B. Lim, Analytical solutions for interconnect stress in board level drop impact, in: 56th Electronic Components and Technology Conference, 2006.
- [20] E. Suhir, On a paradoxical phenomenon related to beams on elastic foundation: could external compliant leads reduce the strength of a surface-mounted device? *J. Appl. Mech.* 55 (4) (1988) 818–821.
- [21] P.A. Engle, Structure analysis for circuit card system subject to bending, *J. Electron. Packag.* (1)(1990) 2–10.
- [22] J.M. Pitarresi, J. Ceurter, Elasticly coupled beams loaded by a point force, in: 13th Engineering Mechanics Conference, Baltimore, MD, June 1999, pp. 13–16.
- [23] M.A. Gharaibeh, D. Liu, J.M. Pitarresi, A pair of partially coupled beams subjected to concentrated force, *IEEE Trans. Compon., Packag. Manuf. Technol.* 7 (8) (2017) 1293–1304.
- [24] A. Perkins, S.K. Sitaraman, Vibration-induced solder joint failure of a ceramic column grid array (CCGA) package, in: 54th Electronic Components and Technology Conference, 2004.
- [25] M.A. Gharaibeh, Q.T. Su, J.M. Pitarresi, Analytical solution for electronic assemblies under vibration, *J. Electron. Packag.* 138 (1) (2016) 011003.
- [26] Spectral Dynamics, STAR Modal 7.0, Spectral Dynamics, Troy, MI. <<http://www.spectraldynamics.com/index.php/products/star/star-modal>>.
- [27] ANSYS, ANSYS 17.0, ANSYS, Inc., Canonsburg, PA. <<http://www.ansys.com>>.

This page intentionally left blank

Stress analysis of stretchable conductive polymer for electronics circuit application

N.A. Aziz¹, A.A. Saad¹, Z. Ahmad¹, S. Zulfiqar¹, F.C. Ani² and Z. Samsudin²

¹*Universiti Sains Malaysia, Engineering Campus, Nibong Tebal, Malaysia*

²*Jabil Circuit Sdn Bhd, Bayan Lepas Industrial Park, Bayan Lepas, Malaysia*

8.1 Introduction

The stretchable electronic circuit (SEC) is a technology which has been improved from a rigid printed circuit board to be bendable, twistable, and stretchable [1,2]. The flexible, soft, and stretchable electronics devices are becoming very common today and use in various applications of health care, energy, and military purposes. The advantages of SEC are flexible for human body application and improve the reliability of the devices subjected to strain [3–5]. Its application is mostly for a sensor like strain sensor, robotic skins, and wearable displays [6–8]. Several techniques were used to pattern the conductors on an SEC, but nowadays, digital printing techniques are commonly used [9]. The stretchable devices are generally also achieved by engineered shapes and elastic substrates. These substrates are intrinsically stretchable and are used to develop the circuit boards due to their unique ability to join with flexible materials and curved surfaces. A lot of methods have been described by various scientists to manufacture a stretchable electronics system, but the most common method is the use of intrinsically stretchable materials.

Stretchable electronics circuit mainly consists of flexible or stretchable substrates [i.e., polydimethylsiloxane (PDMS) and Walopur thermoplastic polyurethane (TPU)], flexible or stretchable conductive ink as a circuit like Ag, Cu, Ag-PDMS, and electronic components (i.e., LED, transistor, resistor, capacitor, and integrated circuit). Elastomers like natural rubber, styrene butadiene rubber, ethylene-propylene-diene monomer, polyurethane, TPU, and predominant PDMS are often used as soft substrates in many electronic devices and reversibly suffered high deformations greater than 200% strain [10]. However, this method shows low electrical mobility and high electrical resistivity of the devices.

Researchers have shown interests in the development of the stretchable circuit and substrate since both elements are the key aspect to control the stretchability of the SEC. The other methods of manufacturing SEC are wavy structural

configuration [11], fractal design [12,13] and mesh, interconnected island [14–16], origami or kirigami structures. These methods are used to improve the tensile capacity of intrinsically stretchable conductors and allow the use of conductive bulk metals in stretchable electronics [17].

The stretchability is controlled by changing two parameters that are stretchable material and stretchable design used for both the substrate and conductor [1,7]. These materials are widely used to print the integrated circuit, and previous research shows interest on its conductivity only which said still can be improved [18]. The stretchability controlled by design shows the stretchable conductor were studied in terms of (1) geometry by controlling the width and thickness and (2) different printing shape of the circuit by introducing horseshoe shape as the best design to reduce the plastic strain. Besides, the substrate design was studied by Ref. [19] that has introduced (3) sandwich structure of substrate material that covers the whole part of the conductor to extend the failure limits of the conductor. However, the previous study shows a lack of information regarding the stretchability control by design for the materials related to our study, which are silver nanoparticles and nanocomposites. The novelty of the research is on the performance of the new formulated silver nanoparticles in combination with PDMS rubber known as Ag-PDMS conductive ink that is still under improvement as a new material to be developed for the stretchable circuit application. The most challenging part in the research is on the reliability of the experimental result in order to characterize the material properties using tensile testing for the new material that is made up from liquid and used as a thin film in an application. Besides, the reliability of the simulation result is also challenging since the experimental technique used to validate the result is crucial in terms of the interpretation of the obtained data.

Fundamental studies were done in this chapter to know the limitation of the SEC in application as a basic circuit geometry and a thermal sensor circuit using both experimental and finite element analysis (FEA). The research is limit to the analysis for Ag-PDMS conductive ink as the conductor material. The preliminary study is on controlling the stretchability by design for Ag-PDMS conductive ink as the circuit material in application as three simple shapes (i.e., rectangular, zig-zag, and horseshoe) and as a complex thermal sensor circuit. Several material models in combination with universal tensile test have been selected to define the material properties of the Ag-PDMS conductive ink. The result is presented in terms of maximum equivalent stress and percentage of equivalent plastic strain for the three simple shapes and the application as a thermal sensor circuit.

8.2 Experimental procedure

8.2.1 Sample preparation

The sample was prepared for universal tensile testing in the form of a small straight specimen. The stress–strain behavior of a material can also be evaluated

by using tensile testing. The obtained stress–strain data were then analyzed to obtain the material constant for the Neo-Hookean model that is used as the input parameter for FEA. There are two types of samples involved in this study, namely substrate and conductive ink. The substrate is made up of silicone rubber known as PDMS as the major substance, and conductive ink is prepared from silver (Ag) flakes as the primer substance with the addition of PDMS for the binder known as Ag-PDMS conductive ink.

PDMS substrate is prepared according to Norhidayah et al. [20]. Briefly, the samples were prepared by mixing PMDS liquid with fume silica solution. A catalyst dibutyltin dilaurate was then added to boost the reaction. The bubbles produced after the reaction were allowed to pass out and then the mixture was poured into the mold. Finally, the substrate was solidified at room temperature and removed after 24 hours. Ag-PDMS conductive ink can also be prepared in a similar way as that of PDMS substrate. The only difference is that Ag flakes are used without the addition of toluene instead of fume silica.

8.2.2 Printing process of circuits

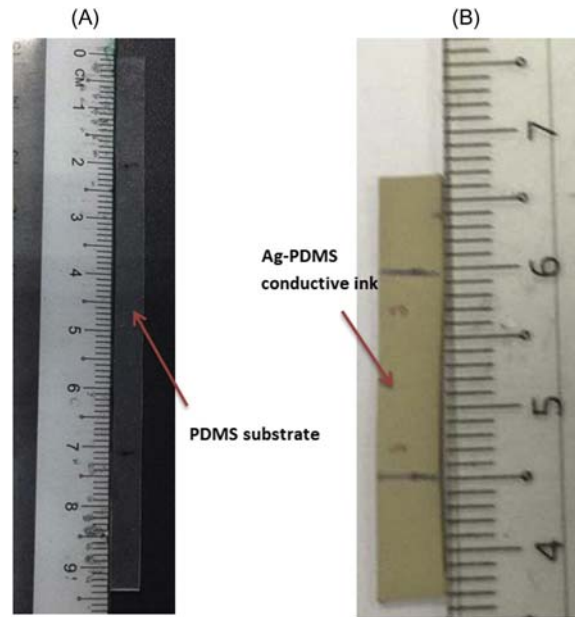
The printing process of a circuit can be done in three main steps:

1. screen mesh fabrication,
2. conductive ink preparation, and
3. printing and curing process.

The screen mesh fabrication is based on the desired circuit pattern. It controls the penetration of the ink and ensures that a uniform thickness of the ink has printed on the substrate. The circuit pattern is then modeled where the top view sets as a reference to estimate the dimensions of the circuit. The conductive ink needs to be prepared before the printing process, and finally the printing and curing process takes place according to the required specification to form a functional circuit.

8.2.3 Universal tensile testing

The five specimens of PDMS substrate and Ag-PDMS conductive ink were cut into strips of $(50 \times 5 \times 0.5) \text{ mm}^3$ size (Fig. 8.1A) and $(15 \times 5 \times 0.5) \text{ mm}^3$ size (Fig. 8.1B), respectively. The American Standard Testing Machine D412 (ASTM D412) was used with some modifications. The testing was conducted under 5-kN load cell and cross-head speed of 10 mm/min at room temperature. The engineering stress–strain data were directly obtained from experimental results. Table 8.1 shows the testing parameters for PDMS substrate and Ag-PDMS conductive ink.

**FIGURE 8.1**

Tensile testing samples for (A) PDMS substrate and (B) Ag-PDMS conductive ink.

Table 8.1 Testing parameters for the universal tensile test of substrate and conductive ink.

Parameter	Substrate	Conductive ink
Specimen type	Straight strip	Straight strip
Specimen size, $L \times W \times t$ (mm ³)	$50 \times 5 \times 0.5$	$15 \times 5 \times 0.5$
Loading rate (mm/min)	10	10
Instron load cell (kN)	5	5

8.3 Stress–strain analysis of substrate and conductive ink

The material properties for PDMS substrate and Ag-PDMS conductive ink were represented by the Neo-Hookean model and multilinear plastic model, respectively. Neo-Hookean model is the simplest model for fully elastic materials (non-linear elastic) and requires the material constant, $C1$ (MPa) while Multilinear plastic model is only suitable for fully plastic materials (nonlinear plastic) and needs Young's modulus, stress, and plastic strain data.

8.3.1 Neo-Hookean model

The curve fitting technique was used to calculate the material constant C_1 from the engineering stress–strain curve developed by FEA software. From the engineering stress–strain data, the value of shear modulus (G) is also obtained as 0.16 MPa. The other method to calculate C_1 is the reverse engineering method that starts with the Neo-Hookean model engineering stress, σ_{eng} (MPa) formula as presented in Eq. (8.1) [21]. Eqs. (8.2) and (8.3) are used to calculate the values of stretch ratio and shear modulus, respectively.

$$\sigma_{eng} = 2C_1 \left[\lambda - \frac{1}{\lambda^2} \right] \quad (8.1)$$

$$\lambda = \varepsilon_{eng} + 1 \quad (8.2)$$

$$G = 2C_1 \quad (8.3)$$

where C_1 is the Neo-Hookean model constant (MPa), λ is the stretch ratio, ε_{eng} is the engineering strain for Neo-Hookean model (mm/mm), and G is the shear modulus (MPa).

8.3.2 Multilinear plastic model

Multilinear plastic model for Ag-PDMS conductive ink requires engineering stress versus plastic strain curve where the plastic strain value needs to be converted from experimental stress–strain data for FEA input parameter. The formulae involve in defining the material properties under the multilinear plastic model are listed in Eqs. (8.4) and (8.5).

$$\varepsilon_{plastic} = \varepsilon_{total} - \left(\frac{\sigma_{eng}}{E_{yield}} \right) \quad (8.4)$$

$$E = \frac{\sigma_y}{\varepsilon_y} \quad (8.5)$$

This plastic model uses the yield stress at 0.20 MPa for Ag-PDMS conductive ink obtained by universal tensile testing. The Poisson's ratio is also assumed as 0.37. The material models and properties for the whole material involved in the research are summarized in Table 8.2.

8.4 Finite element analysis

There are few steps involved in FEA such as modeling of geometry followed by meshing, setting the boundary condition and postprocessing analysis in order to obtain the results of a simulation. The analyses were conducted on general shape geometries and a model of a thermal sensor circuit.

Table 8.2 Material model and properties for the simulation.

Component	Model	Material properties required
PDMS substrate	Neo-Hookean hyperelastic	Engineering stress–strain data $G = 0.16 \text{ MPa}$
Ag-PDMS conductive ink	Multilinear plastic	$E = 1.56 \text{ MPa}$ $\nu = 0.37$ Engineering stress vs plastic strain data at $\sigma_y = 0.20 \text{ MPa}$

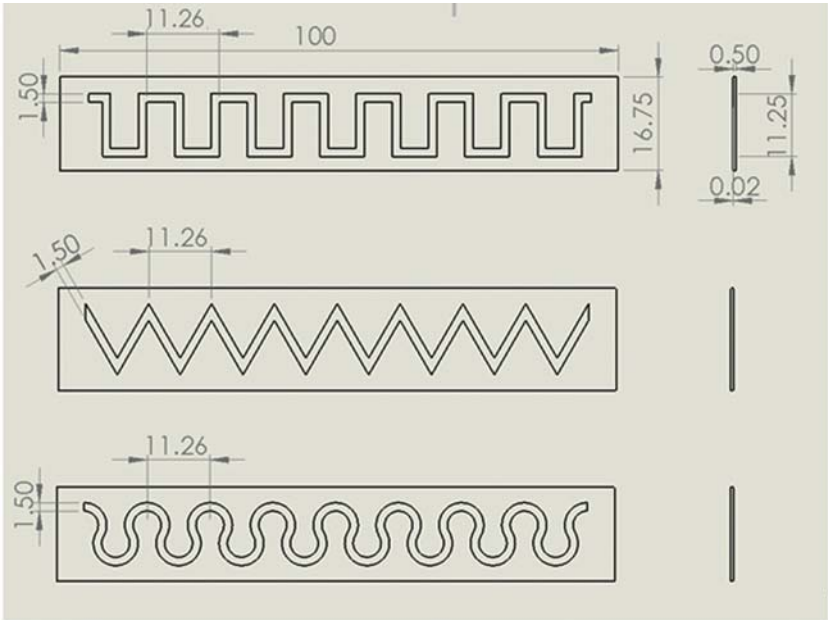
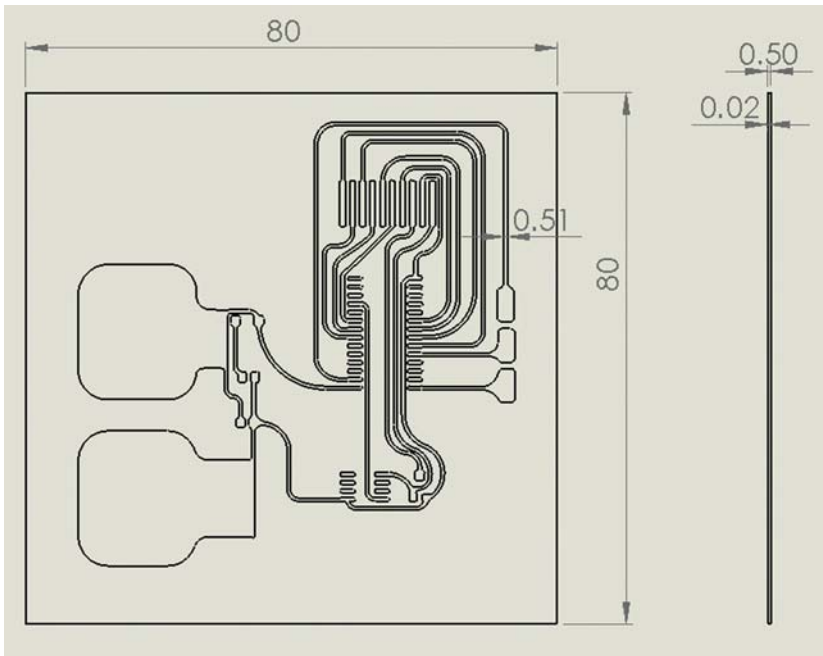


FIGURE 8.2

Image for three different printing shapes with rectangular, zigzag, and horseshoe, respectively.

8.4.1 Modeling and meshing of different printing shapes models

FEA modeling was done in two stages. First, the analyses were conducted on three simple and basic geometries in a circuit that are of horseshoe, zigzag, and rectangular shapes. The models of these geometries are shown in Fig. 8.2. These geometries were created by using Solidwork 2015 (SW 2015).

**FIGURE 8.3**

Geometry of a thermal sensor circuit.

Fig. 8.2 illustrates the three different printing shapes of substrates having an area of $100 \text{ mm} \times 16.75 \text{ mm}$ and 0.5 mm thickness. The conductive ink was modeled as 1.5 mm wide and 0.018 mm thick for all the three shapes.

The second category in FEA modeling is the analysis of a complex thermal sensor circuit, a sensor prototype developed by industry, consisting of a substrate and conductor. The thermal sensor circuit was also modeled in SW 2015. Fig. 8.3 represents the geometry of a thermal sensor circuit made up of $80 \text{ mm} \times 80 \text{ mm}$ area of the substrate with 0.5 mm thickness and 0.018 mm width and 0.51 mm thickness of conductive ink.

After designing, meshing was done by creating several elements in 3-D models. The meshing involves two types of meshes that are global and individual mesh. Global mesh function is used to control the whole model, and individual mesh is restricted for certain part in the model. The meshing setup parameters are summarized in Table 8.3.

The sizing option was used to control the element size in order to optimize the number of elements and shorten the time taken for analysis. The element midside nodes are set as “Dropped” in order to control the meshing element. The body sizing for individual mesh was utilized to control the size of the conductive ink

Table 8.3 Controlled parameters in the meshing process.

Section	Parameter	Details	Value
Global mesh	Sizing	Minimum size	Default
		Maximum size	1.0 mm
Individual mesh	Advanced	Element midside nodes	Dropped
	Body sizing	Element size	0.3 mm

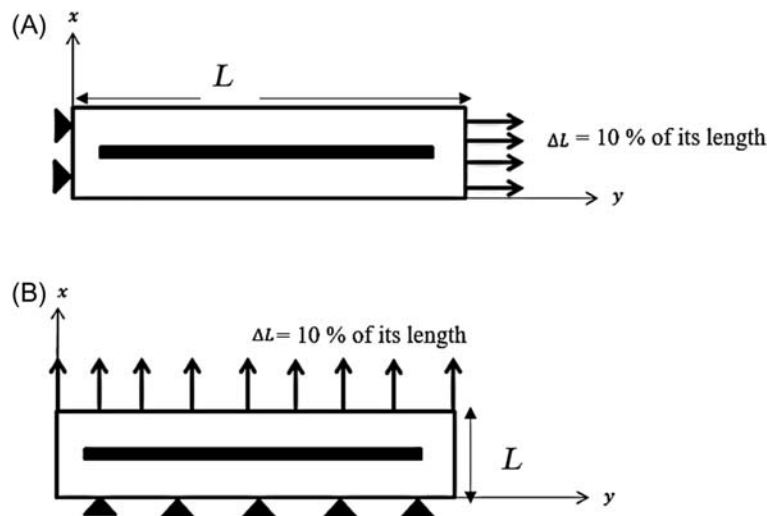


FIGURE 8.4 Schematic diagram for boundary condition used for (A) horizontal and (B) vertical loading direction of a basic geometry circuit.

and set to be as 0.3 mm. The accuracy of results can be increased by changing the size of elements of individual mesh, that is, by controlling the size of conductive ink. Several trials and errors were done to obtain the optimum mesh size.

8.4.2 Boundary conditions

8.4.2.1 Printing circuits

The boundary conditions used for three different circuit printing are schematically shown in Fig. 8.4. In Fig. 8.4A, horizontal loading is applied in the direction of basic geometry circuit and shown that the model is fixed at one end, that is, $y = 0$ mm while displacement (ΔL) is applied at the other end, that is, $y = \Delta L$ mm. The displacements on x -axis and z -axis are set to free. Similarly, Fig. 8.4B

shows that the model is fixed in the horizontal direction at one end ($x = 0$ mm) due to vertical loading and displacement (Δx) is applied at the other end ($x = \Delta L$ mm). The displacements in other y -axis and z -axis are set to free. According to Gonzalez et al. [22], the change in displacement ΔL is set to be 10% of the length of the respective model due to the application of SEC which resulted in a 10% strain.

8.4.2.2 Thermal sensor circuit

The boundary conditions for a thermal sensor circuit are shown in Fig. 8.5 based on three conditions that are uniaxial horizontal loading, uniaxial vertical loading, and biaxial loading.

Fig. 8.5 shows that the model is fixed at the nodes only at the center of the model to avoid model rotation. The load applied is according to Eq. (8.6) until

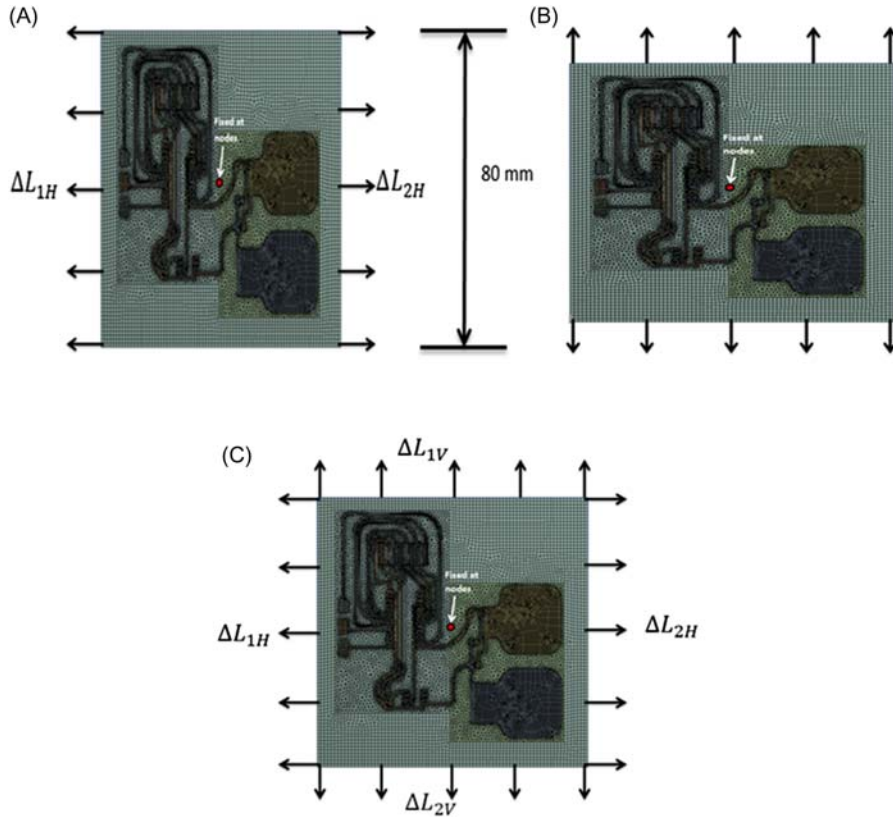


FIGURE 8.5

Boundary condition of a thermal sensor circuit design for (A) uniaxial horizontal loading, (B) uniaxial vertical loading, and (C) biaxial loading with fixed one node.

Eq. (8.8). The total loading, ΔL_{TH} for uniaxial horizontal direction can be calculated from Eq. (8.7) while Eq. (8.8) is used to calculate the total loading, ΔL_{TV} for the uniaxial vertical direction. The biaxial loading can be obtained by adding Eqs. (8.7) and (8.8).

$$\Delta L_{1H} = \Delta L_{2H} = \Delta L_{1V} = \Delta L_{2V} = 40 \text{ mm} \quad (8.6)$$

$$\Delta L_{1H} + \Delta L_{2H} = \Delta L_{TH} = 80 \text{ mm} \quad (8.7)$$

$$\Delta L_{1V} + \Delta L_{2V} = \Delta L_{TV} = 80 \text{ mm} \quad (8.8)$$

$$\Delta L_{TH} + \Delta L_{TV} = 160 \text{ mm} \quad (8.9)$$

8.4.3 Analysis using the simulation process

The analysis using simulation process requires the result validation process before starting an analysis on the actual model. The simple rectangular model was first created and meshed followed by simple boundary conditions shown in Fig. 8.6.

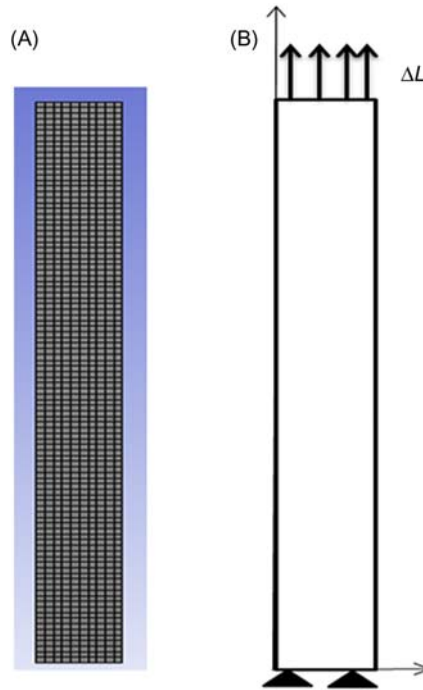


FIGURE 8.6

Rectangular models with (A) mesh and (B) boundary condition for the result validation process.

The stress–strain results were then analyzed for the simple block. The simulation results were compared with the experimental results. The software setup is only valid when the simulation stress–strain results match with the experimental stress–strain data for both substrate and conductive ink.

The result validation process data need to be converted into engineering stress–strain data to give similar result as experimental data. The conversion requires several formulas as listed in Eqs. (8.10)–(8.13).

$$\varepsilon_{true} = \ln(1 + \varepsilon_{eng}) \quad (8.10)$$

$$\varepsilon_{eng} = \exp^{\varepsilon_{true}} - 1 \quad (8.11)$$

$$\lambda = \exp^{\varepsilon_{true}} \quad (8.12)$$

$$\sigma_{true} = 2 \left[\lambda^2 - \frac{1}{\lambda} \right] C_1 \quad (8.13)$$

However, the actual models were analyzed in three different geometries such as straight, zigzag, and horseshoe shapes. The complex thermal sensor circuit consists of a substrate, and conductive ink was only designed for preliminary study. There were three case studies involved that are:

1. Characterization of material properties by using the Neo-Hookean model for PDMS substrate and multilinear plastic model for Ag-PDMS conductive ink.
2. The effect of geometry on the stress–strain behavior of the PDMS substrate and Ag-PDMS conductive ink.
3. Stress–strain analysis for a thermal sensor circuit under
 - a. different loading directions for the PDMS substrate and Ag-PDMS conductive ink, and
 - b. different material for the conductor includes comparison on Copper and Ag-PDMS conductive ink in application with PDMS substrate.

8.5 Results and discussion

8.5.1 Material properties of stretchable electronic circuit material

A uniaxial tensile test was conducted on a prepared PDMS substrate and Ag-PDMS conductive ink at room temperature. Fig. 8.7 demonstrates the experimental results for both materials represented by the engineering stress–strain curves. The maximum stresses of the substrate and conductive ink before failure are 0.49 and 0.37 MPa, respectively. The PDMS substrate contains higher stress at 200% strain while Ag-PDMS contains lower stress at 400% strain. Both materials contain different curve patterns like PDMS substrate shows a nonlinear curve while Ag-PDMS conductive ink has linear and nonlinear curves.

The nonlinear curve of the PDMS substrate can be represented by the hyperelastic model that returns into initial position after load removal. The material

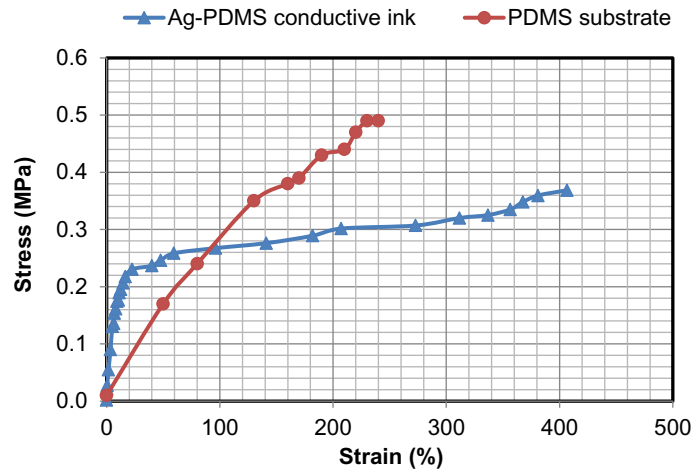


FIGURE 8.7 Stress–strain curve of PDMS substrate and Ag-PDMS conductive ink under universal tensile test condition.

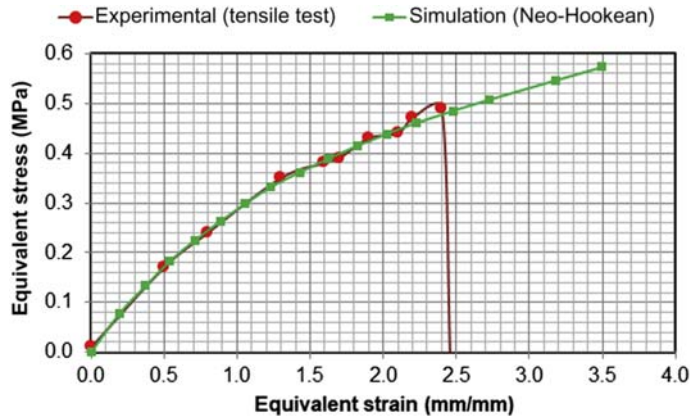
Table 8.4 Material model and required material properties for the simulation.

Component	Model	Material properties required
PDMS substrate	Neo-Hookean	Engineering stress–strain data $G = 0.16 \text{ MPa}$
Ag-PDMS conductive ink	Multilinear plastic	$E = 1.56 \text{ MPa}$ $\nu = 0.37$ Engineering stress vs plastic strain data at $\sigma_y = 0.2 \text{ MPa}$

property for the PDMS substrate is defined under the Neo-Hookean model in ANSYS software. Table 8.4 shows the values of different material properties of the substrate and conductive ink used in Neo-Hookean and multilinear plastic models. The PDMS substrate analysis under the Neo-Hookean model was validated using the experimental data and concluded both results were limited to 200% strain as shown in Fig. 8.8.

8.5.2 Deformation behavior of stretchable electronics circuit

In order to assess the stress–strain behavior of the PDMS substrate and Ag-PDMS conductive ink, three different printing designs (i.e., horseshoe, zigzag,

**FIGURE 8.8**

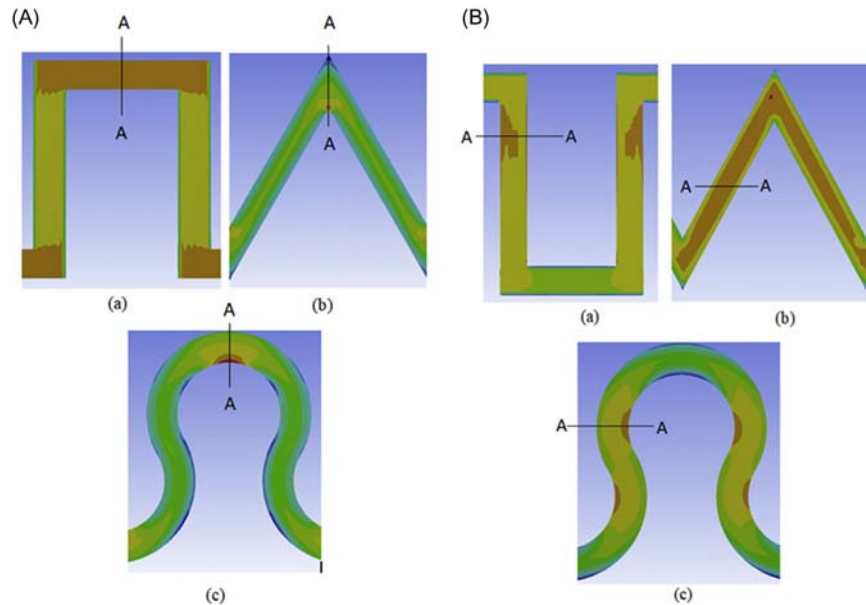
Simulation validation result for PDMS substrate using the Neo-Hookean model.

and rectangular) were analyzed in horizontal and vertical loadings and 10% strain were reported.

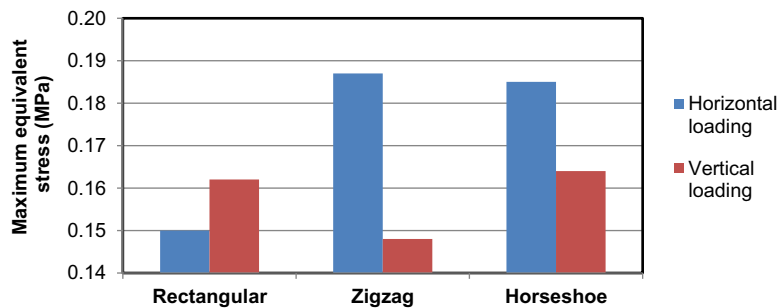
Fig. 8.9 shows the maximum equivalent stress of Ag-PDMS conductive ink for three basic printing circuits in horizontal and vertical displacements.

In horizontal displacement, the equivalent stresses are critical at the horizontal line for a rectangular shape, inner sharp edge for zigzag shape, and inner curve for horseshoe shape. In vertical displacement, the critical equivalent stress area is observed at the lines parallel to the loading at a vertical circuit and less critical for the circuit printing perpendicular to the loading along horizontal circuit for rectangular design. On the other hand, zigzag shape shows high stress for the inner region of the whole slanted lines while horseshoe shows critical equivalent stress at the inner curve of vertical lines that are parallel to the loading. The trends of maximum equivalent stress concentrated parallel to the loadings for the three printing circuits of Ag-PDMS at section A-A for both horizontal and vertical displacements are plotted in Fig. 8.10.

From Fig. 8.10, it is clearly shown that the maximum equivalent stress at section A-A for horseshoe and zigzag in horizontal loading is higher than similar shapes in vertical loading and approximately equal to 190 kPa. The rectangular and horseshoe shapes in vertical loading give the second highest equivalent stress of about 160 kPa. On the other hand, the lowest equivalent stress at section A-A represented by a rectangular shape in horizontal loading and zigzag shape in vertical loading is below than 150 kPa. The horizontal and vertical loadings of three circuit designs have a maximum equivalent stress level in between 190 and 120 kPa under the same loading application. The equivalent stress concentration is affected by the direction of loading where the circuits printing parallel to the loading has higher equivalent stress as compared to the circuit printing

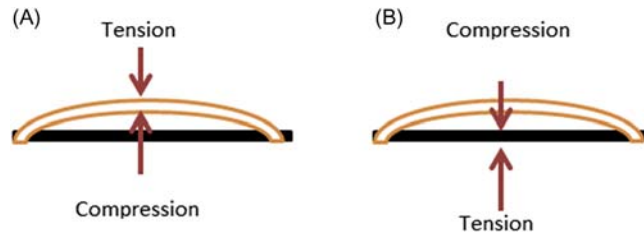
**FIGURE 8.9**

Equivalent stress of Ag-PDMS conductive ink at A-A section for (A) horizontal loading and (B) vertical loading for the three different printing (a) rectangular, (b) zigzag, and (c) horseshoe, respectively.

**FIGURE 8.10**

Maximum equivalent stress at section A-A for horizontal and vertical displacement.

perpendicular to the loading. However, the equivalent stress for the three printing at both section in horizontal loading is still below the yield stress at 200 kPa. The inner sharp edge produces tensile stress while the outer sharp edge creates compressive stress.

**FIGURE 8.11**

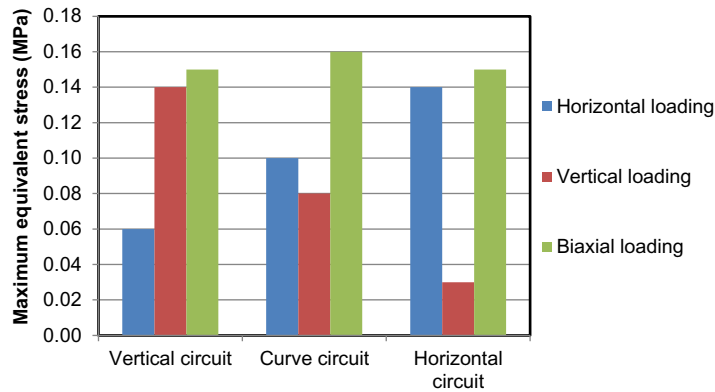
Bending concepts for compression and tension phenomenon. (A) Straight to curve and (B) curve to straight.

Gonzalez et al. [4] observed that tensile stress was produced in parallel circuit direction with loading while compressive stress occurred when the circuit was perpendicular to the direction of loading. In parallel loading, the high equivalent stress is concentrated at one side (i.e., inner curve and inner sharp edge) while the perpendicular shows that the equivalent stress is concentrated uniformly throughout the circuit width. The tension compression phenomenon can be related to the bending concept as shown in Fig. 8.11.

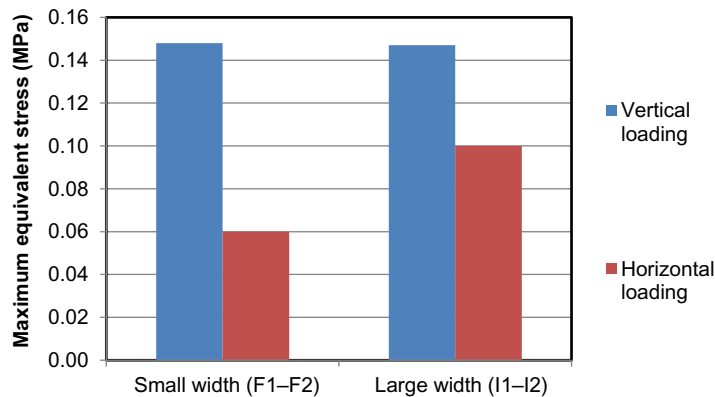
8.5.3 Equivalent stress analysis of a thermal sensor circuit design up to 10% strain

The stress–strain analysis for Ag-PDMS conductive ink is further studied in terms of its application as a thermal sensor circuit. There were two parameters involved in defining the stress–strain behavior of Ag-PDMS conductive ink in a thermal sensor circuit that have different loading directions applied on the combination of different geometries in one design (thermal sensor circuit). There were three loading directions involved, vertical, horizontal (on two sides), and biaxial loading (on four sides), at total 10% displacement for the entire loading. The equivalent stress analysis result for different loading directions on different geometries in a thermal sensor circuit is summarized in Fig. 8.12.

Fig. 8.12 clearly shows that both vertical and horizontal loadings have the highest equivalent stress of 0.14 MPa followed by curvature circuit for circuit printed parallel to the applied displacement. The lowest equivalent stress given by the circuit printed perpendicular is 0.06 MPa for vertical loading and 0.03 MPa for horizontal loading. Biaxial loading results in the highest equivalent stress for the entire three different shapes of about 0.15 MPa. The results indicate that different shape circuit has no effect in reducing the equivalent stress in biaxial loading but it can be reduced when the load applied is perpendicular to the loading direction.

**FIGURE 8.12**

Maximum equivalent stress for different geometry in a thermal sensor circuit at different loading directions at 10% strain.

**FIGURE 8.13**

Maximum equivalent stress for different width circuit at different loading directions in a thermal sensor circuit at 10% strain.

8.5.4 Effect of width in reducing the equivalent stress in a thermal sensor circuit

Thermal sensor circuit analysis is further discussed in terms of the effect of width in reducing the equivalent stress concentrated on the circuit. Fig. 8.12 summarized the result for maximum equivalent stress of different width at different loading directions between vertical and horizontal loadings. Fig. 8.13 shows the results when the displacement applied is parallel to the printed circuit for the

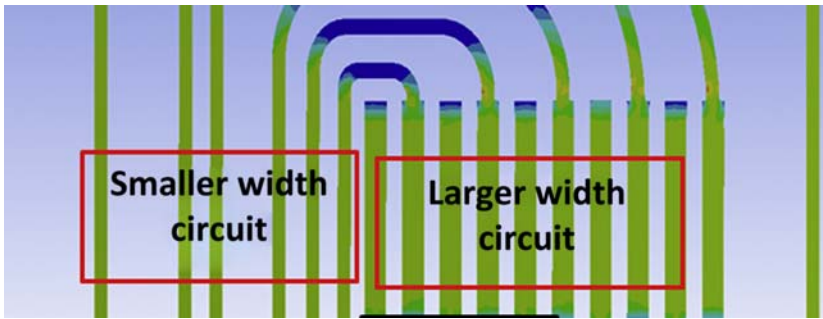


FIGURE 8.14

Color contour of circuit printing at different width geometries.

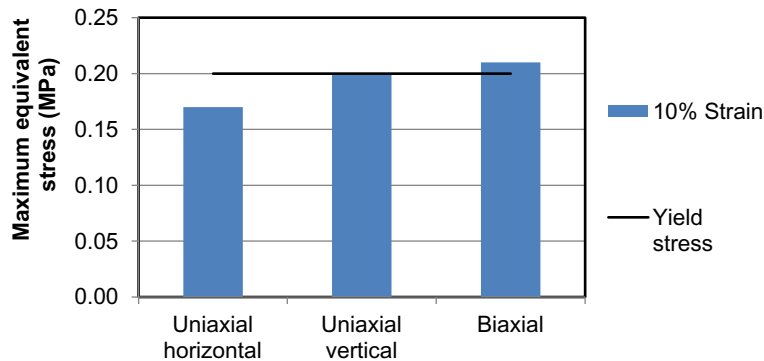
vertical loading while perpendicular to the displacement applied for horizontal loading. By decreasing the value of width, it does not give significant effect in reducing the equivalent stress as results in vertical loading when the circuit is pulled parallel to the circuit shape but it does in horizontal loading when the circuit is perpendicular.

Referring to Fig. 8.14, the simulation result shows a similar color contour at the circuit printing parallel to the loading even they have different width geometries. In addition, the higher equivalent stress level is given by the largest width when the load applied is perpendicular. In conclusion, reducing the width has no significant effect in reducing the equivalent stress when the load applied is parallel to the loading but it does as the circuit is perpendicular. Gonzalez et al. [4] have performed a study regarding the circuit width where smaller width can reduce the percentage of a plastic strain of its horseshoe shape made up of copper material. However, the research is limited to horseshoe shape only without considering the direction of displacement applied.

8.5.5 Equivalent stress limitation when the load is applied up to 10% strain

The displacement below 10% strain indicates that the maximum equivalent stress is still below the yield stress of the material which means that Ag-PDMS circuit is applicable for thermal sensor application. The analysis is extended to see the behavior and limitation of a thermal sensor when the displacement applied is more than 10% strain. Fig. 8.15 shows the results for a thermal sensor circuit at different loading directions when 10%, 20%, and 30% displacement applied.

According to Fig. 8.15, the uniaxial vertical and biaxial loadings give higher equivalent stress reaching the yield stress of material at 0.20 MPa at 10% strain rate. The results indicate that plastic deformation has occurred for both loadings. Uniaxial horizontal loading gives the lowest maximum equivalent stress of about 0.17 MPa.

**FIGURE 8.15**

Maximum equivalent stress for horizontal, vertical, and biaxial loadings at 10% strain.

8.6 Future recommendations

In order to obtain more accurate results, the Neo-Hookean model used for PDMS substrates can be improved by the model based on the conductive ink developed in laboratory and including a damage criterion into the model. The structural integrity of real electronic circuit design can be predicted by developing a more accurate model that will be useful in analyzing the three-dimensional printed products and predict failure of the materials and related structures.

8.7 Conclusion

In this chapter, the stress analysis of PDMS substrate and Ag-PDMS conductive ink of stretchable electronics circuit was carried out by using two models, namely the Neo-Hookean hyperelastic model and multilinear plastic model. The following results were obtained:

- Ag-PDMS conductive ink was characterized under the multilinear plastic model at the yield stress of 0.20 MPa.
- The Neo-Hookean model was used to get material properties of PDMS substrate.
- The stress–strain behavior of the three different geometries of circuit printing shapes (horseshoe, rectangular, and zigzag) under different loading conditions was observed.
- Both horseshoe and zigzag shape circuits gave the highest stress concentration of about 0.19 MPa with no plastic deformation at the inner curve and sharp edge, respectively.
- Among the three shapes of the stretchable circuit, rectangular gives better performance for its lower maximum equivalent stress level of 0.16 MPa.

- The stress–strain behavior of stretchable circuits in the thermal sensor circuit under different loading directions for Ag-PDMS conductive ink also discussed in detail.
- The plastic deformation did not occur in uniaxial horizontal loading and limited to 10% strain but it occurred for uniaxial vertical and biaxial loading. Uniaxial vertical gave 8% strain while biaxial gave less than 1.3% strain plastic deformation.
- On the other hand, plastic deformation for copper material in biaxial loading is higher than Ag-PDMS conductive ink at 7% strain.

Acknowledgments

The authors would like to thank the Collaborative Research in Engineering, Science & Technology (CREST) for research grant P24C1-2015 and Universiti Sains Malaysia Bridging Grant (304/PMEKANIK/6316085). Also, the authors would like to acknowledge the support from ETS Department of Jabil Circuit Penang and Advanced Packaging & SMT Unit of USM.

References

- [1] J.A. Rogers, T. Someya, Y. Huang, Materials and mechanics for stretchable electronics, *Science* 327 (2010) 1603–1607. Available from: <https://doi.org/10.1126/science.1182383>.
- [2] F. Bossuyt, T. Vervust, J. Vanfleteren, Stretchable electronics technology for large area applications: fabrication and mechanical characterization, *IEEE Trans. Compon., Packag. Manuf. Technol.* 3 (2013) 229–235. Available from: <https://doi.org/10.1109/TCPMT.2012.2185792>.
- [3] T. Adrega, S.P. Lacour, Stretchable gold conductors embedded in PDMS and patterned by photolithography: fabrication and electromechanical characterization, *J. Micromech. Microeng.* 20 (2010) 055025. Available from: <https://doi.org/10.1088/0960-1317/20/5/055025>.
- [4] M. Gonzalez, B. Vandevelde, J. Vanfleteren, Design and performance of metal conductors for stretchable electronic circuits, *Circuit World*, 1, 2009, 22–29. Available from: <https://doi.org/10.1108/03056120910928699>.
- [5] D.H. Kim, J.A. Rogers, Stretchable electronics: materials strategies and devices, *Adv. Mater.* 20 (2008) 4887–4892. Available from: <https://doi.org/10.1002/adma.200801788>.
- [6] T. Sekitani, T. Someya, Stretchable, large-area organic electronics, *Adv. Mater.* 22 (2010) 2228–2246. Available from: <https://doi.org/10.1002/adma.200904054>.
- [7] X. Wang, H. Hu, Y. Shen, X. Zhou, Z. Zheng, Stretchable conductors with ultrahigh tensile strain and stable metallic conductance enabled by prestrained polyelectrolyte nanoplateforms, *Adv. Mater.* 23 (2011) 3090–3094. Available from: <https://doi.org/10.1002/adma.201101120>.
- [8] Y. Hu, P. Zhu, T. Zhao, Y. Zhu, X. Liang, R. Sun, et al., Printable and stretchable elastic composites with highly electrical conductivity based on core-shell fillers, in:

- China Semicond. Technol. Int. Conf. 2016, CSTIC2016, pp. 3–5. Available from: <https://doi.org/10.1109/CSTIC.2016.7463939>.
- [9] W. Wu, Inorganic nanomaterials for printed electronics: a review, *Nanoscale* 9 (2017). Available from: <https://doi.org/10.1039/C7NR01604B>.
 - [10] J.-S. Noh, Conductive elastomers for stretchable electronics, sensors and energy harvesters, *Polymers (Basel)* 8 (2016) 123. Available from: <https://doi.org/10.3390/polym8040123>.
 - [11] D.-Y. Khang, J.A. Rogers, H. Lee, Mechanical buckling: mechanics, metrology, and stretchable electronics, *Adv. Funct. Mater.* 19 (2009) 1526–1536. Available from: <https://doi.org/10.1002/adfm.200801065>.
 - [12] Q. Ma, Y. Zhang, Mechanics of fractal-inspired horseshoe microstructures for applications in stretchable electronics, *J. Appl. Mech.* (2016). Available from: <https://doi.org/10.1115/1.4034458>.
 - [13] Y. Su, S. Wang, Y. Huang, H. Luan, W. Dong, J.A. Fan, et al., Elasticity of fractal inspired interconnects, *Small* 11 (2014). Available from: <https://doi.org/10.1002/smll.201401181>.
 - [14] S. Kumar, J.W. Strachan, M.D. Pickett, A. Bratkovsky, Y. Nishi, S. Williams, Sequential electronic and structural transitions in VO₂ observed using X-ray absorption spectromicroscopy, *Adv. Mater.* 26 (2014). Available from: <https://doi.org/10.1002/adma.201402404>.
 - [15] F. Suarez, D.P. Parekh, C. Ladd, D. Vashaee, M.D. Dickey, M.C. Öztürk, Flexible thermoelectric generator using bulk legs and liquid metal interconnects for wearable electronics, *Appl. Energy* 202 (2017) 736–745. Available from: <https://doi.org/10.1016/j.apenergy.2017.05.181>.
 - [16] M. Dickey, Stretchable and soft electronics using liquid metals, *Adv. Mater.* 29 (2017) 1606425. Available from: <https://doi.org/10.1002/adma.201606425>.
 - [17] W. Wu, Stretchable electronics: functional materials, fabrication strategies and applications, *Sci. Technol. Adv. Mater.* 20 (2019) 187–224. Available from: <https://doi.org/10.1080/14686996.2018.1549460>.
 - [18] J. Ding, J. Liu, Q. Tian, Z. Wu, W. Yao, Z. Dai, et al., Preparing of highly conductive patterns on flexible substrates by screen printing of silver nanoparticles with different size distribution, *Nanoscale Res. Lett.* 11 (2016) 412. Available from: <https://doi.org/10.1186/s11671-016-1640-1>.
 - [19] M. Amjadi, A. Pichitpajongkit, S. Lee, S. Ryu, I. Park, Highly stretchable and sensitive strain sensor based on silver nanowire-elastomer nanocomposite, *ACS Nano* 8 (2014) 5154–5163. Available from: <https://doi.org/10.1021/nn501204t>.
 - [20] A.A. Norhidayah, A.A. Saad, M.F.M. Sharif, F.C. Ani, M.Y.T. Ali, M.S. Ibrahim, et al., Stress analysis of a stretchable electronic circuit, *Procedia Eng.* 184 (2017) 625–630. Available from: <https://doi.org/10.1016/j.proeng.2017.04.127>.
 - [21] S.N.A.M. Noor, M.A.F. Khairuddin, J. Mahmud, Biocomposite silicone: synthesis, mechanical testing and analysis, *New Dev. Mech. Mech. Eng.* 1 (2012) 113–117.
 - [22] M. Gonzalez, F. Axisa, M.V. Bulcke, D. Brosteaux, B. Vandevelde, J. Vanfleteren, Design of metal interconnects for stretchable electronic circuits, *Microelectron. Reliab.* 48 (2008) 825–832.

New methodology for qualification, prediction, and lifetime assessment of electronic systems

Bey Tamsamani Abdellatif

Flanders Make—The Strategic Research Center in Manufacturing Industry, Leuven, Belgium

9.1 Introduction

Quantifying accurate reliability at (sub-)system level is not an easy task [1]. Despite the availability of prediction handbooks [2], the accuracy of the obtained results is not guaranteed. For instance, the data used in these handbooks are out-dated, referring to old technologies and assuming stresses that are not always realistic.

Other methods exist which should allow a more accurate reliability estimation, for example, the physics of failure (PoF) prognostics. However, for an industrial end user, following such an approach at (sub-)system level is too expensive. Typical steps to obtain reliability data of one component following PoF prognostic approach would require (1) understanding a given failure mechanism and developing its corresponding PoF model, (2) identifying stress accelerators of this failure mechanism, and (3) planning and implementing an accelerated life test to collect failure data in order to validate the model.

A typical accelerated life test would require failures of components collected during the test time (in the order of months) at different stress levels.

Another approach to get more accurate reliability at (sub-)system level is collecting and analyzing field data. However, this would require a complete process within an organization, by tracking the products in the field and collecting failure information for many years.

Therefore, there is a general need for methods to estimate the reliability of systems in a cost and time efficient way without losing accuracy of the estimates. This is a challenging task especially for long-life systems. For example, it is not cost-effective to collect test data for products with a lifetime of over 20 years [3].

On the other hand, having multiple sources of estimating reliability could be an advantage from engineering point of view allowing the development of a cost-effective and accurate methodology for reliability estimation.

An engineering asset could be seen as a combination of different subsystems, which in turns are composed by components. Such a decomposition could be covered in reliability by logical models such as reliability block diagram (RBD) modeling and/or quantitative fault tree analysis (FTA). These kinds of models associate a block to a specific subsystem or components that have a specific reliability. At that stage, we can deal with separate subsystems/components. The next stage would be to estimate the accurate reliability values for these subsystems/components. The approach we propose will make use of PoF modeling to get more accurate reliability values. Such an approach consists of understanding physically how a failure mechanism would occur in a component/subsystem. This can be modeled by taking into account the material, dimensions of the components, model of the stresses influences on the lifetime, modeling of stress propagation within a subsystem/components, etc.

9.2 Improved reliability assessment method

In this section, we present a methodology to get enhanced and hence more realistic estimations of (sub-)system reliability [1]. A special attention will be given to the PoF modeling underlying every technique in that methodology, illustrated with some examples. The performance of the methodology is evaluated by comparing it to the state-of-the-art approaches and is validated on industrial cases.

The state-of-the methodologies for quantitative estimation of the reliability of products which will be discussed further are (1) prediction handbooks, (2) life data analysis (LDA) or Weibull analysis, and (3) accelerated life testing (ALT).

Fig. 9.1 illustrates the difference between these methodologies in terms of cost and complexity. As could be seen in the graph, cost is related to needed efforts but also to accuracy. Higher cost means higher accuracy while complexity is linked to the integration maturity (concept, prototype, and integration in final systems).

Prediction handbooks are classified in the left bottom part of the graph (low complexity/low cost). This methodology is often applied in the concept phase of the project since no prototype is needed. On the other hand, less preparation is needed as there are prediction tools that can be easily set by using the existing bill of materials (BOM).

The accelerated life tests are somewhere in the middle of the complexity–cost graph. To conduct such tests, you need existing prototype/production samples and need to prepare the tests (test chambers, failure capturing system, and analysis of the data). The life (field) data analysis is set in the top-right corner of the graph (high complexity/high cost). This will definitely give the most accurate reliability estimate because the components/subsystems are working on the final end-use system where the exact stresses are seen. However, getting such data would require a complete process (maintenance policy and people, data collection database, returns

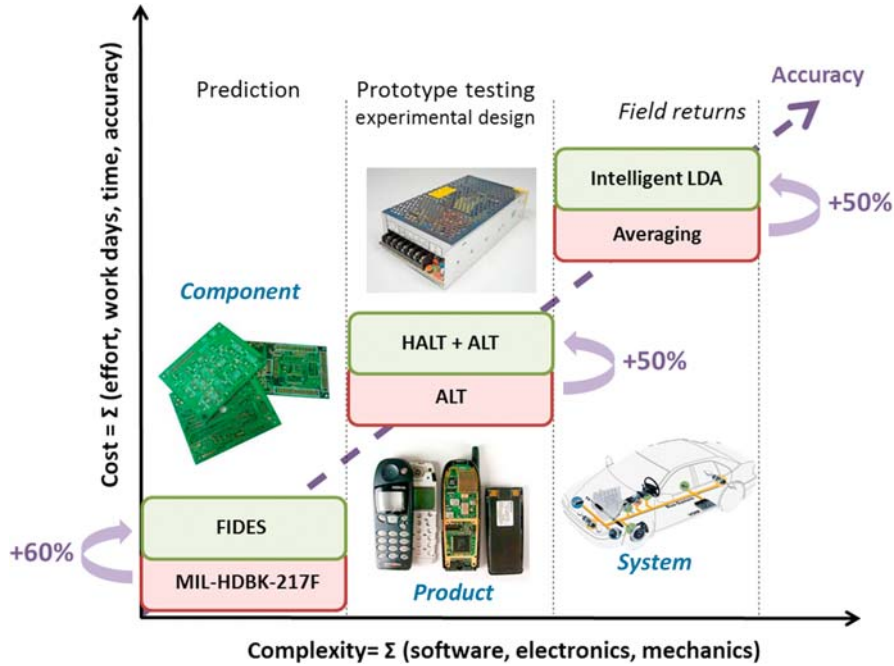


FIGURE 9.1

Methodologies for estimation of the reliability of (sub-)systems shown in relation to their complexity and cost. The recommendations are shown on top of the state-of-the-art approaches (Higher boxes). The respective improvements relative to the state-of-the-art are indicated by the arrow. For each methodology, an application and the applicability are shown.

analyses, etc.). In the following section, we will give more information about the state-of-the-arts of these different techniques and show their limitations.

9.2.1 Prediction handbooks

The military handbook *MIL-HDBK-217F* [MIL-01] composed by the American defense department is still the most widely used handbook for reliability estimation (of electronics) despite of some major concerns in relation to the quality of the results. These concerns mainly result from the empirical nature of the models used to calculate the reliability based on the sum of the weighted failure rate λ :

$$\lambda = \sum_{m=1}^M N_m \lambda_m \pi_{Q_m} \quad (9.1)$$

with $m = 1, \dots, M$ is the number of categories, N_m the number of parts within a category m , λ_m the generic failure rate of a category m , and π_{Q_m} is quality-related factors for a category m taking into account stress factors. It is assumed that the

percentage of failure is constant, and hence the distribution model is exponential. In many cases, however, this assumption is inappropriate, for example, connectors mostly fail as a consequence of fatigue which does not follow an exponential distribution. The failure mechanisms are not properly taken into account, hence the estimation is an underestimation of the field performance. Also the main stress factors and product defects are not taken into account in a relevant way. Moreover, the book is obsolete: the models are based on data collected over 20 years ago, hence it ignores the new technologies. In the end, estimations based on the MIL-217F handbook do result in a worst-case scenario rather than providing a realistic estimation. An alternative to perform reliability prediction taking into account modern electronics technologies (semiconductors, lead-free, processing units, etc.) is to use PoFs insight to understand failure mechanisms and to predict failures under different stress types (for instance under vibration stress which is one of the dominant stresses in many applications).

9.2.2 Life data analysis

LDA estimates the reliability based on data collected from a product at end users (field). For such an analysis, the operational time, the observed failure time, and the number of failed/nonfailed units are required. Typically, mean time between failures (*MTBF*) is determined based on the ratio between the total operational hours (*t*) and the number of observed failures (*N*):

$$MTBF = \frac{\sum_{n=1}^N t_n}{N} \quad (9.2)$$

This simplistic estimate does not converge to the true reliability if for instance the failure rate of the components is changing versus time. A better way to deal with modeling life data is to change the formulae by considering statistical distribution (e.g., using Weibull model [4]).

9.2.3 Accelerated life testing

ALT aims at estimating the probability of failure of a (sub-)system at a given normal user circumstances based on observations under higher levels of stress [5]. The estimation of the lifetime is based on structural and stochastic models.

A structural model describes the relation between the lifetime and the stress level. The model description depends on the type of stress. The Arrhenius model (Eq. 9.3) is a well-known example of a structural model that describes the acceleration factor, *AF*, between the “normal user” temperatures (*T_{use}*) and the “high stress” temperatures (*T_{ALT}*):

$$AF = e^{\left(\frac{\Delta H}{T_{use}} - \frac{\Delta H}{T_{ALT}} \right)} \quad (9.3)$$

ΔH is the activation energy which depends on the failure mechanism and the materials involved. Hence, this parameter describes how the distribution depends on the stress level.

A stochastic model describes the probability of failure given a stress level. The model parameters determine the behavioral characteristics of the system. The Weibull model is a frequently used stochastic model of which the scaling parameters depend on the stress level. As a consequence, the predicted lifetime (plus the uncertainty) given normal stress levels, also depends on the choice of the model structure.

When conducting an ALT, the main question would be at which stress level to test. This has a direct economic impact on the test. Higher AF would lead to a quicker test but can also bring the system to a test zone where nonlinear behavior would occur which is not correlated to the normal use. Later in this section, guidelines will be given to increase the AF while still staying at the relevant range to generate normal failures.

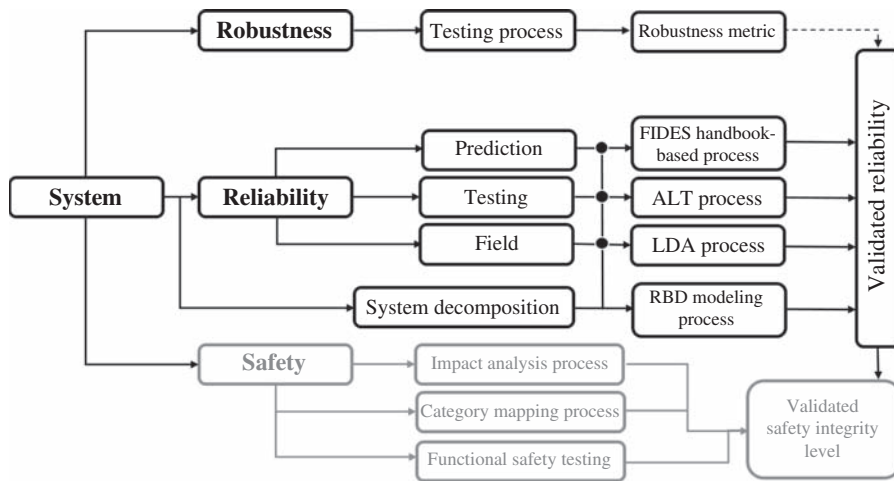
9.2.4 Improved reliability estimation methods

The two main problems arising in the state-of-the art approaches for the estimation of (sub-)system reliability are (1) the approaches do not result in realistic estimates (accuracy) and (2) the approaches are not cost efficient (high needed preparation effort, long test time). To increase accuracy and decrease cost, as typically required from industries, a systematic and integrated methodology has been proposed which enables companies to increase reliability and safety evaluation of their (sub-)system. The methodology is schematically represented in Fig. 9.2. The following subsections only describe the different building blocks of the framework related to reliability. Safety evaluation is out of scope in this chapter.

A summary of the improvements, using these improved reliability estimation methods to assess reliability of some electronic circuits, is presented in Table 9.1.

9.2.5 Prediction handbooks: FIDES rather than MIL-217F

Among others, two more recent prediction handbooks have been identified, which have been selected for further evaluation: IEC TR 62380 [IEC] and FIDES [Fides]. Although IEC TR 62380 is more recent than MIL-217F, it has not been maintained since 2003. As a consequence, the models for the PoF are not adapted for the new technologies. Also, IEC TR 62380 does not take the complete life cycle of the product into account in the reliability evaluation. Where MIL-217F results in worst-case scenario's, IEC TR 62380 results in too optimistic estimations [8]. FIDES prediction handbook, which latest edition has been released in 2009, include reliability information of more recent technologies and considers the full life cycle in the reliability evaluation of a product. It also makes use of different PoF models to describe failure mechanisms. Altogether, we opted to

**FIGURE 9.2**

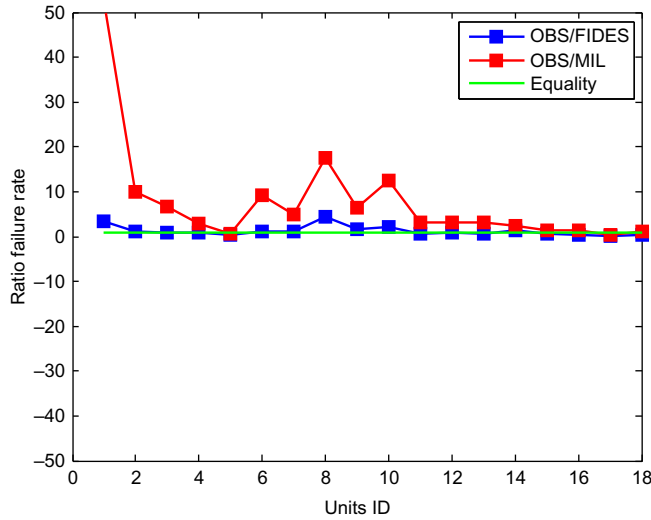
Schematic representation of the systematic and integrated methodology developed and validated for system reliability estimation.

Table 9.1 Overview of observed improvements in either accuracy of the estimated prediction of the respective method or the time reduction by applying the respective method.

Improved accuracy for reliability estimates			Reduced test time
FIDES	X		X
handbook			
ALT			
LDA			
Improvements	Up to 60% compared to MIL-217F	X Up to 50% compared to averaging	Up to 50% compared to traditional testing

obtain predictions from the FIDES handbook: a pilot project where more than 5000 electronic units have been observed in the field for 2 years proved that estimation from FIDES handbook, when process parameters are properly chosen, is much closer to the observed values in the field (~ 1.2 ratio of the observed failure rate estimated by FIDES compared to ~ 7.9 ratio observed estimated by MIL-217). The spread for some observed units is shown in Fig. 9.3.

This rationale is supported by literature [6,7] while Marin and Pollard [8], compared failure rates between FIDES and MIL-217F for a selection of electronic

**FIGURE 9.3**

Comparison reliability prediction from FIDES and MIL-217F compared to measured values from the field.

devices. For a 12 V power supply they report a relative difference in failure rate of over 30% (FIDES relative to MIL-217F).

To predict reliabilities, FIDES uses the following model:

$$\lambda = \lambda_{physical} \prod [Manufact] \prod [Process] \quad (9.4)$$

In contrast to Eq. (9.1), the prediction relies on a combination of (1) the sum of the physical contribution $\lambda_{physical}$ (due to thermal, mechanical, stress) and (2) the quality of the components ($\prod [Manufact]$) and the quality of the complete product cycle process ($\prod [Process]$). Additionally, FIDES provides (freeware) tools to assess system reliabilities. As an example, from FIDES, the model of a ceramic capacitor, the factors contributing to $\lambda_{physical}$ are thermo-electrical stress $\Pi_{Thermo-electrical}$, mechanical stress $\Pi_{Mechanical}$, and thermal cycling stress Π_{TCy} . The respective stress models are function of voltage, temperature, vibration, and time-related parameters. This is shown in Fig. 9.4.

A typical PoF prognostics model to study the thermo-electrical effect, when varying the electric stress, and the PoF prognostics model when varying mechanical stress are shown in Fig. 9.5.

In order to validate the reliability prediction using the FIDES handbook, we performed an estimation of the reliability of the machine controller of a heavy-load vehicle. The improvement in accuracy of the estimated reliability in comparison to the MIL-217F handbook is shown in Table 9.1.

$\prod_{Thermo - electrical}$	In an operating phase: $\gamma_{TH-EL} \times \left(\frac{1}{S_{reference}} \times \frac{V_{applied}}{V_{rated}} \right)_i^3 e^{\frac{11604 \times E_a}{293} \left[\frac{1}{(T_{board-ambient} + 273)} \right]_i}$ In a nonoperating phase: $\prod_{Thermo - electrical} = 0$
$\prod_{TCy}$	$\gamma_{TCy} \times \left(\frac{12 \times N_{annual-cy}}{t_{annual}} \right)_i \times \left(\frac{\min(\theta_{cy}, 2)}{t_{annual}} \right)_i^{\frac{1}{3}}$ $\times \left(\frac{\Delta T_{cycling}}{20} \right)_i^{1.9} \times e^{\frac{1414}{313} \times \left[\frac{1}{(T_{max-cycling} + 273)} \right]_i}$
$\prod_{Mechanical}$	$\gamma_{Mech} \times \left(\frac{G_{RMS}}{0.5} \right)_i^{1.5}$

FIGURE 9.4

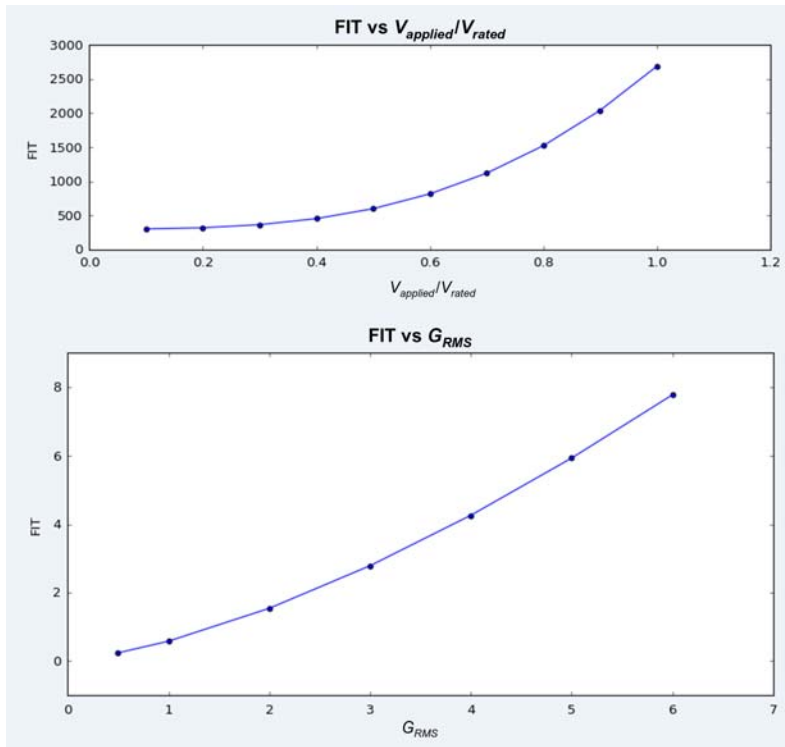
Ceramic failure model as proposed in the FIDES prediction handbook, respectively, for thermo-electrical stress model γ_{TH-EL} is the base failure rate at nominal thermo-electrical stress, $S_{reference}$ is a reference level of electrical stress, $V_{applied, rated}$ applied and rated voltages, E_a the activation energy, $T_{board, ambient}$ the board, and the ambient temperature. γ_{TCy} is the failure rate at nominal thermal cycle stress, $N_{annual-cy}$ is the annual number of thermal cycles, t_{annual} is the annual operation hours, θ_{cy} is the cycle duration, $\Delta T_{cycling}$ the amplitude of a thermal cycle phase, $T_{max-cycling}$ maximum temperature in a cycle, γ_{Mech} the base failure rate at nominal mechanical stress, and G_{RMS} , the applied random vibration stress amplitude.

9.2.6 Intelligent life data analysis rather than real life data averaging

The rationale behind LDA is to estimate the reliability of “all” (sub-)systems on the observations made for a set of (sub-)systems by fitting a statistical model, a Weibull model, to the observed data instead of using averaging as described in Section 9.2.2. The general formulation of this parametric stochastic model is

$$f(t) = \frac{\beta}{\eta} \left(\frac{t-\gamma}{\eta} \right)^{\beta-1} e^{-\left(\frac{t-\gamma}{\eta} \right)^\beta} \quad (9.5)$$

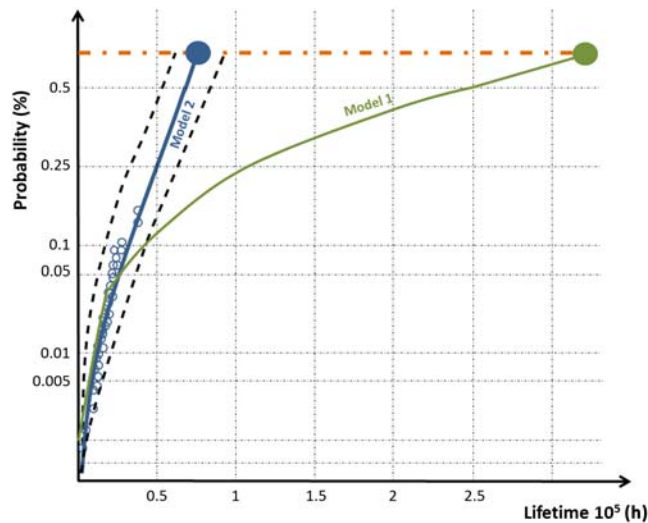
where β , γ , and η are the shape, location, and scale parameters, respectively. In general, γ is set to 0. β values could describe the different zones in the lifetime of the product (commonly called “bathtub” curve). If $\beta < 1$ infant mortality is described meaning that the failure rate is decreasing versus time and a consistent failure is occurring leading to early failures in the field. $\beta = 1$ describes an exponential distribution where the failure rate is constant versus time. This is typically the assumption made in prediction handbooks. $\beta > 1$ describes wear-out zone at end of lifetime where failure rate systematically increases versus time.

**FIGURE 9.5**

PoF prognostics model based on FIDES prediction handbook: (top) for electrical stress: Failures in time (FIT) is plotted versus $V_{\text{applied},\text{rated}}$ applied and rated voltages; (bottom) for mechanical stress: FIT is plotted versus G_{RMS} , the applied random vibration stress amplitude.

As also described in some literature [9], using a wrong model would result in estimation inaccuracies. Fig. 9.6 illustrates the inaccuracies introduced in reliability estimates if nonproper model is used, for instance, using exponential distribution for a wear-out failure. The exponential model results in an inaccurate prediction of reliability (extrapolation of the model to the point that 63.2% of the (sub-)systems failed corresponding to mean-time-to-failure).

Data averaging has been shown not to be an appropriate method to accurately estimate a product's lifetime [10]. To overcome the problem of overestimating the lifetime of a (sub-)system due to the application of simplified models is illustrated in Fig. 9.6; we start the LDA analysis by collecting field data for the selected component. Next, we classify based on the type of failure and per time to failure (TTF). Finally, we fit the data with a relevant model, by a two-parameter Weibull model. Jiang and Murthy [9] already reported the

**FIGURE 9.6**

Model 1 describes the exponential distribution of the data. The model provides an inferior fit of the data. Model 2 describes a parameterized Weibull distribution of the data. The model provides a better fit of the data. The model determines the estimate of the MTBF (at 63.2%, indicated by the horizontal line).

importance of the model parameters and its effect on, for example, TTF and residual lifetime.

In order to check the accuracy of the LDA analysis, two optimization algorithms for median rank (MR) fit were investigated. These methods are maximum likelihood estimator (MLE) and polynomial fit (polyfit). The results were also compared to the averaging method as depicted in Table 9.2. The MR/polyfit algorithm gives the best accuracy (2%), followed by MR/MLE (6.6%) while averaging is lagging much more behind (19%).

9.2.7 [HALT + ALT] rather than ALT

Highly accelerated life testing (HALT), is appropriate to determine relative robustness of products or systems. For example, a newest version of a product would be considered more robust than a previous version if it survives longer HALT test time. Although commonly used by industries in the development phase of a product to evaluate its robustness, HALT does not allow to establish the correlation between the robustness results and the field failures of a product.

In some literature [11], HALT refers to use PoF models at higher stress than normally used. This is different from our HALT terminology. HALT test in this paper refers to performing a test in a HALT chamber where 6DoF pneumatic

Table 9.2 Comparison of different tools to obtain MTBF based on life data.

Tool	Method	η	β	MTBF
Weibull++	MR/MLE	87.95	3.01	78.56
Matlab/Dfittool	MR/MLE	87.95	3.01	78.56
LDA-Tool	MR/polyfit	96.88	1.93	85.93
Traditional	Operation hours/number of failures	100.4	1	100.4
Reference	MR/graph fit	95	2.02	84.17

MLE, Maximum likelihood estimator; MR, mean radian; polyfit, a polynomial fit using the least-square method; these methods refer to the way the tool deals with collected data from devices which did not yet reach the end of their lifetime. η is the scale parameter of the Weibull distribution, β is the shape parameter of the Weibull distribution. Weibull++ is part of Reliasoft software, Dfittool is the part of the Statistics Toolbox of MATLAB.

The reference tool is according to R.B. Abernethy, *The New Weibull Handbook*, 2006.

hammer is used combined with a temperature chamber where temperature rate could reach 30°C/min. On the other side, our terminology of ALT refers to moderate stress levels where PoF models can still be applied.

Other literature [12] claimed that they could estimate reliability of a product based on the HALT chamber—based test. After contacting the authors, it revealed that they developed a methodology where they perform HALT tests on electronic modules and they observe the same designs in the field. After few years, they could find a correlation between HALT data (using a specific test profile) and the field data. Although this is possible, by correlation analysis [13], the assumptions made as need to be clearly understood.

Observations we made from previous HALT tests proved that it is difficult to establish the link with field data. However, HALT test is a very useful tool to learn the user about destruct limits of the tested product. By taking proper safety margins below these destruct limits [14], it would be possible to optimize the maximum stress levels for an ALT test. As a consequence, a lot of test time is gained [15]. A typical stress—life curve is shown in Fig. 9.7. The estimation of the reliability results from clustering the times to failure per failure mechanism. Modeling is equivalent to the description in Section 9.2.6. The obtained time gain for our industrial case is given in Table 9.1.

In order to validate this method, we designed and implemented an ALT for commercial power supplies where we identify (1), the thermal destruct limits of these power supplies and (2), use these limits in the design of ALT. A typical step stress curve to detect this stress levels is shown in Fig. 9.8. Starting from ambient temperature, the stress level decreases in a stepwise to low values to detect the lower limit. The same procedure is then followed by increasing temperature until the high stress limit is identified.

After this step, an ALT test is designed at specific safety margins from these limits. A model was developed to predict the failure rate under a specific condition as depicted in Fig. 9.9.

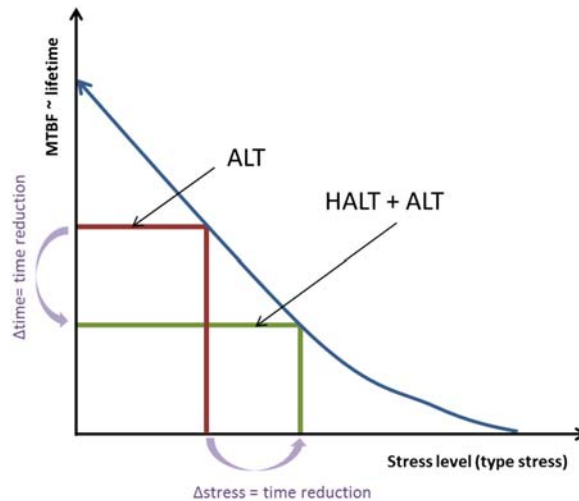
**FIGURE 9.7**

Illustration of the time gain using HALT + ALT testing in reliability prediction versus using only ALT.

From Fig. 9.9, it can be concluded that conducting a test at 100°C for 2000 hour (~ 84 days) would result in 20% failure rate (imaging that we put 100 units in the test chamber, 20 of them would fail after this test period). However, to get the same failure rate (20%) at a test temperature of 50°C, a test duration of more than 10000 (~ 417 days) would be needed. Hence, combining HALT + ALT could result in an acceleration factor of ~ 5 compared to traditional reliability testing (without using HALT information). The real test results prove these predictions.

9.2.8 Reliability block diagram tools and fault tree analysis for complex systems

Complex systems (e.g., cars) could be modeled using RBD and/or quantitative FTA [16]. A typical RBD decomposition model is as shown in Fig. 9.10.

The level decomposition can stop where acceptable reliability information is available. For example, subsystems *I*, *III*, *IV*, *V* reliability data are known from field data. Equipment's *A*, *B*, *D*, *E* reliability data are known from tests from suppliers, assembly *a*, *b*, *c*, *d* reliability data are available from a designed HALT + ALT tests, while reliability of components *E1*, ..., *E6* are predictable from FIDES handbook. This multisource information is perfectly possible to combine in an RBD model and estimate the system's reliability.

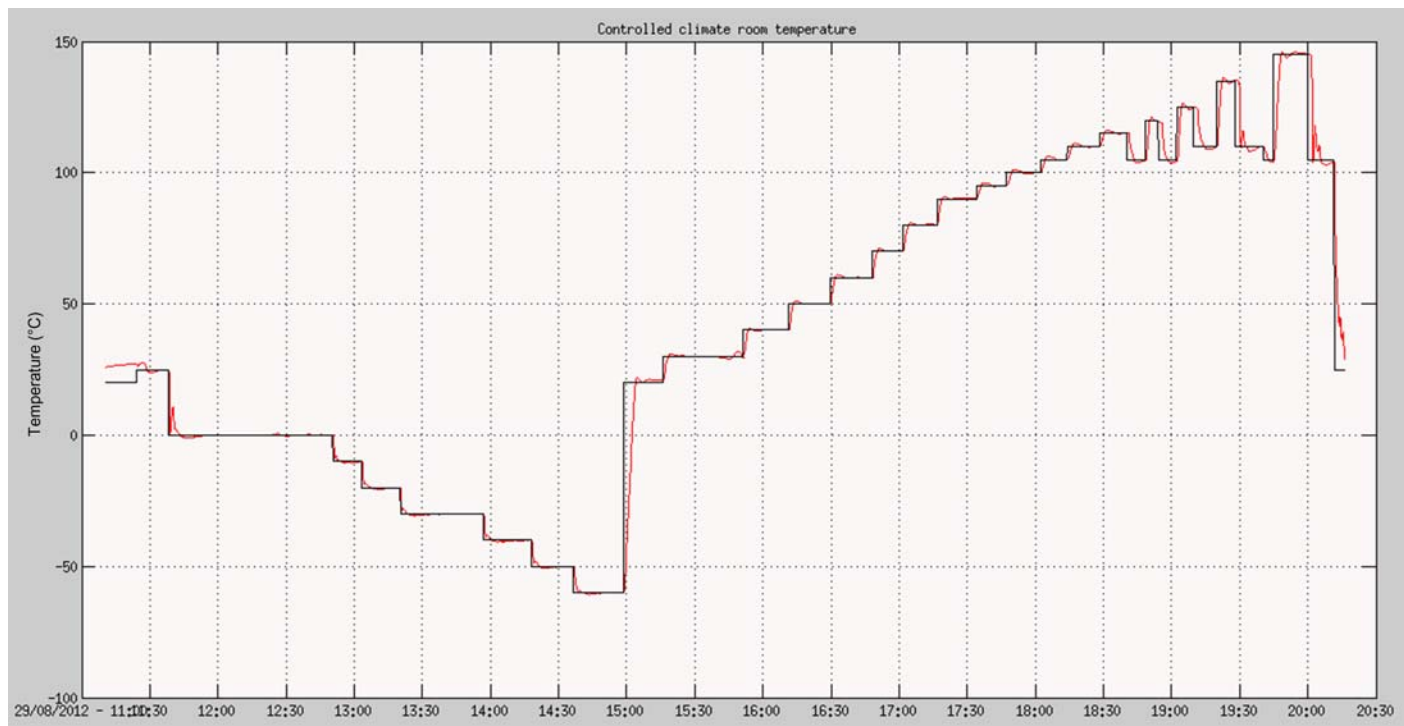


FIGURE 9.8

Step stress to identify thermal stress limits.

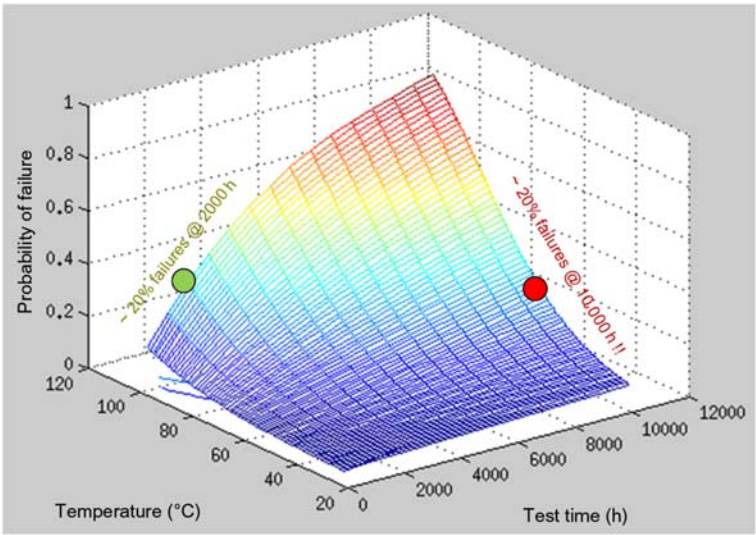


FIGURE 9.9
Prediction of failure rate versus time for an ALT under thermal stresses.

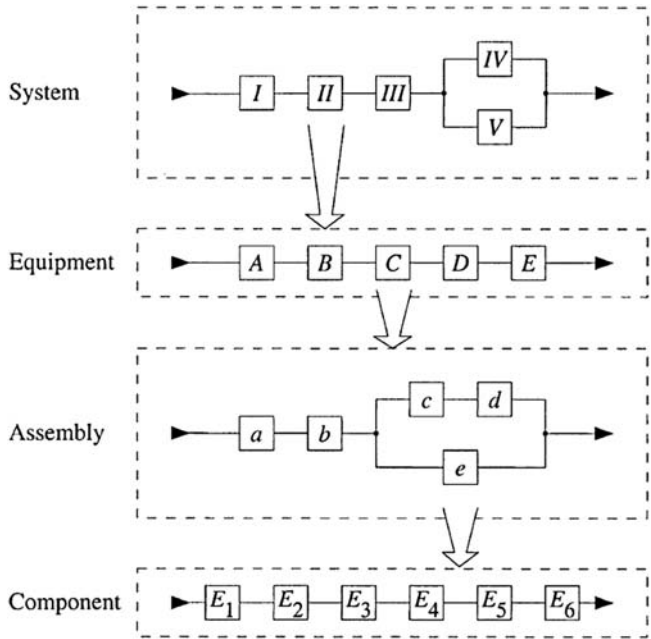


FIGURE 9.10
RBD decomposition of a system.

9.3 Application examples

A typical BOM for a printed board assembly can contain thousands of electronic components. With the broad diversity of the electronic components available nowadays, it is practically impossible to get accurate parameters for all these components and their interconnections. This makes accurate reliability prediction at (sub-)system level not straightforward and incomplete. The constant failure rate statistical component data have been improved by different bodies in recent years as explained in [Section 9.3](#). Combining these data (for noncritical components) with detailed PoF statistical models (for critical components) would give an improved estimate of the reliability at system level without losing completeness. Furthermore, this approach allows for a gradual improvement from the widely used “complete” but inaccurate handbook based methods to a cost-effective, PoF-based methodology to assess the reliability of electronic systems.

In more generic term, to implement PoF-based design for reliability (PoF DfR) in the product’s development process, the use of PoF models requires technology qualification in which the suitability of a component and interconnection technology for a specific application, for example, automotive under-the-hood, is quantitatively evaluated, that is, the PoF models are established for the technology.

Component’s qualification is a part of a technology qualification impacting development cost and time-to-market: the iteration loop “design-prototype-test redo until pass” is costly and time-consuming.

A PoF-based product development cycle is as depicted in [Fig. 9.11B](#); it uses physics-based DfR rules and models that can be applied to new designs and new technologies for which the company has no experience. These physical models are used and quantified in a technology qualification program which evaluates the suitability of an electronic assembly technology (components, substrates, materials, interconnections, etc.) for a range of applications. This reduces the amount of required testing considerably and takes it outside the product development cycle. Testing on prototypes is to a large part replaced by a much faster and more effective virtual prototyping prior to prototype manufacturing. On the other side, traditional approach, as shown in [Fig. 9.11A](#), is experience-based. This experience is rather gained when upgrading designs and learning from previous ones.

9.3.1 Electrolytic capacitors reliability analysis

In this section, we show the application of the combined methodology (conventional + PoF) to assess lifetime and therefore the reliability of components and systems. A validation case based on front light module (FLM) used in automotive applications is demonstrated. To illustrate the methodology and its benefits, the PoF modeling of aluminum electrolytic capacitors used in the FLM are described [\[17\]](#).

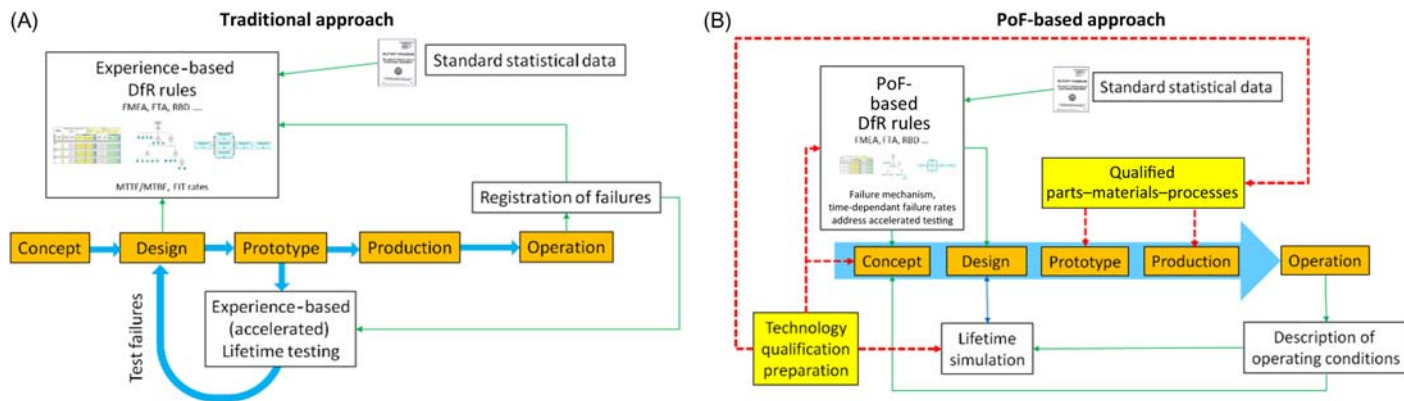


FIGURE 9.11

(A) Traditional product development cycle and (B) PoF-based DfR product development cycle.

The FIDES constant failure rate model for aluminum electrolytic capacitors is given by Eq. (9.6).

$$\lambda = \lambda_{Physical} \times \Pi_{PM} \times \Pi_{Process}$$

With

$$\lambda_{Physical} = \lambda_{0-capacitor} \times \sum_i^{Phases} \left(\frac{t_{annual}}{8760} \right)_i \times (\Pi_{thermo-electrical} + \Pi_{TCy} + \Pi_{mechanical})_i \times (\Pi_{induced})_i \quad (9.6)$$

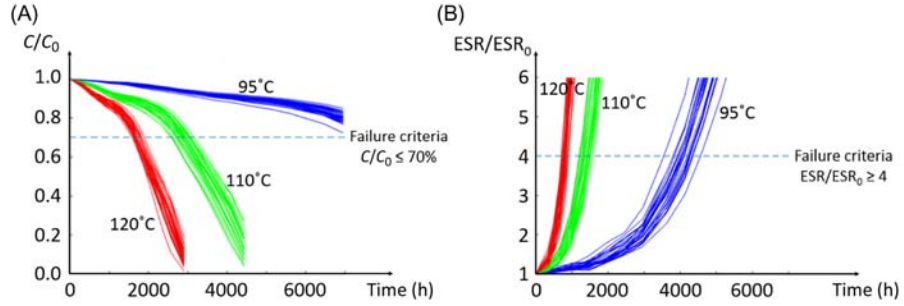
where λ is the overall failure rate of the capacitor; $\lambda_{Physical}$ represents the physical stress contribution to the failure rate; Π_{PM} is the part manufacturing represents the quality and technical control over manufacturing of the component; $\Pi_{Process}$ represents the quality and technical control over development, manufacturing, and usage process for the product containing the component; $\lambda_{0-capacitor}$ is the basic failure rate associated with the component (experience-based); *phases* represent different phases in the mission profile; t_{annual} is the time associated with each operating phase over a year (hours); and Π_{xxx} represents the factors contributing to physical stresses, respectively, in this case thermo-electrical, thermal cycles, and mechanical stresses.

These factors might be derived from known life-stress models where parameters are defined based on experience. More details can be found in Ref. [18], the Π_{xxx} equations for electrolytic capacitors are shown in Table 9.3. Similar models are used for other electronic components.

As reported in literature [19–21], one of the major causes for aluminum electrolytic capacitors failure is the decrease in electrolyte volume due to evaporation of the liquid that is accelerated when the component internal pressure rises due to high core temperatures. The electrolyte content reduction affects the main electrical parameters of the capacitor: (1) the capacitance C and (2) the equivalent series resistance (ESR). As a result of the aging, C is decreasing with respect to the initial value C_0 , while ESR increases with respect to ESR_0 . Failure criteria can be defined according to the final application requirements, for example, $C/C_0 \leq 70\%$, $ESR/ESR_0 \geq 4$.

Table 9.3 Physical stresses factors for aluminum capacitors (FIDES).

$\Pi_{Thermo-electrical}$	In an operating phase: $\gamma_{TH-EL} \times \left(\frac{1}{S_{reference}} \times \frac{V_{applied}}{V_{rated}} \right)_i^3 e^{11604 \times Ea \times \left[\frac{1}{293} - \frac{1}{(T_{board-ambient} + 273)} \right]_i}$ In a nonoperating phase: $\Pi_{Thermo-electrical} = 0$
Π_{TCy}	$\gamma_{TCy} \times \left(\frac{12 \cdot N_{annual-cy}}{t_{annual}} \right)_i \times \left(\frac{\min(\theta_{cy}, 2)}{2} \right)_i^{\frac{1}{3}} \times \left(\frac{\Delta T_{cycling}}{20} \right)_i^{1.9} \times e^{1414 \times \left[\frac{1}{313} - \frac{1}{(T_{max-cycling} + 273)} \right]}$
$\Pi_{Mechanical}$	$\gamma_{Mech} \times \left(\frac{G_{rms}}{0.5} \right)_i^{1.5}$

**FIGURE 9.12**

Example of degradation at $V = V_n$ and different temperatures (95°C, 110°C, and 120°C) (sample size: 30 capacitors per temperature): (A) capacitance C , normalized to the initial value C_0 . (B) ESR, normalized to the initial value ESR_0 .

The development and quantification of a PoF lifetime model for a specific component can be done within a technology qualification program. In the case of aluminum electrolytic capacitors, tests shall be performed to verify the impact of component core temperature to the electrolyte evaporation rate. Sample size shall be adequate to allow statistically relevant failure distribution models.

As an example, the capacitance and ESR as a function of time for sets of 30 capacitors ($V_n = 16$ V, $C_0 = 47$ μ F) aged at different temperatures, and biased at DC nominal voltage with no ripple current, are presented in Fig. 9.12A and B, respectively. As expected, the higher the temperature the faster the degradation of capacitance and the increase in ESR.

Time-to-failure is then extracted for every component, according to the failure criteria, in order to obtain the statistical distribution of failures.

The failure data corresponding to the criterion $ESR/ESR_0 \geq 4$ can be described by Weibull distributions with shape β_i and scale Θ_i , as shown in Fig. 9.13. The three curves can be fit with a statistically significant common shape factor β , confirming the same degradation mechanism as activated at the three stress temperatures. This aspect is crucial to ensure the entire dataset can be used to derive the PoF model. Additionally, the shape $\beta > 1$ confirms that the failure rate is increasing over time, as expected by a wear-out phenomenon. A clear case where the constant failure rate ($\beta = 1$) is not applicable.

Therefore, the reliability function for this capacitor subjected to a constant stress temperature T_{core} assumes the Weibull form Eq. (9.7):

$$R(t) = \exp \left[- \left(\frac{t}{\Theta(T_{core})} \right)^\beta \right] \quad (9.7)$$

A physical model that describes chemical reactions and evaporation phenomena as in this case is the Arrhenius model. The parameters Θ_I for the three stress

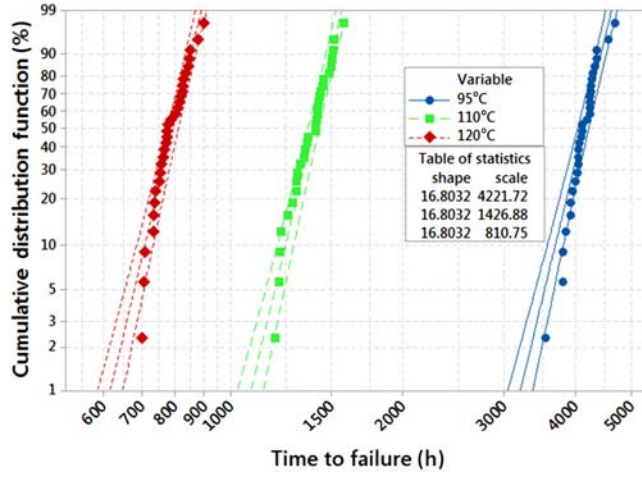


FIGURE 9.13

Example of statistical analysis (three-parameter Weibull): failures extracted from Fig. 9.12B.

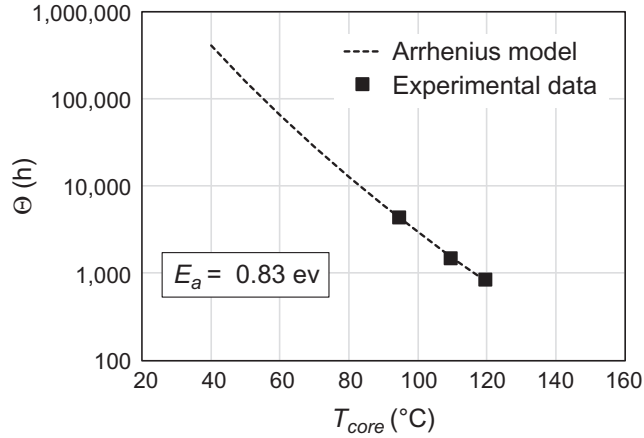


FIGURE 9.14

Arrhenius model (PoF) corresponding to the Weibull scale parameters of Fig. 9.13.

temperatures have been verified to follow Arrhenius relationship, as depicted in Fig. 9.14.

The PoF model for the Weibull scale parameter of Eq. (9.7) as a function of T_{core} can be written as Eq. (9.8)

$$\theta(T_{core}) = \theta_0 \exp \left[\frac{E_a}{k} \left(\frac{1}{T_{core}} - \frac{1}{T_0} \right) \right] \quad (9.8)$$

where E_a is the activation energy of the degradation mechanism (i.e., evaporation process), found to be 0.83 eV in this example, and k is the Boltzmann's constant, 8.62×10^{-5} eV/K. Θ_0 is a reference scale parameter value related to a reference temperature T_0 . Temperatures of Eq. (9.8) are expressed in Kelvin (K). The combination of Eqs. (9.7) and (9.8), together with the parameters obtained within the technology qualification, provides the PoF reliability model for the selected aluminum electrolytic capacitor (9.9):

$$R(T_{core}, t) = \exp \left[- \left(\frac{t}{\theta_0 \exp \left[\frac{E_a}{k} \left(\frac{1}{T_{core}} - \frac{1}{T_0} \right) \right]} \right)^\beta \right] \quad (9.9)$$

9.3.2 Demonstration of the combined methodology on front light module

The methodology is applied to an automotive FLM. A high-level conceptual architecture of this module is shown in Fig. 9.15.

Starting from the BOM of this circuit associated with relevant mission profile and equivalent data provided by FIDES, a first estimation of the reliability was done for a duration of 15,000 hours (corresponding to 5 years' service with an on state of 8 hours a day). The estimated failure rate according to FIDES data is around 6.10^{-6} failure per hour (equivalent to a mean TTF of $\sim 16,7000$ hours). The FIDES estimated failure rate is assumed no change in time as shown in Fig. 9.16 (dashed line with squares).

As we have the exact failure data of the electrolytic capacitor, explained in Section 9.4.1, we adapted the reliability calculation sheet by adding this new information. The estimated FLM's reliability using updated PoF data of capacitor is shown in Fig. 9.16 (dashed line with triangles). It is clear from the graph that

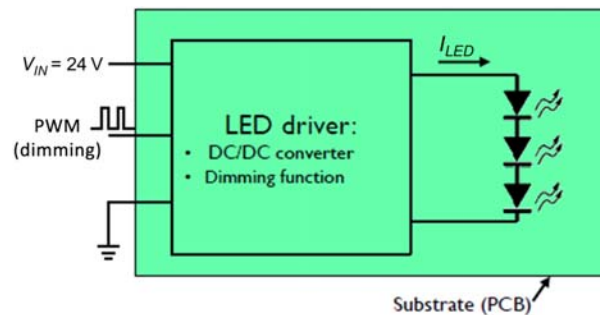
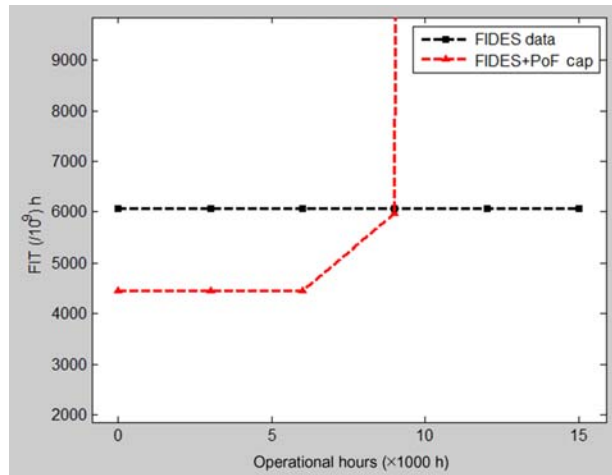


FIGURE 9.15

Front light module circuit diagram.

**FIGURE 9.16**

Comparison failure rate estimation for FLM module using only FIDES data (constant failure rate) and combined with different PoF models for electrolytic capacitor.

the estimated failure rate is not constant anymore in time but increases, which is a consequence of the wear-out nature of the failure of the capacitor. This is also clearly reported in the Weibull parameters estimation (Fig. 9.13) where the Weibull shape parameter $\beta \sim 16$ ($\gg 1$ being the value corresponding to a constant failure rate in time).

It is also clear from the graph that the FLM's failure rate using PoF data is lower than the FIDES value below $\sim 10,000$ hours and only start quickly increasing after 5000 hours. These observations suggest to the designers to take proper design actions depending on the service life of the product (e.g., replacement of parts after a specific service time).

Although demonstrated with limited set of components, the effect of PoF modeling on system's reliability is quite noticeable, which suggests reviewing critical components of the system and provide more accurate PoF data.

9.3.3 Supercapacitors reliability analysis

Supercapacitors are used nowadays in an extensive range of battery-powered devices such as GPS/GPRS transceivers, active RFID tags, industrial PDAs, electronic locks, micro medical pumps, digital cameras, mobile phones, and others. They are also used in vehicle's and machine's subsystems requiring short but robust powerful current pulses. This covers backup, delivery, and leveling of high peak power as well as storing harvested energy [22].

They are designed to meet high-power requirements that cannot be fulfilled by standard batteries. Furthermore, their size and cost are very attractive for industries.

However, selecting the right supercapacitor for a specific application remains a real challenge to industry. The specifications of these components only provide limited information about their lifetime for specific stress values. This information is not enough for industries to design a robust product and avoid high field returns.

In this section, we apply the PoF methodology for qualification and lifetime assessment of electronic systems, to derive PoF models for supercapacitors at different stresses relevant for some industrial applications. It is expected from these models to better understand the performance of supercapacitors at different stresses and to predict accurate lifetime of supercapacitors allowing industry to robustly design their products and avoid high field returns.

Supercapacitors are classified between electrolytic capacitors and batteries. Their operating principle is based on the accumulation of electric charges with opposite polarities at the interface between a porous electrode and an electrolyte, which is equivalent to a capacitor with a very thin dielectric and a very large surface area. This structure requires no chemical reaction to occur but only charge transfers, which makes the cycle life of these components much larger than the batteries [23].

Thanks to the absence of the electrochemical reaction inside a supercapacitor, this latter is not affected by cycling which may easily reach high values ($> 500,000$ cycles) without losing their energy storage capability. This makes the main lifetime issues of supercapacitors related to their longevity versus operating voltage and temperature. The voltage and temperature stresses accelerate the supercapacitors degradation in time.

Although this aging phenomenon is extensively described in literature [24], the translation toward the expected performance and lifetime of these supercapacitors are not critically reviewed considering end user's requirements.

Through the PoF methodology for qualification and lifetime assessment of electronic systems, as described in Section 9.3 the complete development cycle of the product is considered, where end user's requirements and return of experiences are addressed.

In this section, this PoF methodology is applied for qualification and life assessment of supercapacitors. This include (1) the design and implementation of a life test, (2) methods for performance parameters estimation and effects of components from different batches, and (3) design rules for reliability taking the life tests results into account.

9.3.3.1 Supercapacitors failure modes

By reviewing the state of arts [24,25], it becomes clear that supercapacitors have two degradation patterns. One is degradation of the electrochemical system (such as electrode or electrolyte) caused by applying voltage, second degradation is

drying up by the evaporation of electrolyte mainly caused by temperature. In both cases, ESR increases and capacitance decreases. The final failure is open mode by increasing internal resistance.

9.3.3.2 Design of an accelerated life test for supercapacitors

In the PoF methodology used in this analysis, qualification of electronic components should be done in advance to or in the early stage of the product development cycle, as illustrated in Fig. 9.11. Among other qualification techniques, ALT is the most convenient technique to build accurate PoF models. As supercapacitors are used in critical systems, accuracy of their lifetime is quite important. Therefore, in this section, an ALT test was performed to study the degradation signatures of supercapacitors in time and build their PoF models.

For this ALT design, economical and accuracy aspects are highlighted. Therefore, comparison of these two aspects using research (expensive setup and long test) and industrial (economical setup and short test) design of experiments and setups is highlighted.

9.3.3.2.1 Design of experiments

As temperature and voltage are the main stresses for the supercapacitors, the focus for the DoE is made on these two stresses.

For the long test design, 48 supercapacitor samples were considered at each of the 9 different stress combinations (voltage, temperature), resulting in a total of 432 tested supercapacitors. The long test lasts for more than 5000 hours.

The short test only considers 5 supercapacitors samples subject to each of the 9 stress combinations (voltage, temperature), resulting on 45 tested supercapacitors per brand. In total 7 brands were tested, which gives a total number of 315 tested supercapacitors. The short test only lasts for 1500 hours.

These two test (long and short) topologies would allow us to compare the PoF models that would be built using the two datasets, leading to conclusions on accuracy assessment with economical tests.

In addition to the constant stressors, cyclic voltage stress was considered for some samples to stress them in similar way as they are stressed in the final applications. This would allow a comparison of constant versus cyclic voltage stresses.

9.3.3.2.2 Test setup

The research (expensive) test setup consists of nine electronic boards, each populated by 48 supercapacitors of 10 F. These boards are connected through custom designed main boards, that guarantee current balancing between the supercapacitors and emergency stopping in case of excessive overvoltage, to two energy storage testing systems (PEC SBT 0650 and PEC SBT 8050). These testers contain stable high-power supplies and automatically controlled electronic loads and guarantee the voltage stresses in a constant and a cyclic way. The electronic boards with supercapacitors are tested in two different ovens (Heratherm OMS 180), respectively at 65°C and 85°C.

The industrial (economic) test setup makes use of common measurement equipment and a test PCB. This setup is made in a way that automation is possible to reduce cost and fully benefit the results, using a switchboard. The test is setup in such a way that capacitors are stressed during a period and measured at an interval inspection. The test PCB contains 15 capacitors with different operating voltages (100%, 90%, and 80%) per group of five capacitors. They are tested in three different temperatures of 55°C, 60°C, and 65°C.

9.3.3.3 Supercapacitors performance parameters and failure criteria

ESR increases in time and capacitance decreases in time are quite known indicators of electrolyte-based capacitor degradation. This includes electrolytic capacitors and supercapacitors. While for the former one, ESR could be easily measured by standard impedance meters (with measurements range from few hundreds m Ω to couple of Ω), the latter's ESR is not possible with these types of impedance meters due to the low ESR values (few tens of m Ω).

Another measurement method based on charging/discharging the supercapacitors with specific loads and with constant current would allow ESR and capacitance estimation of supercapacitors even within a small range of ESR values. This estimation method is described in different documents: IEC62576: Electric double-layer capacitors for use in hybrid electric vehicles—test methods for electrical characteristics [26], Maxwell test method [27]. Two approaches could be followed to retrieve ESR estimates (1) directly from voltage drops in the profile as shown in Fig. 9.17 (e.g., ΔV_1), (2) through a fitted model to the discharge slope as shown in dotted line in Fig. 9.17 resulting to ΔV_2 .

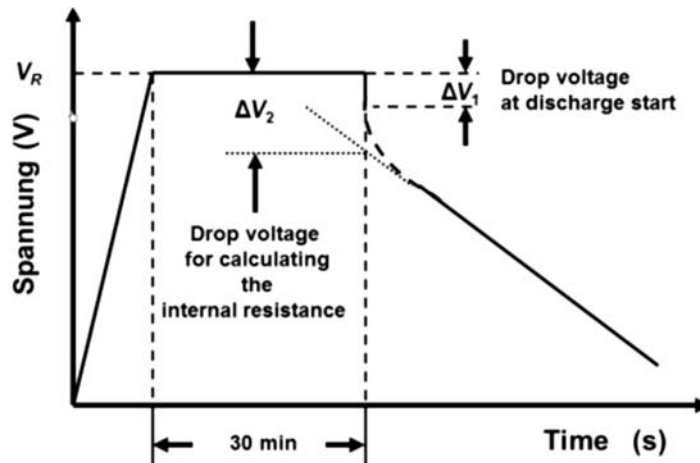


FIGURE 9.17

Test profiles proposed by IEC 62576.

For both methods, the capacitance is estimated using Eq. (9.10):

$$\frac{\Delta V}{\Delta t} \approx -\frac{1}{C} \quad (9.10)$$

where ΔV , Δt , C , respectively, denotes voltage variation in the discharging slope, equivalent time variation, and capacitance.

The ESR can be estimated using Eq. (9.11) or (9.12) depending on method 1 (step-based) or method 2 (fitted line).

$$ESR_1 = \frac{\Delta V_1}{I} \quad (9.11)$$

$$ESR_2 = \frac{\Delta V_2}{I} \quad (9.12)$$

where ESR , $\Delta V_{1,2}$, I , respectively, denotes for equivalent serial resistance of the supercapacitors, voltage drop at discharge start from measured data or using fitted model as depicted in Fig. 9.17, and constant current used in the discharge state.

To get an idea of the deviations between these different methods from our initial measurements (before degradation), the mean and std values of ESR and capacitance estimates, using the two methods, are given in Table 9.4.

From this analysis, it is observed that ESR estimates using the two methods are quite different ($> 15\%$). Furthermore, by comparing measurements made at different test time (during degradation), it appears that the std using the fitting method increases in time while the std using step-based method is relatively constant. These variations are shown in Fig. 9.18.

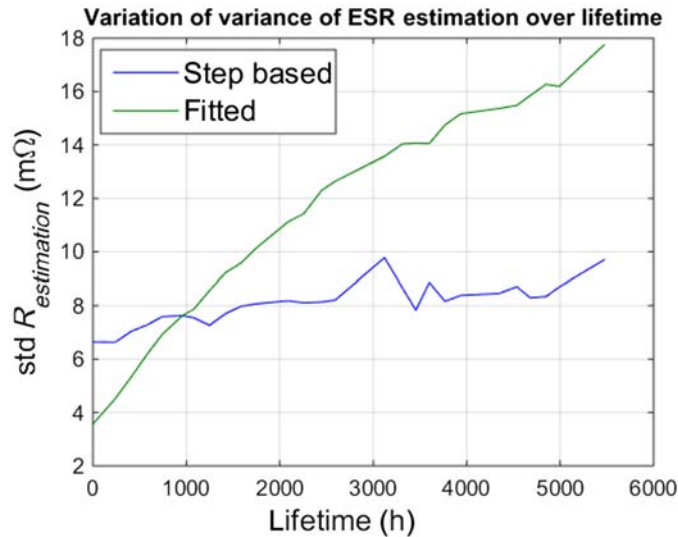
As, in our further analysis, we look to relative changes in time w.r.t. initial values, we want to avoid the systematic error induced by the fitting method.

By applying the step-based estimation method on collected data and normalizing this data to the initial measurements, the degradation curves using both capacitance and ESR can be retrieved. Some examples are shown in Fig. 9.19 where x -axis represents the test time in hours and y -axis, respectively, represents relative capacitance values w.r.t. to initial capacitance value (top) and ESR values w.r.t. to initial ESR value. The data shown in Fig. 9.19 are collected under the following stress condition (2.7 V constant voltage, 65°C).

The failure criteria would depend on the final industrial application. In this example we trigger a failure when the capacitance decreases 30% from its original value, or when ESR doubles. However, these two failure criteria are not

Table 9.4 Performance parameters estimation.

	Average	Standard deviation
ESR (small step) ($\text{m}\Omega$)	85.48	3.74
ESR (fitted line) ($\text{m}\Omega$)	103.46	1.84
Capacitance (F)	10.49	0.51

**FIGURE 9.18**

Variation of std of ESR estimates in time using fitting method (graph with higher range) and step-based method (graph with lower range).

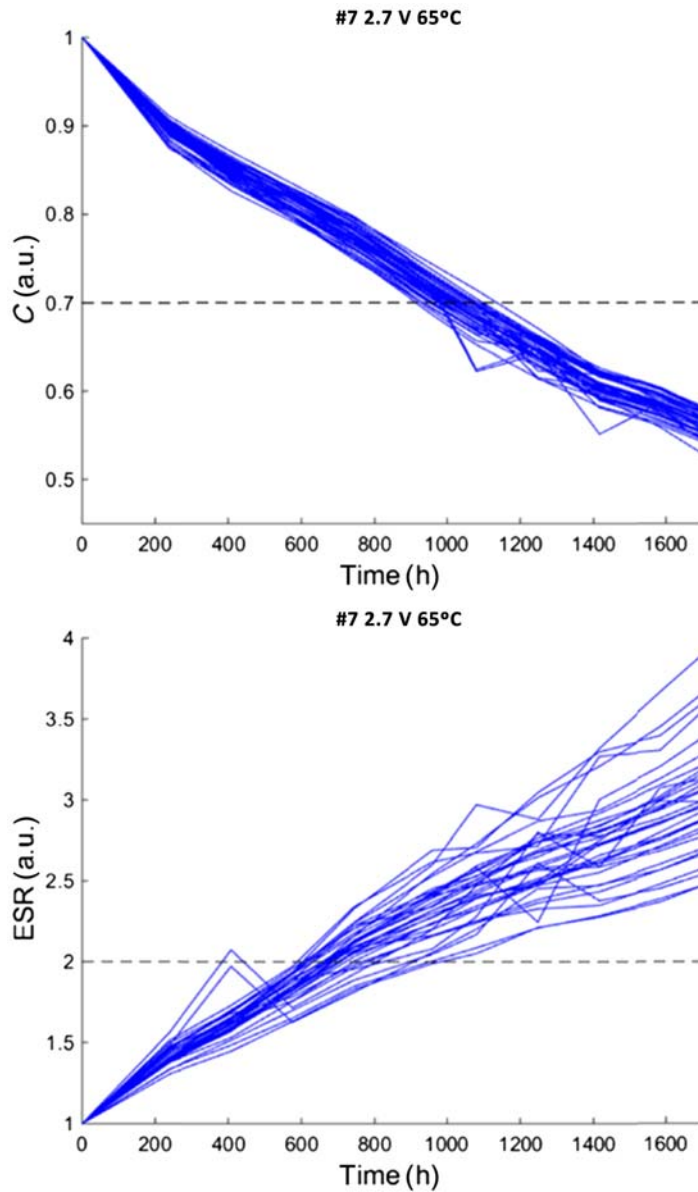
matched one to one. The failure times based on the graphs of Fig. 9.19 are collected and statistically analyzed.

A Weibull model [28] is fitted to the failure data collected respectively from capacitance and ESR degradations. The results are shown in Fig. 9.20, where the x -axis represents the TTF in hours and y -axis represents the failure probability density (top graph) and cumulative probabilities (bottom graph).

The results shown in Figs. 9.19 and 9.20 confirm that the failure criteria based on ESR degradation ($\times 2$) or on capacitance degradation (-30%) do not result on the same TTF. Furthermore, Fig. 9.20 highlights that the failure data do not follow perfectly the Weibull distribution, which can be explained by (1) the observations given in Section 9.5 and (2) noise level in ESR failure values.

Note that the ESR estimates accuracy is lower than capacitance estimates accuracy (shown in Fig. 9.19 with noisy ESR degradation signals, and essentially due to low ESR values ($\pm 75 \text{ m}\Omega$) which are strongly influenced by connector's resistances), but this does not fully explain the difference in TTF. Therefore, choosing one failure criteria or another would have a consequence into TTF and as a result on the PoF model.

The choice of the right failure criteria should then be application specific and supported with a failure mode and effect analysis (FMEA) [29] to check the criticality of these failure criteria on the system level.

**FIGURE 9.19**

Degradation curves of supercapacitors in time (top) capacitance, (bottom) ESR. The dashed line indicates failure threshold for capacitance (30% degradation from initial value) and ESR (two times initial value).

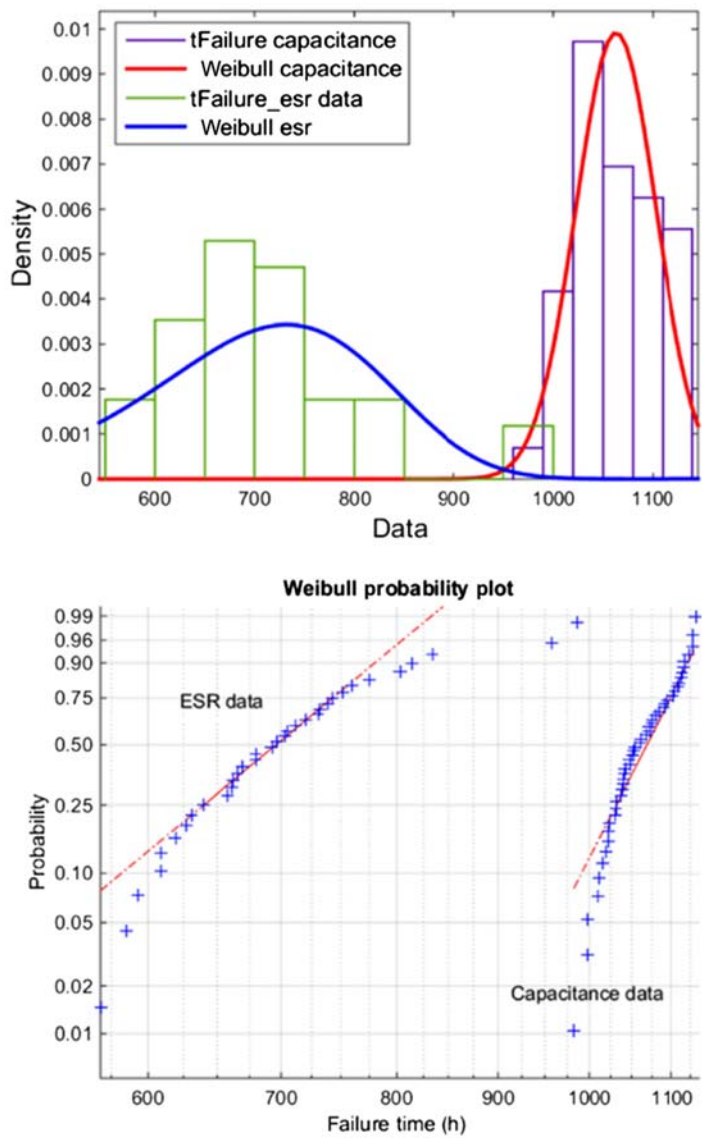


FIGURE 9.20
Weibull distribution of failure time using capacitance failure criteria and ESR failure criteria, top: probability density, bottom: cumulative probability.

9.3.3.4 Physics of failure models

9.3.3.4.1 Degradation analysis based on data collected from research (expensive) setup

Before starting the PoF modeling, it is important to highlight some observations made when collecting the degradation data using the research setup. As these data-sets were collected for a long test time, it gives more insight into the degradation behavior. The degradation signatures, from the different test boards (from 1 to 9, each equipped with 48 supercapacitors), are presented, as ESR versus capacitance, in Fig. 9.21. From this figure, it is clear that the degradation rate of the supercapacitors changes versus the applied stresses.

For instance, the test board nr. 2, which is overstressed w.r.t. maximum specified ratings (2.3 V/85°C), shows a very quick degradation following a linear curve. Test board nr. 7 and board nr. 1 are stressed at maximum specified ratings and show relatively linear degradation where less linearity (slight change in degradation rate) is observed for board 1 suggesting thus a change of the failure mechanism rate. This effect is more highlighted when the stresses become lower, for instance board nr. 8, which is exposed to a cyclic voltage stress (min. 1.35 V

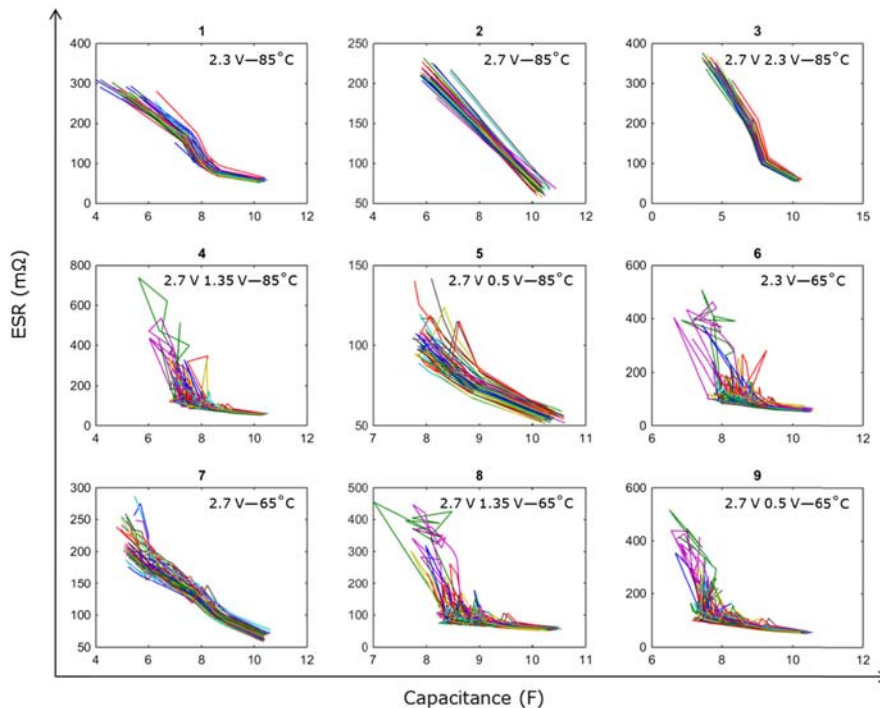
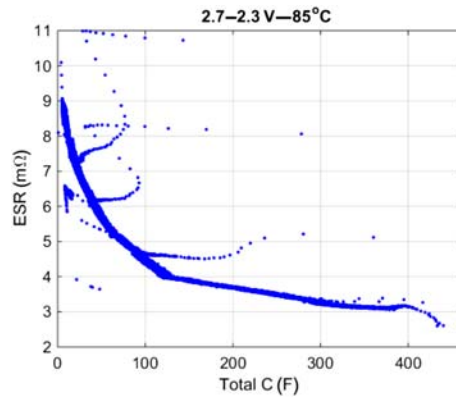


FIGURE 9.21

Degradation signatures of different supercapacitors at different stresses.

**FIGURE 9.22**

Degradation of supercapacitors bank of board nr. 3 based on continuous measurements.

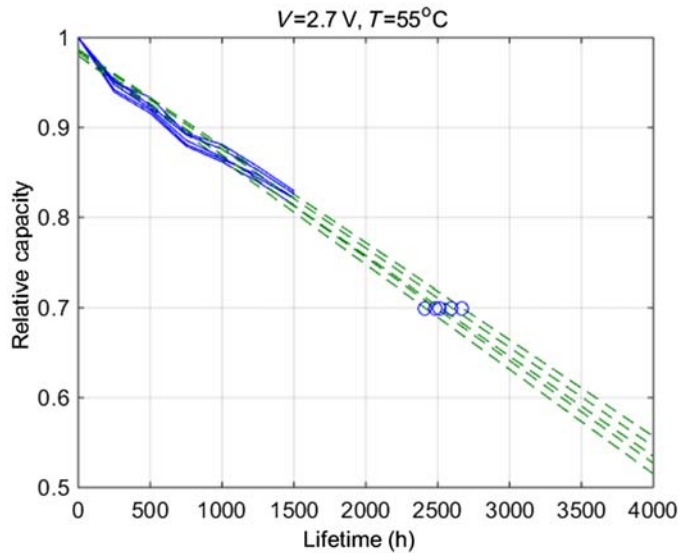
and max. 2.7 V) and a temperature of 65°C. This also suggests that voltage cycle has the same degradation effect as if the supercapacitors are stressed at the equivalent constant average voltage, with a remark that ESR values are relatively higher under cyclic voltage stresses.

With regard to literature [30], this difference in degradation rates is explained as the porosity of the electrode surface of the supercapacitors that is varying quickly in the beginning since the first impurities generated by the current extraction quickly fill part of the macro pores. These pores form a blocking element of the electrode–electrolyte surface contact which leads to an increase of the length of the micro-contact paths of the electrode–electrolyte interface, the conductivity, and the ion mobility increase as well, and they are reducing the negative effect associated with the extraction current, resulting into a decrease of the degradation rate.

The degradation signatures shown in Fig. 9.21, where measurements of the capacitance and ESR are done once per week along the test period, is confirmed with other continuous logged measurements for the boards subject to cyclic voltage stress (nr. 3, 4, 5, 8, and 9). These voltage cycle stresses are continuously logged from the energy storage testing systems, thus allowing estimation of the equivalent capacitance and ESR of components connected to a single channel. The results are shown, for the equivalent total capacitance of the 48 supercapacitors of board nr. 3, in Fig. 9.22.

9.3.3.4.2 Degradation analysis based on data collected from economical (cost-effective) setup

The economical test was stopped after 1500 test hours. These data from nonfailed units is then considered as suspended (right censored) data. In order to estimate

**FIGURE 9.23**

Prediction of TTF based on censored data.

the TTF based on these datasets, models are fitted to the data to predict the TTF. An example of this prediction is illustrated in Fig. 9.23.

In this example the linear fit was chosen although it is contradicting the conclusions from previous section suggesting potentially a change of degradation rate as the stresses are within specs. Consequently, the TTF estimates are supposed to be conservative, which would also result in a conservative PoF model. Other fitting models were not investigated.

The collected data allow us to build different PoF models, where voltage, temperature, and combined effect can be studied. In Table 9.5, some of these PoF models are proposed.

9.3.3.4.3 Physics of failure modeling

Starting from the TTF data collected, as described above, we could build PoF models that would describe the probability of failures versus the applied stress. The data collected so far, especially through the research setup, would allow us to do different analyses as described in Table 9.5. Depending on the final application, one or another model might be more relevant to the end-use stresses allowing thus qualifying the supercapacitors with regards the final stresses.

However, we will focus in this section on demonstrating the PoF model 1 to study the effect of temperature on the supercapacitors lifetime. The same approach can be followed to further build other PoF models.

Table 9.5 Some possible PoF models that can be built with the designed experiments.

PoF	Test	Description
PoF 1	#1/#6	PoF @2.3 V—effect of temperature
PoF 2	#2/#7	PoF @2.7 V—effect of temperature
PoF 3	#1/#2	PoF @85°C—effect of voltage
PoF 4	#6/#7	PoF @65°C—effect of voltage
PoF 5	#4/#8	PoF @50%DoD—effect of temperature
PoF 6	#5/#9	PoF @80%DoD—effect of temperature
PoF 7	#3/#4/#5	PoF @85°C—effect of DoD
PoF 8	#8/#9	PoF @65°C—effect of DoD

As a second target in this section, we will compare the PoF built using expensive and economic datasets.

The Weibull probability plots for both datasets are given in Fig. 9.24. Note that the failure data shown in the top graph correspond to PoF1 data in Table 9.5 where the applied stresses are (constant voltage 2.3 V, 65°C/85°C temperatures), while the data from bottom graph are collected from economic tests stressed at (2.4 V, 55°C/65°C).

Although the failure data are collected from different setups, the Weibull plots from the two graphs are quite aligned, thus confirming the good quality and accuracy of collected data.

In case of similar failure mode, the Weibull plots at different stresses should be parallel, which means that they have similar shape factor.

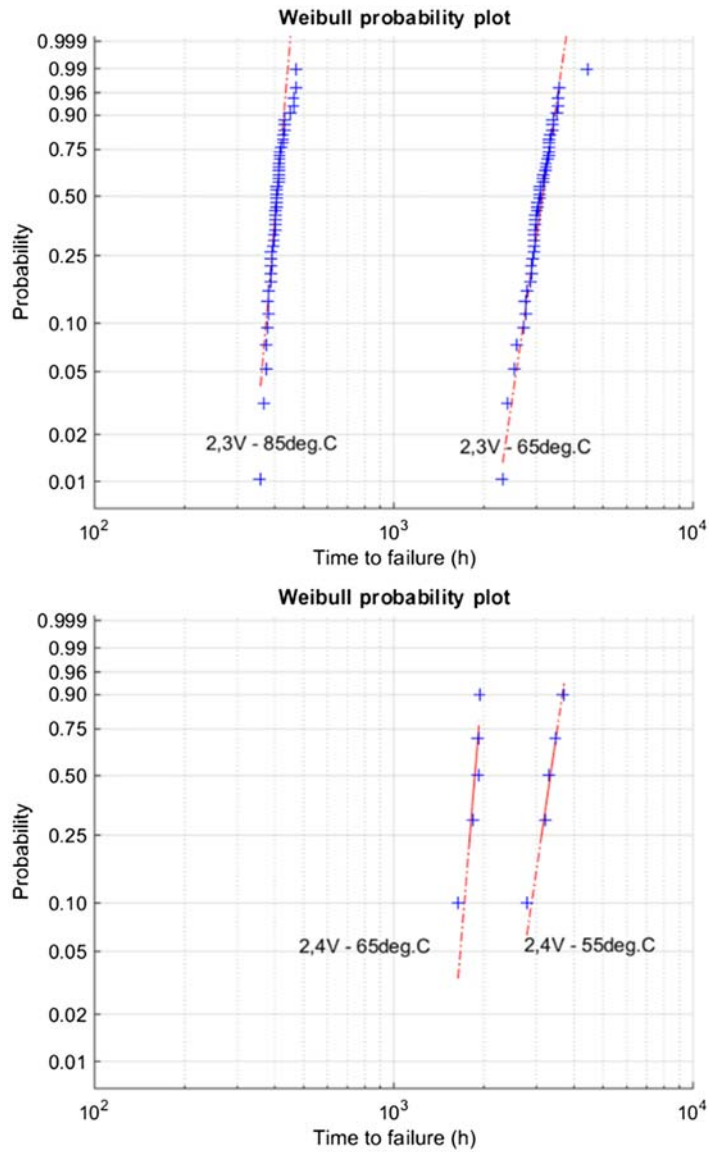
In our case, these Weibull plots are qualitatively parallel, but with a close zoom, there are some changes in the data trends (only with long tests) supporting the discussion above about degradation rate change. However, we strongly believe that the failure mode is the same for all the tests shown in Fig. 9.24 and that is the evaporation of electrolyte, as described in Section 4.3.1.

For a temperature dependent failure mode, the dependency of temperature is expressed with an Arrhenius formula. This formula is derived from the Arrhenius reaction rate equation proposed by the Swedish physical chemist Svante Arrhenius in 1887 [7].

The acceleration factor at any temperature is expressed as

$$AF = \frac{\eta(T_0)}{\eta(T)} = \exp\left(\frac{E_a}{K} \times \left(\frac{1}{T_0} - \frac{1}{T}\right)\right) \quad (9.13)$$

where AF , η , T_0 , T , E_a , K , respectively, denotes, acceleration factor, scale (life), use temperature, test temperature, activation energy, and Boltzmann's constant. Next to the estimated life, the activation energy is an important parameter to be

**FIGURE 9.24**

Weibull plots for expensive (top graph) and economic (bottom graph) failure datasets.

estimated as it is a measure of the effect that temperature has on the reaction of the failure mechanism.

The Arrhenius model is fitted to dataset from research setup and the results are shown in Fig. 9.25. From the graph, the failure distributions at the two test temperatures can be seen respectively at 85°C (358.15K) and 65°C (338.15K). Based on this model, different parameters can be estimated as described in Eq. (9.13).

The estimated scale parameter (η –life) at use temperature (in our case 40°C) and estimated activation energy (E_a) are given in Table 9.6. The activation energy is a typical value for the failure mode as it determines the slope of the Arrhenius graph.

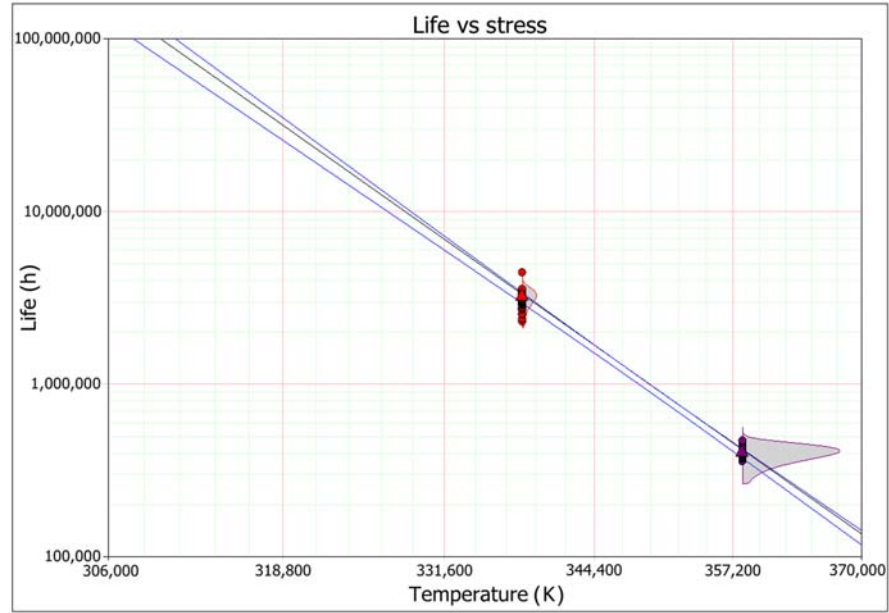


FIGURE 9.25
Arrhenius model applied to research dataset.

Table 9.6 Estimated PoF parameters using Arrhenius–Weibull PoF model.

η	50,000 h
E_a	0.9 eV

9.4 New trends to improve reliability analysis

I would like in this section to start with two observations that could change, revolutionarily in coming years, the way reliability of electronics is done today.

The first observation is that electronic components nowadays are formed from complex interconnections (e.g., MEMS electronics [31]). These interconnections are wearing out due to stresses (e.g., vibration, thermal cycling, or their combinations). This means that the failures of these components become mainly wear-out failures (as of “electromechanical” systems), in contrary to old electronic components that are vacuum-based and where their failures could be seen as random failures.

The second observation is that electronic chips/PCBs architecture is more and more modular and can be easily adapted to include a build-in system to monitor a specific component and/or to record continuously stress data such as temperature.

These two observations form opportunities to accurately assess the reliability of electronic components and as a consequence electronic systems: while the former observation suggests that these electronic systems are continuously aging opening an opportunity for condition monitoring of these systems. Condition monitoring is a well-established technology used in electromechanical systems [32].

While the later observation suggests cost-effective “online condition monitoring” where the data are continuously logged/transferred for more accurate estimation of the final use stress and/or the condition of the electronic asset. Here the cost effectiveness does not only mean the cost of the build-in module but also the cost of the infrastructure to remotely transfer the data and/or interconnect different systems together or in a network. This is possible today, thanks to the revolutionary Internet of things technologies [33].

These two ingredients—final use stress and the current condition of the asset—are the basic ingredients to assess reliability of a system by using dedicated models and methods as explained in previous sections. The advantage compared to previous analysis is that now we will have exactly the distance to failure that would allow to preform accurate LDA and the exact stress level that would allow to perform life-stress analysis.

In the coming sections, I would highlight some advantages and examples of using the exact stress level (mission profile) and the exact condition of the asset, with regards to conventional approximations.

9.4.1 Mission profile

The failure rate of an electronic equipment depends on two factors: (1) design complexity and (2) stress (inherent) reliability of the components which is related to used technology.

In electronic equipment, the most prominent stresses are temperature, voltage, vibration, and temperature rise due to current. The effect of each of these stresses on each of the components must be considered. In order to achieve good reliability, various derating factors have to be applied to these stress levels. The derating has to be traded off against cost and size implications. Great care and attention to detail is necessary to reduce thermal stresses as far as possible. The layout has to be such that heat-generating components are kept away from other components and are adequately cooled. Thermal barriers are used where necessary and adequate ventilation needs to be provided. The importance of these provisions cannot be overstressed since the failure rate of some components will double for a 10°C increase in temperature. Note that decreasing the size of a unit without increasing its efficiency will make it hotter, and therefore less reliable [34].

Improved reliability analysis, in last years, is referred to as mission profile aware reliability and are largely used in aerospace and automotive applications [35]. For instance, a mission profile for a navigation computer onboard a helicopter consists of different phases, where at each phase stress types and levels are identified: Event 1: “Start the navigation computer” (change to temperature stress), Event 2: “Start the engine” (change to vibration stress), Event 3: “Takeoff” (change to vibration and temperature stress), Event 4: “Landing” (change to vibration and temperature stress), Event 5: “Stop the engine” (change to vibration stress), Event 6: “Switch the computer off” (change to temperature stress), and return to the parking phase.

While current methods, like in FIDES, assume specific values of stress levels based on premeasurements and experiences, online measurement of these stress levels represent a breakthrough for accurate stress estimation, to deal for instance with variable operations, and therefore one step forward an accurate reliability analysis.

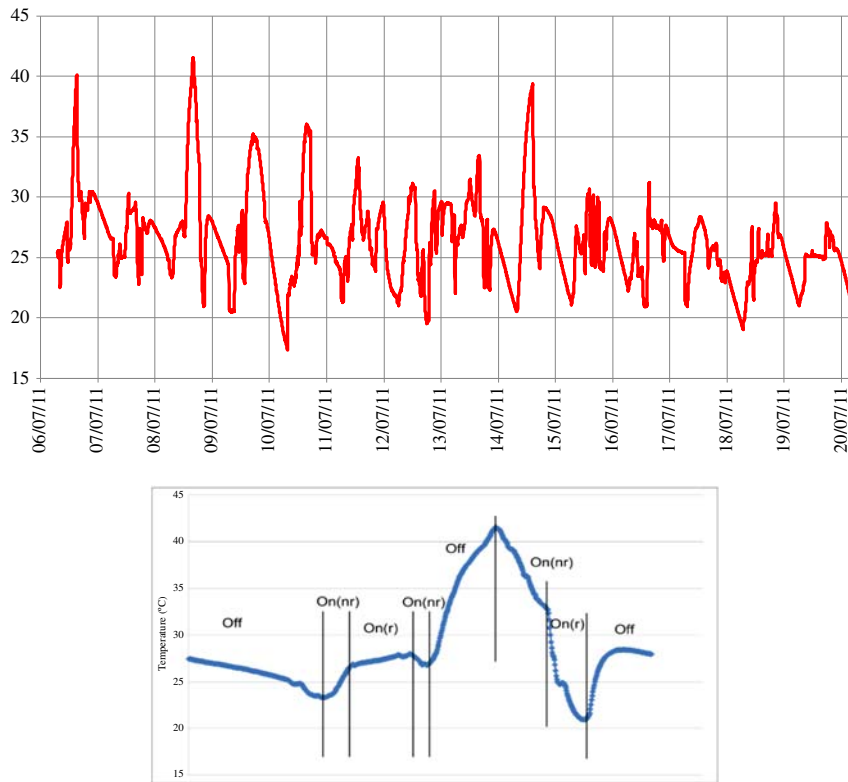
Temperature cycles measured onboard a train in an electronic module are shown in Fig. 9.26. Detailed analysis of 1-day cycle allows to estimate off and on state even when the train is running (indicated with [r] in the graph) or when it is not running (nr).

These phases are used in a reliability tool to estimate the reliability of the module with a constant temperature approximation and using the measured cycles as shown in Table 9.7.

The estimated failure in time (FIT inversely proportional to reliability), with the two methods are quite different and show more than 66% improvement using the exact measured temperature cycles.

9.4.2 Online condition monitoring—case study

Aging electrical systems are prevalent in today’s society [36]. They are abundantly present in buildings, aircraft, and transportation systems, consumer products, industrial machinery, etc. Wiring and connections failures could be seen as dominant failures in such electrical systems and are the most significant potential

**FIGURE 9.26**

Temperature profile of an electronic module onboard a train. Top: different temperature cycles along 15 days. Bottom: detailed analysis of 1-day temperature profile.

causes of catastrophic failures and maintenance cost in these systems. Furthermore, airline crashes attributed to aging wires and connections have brought this issue into the public eye [37]. A wiring system does not only distribute electrical power but also provides control and information links between multiple systems and subsystems. The components to make up the wiring system include power and control conductors, signal and instrumentation conductors, fiber-optic cables, connectors, circuit breakers, relays, power distribution, and control panels, etc. Failure of any of these components can disable the functioning ability of a system [38]. Different techniques exist which allow to detect, or monitor the aging of wires and connectors and sometimes localizing the defects location. Reflectometry is commonly used for detecting faults in a wiring harness and localizing their positions [39]. Different reflectometry methods exist, such as time domain reflectometry (TDR), frequency domain reflectometry, etc. [40,41]. The reflectometry methods are often used when the wiring system is not powered.

Table 9.7 Estimated failure in time (FIT) using approximated constant temperature (top table) and exactly measured temperature cycles (bottom table).

Λ Total		1.800,75 FIT				
			Results			
Model	Component type	Component name	Λ Total	Quantity	Λ Unit.	%
ELECTRONIC COMPONENT	ALUMINIUM CAPACITOR [ECAC]	Polyster cap	273,7	6	45,6	15%
ELECTRONIC COMPONENT	ALUMINIUM CAPACITOR [ECAC]	electrolytic cap	317,7	10,000	31,8	18%
ELECTRONIC COMPONENT	Ceramic capacitors [ECCC]	Ceramic cap	1015,2	15,000	67,7	56%
ELECTRONIC COMPONENT	Connectors [ECCO]	connector	2,1	1,000	2,1	0%
ELECTRONIC COMPONENT	Discrete semiconductors [ECD5]	diode	157,4	4,000	39,3	9%
ELECTRONIC COMPONENT	Integrated Circuits [ECIC]	IC	15,6	1,000	15,6	1%
ELECTRONIC COMPONENT	Light Emitting Diode (LED) [ECLE]	led	1,0	1,000	1,0	0%
ELECTRONIC COMPONENT	Magnetic Components: Inductors and Transformers	hf-coil	1,2	6,000	0,2	0%
ELECTRONIC COMPONENT	Magnetic Components: Inductors and Transformers	transfo	1,5	1,00	1,5	0%
ELECTRONIC COMPONENT	Resistors [ECRE]	resistor metal film	4,4	5	0,9	0%
ELECTRONIC COMPONENT	Resistors [ECRE]	resistor power film	11,0	4	2,8	1%

Λ Total		608,89 FIT				
			Results			
Model	Component type	Component name	Λ Total	Quantity	Λ Unit.	%
ELECTRONIC COMPONENT	ALUMINIUM CAPACITOR [ECAC]	Polyster cap	127,0	6	21,2	21%
ELECTRONIC COMPONENT	ALUMINIUM CAPACITOR [ECAC]	electrolytic cap	142,3	10,000	14,2	23%
ELECTRONIC COMPONENT	Ceramic capacitors [ECCC]	Ceramic cap	211,5	15,000	16,1	40%
ELECTRONIC COMPONENT	Connectors [ECCO]	connector	1,4	1,000	1,4	0%
ELECTRONIC COMPONENT	Discrete semiconductors [ECD5]	diode	20,5	4,000	5,1	3%
ELECTRONIC COMPONENT	Integrated Circuits [ECIC]	IC	6,7	1,000	6,7	1%
ELECTRONIC COMPONENT	Light Emitting Diode (LED) [ECLE]	led	2,9	1,000	2,9	0%
ELECTRONIC COMPONENT	Magnetic Components: Inductors and Transformers	hf-coil	3,9	6,000	0,6	1%
ELECTRONIC COMPONENT	Magnetic Components: Inductors and Transformers	transfo	6,1	1,00	6,1	1%
ELECTRONIC COMPONENT	Resistors [ECRE]	resistor metal film	17,9	5	3,6	3%
ELECTRONIC COMPONENT	Resistors [ECRE]	resistor power film	38,8	4	9,7	6%

This makes the detection of intermittent connection problems not possible. These intermittent failures only occur in live (active) systems and often when the system is exposed to an external stress (e.g., temperature swings, vibration), for instance in a car. They can have a very short duration (down to μs) [42]. Such intermittent connection faults can be fatal in some applications because they are not detectable offline and when happening, online, they may lead to a loss of system functionality in critical conditions (e.g., for safety critical systems). Therefore, it would be logical to develop techniques which can detect, monitor, and if possible localize these faults in live systems. Although some techniques belonging to the reflectometry family can deal with intermittent faults, for instance spectral TDR, these techniques require expensive components (e.g., high-frequency generator, mixer, etc.) which makes these techniques not suitable for industrial and automotive applications with their low-cost requirements. Other alternative technologies for connections defects detection and localization consist of impedance measurements (LCR as a reference to inductance [L], capacitance [C], and resistance [R] measurements) of the wire and/or the connector. LCR meters exist already for a long time to perform equivalent impedance measurements of an electrical circuit [43]. Some papers used this principle to develop a low-cost equipment to measure equivalent capacitors or inductances of a circuit which will be associated with open or short connection faults [44]. The method used in that paper does not work in live systems. Using impedance measurement technique for detecting impedances in live circuits has its own challenges and limitations (e.g., used frequencies, needed filters, change of impedance due to additional measurement equipment, etc.). An analysis of such impedance measurement in live mains has been done using expensive lab equipment's [45].

In this section we investigate the detection and localization of intermittent connection faults in live industrial applications. We propose two different low-cost methods (using commercial off-the-shelf components) for this purpose that allow:

1. detection and localization of intermittent connection faults using an extended impedance measurement technique and
2. detection of intermittent faults based on applying a band-pass filter on measured signals from a circuit.

9.4.2.1 Concepts for connection defects detection, prognostics, and localization

In this section the concepts of the two proposed methods will be explained.

9.4.2.1.1 Method for connection defects detection and localization—based on online impedance measurements

Impedance measurement can be used to detect different connection faults by estimating the additional impedance, due to connection faults, to the equivalent impedance of a circuit. The type of the fault can be differentiated by looking to

the imaginary part of the impedance (capacitive in case of open connection fault or inductive in case of short connection fault). Different methods exist depending on the used diagnostic circuits [46]: bridge method where a bridge of impedances (including the impedance of the device under test) is used to estimate a balance point, resonant method where a circuit is adjusted to resonance by adjusting a tuning capacitor, I–V method to calculate the unknown impedance by measuring the current and voltage across the impedance. We adopted the I–V method in this work which we adapt to be suitable for working in a live (powered) system. The basic schematic is shown in Fig. 9.27. To make it suitable for online usage, the diagnostics circuit parameters need to be selected such that it has no influence on the original sensor measurements. These parameters are related to the transmitted sine wave that is superimposed to the original circuit signals. The frequency of the transmitted sine wave should be selected beyond the bandwidth of the electrical circuit such that it is not influencing it is working. The amplitude of the transmitted sine wave should be small enough to avoid disturbing the circuit but large enough to be detected. These parameters were tuned properly to the target applications.

The diagnostic circuit would provide diagnostic voltage and current which should be analyzed both in amplitude and phase to extract the equivalent impedances (capacitive for open and inductive for short). An application specific example of a signal captured when using a resistive load is depicted in Fig. 9.28. An algorithm has been developed which conditions the measured signals by fitting automatically a sine wave to the measured signals and use these fits to reliably estimate the amplitude and the phase of voltage and current. Using these two parameters, the resistive and capacitive parts of the equivalent impedance can be calculated. In this examples an equivalent resistance of $1063\ \Omega$ and an equivalent

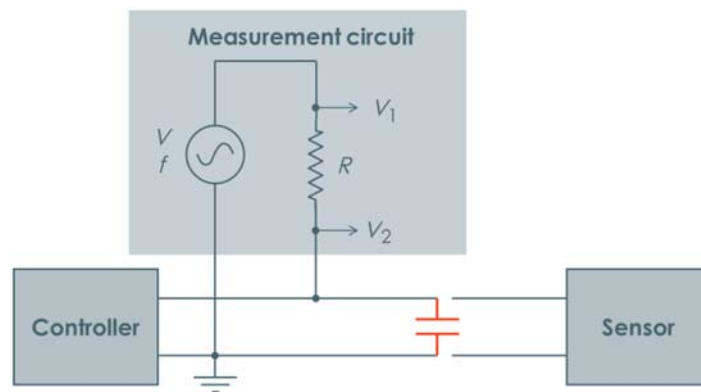
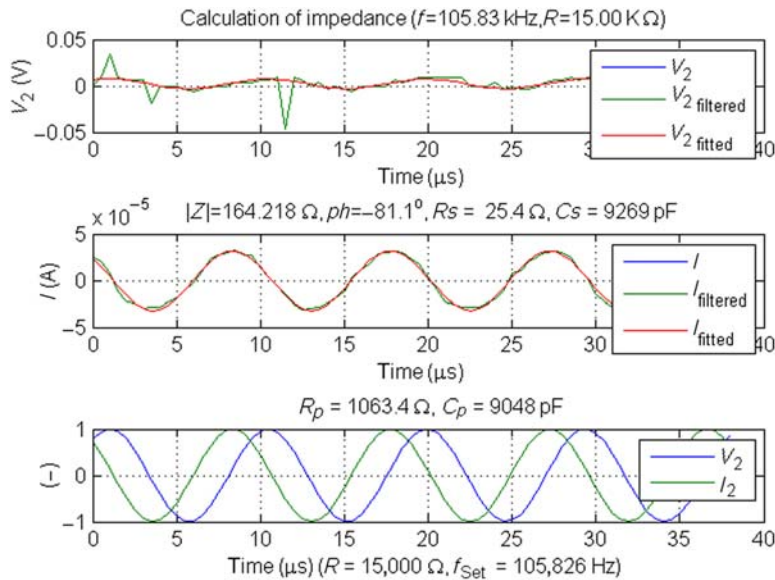


FIGURE 9.27

Impedance measurement circuit used to diagnose open connection fault in an industrial circuit.

**FIGURE 9.28**

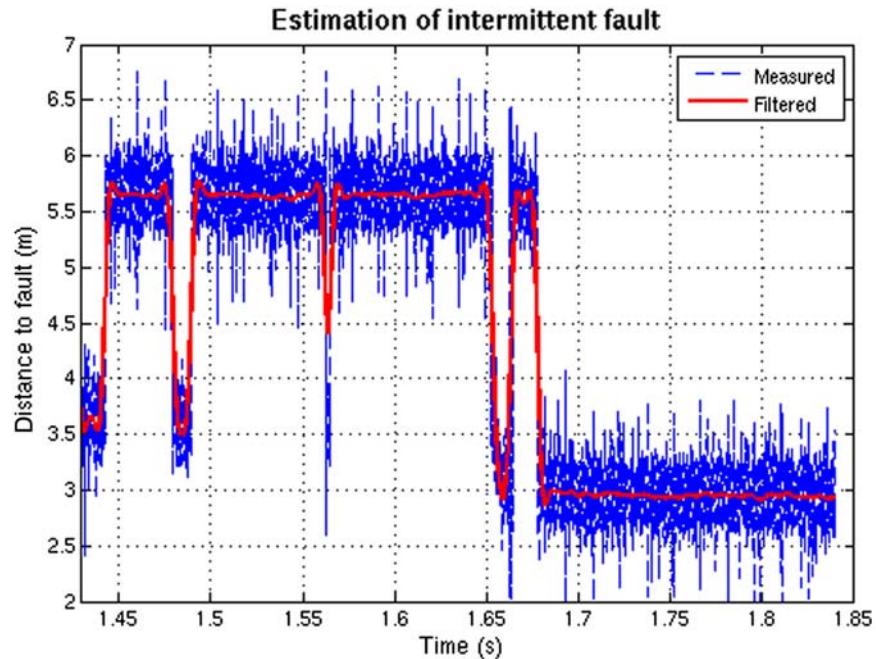
Recorded signals and curve fitting and equivalent impedance estimation ($|Z|$: equivalent estimated absolute impedance, ph : estimated phase between current and voltage, R_s , C_s : Equivalent series resistance and capacitance), R_p , C_p : equivalent parallel resistance and capacitance).

capacitor of 9048 pF, which corresponds to the load impedance, is found. Assuming now that a connection problem occurs between the controller and the sensor, the online estimated resistance and capacitance will be different and would be proportional to the length of the wire where the fault occurs allowing thus localization of the fault. If instead of open, a short fault will occur, an inductance will be estimated instead of a capacitance value.

Considering two wire's lengths of respectively 3 and 5 m between the control unit and the sensor (by placing two sensors/switches in parallel at different distances), the estimation of the distance to fault is done using our technique online and the results are shown in Fig. 9.29. The intermittent duration of the faults that can be detected is limited by the used acquisition system (ms in the graph). The accuracy of the fault localization reaches $2\sigma \pm 30$ cm, which in many industrial applications is sufficiently accurate to determine the location of the defected connector.

9.4.2.1.2 Method for connection defects detection—based on band-frequency filtering

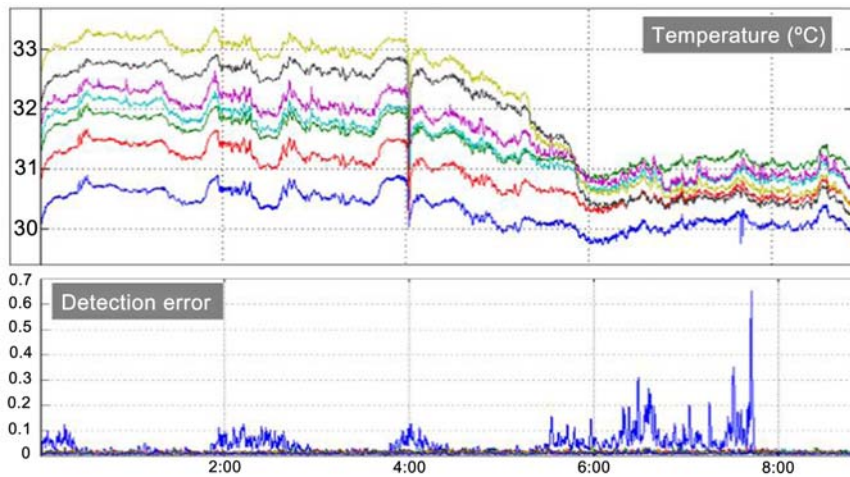
In various applications, localizing the connection fault positions is not necessary. In these cases, detecting the connection faults will be sufficient. Based on

**FIGURE 9.29**

Estimation of distance to fault for an intermittent open connection fault.

connector tests we performed for different connectors, and as reported in different literature studies [47], the measured signals of an electric circuit with a degrading contact resistance would present relatively high-frequency noises. These variations of fault amplitude and duration can be explained by the number of irregularities in the surfaces of the connectors, which due to thermal changes and/or vibration of the connectors introduce intermittent loss of contact which increases versus time. By analyzing carefully, the characteristics of these contacts noises on different industrial applications, it has been concluded that an algorithm based on band-pass filtering, using generic parameters, could be used to detect these noises whose amplitude is correlated to contact degradation level. This is very advantageous for industrial applications since no external diagnostics hardware would be needed.

The developed algorithm is validated on different signals coming from temperature sensors as illustrated in Fig. 9.30. The algorithm successfully detects the connection faults in a very early stage as illustrated by the increasing detection signal in the bottom graph. Initially the behavior is quite intermittent but after some time the faults can be permanently captured.

**FIGURE 9.30**

Detected connection fault in one of the temperature sensor's signals. Top: temperature signals. Bottom: detection signal from connection's faults detection algorithm.

9.5 Summary

We presented in this chapter an improved product reliability quantification methodology where PoF-based prognostics modeling were highlighted. This methodology is a combination of mainly three improved methods based on (1) improved prediction handbook, (2) improved ALT, and (3) improved LDA. The reported improvements compared to state of the arts and common practices are quite high and reach at least 50%. These improvements are immediately understood when you compare the parameters/assumptions of current methods to the ones we proposed. Often noncorrect assumptions are made (e.g., constant failure rate), and/or methods for outdated technologies or processes are applied. By means of PoF modeling, proposed in the improved methods, we introduced more physical insight in the analyses yielding better reliability prediction estimates.

However, the PoF models used in the proposed methodologies are approximates to the use cases we investigated and will always contain parameters that need to be tuned by guidelines (e.g., process guidelines proposed by FIDES) or by getting more experiences with the product studied (e.g., material parameters used in life-stress models). Having a generic PoF model that would take all details of product construction and interaction of components is practically very difficult, if not impossible, to get.

This methodology was developed with the mind-set to serve industrial processes. Therefore, a set of practical user friendly tools were developed and which some of have been presented in this section.

The reliability monitoring is an interesting topic to save market shares and liability of manufacturing companies; therefore it needs the right tools to allow accurate and economical estimates for such a monitoring. We believe this presented methodology has the potential to contribute to achieve these goals.

In order to illustrate the proposed PoF-based methodology, reliability assessment and qualification of some important electronic components were illustrated.

Electrolytic capacitors and supercapacitors performance and lifetime assessment and qualification are very important for choosing the right component for a specific application.

We demonstrated a PoF-based methodology to study the performance and lifetime of electrolytic capacitors and supercapacitors.

We tackled different challenges in this assessment process. The first one is related to the accuracy of ESR estimates where the accuracy depends on the estimation method. The second is related to the degradation analysis and changes in degradation rates versus the applied stresses. The third is related to the definition and effect of the failure criteria on the final lifetime estimation, where TTF values depend on which failure criteria is chosen. The last, but not least, is related to the accuracy of PoF model to predict lifetime at different stress conditions and with different test settings. These analyses form a qualification framework that fits in our overall PoF assessment methodology for electronic components.

We also demonstrated that results from the economic datasets are in line with results from extensive datasets. This would allow designers to consider this qualification approach as the qualification test is realistically short in time and economically cost-effective.

Another important source of defects, connections problem, was also tackled. Feasibility of prognostics of connection defects (faults) in electronics modules has been shown both for detection and localization. Impedance based measurement techniques have been demonstrated with possibility to be applied in live powered systems. Two different cases with equivalent resistive and complex impedances have been considered for validating this technique. Accuracy in the order of 30 cm has been achieved for localizing the connection fault. For applications where no alteration of the circuit is possible, by adding a diagnostics hardware, we developed an algorithm based on band-frequency filtering to highlight connection faults and detect them in a very early stage. The validation of this algorithm on temperature sensors signals proved the capability of this algorithm to detect the faults in a very early stage and proved also that this algorithm is generic. These methods show a high potential of connection faults detection and localization for industrial applications.

The collected condition of the connection in the electronic system provides accurate information for further reliability analysis. Actually, the data can easily be transformed into failure data, for instance by applying a prognostic model, using a user-defined threshold. Based on these failure data, LDA can be applied to derive statistical analysis including reliability of the asset.

9.6 General observations and conclusion

Reliability analysis of an electronic system can be misleading if the correct methodology is not used. This wrong analysis can be catastrophic in some cases, especially for safety critical systems.

Different methodologies can be found to perform reliability analysis with their pros and cons. Between outdated reliability handbooks and expensive accelerated life tests, a new methodology is proposed that can bring economic benefits with acceptable accuracy.

It is important here to remind that these methodologies are only helping the different stakeholders in an organization to correctly perform reliability assessment. Most importantly is that reliability assessment and control should be adopted as a process in an organization, meaning that reliability analysis should be done at every step of the product life cycle.

In different industrial cases, after the initial failure assessment of an electronic system, for example, through an FMEA in the design or prototyping phase, the critical components are highlighted. These components that need special attention are often very limited. This would allow to smartly chose the right method to assess reliability of the complete system. For example, use handbooks to get approximative reliability values for noncritical components, and use accelerated life test with more accurate reliability estimates for critical components. We highlighted different possibilities to end up with an economic reliability assessment, as the cost to get an accurate reliability in a system level can be very high.

Although historically present in electronic systems, electrolytic capacitors, with their long time usage in industry, remain critical in many applications. Their reliability is still a challenge to industries. This challenge comes from the large diversity of these components available in the market, which may slightly differ in technologies and/or production processes. Such a variation is enough to make a difference of 20%–40% in lifetime under use conditions. These large variations motivate the analysis done with these components, and shown in this chapter, where ALT using components from large number of suppliers was performed and results show these diversities in terms of lifetime. It is recommended to industries to think about economical accelerated tests, for instance based on the proposed methodology, to test the diversity and come to more precise lifetime estimates, especially when these components are used in critical applications.

From the other side, new emerging electronic components, like supercapacitors, are more and more used by industry. However, there is a strong lack and misinterpretation of the reliability data available in the datasheet, for example what are the exact charge/discharge cycles in the use temperatures that are often different from the ones described in the datasheets. We demonstrated in this chapter two critical stress parameters (voltage and temperature) for these components and propose an analysis method in order to get a better estimate of lifetime under

the use values of these stresses. An economical test methodology was proposed and is strongly recommended to end users industries.

Beside the failure analysis done by industries, often in the design phase, where the critical components are identified, another important source of reliability/lifetime of electronics are the field data called also return of experiences.

Although not properly collected by most of the industries, mainly due to its high associated cost, it was observed from data coming from different machine builders who propose service contracts that a very large amount of failures are coming from connection problems. This supports the analysis presented in this chapter about electronic connections defects.

These defects are quite challenging due to their intermittent nature, meaning that the connection problem could occur, for a short time, when the system is operational (e.g., intermittent loss of contact). The proposed methodology consists of online monitoring which is highlighted as an example of how a condition monitoring system could be used as a source of inputs for reliability analysis.

With the fourth revolution of industry (Industry 4.0) and the era of interconnected systems, the online monitoring is expected to explode in electronic systems in coming years.

This will revolution the way reliability is approached: by recording the exact mission profile, discussed in this chapter, where the exact stress types and values are recorded, and the condition (health) of the electronic asset collected through a condition monitoring system, lifetime estimates become a custom feature of the electronic asset.

When interconnected to other systems, and/or to the cloud, this information can be used to accurately assess reliability of the interconnected fleet. This represents a kind of stressed systems that when configured correctly could be seen as a replacement of the traditional reliability demonstration test with the advantage of low-cost and correct stresses values.

But of course this would need some years before the relevant data are collected and deployed.

Acknowledgments

I gratefully thank different colleagues who contributed to the work presented in the different sections of this chapter. This work comes from different Flanders Make projects funded by the Flemish government through VLAIO.

References

- [1] A. Temsamani et al., Improved Product Reliability Quantification Methodology Making Use of Physics of Failure Based Prognostics, PHME (2016).

- [2] Handbook of Reliability Prediction for Electronic Components (MIL-HDBK-2017 (1995), IEC 62380 (2004), FIDES (2009)).
- [3] J.H. Wohlgemuth, S. Kurtz, Using accelerated testing to predict module reliability, 37th IEEE Photovoltaic Specialists Conference (PVSC), 2011, IEEE, 2011.
- [4] R.B. Abernethy, The New Weibull Handbook, fifth ed., 2006, 350.
- [5] Y. Lu, H.T. Loh, A.C. Brombacher, E. den Ouden, Accelerated stress testing in a time-driven product development process, *Int. J. Prod. Eco.* 67 (1) (2000).
- [6] P. Charpenel, F. Favenel, R. Digout, M. Giraudeau, M. Glade, J. Guerveno, et al., The right way to assess electronic system reliability: FIDES, *Microelectron. Reliab.* 43 (2003) 1041–1404.
- [7] F. Bayle, A. Mettas, Temperature acceleration models in reliability predictions: justification & improvements, *Proceedings of Annual Reliability and Maintainability Symposium (RAMS)*, 2010, IEEE, 2010.
- [8] J.J. Marin, R.W. Pollard, Experience report on the FIDES reliability prediction method, *Proceeding of Annual Reliability and Maintainability Symposium*, IEEE, 2005.
- [9] R. Jiang, D.N.P. Murthy, A study of Weibull shape parameter: properties and significance, *Reliab. Eng. Syst. Safe.* 96 (12) (2011).
- [10] D. Ryu, S. Chang, Novel concepts for reliability technology, *Microelectron. Reliab.* 45 (3) (2005).
- [11] T. Ashburn, D. Skamser, Highly accelerated testing of capacitors for medical applications, in: *Proceedings of the 5th SMTA Medical Electronics Symposium*, 2008.
- [12] H. McLean, From HALT results to an accurate field MTBF estimate, *Proceedings of Reliability and Maintainability Symposium (RAMS)*, 2010 *Proceedings-Annual*, IEEE, 2010.
- [13] D.W. Coit, J.L. Evans, N.T. Vogt, J.R. Thompson, A method for correlating field life degradation with reliability prediction for electronic modules, *Qual. Reliab. Eng. Int.* 21 (7) (2005).
- [14] W. McLean, HALT, HASS, HASA explained, 2009, Asq Pr; Revised edition (May 13, 2009).
- [15] S.J. Shi, W. Zhao, Research on pre-HALT analysis and the application of test data in MTBF evaluation, in: *8th International Conference on Reliability, Maintainability and Safety*, 2009, ICRMS 2009, IEEE, 2009.
- [16] W. Wang, J.M. Loman, R.G. Arno, P. Vassiliou, E.R. Furlong, D. Ogden, Reliability block diagram simulation techniques applied to the IEEE std. 493 standard network, *IEEE Transact. Industry Appl.* 40 (3) (2004).
- [17] A. Temsamani, et al., Improved and accurate physics-of-failure (PoF) methodology for qualification and lifetime assessment of electronic systems, *Microelectron. Reliab.* 76 (2017) 42–46. ISSN: 0026-2714.
- [18] FIDES handbook, <http://www.fides-reliability.org/>.
- [19] Gasperi M.L., Life Prediction Model for Aluminum Electrolytic Capacitors, *IAS1996*, IEEE. 3 (1996) 1347–1351.
- [20] K. Abdennadher, et al., A real-time predictive-maintenance system of aluminum electrolytic capacitors used in uninterrupted power supplies, *IEEE Trans. Ind. Appl.* 46 (4) (2010).
- [21] C.S. Kulkarni, et al., Physics based degradation models for electrolytic capacitor prognostics under thermal overstress conditions, *Int. J. Prog. Health Manage.* (2013) 1–17.

- [22] A. Tamsamani, et al., Physics-of-Failure (PoF) methodology for qualification and lifetime assessment of supercapacitors for industrial applications, *Microelectronics Reliability* 88 (2018) 54–60. ISSN: 0026-2714.
- [23] H. Barde, Supercapacitors: applications and requirements, in: *Proceedings of the European Space Components Conferences ESCCON 2000*, vol. 439, 21–23 March 2000, p. 79.
- [24] Murata supercapacitor Technical note, document No. C2M1CXS-053N, Copyright © Murata Manufacturing Co.
- [25] HC Series Ultracapacitors, document number: 1013793.9 Maxwell Technologies.
- [26] IEC62576: Electric double-layer capacitors for use in hybrid electric vehicles—test methods for electrical characteristics, Edition 2.0 2018-02.
- [27] Application note 1007239, Test procedures for capacitance, ESR, leakage current and self-discharge characterizations of ultracapacitors, Maxwell Technologies, Inc., June 2015.
- [28] *The Weibull Distribution: A Handbook*, H. Rinne, CRC Press, 20 November 2008.
- [29] How to conduct a failure modes and effects analysis (FMEA), a white paper issued by Siemens PLM software.
- [30] D. Torregrossa, M. Paolone, Modelling of current and temperature effects on supercapacitors ageing. Part I: Review of driving phenomenology, *J. Energy Storage* (2015). EST 42 No. of Pages 10.
- [31] *An Introduction to MEMS (Micro-electromechanical Systems)*, Prime Faraday Technology Watch, ISBN 1-84402-020-7, January 2002.
- [32] A. Tamsamani et al., Prognostics for optimal maintenance (POM): an integrated solution from data capturing to maintenance decision, in: *Proceedings on the 24th International Congress on Condition Monitoring and Diagnostics Engineering Management*, 2011.
- [33] F. Samie, L. Bauer, J. Henkel, IoT technologies for embedded computing: a survey, in: *2016 International Conference on Hardware/Software Codesign and System Synthesis (CODES + ISSS)*, Pittsburgh, PA, 2016, pp. 1–10.
- [34] Reliability in electronics, Technical article, XP, <https://www.xppower.com/Portals/0/pdfs/Reliability.pdf>.
- [35] M. Musallam, C. Yin, C. Bailey, M. Johnson, Mission profile-based reliability design and real-time life consumption estimation in power electronics, in: *IEEE Transactions on Power Electronics*, vol. 30, no. 5, May 2015.
- [36] A. Tamsamani et al., Prognostics of connection defects in electronics modules, in: *European Conference of the Prognostics and Health Management Society*, 2016.
- [37] C. Furse, Y.C. Chung, C. Lo, P. Pendayala, A critical comparison of reflectometry methods for location of wire faults, *Smart Struct. Syst.* 2 (1) (2006) 25–46.
- [38] S.J. Kiptinness, *An Analysis of the Conventional Wire Maintenance Methods and Transition Wire Integrity Programs Used in the Aviation Industry*, The Faculty of the Department of Technology Digital Commons @ East Tennessee State University, 2004.
- [39] D. Lynch, NASA hybrid reflectometry project, in: *6th Joint FAA/DoD/NASA Aging Aircraft Conference*, September 16–19, 2002, pp. 1–9.
- [40] C. Sharma, et al., Lower power STDR CMOS sensor for locating faults in aging aircraft wiring, *IEEE Sensors J.* 7 (1) (2007) 43–50. 2007.
- [41] P. Smith, Analysis of spread spectrum time domain reflectometry for wire fault detection, *IEEE Sensors J.* 5 (6) (2005) 1469–1478. 2005.

- [42] P. Kuhn, Locating hidden hazards in electrical wiring, in: Aged Electrical System Research Application Symposium, Chicago, IL, October 18–19, 2006.
- [43] Agilent, 4262A LCR Meter Operating and Service Manual, 1983.
- [44] Y.C. Chung, et al., Capacitance and inductance sensor circuits for detecting the lengths of open and short circuited wires, *IEEE Trans. Instrum. Meas.* 58 (8) (2009) 2495–2502.
- [45] M. Coenen et al., Live main impedance measurement and analysis, in: Proceedings of the 2013 International Symposium on Electromagnetic Compatibility (EMC Europe), 2–3 September 2–3, 2013, pp. 114–119.
- [46] Agilent, Impedance Measurement Handbook, A guide to measurement technology and techniques, fourth ed., Agilent Technologies.
- [47] J.H. Lau, et al., Effects of fretting corrosion on Au-Sn plated contacts in electronic cable interconnects, *JSB Tech.* (2013) 1–12.

This page intentionally left blank

PART

Textiles industries

2

This page intentionally left blank

Failure of yarns in different textile applications 10

Radostina A. Angelova

Technical University of Sofia, Sofia, Bulgaria

10.1 Introduction

Yarn strength is probably the most important parameter of both staple fiber yarns and filaments that is controlled among all mechanical properties of the yarns. It is defined as the breaking force of the yarn, commonly measured in Newtons (N) or centinewtons (cN). The ratio between the yarn's breaking force and its linear density (measured in tex) gives another largely used parameter for assessing the strength of the yarn—the yarn tenacity, expressed in cN/tex. The tenacity gives the possibility to compare the strength of yarns with different thickness.

The phenomenon of spun yarn failure is strongly preconditioned by the yarn structure, determined in turn by the spinning method, applied to consolidate the fibers bundle into a yarn. It is the twisting of the fibers bundle that gives the strength to the final yarn.

Five spinning methods exist today that are used for industrial spinning of natural and man-made fibers: ring, rotor, friction, hollow-spindle, and air-jet. Only the ring spun yarns have a helical structure along their length, thus using to a maximum extent the strength of the fibers in the cross section (Fig. 10.1). The helical path of the fibers is visible. The same is the path of the single yarns, when they are twisted to form a twofold yarn (Fig. 10.2).

Rotor spun yarns show core—sheath structure, where only the core possesses a helical twisting of the fibers, while the sheath is constituted of individual fibers or ribbons of fibers, wrapped around the core (Fig. 10.3). Air-jet spun yarns have a fasciated structure: a core of parallel fibers around which a small number of surface fibers are twisted [1] (Fig. 10.4).

Wrap yarns, spun by using hollow-spindle spinning method, have a core of parallel staple fibers, consolidated and strengthened by a twisting thread (staple fiber yarn or filament) [2] (Fig. 10.5). Friction spun yarns appear visually like ring yarns, but their internal structure is entirely different: it consists of fibers with poor orientation and loose packing in the yarn's cross section [3].

The yarns, being twisted fiber structures, possess a unique failure mechanism: the force, which leads to the break of the yarn, provokes the strengthening of the yarn simultaneously. When strained, fibers in the yarn stretch a segment by

**FIGURE 10.1**

The helical structure of a ring spun yarn: microscopic view ($\times 40$) of the longitudinal structure (cotton-polyester yarn, 15 tex).

segment, due to the lateral constraint. At the same time, the yarn strength increases due to the increment of the lateral pressure.

The strength of the yarn is not an intrinsic property but depends on both the structure and loading environment [4]. However, the strength of a spun yarn can never reach the strength of the fibers' bundle. The reason is that during the yarn's failure some of the fibers break, but others slip. It is the yarn structure (fibers alignment and packing as well as fibers distribution in the yarn's cross section) that preconditions the proportion of the broken, and the slipped fibers. The specific morphology of the spinning method determines the fiber orientation in the yarn cross section and alongside the yarn. It is the directional orientation of the individual fibers that specifies the mechanical properties of both yarns and fabrics made of them; therefore the monitoring of the fiber orientation in textiles is of particular importance [2,5].

The strength of yarn depends on the strength of its weakest point. While subjected to a tensile load, the yarns are compressed radially. With the increment of the generated tension, some fibers start to break, usually at the yarn's weakest cross section. Broken fibers may be both weaker fibers, compared to the other fibers in the bundle or, depending on the structure of the yarn (ring, rotor,

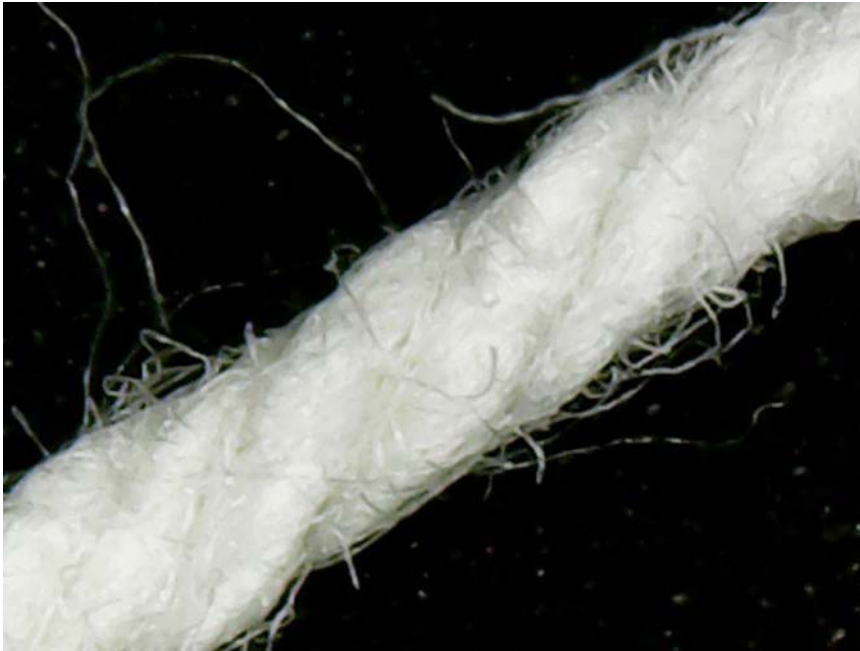


FIGURE 10.2

The helical structure of a twofolded ring spun yarn, microscopic view ($\times 40$) of the longitudinal structure (acrylic 50 tex).

and air-jet), be more strained, compared to the other fibers (e.g., the peripheral fibers in the ring spun yarns). The interfiber friction is also of crucial importance: even the moderate changes in the frictional forces between the fibers (e.g., through twist) can lead to a sizable increment of the yarn strength [6].

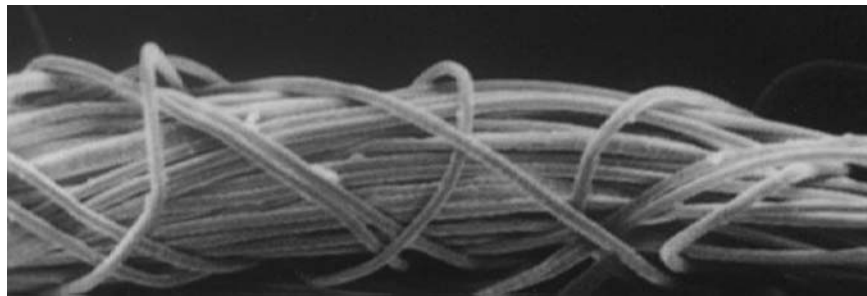
The further increment of the tensile load depends on the strength of the rest of the fibers at the weakest point of the yarn. Before the yarn failure, slippage of some fibers appears, while other fibers grip one to another. The gripped fibers break as soon as they reach their breaking elongation that is strongly affected by the continuous tensile load and its concentration in the continuously decreasing cross section of the yarn. The complete yarn failure occurs in a mixed mode of a simultaneous fibers' slippage and fibers' breakage [7].

The yarn's stress–strain characteristics can illustrate the mechanism of yarn failure. Fig. 10.6 presents the typical stress–strain curve of the yarn under tensile load:

- A linear dependence between the load and the yarn strain exists only in the region I, where small stresses can be observed. In this region, the slippage of the fibers is prevented by the interfiber friction.

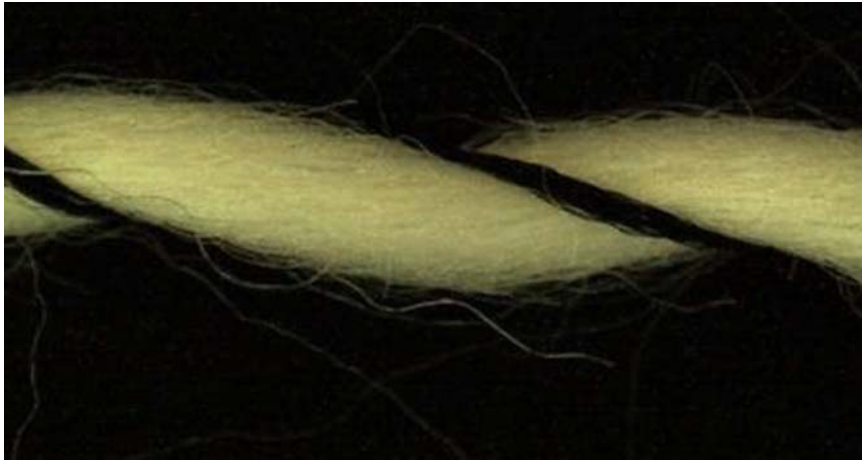
**FIGURE 10.3**

Rotor spun yarn: microscopic view ($\times 40$) of the longitudinal structure (cotton-polyester, 20 tex).

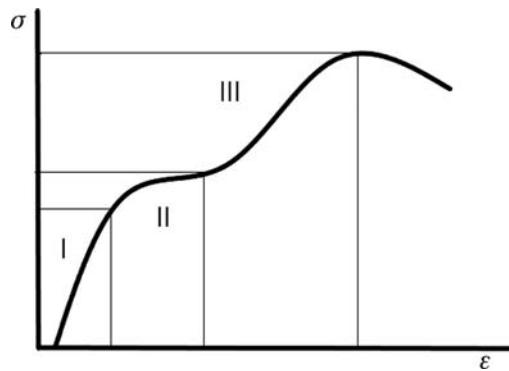
**FIGURE 10.4**

Air-jet spun yarn: SEM view of the longitudinal structure (wool-polyester, 20 tex).

- After increasing the stress in region II, some of the fibers in the yarn cross section start to slip. Thus under a relatively short interval of the stress increment, the strain of the yarn is substantially augmented.
- In region III both fiber slippage and fiber breakage occur until the fiber yarn failure appears.

**FIGURE 10.5**

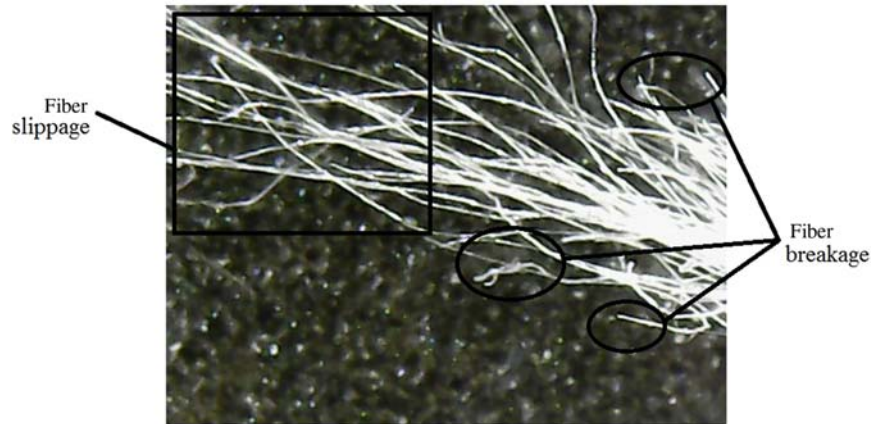
Wrap spun yarn: microscopic view ($\times 20$) of the longitudinal structure (acrylic-polyester, 36 tex).

**FIGURE 10.6**

Stress—strain curve of a staple fiber yarn.

10.2 Staple yarn failure depending on the spinning method

The described theory of yarn failure has been developed for the structure of a ring spun yarn, where each yarn's cross section is twisted relatively to the previous one at a given angle. The yarn failure is a combination of slippage and breakage of the fibers. Fig. 10.7 shows a microscopic view of the yarn failure where the typical breakage of the peripheral fibers, followed by the slippage of the central fibers in the yarn can be observed.

**FIGURE 10.7**

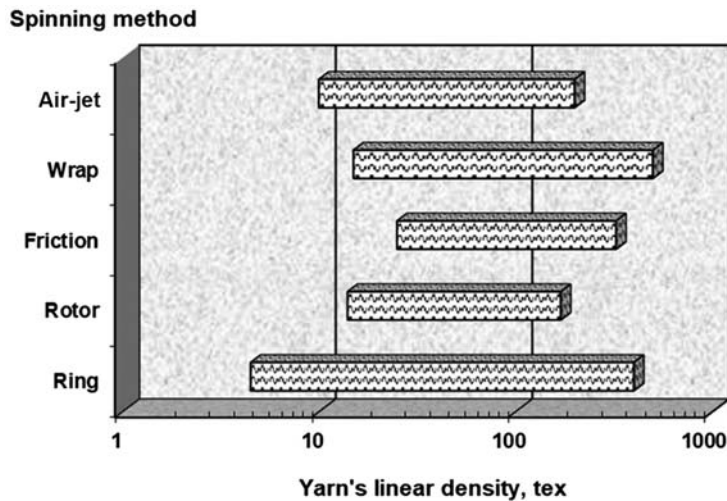
Broken end of a staple fiber yarn with fiber breakage and fiber slippage.

Different processes could appear, however, depending on the particular spinning system, used to produce the yarn and its consecutive yarn structure. The share of the broken and slipped fibers would also be different. For example, the structure of a wrapped yarn, obtained by hollow-spindle machines, preconditions not two, but three different modes of yarn failure: due to the breakage of the wrapper, slippage of the core fibers, and the breakage of the core fibers [2].

The weakest point of the yarn is usually the cross section with a minimum number of fibers. Ghosh et al. [7] developed a model of the yarn failure, pretending to be universal for every type of spun yarns, as the model is based on the failure behavior of the yarn under tensile load. However, the input values of the model involve parameters, which measurement is very tedious, for example, the proportion of the slipped and broken fibers. Besides, the meaning of such a model is to be applied for prediction of the yarn failure, without real yarn break (an even before the yarn spinning). However, the model input data require parameters, which can be achieved only after experimental measurements of the yarn break.

Ring spun yarns are considered a golden standard for yarn quality. The common strategy when assessing particularities of a different type of yarn is to compare the “unconventional” yarn with the respective ring yarn (with the same fibers composition and linear density) [8].

Rotor, friction, air-jet, and wrap spinning methods are unbeatable regarding machine productivity. The ring spinning method, however, is leading in the production of both high-quality yarns and fine yarns (below 10 tex). Only the air-jet spinning method can be a partial competitor to the ring spinning in terms of fine yarns manufacturing as the finest air-jet spun yarns reach as low as 10 tex (Fig. 10.8).

**FIGURE 10.8**

Range of linear density of the yarns, depending on the spinning method.

Being different from the structure of the ring spun yarns, the structure of the rotor, friction, air-jet, and wrapped yarns causes a significant criticism concerning the yarn failure. The reason is that all they miss is the helical path of the fibers, which is equal for all cross sections depending on the twist during the ring spinning. Only the wrapped yarns can compete with the ring spun yarns in terms of strength, due to the presence of the wrapper component that twists around the core following a helical path as in ring spinning. The proper design of the wrapper concerning its material, type, and magnitude of the wrap twist can effectively manipulate the wrapped yarn failure, thus reaching the strength of a ring spun yarn.

Fig. 10.9 presents the relative strength of the yarns, depending on the spinning method. The failure strength of the ring yarn is considered 100% and the strength of the other types of yarns is expressed as a percentage of it. As it can be seen, none of the other spinning methods can reach the strength of the yarns, produced by ring spinning.

The morphology of the particular spinning method reflects the structure of the yarn regarding the regularity of the linear density. The higher number of thin and thick places in the yarn cross section provokes the yarn's failure at lower tensile loads. Fig. 10.10 compares the regularity of the yarn concerning the linear density: the uniformity of the ring spun yarns (considered again as 100%) can only be achieved by rotor and wrapped spun yarns in some cases. Air-jet and friction spun yarns are the most irregular, compared to the others.

The yarn failure is also related to the number of the compound fibers in the yarn cross section. The difference between the five modern spinning methods is

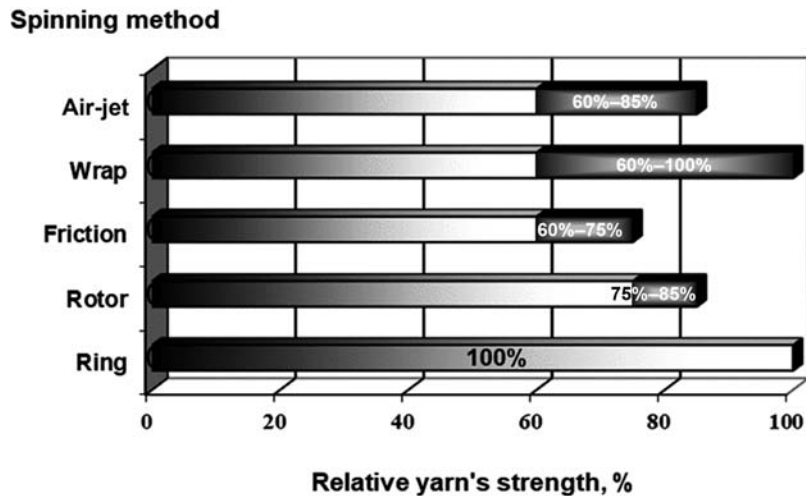


FIGURE 10.9
Relative strength of the yarns, depending on the spinning method.

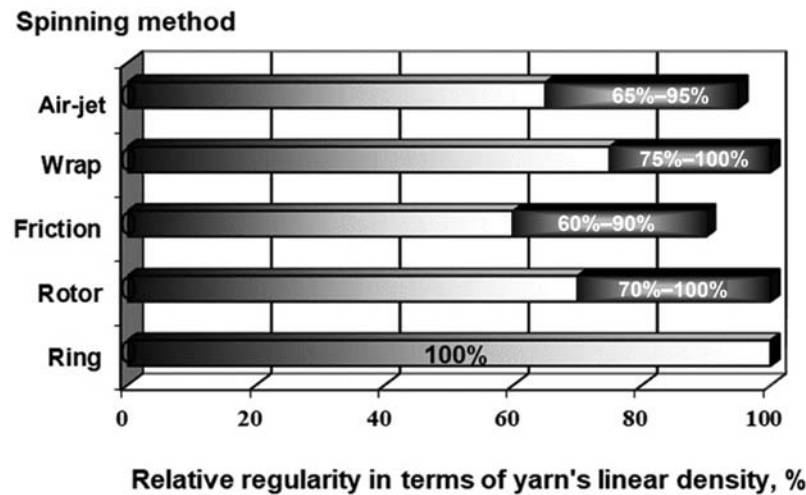
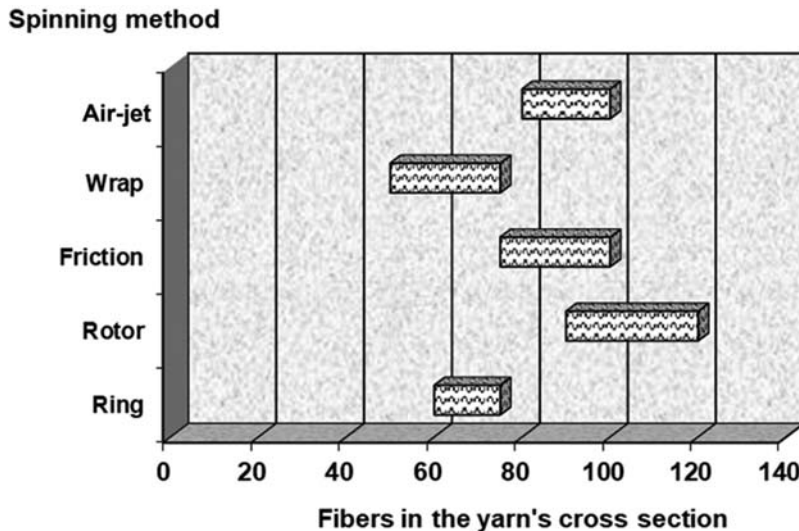


FIGURE 10.10
Relative regularity of the yarn in terms of their linear density, depending on the spinning method.

not very high, as shown in Fig. 10.11. Wrapped yarns can be produced with the lowest number of fibers in the cross section, as the wrapper is the element in their structure, which strengthens the yarn much more than the low twisted fibers in

**FIGURE 10.11**

Minimum number of fibers in the cross section of the yarns, depending on the spinning method.

the core of the yarn. The production of rotor spun yarns is related to the highest number of fibers in the cross section, which is preconditioned by the worse orientation of the fibers alongside the yarn. Thus to reach the required yarn strength, necessary for further processing on weaving and knitting machines, the number of fibers in rotor yarn's cross section is 1.5–2 times higher as an average, compared to ring spun yarns.

10.3 Yarn failure depending on the gauge length

To evaluate the yarn failure, standard tensile tests are applied. However, during manufacturing and in a real application, the fabrics and their compound yarns experience strains and stresses, which differ from the loads during the standard yarns' tensile tests. As a result, the tensile strength parameters of the yarns, obtained preliminary, do not necessarily reflect their behavior in the textile macrostructure.

The influence of the gauge length on the yarn failure has been largely studied. It is known that the balance between the fibers that break and fibers that slip in the yarn under tensile load vary with the gauge length. The shorter gauge length during the yarn's tensile test gives better correlation with the yarn failure in the fabric than the standard gauge length [9].

At the same time, the influence of the yarn structure, preconditioned by the spinning system, is also present. Gosh et al. [7] have found that at standard gauge lengths ring spun yarns have the shortest lengths of the failure zone, compared to the rotor, friction, and air-jet spun yarns. The internal arrangement of the fibers in the yarn is with better migration and very good interlocking between them gives the ring spun yarns an advantage compared to the other types of yarns. In fact, the ring spun yarn structure leads to obtaining of best yarns' failure resistance at standard gauge lengths.

The decrement of the gauge length below the staple fiber length provokes an increase of the strength of ring, rotor, friction, and air-jet spun yarns [7,10]. Being gripped by the two jaws of the testing instrument the two ends of most fibers are firmly positioned and cannot move toward the neighboring fibers. Thus the yarn failure appears much more due to fiber breakage than due to fiber slip-page. The result is that the yarn strength increases and tends to the fiber strength (which is always higher) and correlates better with the strength of the fabrics, produced with them [9].

The comparison between several types of yarn's structures shows that at gauge length well below the staple fiber length air-jet spun yarn's failure appears at greatest loads, compared to the ring, rotor, and friction spun yarns. The very high proportion of core fibers in the structure of the air-jet spun yarns that are parallel to the yarn axis contribute a lot to the yarn strength under tension. By analogy, the friction spun yarn's failure appears at lowest loads, as its inner structure involve chaotic, looped fibers. Investigating the influence of the gauge length on the yarn failure Hussain et al. [11] have found that there is no significant difference between ring and rotor spun yarns at relatively short gauge lengths.

10.4 Yarn failure depending on the strain rate

Yarn failure occurs faster when the extension rate increases. The elongation of the yarn at the moment of the yarn failure is

$$E = \frac{100Vt}{60l} \quad (10.1)$$

where E is the breaking elongation of the yarn, %; V is the extension rate, mm/min; t is the time to yarn failure, s; and l is the test length of the yarn, mm.

It was considered that the rate of strain and the yarn failure load have a linear relationship, but later investigations on the topic have found that the tenacity of the yarns reaches a peak at a certain strain (around 20 cm/min) and then decreases [12]. The conclusions were drawn after measuring the strength of ring and rotor spun yarn structures at strain rates of 1, 10, 20, 50, and 100 cm/min.

The effect of the strain rate on the yarn failure can be explained by the mechanics of the influence of the strength of the fibers on the strength of the spun yarn. When the yarn is under tension, the transverse forces increase the

tension between the fibers, provoking higher frictional resistance. Depending on the twist and the packing density of the yarn, which in terms determines the position of the fibers in the yarn structure, the fibers can realign to a certain extent. The result is their better contribution to the tensile resistance of the spun yarn.

The load at low strain rate gives enough time to more fibers to relatively move in relation to their neighbor fibers. As a consequence, the yarn failure occurs much more due to the fibers' slippage than to the fibers' breakage. With the increment of the strain rate, the influence of the load becomes catastrophic, and the fibers' breakage dominates the yarn tenacity. The brief time is not sufficient for the realignment of the fibers, and the yarn tenacity decreases. Balasubramanian and Salhotra [12] suggest that an optimum strain rate has to be found for the appearance of maximum yarn tenacity.

10.5 Modeling of the yarn failure

The failure of yarns has been largely studied, as the research on the mechanical properties of yarns dates back at least a century ago. In the last three decades, the modeling of the yarn failure/strength, among other properties of the yarns, became one of the most critical tasks of the research in the field of textiles. Several theories were applied to evaluate the yarn strength (yarn failure) [13,14]:

- the force method based on the theory of the weakest link and the strain distribution in the yarn;
- the energy method (with several modifications), which uses the principle of the minimum potential energy;
- the finite-element method, quite complicated, with severe difficulties to take the nonlinear effects of the fibers into account; and
- the method, based on the short-path hypothesis, etc.

Unfortunately, most of the models for prediction of the yarn failure do not give the necessary accuracy of the theoretical results to be widely applied for distinct types of yarns.

The approach based on artificial intelligence is also applied to model the non-linear relationship between the fiber material and the yarn tensile behavior. Artificial neural networks and neuro-fuzzy models were used to predict yarn strength among other yarn properties [15,16]. Statistical, mathematical, and artificial neuro networks were compared as the approach for modeling the yarn failure elongation [17] and yarn tenacity [18].

Analyzing the mechanics of the yarn's failure Pan [4] claimed that at high-twist levels there is a minor difference between the approach for calculating the strength of staple fiber yarns and multifilaments. When the aim is yarn failure prediction, the only difference between them is the length of the fibers (or monofilaments). Developing a model, based on Daniel's statistical model for a bundle

with parallel fibers and new theoretical considerations, they prove that at high-twist levels a staple fiber yarn can be treated as a multifilament (continuous filament) yarn.

Support vector machine models were proposed by Yang et al. [19] for predicting the yarn properties. The statistical learning theory was successfully applied for the prediction of the joint influence of the variability in raw materials and the machine variables during the multistage processing on the yarn failure. Furferi and Gelli [14] applied a feed forward back propagation model for prediction of the yarn strength by the parameters of the roving. The prediction error was less than 4% compared to a multiple regression model.

In the recent decades the market demand for fabrics with technical applications provoked a paradigm shift in the requirements for the properties of the woven textiles: nowadays their functional properties dominate over the aesthetic properties in many textile applications. Fabric failure may be the decisive parameter for the quality of the woven macrostructures; therefore the prediction of their tensile strength became an important research task. Statistical modeling and artificial neural networks were widely applied.

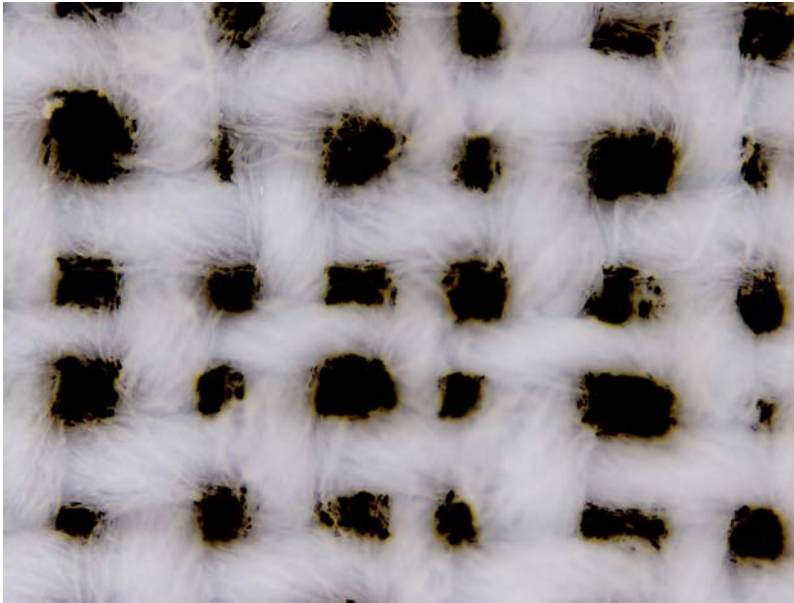
Artificial neuro networks and Taguchi design of experiment were successfully used to predict the relationship between parameters of fibers, yarns, and woven macrostructure on the fabric failure [20]. A back propagation artificial neuro network has been used for prediction of the tensile strength of a plain woven fabric by Majumdar et al. [21]. A multiple linear regression technique was applied to assess the effectiveness of the neuro network modeling. It was found that the strength of the warp yarns and the yarn density in the warp direction have the most significant role for the failure of the woven macrostructure. Recently, a mind evolutionary neural network was applied to predict the tensile behavior of cotton spun yarns [22]. A neural network coupled with response surface method and based on Grey Wolf Optimizer was also used in modeling of the failure of ring spun yarns (Sirospun process) [23].

10.6 Yarn failure in fabrics and composite structures

10.6.1 Yarn failure in fabrics

Most of the yarns are used for weaving or knitting of fabrics, representing the mesostructure of the woven (Fig. 10.12) or knitted (Fig. 10.13) macrostructures. The exploitation of the fabrics differs from the tests, applied to the compound yarns, including the tensile tests. During wearing, storing, and washing, textiles are subjects to specific mechanical stresses [24]: they can be exposed to strain forces of 20% or more [25], load of thousands of Newtons per meter [26], and forces that make them bend to radii of less than 1 mm [27].

The yarn failure in fabrics, woven into plain and twill weaves has been studied in Ref. [28]. Yarns were produced on ring, rotor, and air-jet spinning machines

**FIGURE 10.12**

Microscopic view of a woven macrostructure.

and their failure under uniaxial tension in similar woven macrostructures was experimentally tested. It was found that the magnitude of the fabric strength exceeded the tensile strength of the single yarn. The results are valid especially for dense fabrics, where the yarns experience more extended contact pressure zones alongside their length, thus decreasing the chance for in-fabric yarn failure.

In fabrics with high warp and weft density, most of the isolated in-fabric yarn failures appear at the bend locations between the threads of the two perpendicular systems of yarns. Rotor and ring spun yarns break abruptly, which results in a very short yarn failure zone. The same type of break was observed for yarn failure at near zero gauge length [28]. The in-fabric failure of the air-jet spun yarns was quite similar to that of ring and rotor spun yarns.

In woven macrostructures, with lower warp and weft density, the in-fabric yarn failure is similar as topology and mechanics to the single yarn failure (out of the woven macrostructure), with additional pressure in the lateral direction. The longer distance between the yarns does not provoke jamming of the cross yarn; thus the yarn failure could appear at different places, not necessarily at the banding point, where the highest local fiber strain would occur in dense fabrics.

One interesting direction to use the common staple yarn structure and tensile properties in new areas like the functionalized textiles is the recently proposed technology for hybrid yarns [29]. Nanofibers are applied in the inner structure of untwisted staple yarns, and after the retwisting of the yarn both textile fibers and

**FIGURE 10.13**

Microscopic view of a knitted macrostructure.

nanofibers are integrated together. The tensile strength of the hybrid yarn is enough to further be manufactured into woven or knitted macrostructure, giving floor for obtaining of functionalized textiles of a new type.

10.6.2 Yarn failure in composite structures

The composite structures consist of two or more materials with considerably different properties. One of the materials is the binder or matrix, which binds together and surrounds the components of the other material (fibers) that is called reinforcement. The result is a composite material with unique properties and applications.

Fiberglass is an example of the first modern human-made composite materials: the matrix is plastic, reinforced by the glass, formed as fine threads that are usually woven into a glass fabric (Fig. 10.14). Glass fibers, being made of brittle material (Fig. 10.15), would break without the protection of the matrix. On the other hand, the plastic binder increases its strength extremely, acting together with the glass fibers and using their very high sturdiness.

Carbon fibers are used as a substitute for the glass fibers, as they are stronger, but lighter in weight (Fig. 10.16). Their high price makes them still applicable in some expensive market niches like aircraft structures or special sports equipment.

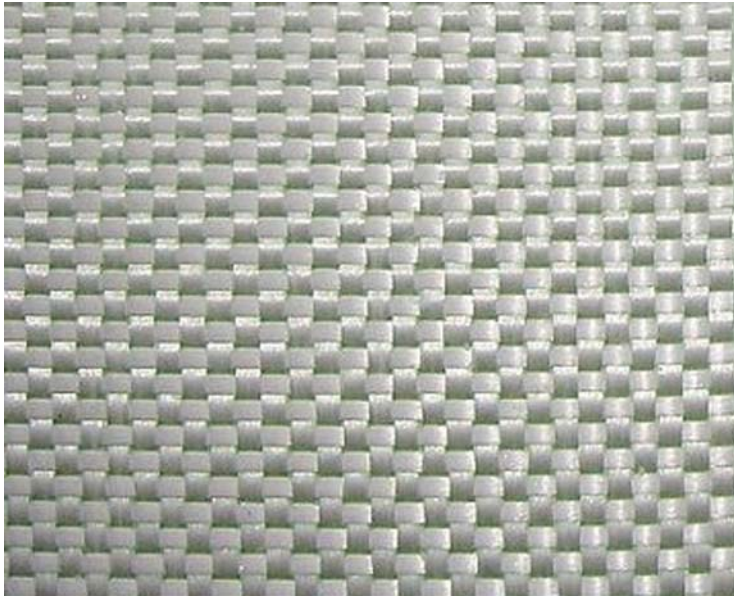


FIGURE 10.14

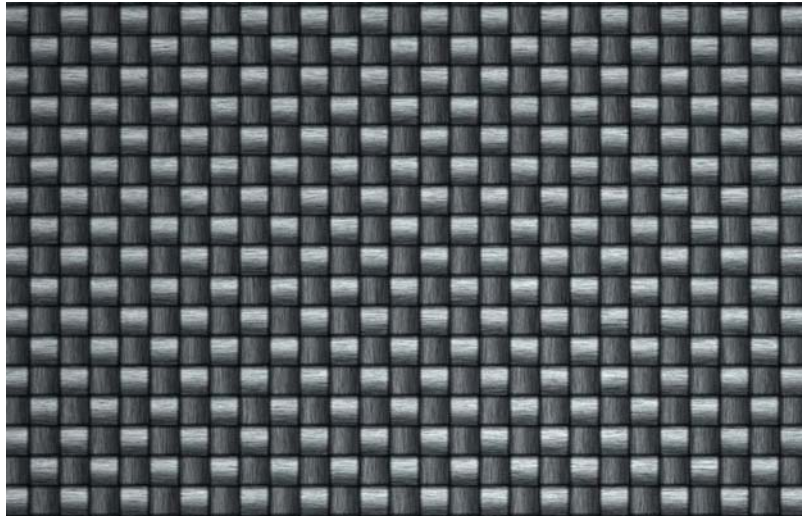
Fiberglass woven macrostructure.



FIGURE 10.15

Fiberglass material.

Carbon nanotubes (CNTs) are the next generation of carbon fibers, involved in the production of composite materials that are even lighter and stronger than the ordinary carbon fiber reinforced composites. Despite their very high price,

**FIGURE 10.16**

Carbon woven macrostructure.

the CNTs are very promising for construction of cars and aircrafts that are lighter in weight and will consume less fuel.

Several studies were dedicated to the development of models for prediction of the strength of the fabric platform in fabric reinforced composites, as well as the strength of the resulting composite materials. Some of them started with describing the yarn failure in the compound textile platform. Ko and Pastore [30,31] developed a model of the yarn orientation in a three-dimensional (3D) braided preform to estimate the influence of the yarn strength on the strength of the textile platform. The last step was to predict the composite strength.

Geometry model of braided fabric and the maximum strain energy criterion were applied to predict yarn failure in a reinforced composite [32]. A simple linear model for yarn bending was also applied together with the model of the fabric platform to predict the yarn failure and the resulting strength of a two-dimensional (2D) triaxial braided composite material [33]. Naik [31] proposed a general purpose technique for analysis of both woven and braided fabric reinforced composites. It allowed predicting the failure initiation, the progression of the composite damage, and strength of a 2D textile platform.

Recent studies involve both numerical and experimental investigation of the low velocity failure in woven composites with polymer matrix [34], global and local determination of the failure in composites, based on interlock woven fabrics [35], and application of fiber-based continuum analysis for investigation of the mechanical behavior of 3D woven and braided composites [36].

The review paper of ElAgamy and Laliberté [37] gives a valuable insight into the development of the geometrical modeling of composites, reinforced with

textiles. It presents the geometrical models of textile structures, used for predicting the yarn and fabric behavior, including their failure. One- and two-dimensional models for modeling of textile reinforced composites are discussed, together with the software codes available today for modeling of composite's structure and behavior. The general conclusion is that there is still not a single model, which can be applied for weave structures of different dimensions in any deformed state, as most of the models, though different in their input strategies, complexity, and output results, are restricted to specific textile structures or output data.

10.6.3 High-strength yarn failure

High-strength fabrics are used for protective clothing for military personnel and law enforcement, as well as in different protective applications, for example, turbine fragment containment barriers. The research on aramid and other high-strength fibers, as well as on fabrics made of them and their applications, is extensive in both experimental field and numerical modeling.

The failure of high-strength yarns is rate dependent to a great extent. The rate-sensitive mechanical properties of Kevlar yarns were studied in Ref. [38]. The research of Dooraki et al. [39] has found that failure strengths of Kevlar yarns show limited dependence on the strain rate, while products from Zylon and Twaron have a more significant strain rate dependency. Despite the availability of data for the failure of single fibers, they cannot be scaled up or extrapolated for the yarns consisting of many fibers or the 2D or 3D woven or knitted textiles. It also results that the strain rates, measured in static experiments, are orders of magnitude smaller than the strain rates, which are observed in ballistic tests.

The statistical nature of the properties of the yarns from high-strength fibers results in the probabilistic behavior of the fabrics made of them. For example, the projectile can engage a set of weaker or stronger set of yarns at the impact site, which can result in penetration (with different velocities) or nonpenetration at all [40]. Several methods for dynamic tensile tests of high-strength fibers and fabrics are available, and the research on the dynamic phenomena and the influence of various factors on both the tensile failure of fibers, yarns, and fabrics and the measurement parameters on the results obtained is continuing. A lack of general agreement on the methodologies applied for dynamic tests is observed [41]. Different techniques for experimental measurement of high-strength materials are reported in the literature: split Hopkinson pressure bar, flywheel facilities, falling weight devices, servo-hydraulic machines, etc.

An analytical model of the single Kevlar yarn ballistic impact, based on the momentum theory, was proposed in Ref. [42]. Composites, reinforced by Spectra polyethylene fibers, were investigated in terms of their penetration failure [43]. It was found that the ratio of the yarn failure in the fabric to that of the composites correlated with the energy absorption ability of the composites. The viscoelastic

behavior of Nomex, Kevlar, and polypropylene yarns was studied in Ref. [44], and the specific nonlinear effects and their sources were discussed.

Shin et al. [45] studied the cut resistance of high-strength yarns, under tension-shear load. Among the three investigated yarns it was found that the cut resistance of Zylon (polybenzobisoxazole) yarns was higher than that of spectra (polyethylene) and Kevlar (aramid) for all investigated slice angles, the sharpness of blades and pretension values. The reduction of the yarns' cut resistance with the increment of the yarn pretension was also observed and analyzed.

Numerical study on the combined effect of the yarn tensile strength and the friction between the yarns in the fabric as well as fabric clamping and projectile impact location on the probabilistic impact response of woven Kevlar fabrics was presented in Ref. [46]. As the tensile failure of a Kevlar yarn is statistical in nature, it was modeled in the study by Weibull strength distribution.

The target boundary conditions are of particular importance for fabrics ballistics effectiveness. Shin et al. [45] have found that a gripped on four corners fabric made of high-strength yarns (Zylon and Spectra) absorbs more energy; in addition to the yarn failure, a corner-hole failure appears as well. The study also concluded that the ballistic fabrics absorb more energy when the yarns are oriented at 45 degrees to the edges in comparison to fabrics where the yarns are parallel to the edges.

10.6.4 Failure of yarns from brittle high-performance fibers

Carbon and glass fibers are high-performance, brittle fibers, frequently used in structural engineering. The problem with their utilization is that every step in the production process may lead to failure of the fiber bundle. Only a few fibers in the yarn could be damaged, which will not lead to yarn failure and breaking of the production process, but this microdamage will reduce the strength and other bearing parameters of the yarn, the fabric and the composite material as the final product.

The increased application of these materials requires tools for analysis and simulation of the process parameters and their influence on the behavior of the final product. The simulation models are based on the description of the composite mesostructure: modeling of the single yarn [47]. Simulation models based even on the microstructure, for example, modeling of every single filament in the yarn, are also available [48,49].

The relative fiber volume preconditions the strength of the brittle fibers; therefore a statistical approach is used for development of the models. Kun et al. [50] proposed fiber bundle models, based on the interaction of the fibers and Weibull distribution failure criteria. Actually, during the last 20 years, such models have been used to describe materials degradation in a broad class of applications, even to earthquakes and social phenomena [51,52]. Commercial numerical codes, based on finite elements method like ABAQUS, have implemented material models with Weibull-distributed failure criteria [53,54].

Besides the microscale modeling, Döbrich et al. [49] have done experimental tests for the yarn failure of a single carbon filament and carbon multifilaments. It was found that both the failure strength and the failure elongation of the multifilament decreased in comparison with the failure strength and elongation of the single monofilament. The gauge lengths of the two tests were different: smaller for the single carbon monofilament (20 mm) and longer for the multifilaments (500 mm), which could provoke higher failure strength to be obtained (as discussed earlier). Another point for argument after the tests was the decrement of the failure elongation in case of the carbon multifilament (from 2.22% to 0.77%), a matter of the Weibull-distributed failure behavior [40].

Hybrid yarn manufacturing is a way to incorporate brittle fibers in high-performance structures, which have better tensile performance in the resulting composite materials. Hasan et al. [55] reported research on the properties of hybrid yarns, made from carbon filament yarns and polyamide filament. It was found that the adhesion properties of the carbon filament and the composite matrix are improved, thus showing good potential for application in textile reinforced thermoplastic composites.

10.6.5 Carbon nanotubes yarn failure

Yarns from CNTs can be applied for reinforcement of lightweight composite structures [56]. These yarns consist of hundreds to millions CNTs in the cross section and can be twisted, reaching a strength that is much higher than the strength of Kevlar and graphite yarns: the breaking energy for CNTs yarns is from 110 to 975 kJ/kg while the same value for Kevlar yarns is around 33 kJ/kg [57].

The twisting of CNTs yarns makes them more regular, allows them to contract radially when stressed and to transfer the load more efficiently. The yarn failure due to an extension to break is ductile-like, showing evidence for nanotubes fraction, pull-out, and sliding [56]. At the same time, the CNT yarns show very little unraveling, compared to conventional staple fiber yarns, when elongated to failure. The difference is supposed to be as a result of the extremely high contact area between the CNTs [58], which is not valid for the interfacial surface between the fibers in the conventional yarns.

Like conventional ring spun yarns, CNT yarns are as better as lower is the dispersion in their strength. The increment of both the CNT yarn's diameter and the twisting angle decreases the dispersion in the measured values of the yarn failure under tension. The weakening effect from the gauge length increment becomes smaller.

The composites, reinforced with CNTs yarns, have lower strength than the yarns [56]. Despite of this, the same rule is valid as for the fabrics from conventional spun yarns: stronger composites are manufactured from stronger CNTs yarns.

Different models have been developed to predict the yarn failure of CNT yarns. Beyerlin et al. [56] applied the Monte Carlo method for the prediction of the relationship between both the yarn's nanostructure and its tensile strength and CNTs yarns' strength and the failure of composites, reinforced with them. Beese et al. [59] used a Monte Carlo based model for predicting the correlation between the CNTs yarns failure and their porosity and alignment.

10.6.6 Yarn failure in electronic textiles

Electronically functionalized textiles offer clear benefits and have several commercial applications. They can be found in medicine, where they measure physiological parameters and even react, giving signals for people's action, give medication, etc. The incorporation of visual effects or light sources gives the textiles the functionality to give signals or to be used for communication. Electronic smart textiles are also used for ensuring the thermophysiological comfort of people [60].

The electronic textiles involve textile phase and electronic components, such as sensors and actuators, organic photovoltaics, and light emitting diodes [61]. The circuit of the system is built by using conductive yarns, among the regular fibrous yarns. The presence of conductive yarns allows several new functionalities to be attached to the textiles and used widely in different applications: from monitoring of vital functions of the body to fashion design.

The conductive yarns are incorporated into the classical textile macrostructure mainly by weaving [27,62], knitting [63], embroidery, [64], stitching, and lamination [61].

During the weaving and knitting processes, the conductive yarns are mixed with the regular threads, following the same pattern. The inflexible components of the electronic textile (e.g., sensors) are mounted on a later stage. If the conductive yarn is stitched to the fabric, it is guided by the needle and fixed on the textile layer by the counter thread (that can also be conductive). In the case of embroidery, a separate, not conductive thread fixes the conductive yarn on the fabric's surface.

The more the field of application of electronic textiles is expanding, the more the market demand to produce well-designed, comfortable, and durable terms of care and exploitation items grows. In this sense, the conductive yarn performance and failure become very important, as it creates a functional unit or assists the interconnection between two or more functional units [65].

Different types of conductive yarns are used to produce the circuits of the electronic textiles [61]:

- polyfilament yarns, wrapped with a metal foil;
- core yarns made of a polymer core and fiber coated with a silver coating (e.g., silver plated polyamide yarn); and

- fully conductive yarns of a copper core and additional protective coating made of noble metals (silver).

The conduction abilities and the strength of the conductive yarns vary. If the yarn failure appears at low tensile load, the embroidery is the preferable technology instead of stitching.

A distinctive feature when incorporating the conductive yarn into the textile macrostructure through weaving is that the conductive yarns are deformed on the loom due to the crossing of the warp and weft threads. de Vries and Peerlings [24] studied the failure of metallic filaments (consisted of 20 silver plated copper yarns) in connection with the linear density of the regular threads in the weave (polyester staple fiber yarns) and the weft density of the fabric. It was proved that the conductive yarns' failure appears at lower values than the electrical failure, so the results question the need for an electrical test to determine the conductive yarn maximal elongation. The study [24] goes further, proposing to omit the mechanical test for the yarn failure as well because it proposes a model that allows predicting the extensibility of the conductive yarn by the linear density of the regular (nonconductive) yarns in the woven macrostructure and its weft density (picks/cm).

10.7 Conclusion and future trends

There is no doubt that modern studies in the field of yarn failure are dedicated mainly to yarn strength issues in sophisticated, hi-tech structures such as textile reinforced composites, functional textiles, and electronic textiles. Far fewer studies today are focused on theory, experimental research, mathematical modeling, and computer simulation of classic fibrous yarns produced by classic spinning technology and used in traditional textiles. This trend is fully explained by the rapid development of innovative technologies and the ever-expanding range of products that contain textile components (and in particular yarns) and has different hi-tech applications. Studies on hybrid yarns, high-strength yarns, and conductive yarns are just some of the challenges that modern research on yarn failure is facing.

At the same time, the review of publications on yarn failure in different textile applications shows that there is still no universal model describing the structure of a staple fiber yarn, obtained through different spinning methods. A general model of yarn strength also lacks for both staple fiber yarns and filaments. The developed powerful computer codes to simulate the complex hierarchical structure of fabrics and composites use practically imperfect mathematical models of their mesostructure (yarns and filaments), thus introducing further simplifications into the 2D and 3D simulations of the fabrics and composites, leading to inaccuracies in the final numerical results.

In this sense, methods based on artificial intelligence contain perhaps the highest potential for a correct, near-realistic description of the whole set of factors that affect the yarn's strength, structure, and other properties that are important to their different applications.

Finally, but not least, this, though a brief review of the studies on yarn failure shows that research issues that once required good knowledge of classic textile technology, yarn strength testing methods, and a well-functioning dynamometer, today require detailed knowledge in many different areas of science, which are often too far away from textile science and practice. On the one hand, this necessitates a paradigm shift in classical textile training and, on the other hand, shows that the interdisciplinary, collective work of research teams is not only necessary but also inevitable.

References

- [1] R.A. Angelova, Air-jet spinning, in: *Advances in Yarn Spinning Technology*, 2010, pp. 315–344.
- [2] R.A. Angelova, Hollow spindle spinning, in: *Advances in Yarn Spinning Technology*, 2010, pp. 345–364.
- [3] R.A. Angelova, *Modern Spinning Methods*, Avangard Publ. Sofia, Bulgaria, 2010.
- [4] N. Pan, Prediction of statistical strengths of twisted fibre structures, *J. Mater. Sci.* 28 (22) (1993) 6107–6114.
- [5] M. Tunák, J. Antoch, Monitoring homogeneity of textile fiber orientation, *Text. Res. J.* 88 (11) (2018) 1226–1243.
- [6] R.M. Broughton Jr, Y.E. Mogahzy Jr., D.M. Hall Jr, Mechanism of yarn failure, *Text. Res. J.* 62 (3) (1992) 131–134.
- [7] A. Ghosh, S.M. Ishtiaque, R.S. Rengasamy, Analysis of spun yarn failure. Part I: Tensile failure of yarns as a function of structure and testing parameters, *Text. Res. J.* 75 (10) (2005) 731–740.
- [8] A.A. Merati, M. Okamura, Hollow yarn in friction spinning. Part II: Yarn structure and deformation under axial tension and lateral forces, *Text. Res. J.* 71 (5) (2001) 454–458.
- [9] M.L. Realff, M. Seo, M.C. Boyce, P. Schwartz, S. Backer, Mechanical properties of fabrics woven from yarns produced by different spinning technologies: yarn failure as a function of gauge length, *Text. Res. J.* 61 (9) (1991) 517–530.
- [10] W. Oxenham, R.Y. Zhu, G.A.V. Leaf, Observations on the tensile properties of friction-spun yarns, *J. Text. Inst.* 83 (4) (1992) 621–623.
- [11] G.F.S. Hussain, R.P. Nachane, I.K. Krishna, B. Srinathan, Weak-link effect on tensile properties of cotton yarns, *Text. Res. J.* 60 (1990) 69–77.
- [12] P. Balasubramanian, K.R. Salhotra, Effect of strain rate on yarn tenacity, *Text. Res. J.* 55 (1) (1985) 74–75.
- [13] T. Liu, K.F. Choi, Y. Li, Mechanical modeling of singles yarn, *Text. Res. J.* 77 (3) (2007) 123–130.
- [14] R. Furferi, M. Gelli, Yarn strength prediction: a practical model based on artificial neural networks, *Adv. Mech. Eng.* 2 (2010) 640103.

- [15] S.A. Malik, A. Farooq, T. Gereke, C. Cherif, Prediction of blended yarn evenness and tensile properties by using artificial neural network and multiple linear regression, *Autex Res. J.* 16 (2) (2016) 43–50.
- [16] G.G. Ahmad, Using artificial neural networks with graphical user interface to predict the strength of carded cotton yarns, *J. Text. Inst.* 107 (3) (2016) 386–394.
- [17] P.K. Majumdar, A. Majumdar, Predicting the breaking elongation of ring spun cotton yarns using mathematical, statistical, and artificial neural network models, *Text. Res. J.* 74 (7) (2004) 652–655.
- [18] A. Guha, R. Chattopadhyay, Jayadeva, Predicting yarn tenacity: a comparison of mechanistic, statistical, and neural network models, *J. Text. Inst.* 92 (2) (2001) 139–145.
- [19] J.G. Yang, Z.J. Lv, Q. Xiang, Yarn properties prediction using support vector machines: an intelligent reasoning method, in: *Fourth International Conference on Fuzzy Systems and Knowledge Discovery, FSKD 2007*, IEEE, vol. 1, 2007, pp. 696–701.
- [20] M. Zeydan, Modeling the woven fabric strength using artificial neural network and Taguchi methodologies, *Int. J. Cloth. Sci. Tech.* 20 (2) (2008) 104–118.
- [21] A. Majumdar, A. Ghosh, S.S. Saha, A. Roy, S. Barman, D. Panigrahi, et al., Empirical modeling of tensile strength of woven fabrics, *Fiber Polym.* 9 (2) (2008) 240–245.
- [22] Z.G. Wu, K.J. Zhang, C.W. Yu, Cotton yarn quality prediction based on mind evolutionary neural network, *DEStech Transact. Eng. Technol. Res. (MCEE)* (2017) 632–640.
- [23] E. Hadavandi, S. Mostafayi, P. Soltani, A Grey Wolf Optimizer-based neural network coupled with response surface method for modeling the strength of siro-spun yarn in spinning mills, *Appl. Soft Comput.* 72 (2018) 1–13.
- [24] H. de Vries, R. Peerlings, Predicting conducting yarn failure in woven electronic textiles, *Microelectron. Reliab.* 54 (12) (2014) 2956–2960.
- [25] C. Mattmann, T. Kirstein, G. Tröster, A method to measure elongations of clothing, in: *Proceedings of the 1st International Scientific Conference Ambience*, vol. 5, September 2005.
- [26] R.M. Crow, M.M. Dewar, Stresses in clothing as related to seam strength, *Text. Res. J.* 56 (8) (1986) 467–473.
- [27] K. Cherenack, C. Zysset, T. Kinkeldei, N. Münzenrieder, G. Tröster, Woven electronic fibers with sensing and display functions for smart textiles, *Adv. Mater.* 22 (45) (2010) 5178–5182.
- [28] M.H. Seo, M.L. Realff, N. Pan, M. Boyce, P. Schwartz, S. Backer, Mechanical properties of fabric woven from yarns produced by different spinning technologies: yarn failure in woven fabric, *Text. Res. J.* 63 (3) (1993) 123–134.
- [29] N. Korkmaz Memiş, G. Kayabaşı, D. Yılmaz, Development of a novel hybrid yarn production process for functional textile products, *J. Ind. Text.* (2018). 1528083718766847.
- [30] F.K. Ko, C.M. Pastore, Structure and properties of an integrated 3-D fabric for structural composites, *Recent Advances in Composites in the United States and Japan*, ASTM International, 1985.
- [31] R.A. Naik, Failure analysis of woven and braided fabric reinforced composites, *J. Compos. Mater.* 29 (17) (1995) 2334–2363.

- [32] F.K. Ko, Three-dimensional fabrics for composites, *Textile Structural Composites, Elsevier Science Publishers*, 1989, pp. 129–171.
- [33] J.E. Masters, M.J. Fedro, P.G. Ifju, An experimental and analytical characterization of three triaxially braided textile composites, in: *Proceedings of the Third NASA Advanced Composites Technology Conference*, 1992, pp. 263–286.
- [34] A. Elias, F. Laurin, M. Kaminski, L. Gornet, Experimental and numerical investigations of low energy/velocity impact damage generated in 3D woven composite with polymer matrix, *Compos. Struct.* 159 (2017) 228–239.
- [35] T.L. Chu, C. Ha-Minh, A. Imad, Analysis of local and global localizations on the failure phenomenon of 3D interlock woven fabrics under ballistic impact, *Compos. Struct.* 159 (2017) 267–277.
- [36] H. Ahn, W.R. Yu, Mechanical analysis of 3D braided and woven composites using fiber-based continuum analysis, *Compos. Struct.* 160 (2017) 1105–1118.
- [37] N. ElAgamy, J. Laliberté, Historical development of geometrical modeling of textiles reinforcements for polymer composites: a review, *J. Ind. Text.* 45 (4) (2016) 556–584.
- [38] B. Parga-Landa, F. Hernandez-Olivares, An analytical model to predict impact behaviour of soft armours, *Int. J. Impact. Eng.* 16 (3) (1995) 455–466.
- [39] B.F. Dooraki, J.A. Nemes, M. Bolduc, Study of parameters affecting the strength of yarns, in: *Journal de Physique IV (Proceedings)*, EDP Sciences, vol. 134, August 2006, pp. 1183–1188.
- [40] G. Nilakantan, J.W. Gillespie Jr, Ballistic impact modeling of woven fabrics considering yarn strength, friction, projectile impact location, and fabric boundary condition effects, *Compos. Struct.* 94 (12) (2012) 3624–3634.
- [41] D. Zhu, B. Mobasher, S.D. Rajan, Dynamic tensile testing of Kevlar 49 fabrics, *J. Mater. Civil Eng.* 23 (3) (2010) 230–239.
- [42] X. Chen, F. Zhu, G. Wells, An analytical model for ballistic impact on textile based body armour, *Compos. Part B Eng.* 45 (1) (2013) 1508–1514.
- [43] T.F. Walsh, B.H. Lee, J.W. Song, Penetration failure of Spectra® polyethylene fiber-reinforced ballistic-grade composites, *Key Engineering Materials*, vol. 141, Trans Tech Publications, 1998, pp. 367–382.
- [44] F.J. Wortmann, K.V. Schulz, Non-linear viscoelastic performance of Nomex, Kevlar and polypropylene fibres in a single step stress relaxation test: 2. Moduli, viscosities and isochronal stress/strain curves, *Polym. (Guildf)*. 36 (12) (1995) 2363–2369.
- [45] H.S. Shin, D.C. Erlich, J.W. Simons, D.A. Shockey, Cut resistance of high-strength yarns, *Text. Res. J.* 76 (8) (2006) 607–613.
- [46] G. Nilakantan, M. Keefe, E.D. Wetzel, T.A. Bogetti, J.W. Gillespie Jr, Effect of statistical yarn tensile strength on the probabilistic impact response of woven fabrics, *Compos. Sci. Technol.* 72 (2) (2012) 320–329.
- [47] F. Loix, P. Badel, L. Orgéas, C. Geindreau, P. Boisse, Woven fabric permeability: from textile deformation to fluid flow mesoscale simulations, *Compos. Sci. Technol.* 68 (7–8) (2008) 1624–1630.
- [48] I.G. Lепенies, M. Richter, B.W. Zastrau, A multi-scale analysis of textile reinforced concrete structures, *PAMM* 8 (1) (2008) 10553–10554.
- [49] O. Döbrich, T. Gereke, M. Hengstermann, C. Cherif, Microscale finite element model of brittle multifilament yarn failure behavior, *J. Ind. Text.* (2016). 1528083716674908.

- [50] F. Kun, F. Raischel, R.C. Hidalgo, H.J. Herrmann, Extensions of fibre bundle models, *Modeling Critical and Catastrophic Phenomena in Geoscience*, Springer Berlin Heidelberg, 2006, pp. 57–92.
- [51] R. da Silveira, An introduction to breakdown phenomena in disordered systems, *Am. J. Phys.* 67 (12) (1999) 1177–1188.
- [52] D.L. Turcotte, M.T. Glasscoe, A damage model for the continuum rheology of the upper continental crust, *Tectonophysics* 383 (1–2) (2004) 71–80.
- [53] S.U. Zhang, B.W. Lee, Fatigue life evaluation of wire bonds in LED packages using numerical analysis, *Microelectron. Reliab.* 54 (12) (2014) 2853–2859.
- [54] D. Zhang, M. Abendroth, M. Kuna, J. Storm, Multi-axial brittle failure criterion using Weibull stress for open Kelvin cell foams, *Int. J. Solids Struct.* 75 (2015) 1–11.
- [55] M.M.B. Hasan, E. Staiger, M. Ashir, C. Cherif, Development of carbon fibre/polyamide 6, 6 commingled hybrid yarn for textile-reinforced thermoplastic composites, *J. Thermoplast. Compos.* 28 (12) (2015) 1708–1724.
- [56] I.J. Beyerlein, P.K. Porwal, Y.T. Zhu, K. Hu, X.F. Xu, Scale and twist effects on the strength of nanostructured yarns and reinforced composites, *Nanotechnology* 20 (48) (2009) 485702.
- [57] F. Vollrath, D.P. Knight, Liquid crystalline spinning of spider silk, *Nature* 410 (6828) (2001) 541.
- [58] M. Zhang, K.R. Atkinson, R.H. Baughman, Multifunctional carbon nanotube yarns by downsizing an ancient technology, *Science* 306 (5700) (2004) 1358–1361.
- [59] A.M. Beese, X. Wei, S. Sarkar, R. Ramachandramoorthy, M.R. Roenbeck, A. Moravsky, et al., Key factors limiting carbon nanotube yarn strength: exploring processing-structure-property relationships, *ACS Nano* 8 (11) (2014) 11454–11466.
- [60] R.A. Angelova, *Wearable technologies for helping human thermophysiological comfort, Examining Developments and Applications of Wearable Devices in Modern Society*, IGI Global, 2018, pp. 203–231.
- [61] M. de Kok, H. de Vries, K. Pacheco, G. van Heck, Failure modes of conducting yarns in electronic-textile applications, *Text. Res. J.* 85 (16) (2015) 1749–1760.
- [62] E. Bonderover, S. Wagner, A woven inverter circuit for e-textile applications, *IEEE Electr. Device Lett.* 25 (5) (2004) 295–297.
- [63] J.T.F. Ding, X. Tao, W.M. Au, L. Li, Temperature effect on the conductivity of knitted fabrics embedded with conducting yarns, *Text. Res. J.* 84 (17) (2014) 1849–1857.
- [64] J.S. Roh, Y.S. Chi, J.H. Lee, S. Nam, T.J. Kang, Characterization of embroidered inductors, *Smart Mater. Struct.* 19 (11) (2010) 115020.
- [65] Y.T. Chui, C.X. Yang, J.H. Tong, Y.F. Zhao, C.P. Ho, L.L. Li, A systematic method for stability assessment of Ag-coated nylon yarn, *Text. Res. J.* 86 (8) (2016) 787–802.

This page intentionally left blank

Textile failure analysis and mechanical characterization using acoustic emission technique

11

Carlos Rolando Rios-Soberanis

Centro de Investigación Científica de Yucatán (CICY), Mérida, Mexico

11.1 Introduction

It is very well known that architecture/geometry of textiles has considerable effects on their mechanical behavior being of significant interest for the satisfactory performance of these fabrics as reinforcement in composite materials. For example, textile dimension decreases/increases when the cloth is strained, cloth shrinks when the fibers swell on wetting, and sites of stress concentrations magnify when textiles are stretched by external stresses. Consequently, the stress–strain behavior of the constituent fibers and the fabric structure are the main factors that influence the short-term mechanical properties. Studies determining the effect of fabric geometry have supported numerous parameters to be considered when manufacturing a textile for any application, such as the prediction of the maximum mechanical capabilities by taking in account the main acting directions, dimensional properties, textile behavior on site, among others. Additionally, the geometric structure developed in the fabric depends on the fiber entanglement, fiber orientation in the web, and needling process parameters. Such arrangement will dictate, at the end, the performance behavior of the final textile. The failure mechanisms, then, will be governed by the textile construction and nature. This emphasizes the importance of evaluating the textiles mechanical behavior in order to understand its limits by identifying its mechanical sequence events during the application of external stresses. Acoustic emission (AE) technique exhibits high potential to characterize failure mechanisms in composite materials. In textile-reinforced composites, such damage events can be associated with architecture/geometry of textiles interweaving. In order to correlate AE signals to their corresponding failure mechanisms, signals are to be obtained from textile/epoxy-laminated composites subjected to tensile tests. Several mechanisms take place at different stages of the loading process (e.g., delamination, fiber/matrix interfacial debonding, matrix cracking, fiber/matrix friction and rubbing, and fiber breakage). AE signals perceive the energy of elastic waves when

released by a composite damage event; hence, such energy can be correlated to a specific mechanical event and, at the same time, to a textile geometric interweaving structure.

11.1.1 Textiles architecture

Nowadays, the textile industry has developed advanced technologies allowing the fabrication of fabrics with complex structures and geometries that provide them with particular properties. Several factors can interact in the manufacturing of fabrics such as the distribution of fibers in two or three dimensions as well as combinations of different types (even in nature: i.e., ceramic metallic or polymeric) of fibers in the same textile (named commingled textile). Fabrics can be manufactured by different techniques depending on the architecture they will have at the end. Such techniques lead to knitted, woven, and nonwoven textiles architecture.

Fig. 11.1 exhibits the most common raw materials used for textile manufacturing depending on the nature of the fibers and a wide classification of fabric architecture. Textile materials are characterized by the distinct hierarchy of interweaving geometry structure as stated by Hearle [1] and Lomov et al. [2]. It is indicated that the description of the nature of textiles based on its geometry defines the mechanical behavior and the mechanisms of failure. Therefore the hierarchal sequence in textiles conformed by fiber, yarn, and interweaving are of vital importance to take full advantage of fabric versatility.

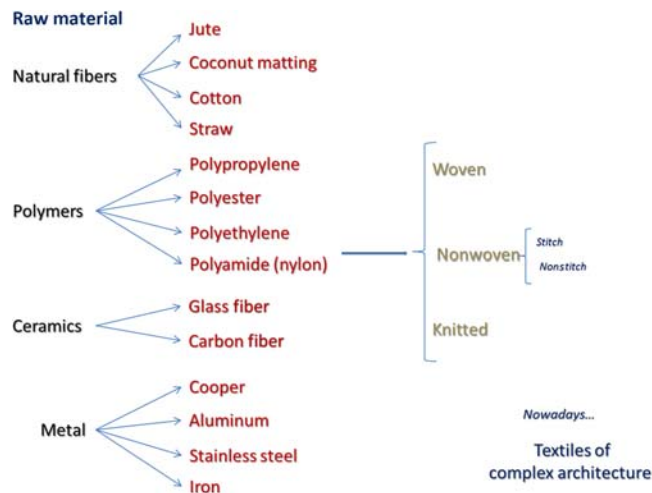


FIGURE 11.1

Most common raw materials for textiles manufacturing.

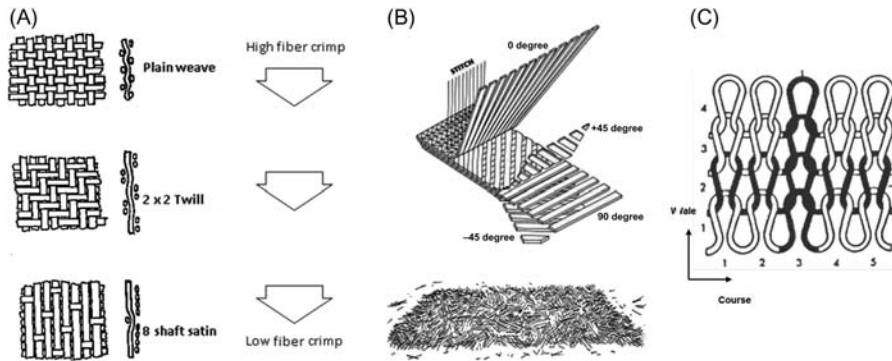


FIGURE 11.2

Textiles architectures: (A) woven, (B) nonwoven: stitched and nonstitched (*mat*), and (C) knitted.

A general accepted textile architecture classification, displayed in Fig. 11.2, can be considered as follows.

Woven: Woven fabrics are fabricated on looms in a wide variety of weights, weaves, and widths. Bidirectional woven fabrics provide good strength in the 0- and 90-degree directions. They allow easy to handle and provide biaxial strengthening when used as reinforcement; however, the waved points, where the fibers are crimped as they pass over and under one another, can act as stress concentration sites when loads can generate damage initiation.

Nonwoven: Such textiles are considered lacking of crimped points since the yarns do not cross over each other and they can be arranged randomly or unidirectionally oriented. Nonwoven fabrics can be subclassified as stitched and nonstitched based on the yarns arrangement. Nonstitched materials are commonly known as mats that are nonwoven fabrics providing equal strength in all directions. These fabrics come in two distinct forms: chopped and continuous strand. Chopped mats contain randomly distributed fibers that are held together with a chemical binder and use to be inherently weaker than continuous strand mats. On the other hand, stitched fabrics are created by placing yarns into the desired alignment and stitching them together. This process allows great flexibility in yarn alignment because the yarns can be laid in practically any arrangement, including orienting all strands in one direction. Moreover, the proportion of yarn in any direction can be selected at will [3].

Knitted: In the textile industry, there is a large range of fabric structures that can be classified as knitted. In general, these fabric architectures may differ in appearance but they are all made up of interlocking loops of yarn. Knitted fabric possesses excellent drapability that makes them to be considered in the manufacture of parts of complex shapes (e.g., parts with double curvatures). The tailorability of these components is varied and depends on the knitted fabric type and

architecture to suit specific structural requirements. The fabric themselves are normally manufactured through modern techniques developed by the textile industry and several knitting geometry configurations are possible to achieve. Since the fabric yarns are oriented in a repeating series of intermeshing loops, the direction of the fibers is changing continuously in three dimensions.

Textiles mechanical properties are intimately related to its architecture, which is simply the geometry of the yarns interweaving. Generally, for structural and engineering applications, the main properties that are normally taken in consideration are stiffness, strength, and resistance to damage growth. However, due to its complicated microstructure, appreciation of mechanical properties of textile materials is still not completely understood especially when architecture exhibits complex conformation. The influence of the variability of the fabric internal geometry on the mechanical properties is estimated depending on the orientation where the external applied stresses act [4].

Textiles are versatile materials used widely in diverse applications from clothing to engineering. Mechanical properties, as in all materials, are prevailing to emphasize when choosing a textile for a determined enforcement. Typical textile mechanical characterization covers, among others, stretching (tension), tear, opening size, nominal thickness, mass per area, punctures strength, etc. All are conditioned by standard designation test method (ASTM). However, to study stress concentration sites that lead to crack initiation and propagation until fabric fracture is quite complicated. Rios et al. [5,6] analyzed fracture damage mechanisms of knitted textile by placing the cloth into epoxy resin employing a simple wet layup process. The result is a composite material, which is completely transparent. Therefore the cloth reinforcement architecture can be seen clearly through the composite and it allows the cracks to be observed running along the sample easily, thus the initiation sites and further processes can be related to the textiles geometry.

11.1.2 Textiles-reinforced composites

Composite materials reinforced with textiles have been widely investigated in the last three decades, and in the recent years, they have received increasing interest from the composite materials community [7–10].

Textile technology is becoming highly applicable in a wide range of new applications such that it is replacing current metal technology in several fields even modifying old traditional composite manufacture processes, such as autoclave and prepregging. The main reason is the potential of the textile-based composite materials for reducing manufacturing costs and improving processability as well as having minimum material wastage and reducing production time. On the other hand, their mechanical properties can be tailored to increase and enhance their engineering performance. In the last years of the 20th century, conferences devoted to composite materials had burgeoning specialized sessions on textile reinforcements [11].

Fabric-reinforced “textile” composites potentially have better out-of-plane stiffness, strength, and, toughness properties than tape laminates. They are also amenable to automated and efficient manufacturing techniques. However, the architecture of a textile composite is complex, and therefore, the parameters controlling its mechanical properties are numerous. Considerable research has been directed at understanding the behavior of composites made using a textile-manufacturing technique in an attempt to gain industrial acceptance [12,13]. Over the past years, the textile-manufacturing industry has developed the ability to elaborate net-shaped fabrics using highly automated techniques, such as stitching, weaving, braiding, and knitting. In the manufacture of preforms for advanced composite materials, textile technology has been under intensive investigation due to the potential of these to produce low-cost high-quality structures with improved mechanical performance.

Investigations concerning to damage development and fracture mode are important for two reasons: first, in order to give confidence in the wider use of these materials and, second, to gain a better understanding of damage accumulation, which is intimately related to their energy-absorbing characteristics.

11.1.3 Acoustic emission technique

In order to predict the mechanical behavior of a material during its service life, it is important to evaluate its mechanical response under different types of external stresses by studying the initiation and progression of damage along the effects induced by external environmental factors and degradation. The onset of damage is related to the structural integrity of the component and its fatigue life. For this, among other reasons, nondestructive techniques such as AE have been widely used at the present time for almost any kind of materials characterization. This method has demonstrated excellent results on detecting and identifying initiations sites, cracking propagation, and fracture mechanisms of polymer matrix composite and ceramic materials

The understanding about damage generation from initiation and propagation until total fracture is of great importance for the materials design, development, and application. Microstructural failure mechanisms are strongly dependent on several factors in any material such as the nature of the components, reinforcement/matrix interface, volume fractions, reinforcement geometry, etc. Several studies have proved the efficacy of AE technique by identifying different types of damage mechanisms; for instance, fiber-reinforced composites exhibit matrix damage (e.g., matrix microcracking, coalescence of microcracks, matrix/matrix friction), interfacial debonding (e.g., fiber/matrix, bundle/matrix), fiber/matrix and fiber/fiber friction, and fiber and bundle breaks [14–16].

The AE technique is based on the detection of acoustic waves from ultrasonic level generated by the process of a rapid propagation of a microflaw or another kind of mechanical energy source [17,18]. Piezoelectric sensors perceive the emitted signal from the damage notation site by the surface dynamic movement and

convert it in an electrical response. Generally, a frequency between 50 kHz and 1 MHz is used since lower frequencies are related to external noise produced by environmental racket including equipment vibrations.

Undoubtedly, the acoustic signal response is very versatile due to the wide variety of parameters that can be identified. Fig. 11.3 exhibits the obtained data through the AE technique where it is possible to correlate the registered signals to the failure mechanisms events of a given material identifying damage development stages, such as microcracks, delamination, interface failure, matrix cracking, fiber breakage, etc. The most important setting for the reliable acquisition of AE information is the threshold value since only signals that cross the predefined threshold level are registered and evaluated. By this means, acoustic signals emitted by the material under stress (e.g., tension, compression, and bending) are isolated from external noise due to equipment vibrations [19].

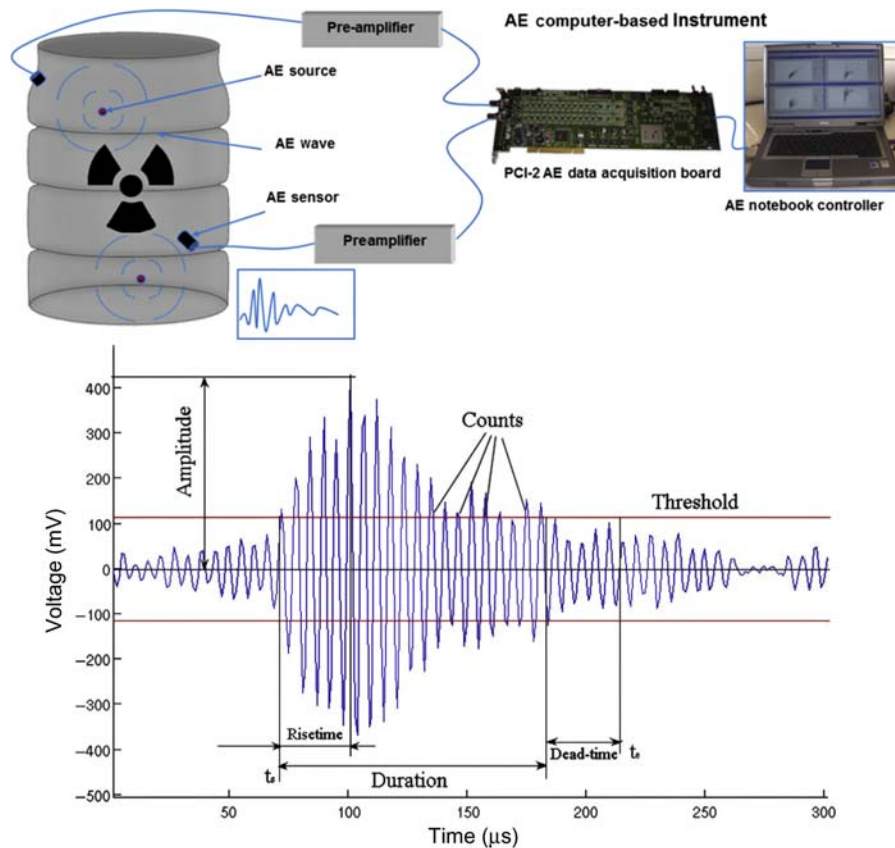


FIGURE 11.3

Typical parameters of AE signals.

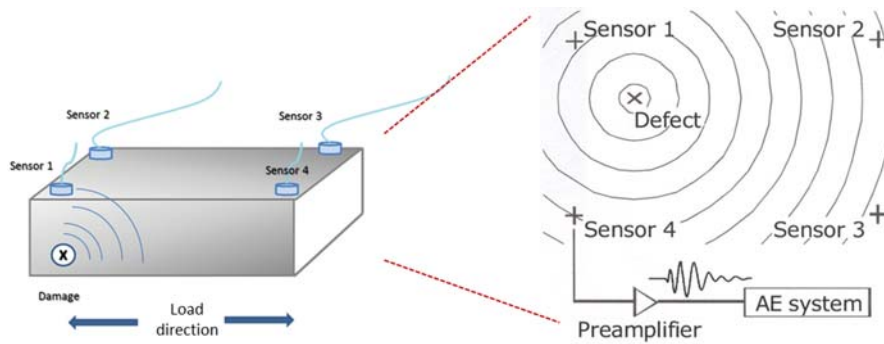


FIGURE 11.4

Damage source location by using an AE technique.

When a material is stressed, there is a natural opposition to deformation (resistance), once a certain point is reached; system discontinuity appears in the materials as microdamage. When damage initiates, energy is released, in consequence, elastic/mechanical waves are transported through the bulk in a velocity that depends on the material's nature. Such ultrasound signals are collected by the AE sensors. Therefore one of the most important features of AE technique is the ability to localize the source of a damage event [20]. Following the evolution of source locations, the technique can lead to a better insight into the materials behavior under load resulting in the four-dimensional image of defect developments. By using the adequate number of sensors, covering an area of the analyzed material, mechanical waves will take an arrival time to get the sensors, and extrapolating these arrival times, the source can be found (Fig. 11.4).

AE is defined by ASTM E 610 as "...the class of phenomena whereby transient elastic waves are generated by rapid release of energy from localized sources within a material, or the transient elastic waves so generated" and by The American Society For Nondestructive Testing as "... the elastic energy that is *spontaneously* released by materials when they undergo deformation."

11.1.4 Textiles mechanical and damage characterization

Several researchers, from different applicative and characterization perspectives, have studied mechanical behavior for textiles. In our personal circumstance, fabrics have been analyzed depending on its application or their architecture/geometry structure; therefore three different scenarios will be discussed next.

Case 1—Damage analysis in knitted fabric

Among textile techniques, knitting is particularly suited for the manufacture of composites with elaborated three-dimensional shapes since structures with complex profiles can be very difficult and expensive to manufacture using standard

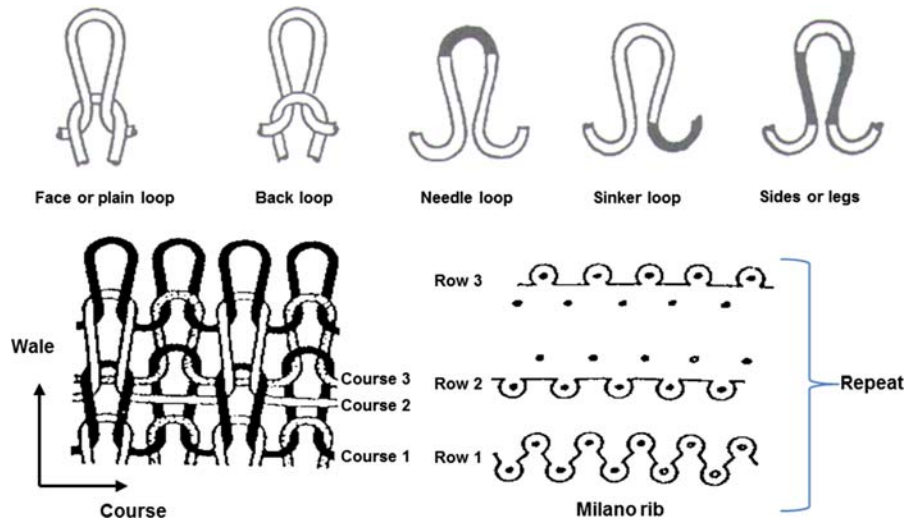
prepreg or wet layup technology. This advantage of knitted fabric (i.e., the ability to be shaped) is due, as it is well known, to its fiber architecture. The use of knitting technology with advanced fibers, such as glass, carbon, and aramid, to produce near-net-shape fabrics has received increasing interest in recent years from the composite materials community. Knitted fabrics have the potential of being used in engineering structures with complex shapes in conjunction with a suitable liquid molding technique, such as resin transfer molding, due to their excellent drapeability and manufacturability. Composites of this type, based on knitted fabrics, are a relatively new class of materials and extensive analysis in the area of damage development has not yet been carried out.

Such investigations are important for two reasons: first, in order to give confidence in the wider use of these materials and, second, to gain a better understanding of damage accumulation, which is intimately related to one of the major advantages of knitted fabric composite (i.e., their energy-absorbing characteristics).

Many authors have commented on the development of damage during tensile loading of knitted-fiber composites. There has been general agreement that (1) for loading in the wale direction, the initiation of damage occurs due to fiber bundle/resin matrix interface failure at the needle or sinker loops, with subsequent linking of this damage to form long cracks; and (2) for loading in the course direction, the damage initiates from the sides or legs of the loops [21–23]. However, in most studies to date, it has only been possible to draw conclusions based on analysis of the final fracture surfaces of the composites.

The architecture of knitted fabrics involves many important factors that contribute to their final behavior, including loop length and loop shape. Knitted loops are arranged in rows and columns, roughly equivalent to the weft and warp of woven structures and are termed courses and wales, respectively. A course is a horizontal row of loops produced by adjacent needles during the same knitting cycle. A wale is a vertical column of loops produced by the same needle knitting at successive knitting cycles, and thus intermeshing each new loop through the previous loop. The wale direction is stiffer, whereas in the course direction, the flexibility is higher. Such orientations are strategic to understand the fabric mechanical behavior. Fabric architecture used in this research is schematized in Fig. 11.5. In the Milano weft rib structure made up of glass fiber, each repeat consists of three courses—two rows of single threads, knitted together by a row of 1×1 rib. Consequently, the resultant fabric is balanced, that is, both the face and the back surfaces are identical in construction. In courses 2 and 3, the fiber tows are knitted only in one face, either front or back, whereas course 1 (the rib thread) holds together courses 2 and 3.

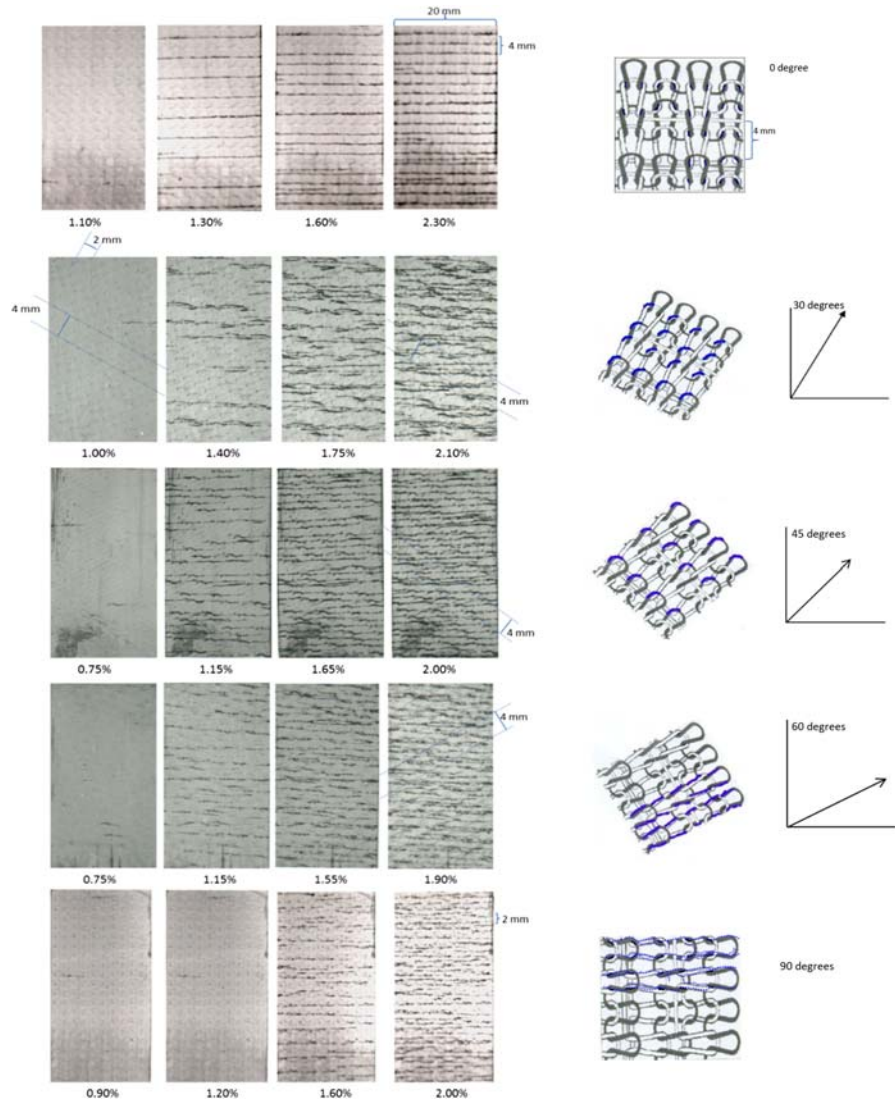
In order to observe the damage development and the architecture effect on cracking progression, a textile-reinforced composite was manufactured. The process consisted in placing a single cloth of Milano weft-knit textile produced from E-glass yarns to be impregnated with an epoxy resin and cured at high temperature (wet layup). This technique produced highly transparent, void-free laminates with a fiber volume fraction of $13\% \pm 0.5\%$. The single fabric in the composite

**FIGURE 11.5**

Schematic diagrams of the Milano weft-knitted fabric structure.

can be easily observed through the composite so crack progression can be monitored and correlated to its geometry. Laminates were made with the knitted fabric oriented at different angles by varying the direction of the fabric in order to cut off samples at 0-, 30-, 45-, 60-, and 90-degree angles. Coupons were mechanically tested on tensile mode and AE sensors were attached to the surface of the samples in order to identify ultrasonic wave signals coming from microdamage events. Fig. 11.6 exhibits the damage sequence and cracking progression of the knitted composites under tensile stress for each angle tested. It is clear that the cracking pattern that it develops is intimately connected with the fabric architecture along the orientation of the acting stress. Sequence of events indicates that for wale direction (0 degree), significant cracks appeared first at 1.10% strain in the plane, where the float stitch of courses 2 and 3 are together possibly due to the strain magnification caused by adjacent tows. These cracks have a spacing of 4 mm, which is the dimension of the repetitive unit in the knitted fabric. When these sites have been used, then at a higher strain, cracks develop where the rib holds threads 2 and 3, giving a final crack spacing of 2 mm, which is the wale direction dimension of a single loop. Onward, from 30, 45, and 60 degrees, damage onset decreases reducing mechanical properties and crack density augments since flaws can run freely along the loops legs. Knitted fabric crossover yarn points seems to be dictating the path for cracking development.

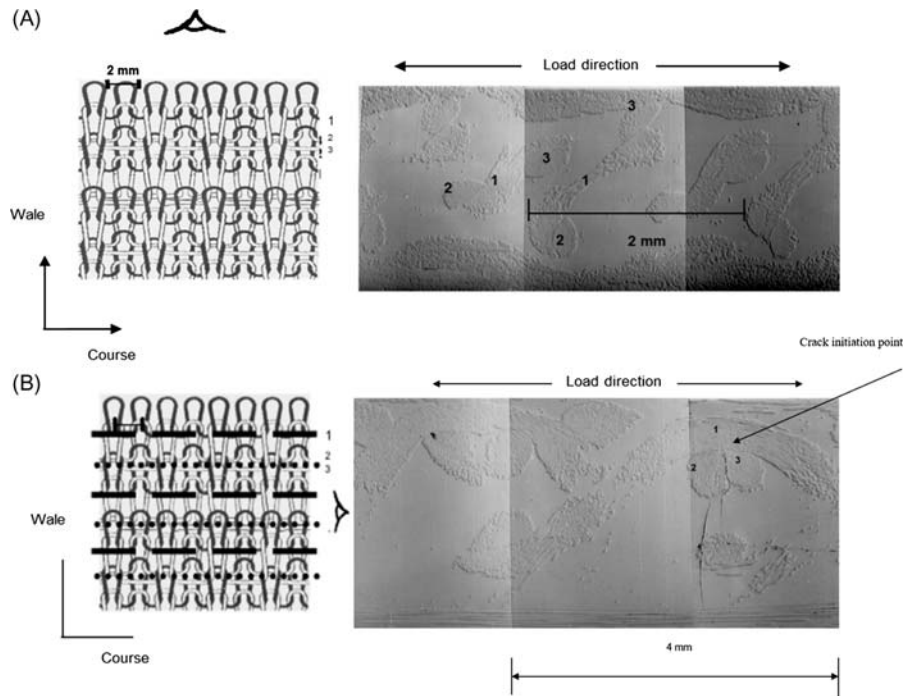
Crack density increases fairly uniformly for 90-degrees sample since cracks are able to propagate freely and grow following the legs of the loops. As the cracks curve to follow the needle, or head, of the loops, they cross a loop and, hence, take on a branched appearance. In this orientation, it is easy for the

**FIGURE 11.6**

Crack development for the samples with the fabric layer in the 0-, 30-, 45-, 60-, and 90-degree directions.

developing matrix cracks to link up as they grow. All these sites coalesce to propagate the cracks along the sample perpendicularly to the load direction by running on the sides or legs of the loops.

Sections from the tested samples were cut out using the diamond saw, mounted in epoxy resin, and polished in order to obtain information about

**FIGURE 11.7**

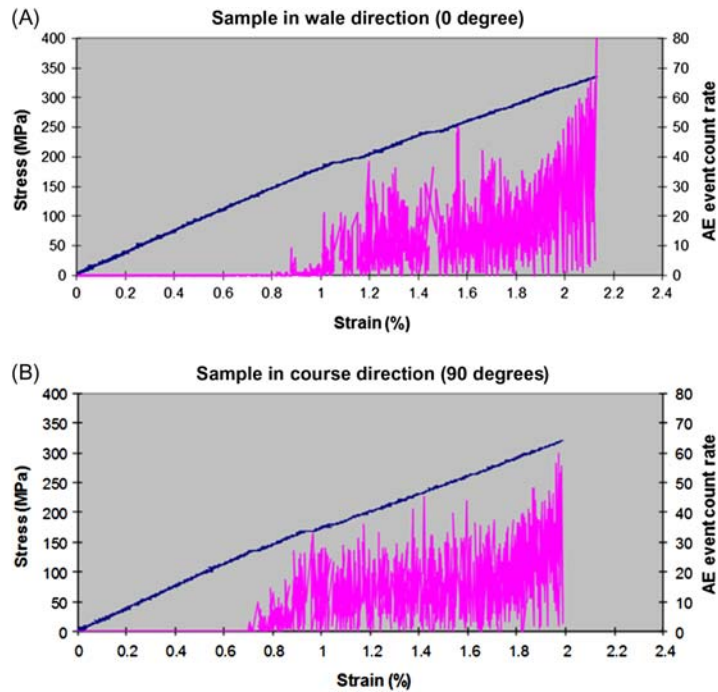
Structure of the knitted fabric composite (A) viewed perpendicular to the load direction and (B) viewed parallel to the load direction.

damage initiation and cracking development in relation to the knitted fabric architecture. Samples were taken both parallel and perpendicular to the loading direction (Fig. 11.7). Crack initiation points can be observed in the crossover yarns. Damage first appears as fiber/matrix interface failure.

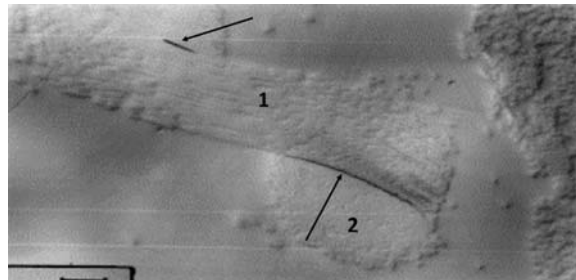
AE technique assisted the damage onset identification and the location of the microcracks. Fig. 11.8 exhibits stress–strain AE events curves for samples tested at 0 and 90 degrees. AE signals are early detected at 90 degrees ($\sim 0.7\%$) in comparison with samples at 0 degree and, in both cases, elastic waves are sensed before significant damage is noticed indicating that such flows are at microlevel. When the stress is increased, energy and amplitude signals are higher correlated to fiber/matrix debonding/friction, matrix cracking, and fiber fracture.

Samples were transversally cut when first AE signal was detected in order to observe the composite cross-sectional area into microscope to relate crack onset to textiles architecture. It was found that microdamage initiated in the crossover yarn points of the knitted fabric (Fig. 11.9).

The influence of the knitted fabric architecture in determining the damage development in the knitted textile reinforce composites was shown in the results.

**FIGURE 11.8**

Stress—strain AE events for textile composite at (A) 0 and (B) 90 degrees.

**FIGURE 11.9**

Micrograph showing microdebonding between courses 1 and 2 crossover point in a coupon tested in the wale direction.

The identification of the predamage as occurring in the crossover points in the knitted fabric architecture was identified and determined by the transparency of the coupons, together with the employment of the AE technique. It was demonstrated that the most important sites for predamage occur in the fabric planes where the float stitch of courses 2 and 3 are held together. In all the samples, the

development of the matrix cracks was observed to initiate at these crossover points perpendicular to the load direction and independent of the angular orientation.

Case 2—Damage sequence in multiaxial noncrimp fabrics

Noncrimp fabrics (NCFs) have fulfilled the requirements of manufacturing multiaxial textiles by knitting threads in order to bind together multiple layers of large unidirectional yarn bundles; besides, they combine a perfect placement of the reinforcing fibers with easy, inexpensive, and automated manufacturing to produce textiles-reinforced composites [24,25]. Mechanically speaking, the threads do not bear the loads (external stress), but hold uncrimped yarns in place prior to the infusion of resin. However, binding threads can be considered as stress concentrators since they stitch all the layers crossing them transversally. By this reason, mechanisms of failure in the NCFs may generally be linked to crimp in the tows.

A great advantage in NCFs manufacture is that they are textile preforms with multiaxial layers of fiber bundles stitched together in directions desired for structural design. During its construction, NCFs are stacked in an established sequence in order to provide multiaxial strengthening at different angles. The architecture of the NCFs implies that these materials are heterogeneous not only at the micro-scale but also on the mesoscale (fiber bundles) [26].

The form and architecture of the fibrous reinforcement is perhaps the single most important component determining both the performance and cost in a textile composite material. It is very well known that any reliable damage tolerance analysis (experimental and model estimations) for textiles-reinforced composite structures must involve criteria based on failure mechanisms where fabric architecture/geometry plays a very important role in its mechanical performance.

In this experimental case 2, an attempt to contribute to the understanding of the damage and degradation process in tension load of E-glass fiber multiaxial non-NCF-reinforced epoxy composites is discussed. The particular interest of this study is the development of damage during tensile loading in order to identify the initiation sites and cracking progression until total fracture of the material to be related to textile reinforcement architecture.

A multiaxial E-glass textile exhibiting a mass per unit area of $972 \pm 5\%$ g/m² and a $[0, +45, 90, -45]$ degrees stacking sequence was used as a reinforcement in the epoxy resin matrix composite. The layers have relative mass fractions as indicated in Table 11.1 and are stitched together with a polyester multifilament binding yarn. The unidirectional fiber bundles in the four planes are held together by a fine polyester thread in both sides. In the outer layer, where fibers are running at 0 degree, the polyester (PES) thread is knitted in a zig-zag pattern and in the other side (outer layer at -45 degrees) is knitted at 0 degree.

Fig. 11.10 illustrates both sides of the Z-pinned multiaxial textile. Epoxy system chosen was completely transparent when cured allowing visualization of the textiles architecture. Using one layer of textile and epoxy resin system as a

Table 11.1 Technical specification of the textile.

Layer orientation (degrees)	Weight (g/m ²)	Composition
0	354	E-glass
+45	200	E-glass
90	207	E-glass
−45	200	E-glass
Knit yarn	11	Polyester
Total	972 ± 5%	

matrix, a composite with a fiber volume fraction of about 18% was manufactured by wet layup process. Laminas were elaborated taking in account the two principal direction layers that conform the multiaxial textile (0 and 90 degrees).

A Shimadzu universal machine was used to carry out mechanical tests in tension. The textile direction in question was parallel to the load direction in which the samples were loaded to progressively higher strain at a cross-head speed of 0.5 mm/min. AE sensors were attached to the sample, as shown in Fig. 11.11A, to obtain the signal in order to be related to damage development and mechanisms of fracture.

During tensile loading, in each stress–strain curves, several discontinuities related to significant cracks developed transversally to the sample were observed. At a higher strain, discontinuities were more frequent; the higher the load, the higher amount of cracks are formed. Samples at 90 degrees exhibited slightly higher crack density (cracks per area). Apparently, textile geometry (stacking sequence) does not affect significantly the mechanical parameters values; however, damage development and propagation along samples depending on the loading direction are influenced by the polyester knit yarn structure (Fig. 11.12).

The load (N)–time (s)–amplitude curves for samples when the textile is oriented at 0 degree is presented in Fig. 11.12A. AE signals detected over 280 seconds are intimately related to the first deflection in the curve, which is also correlated to the appearance of the first significant damage that emits high energy. This damage is associated to matrix cracking in the composite. At 90-degree direction (Fig. 11.12B), it is possible to observe more small discontinuities related to the AE signals that are identified as matrix cracking. Energy is augmented rapidly as the more cracking damage occurs. In all the samples tested in tension (0 and 90 degrees), no fiber breakage was observed or AE detected until total fracture happened. Samples at 90 degrees exhibited an early damage development in comparison with 0 degree. Lower damage initiation at 90-degree samples was apparently due to the major amount of unidirectional glass fibers at 0-degree layer (denser), which is positioned perpendicular to the applied stress and allows more transversal matrix cracks to be developed along the coupon.

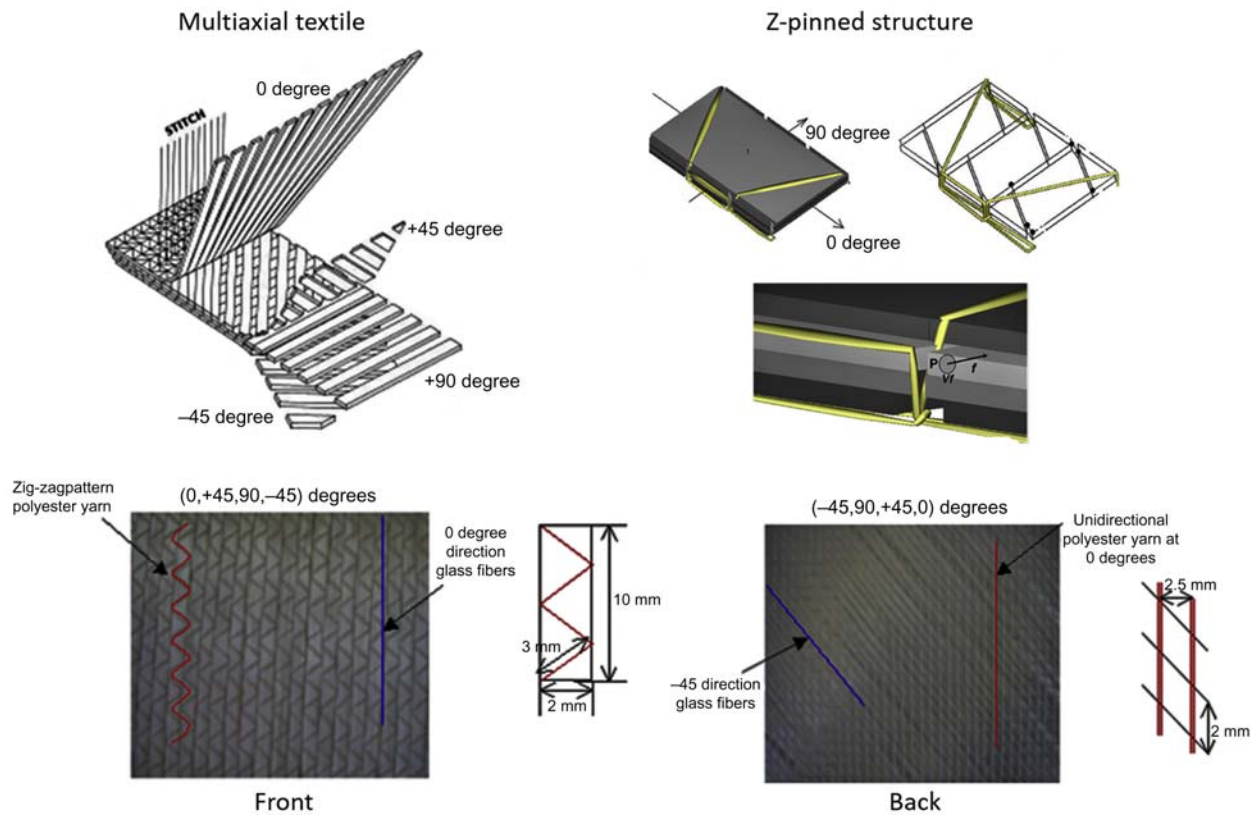
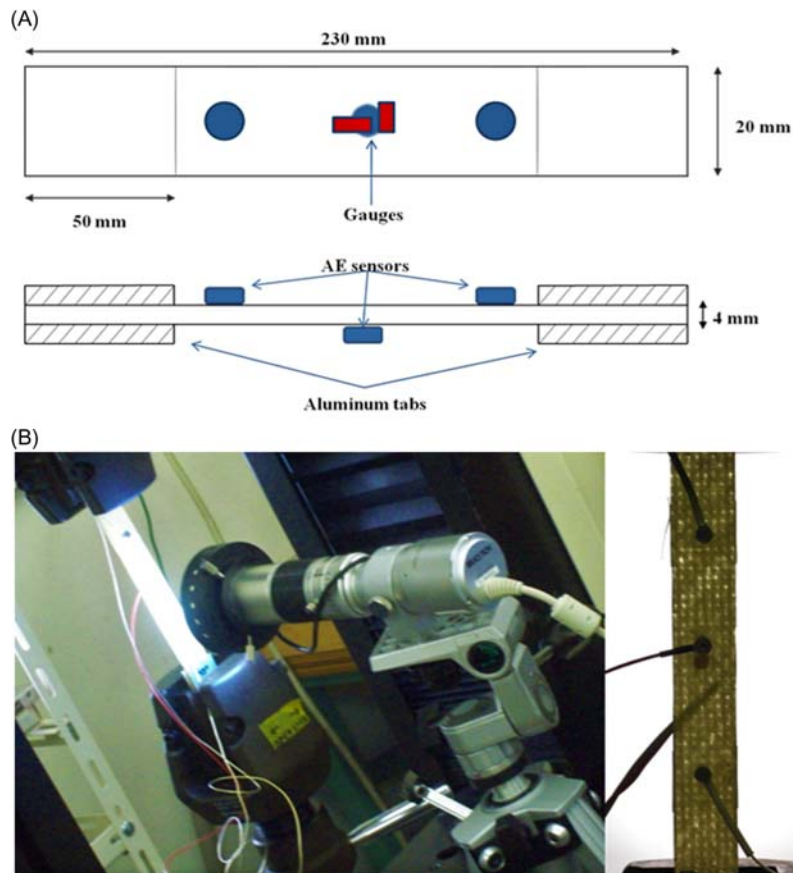


FIGURE 11.10

Z-pinned multiaxial textile architecture/geometry.

**FIGURE 11.11**

(A) Sample dimensions and AE sensors locations. (B) Load recording in tension test.

Case 3—Mechanical properties of geotextiles: effect of interweave geometry

In order to properly characterize mechanical parameters such as deformation patterns in textile materials and to model individual yarn behavior, the size, shape, and mesolevel deformation locations of the yarns need to be verified as a function of applied strain. The mechanical properties of geotextiles are mainly dominated by the nature of fiber used and the weaving parameters; therefore it is necessary to exhibit the correlation between the applied external deformations, microscopic flaws, and mesolevel changes with the purpose of describing the changes in the internal structure of the system subjected to different external applied stresses. Nowadays, geotextiles are widely used in geotechnical, environmental, hydraulic, and a diverse range of engineering applications [27,28]. Textile

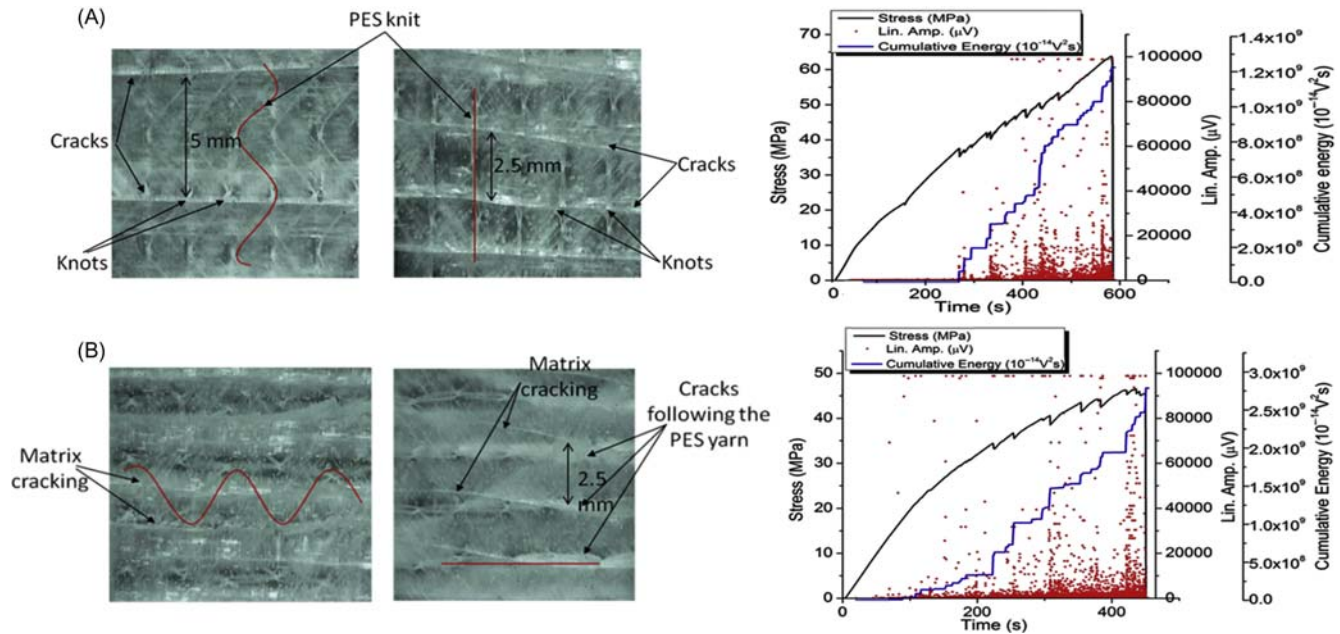


FIGURE 11.12

(A) Textile oriented at 0 degree. (B) Textile oriented at 90 degrees.

manufacture technology allows obtaining fabrics with different kind of interweaving from simple-to-complex structures; such textile geometry plays a vital role in the mechanical capabilities.

Through the identification of the initiation sites, the coalescence of cracks is possible to correlate the damage with the textile geometry and its effect on the mechanical properties. Such variations are intimately correlated to the textiles structure. Additionally, fractography is frequently used as a tool to identify governing mechanisms and link them to the material internal configuration.

Two geotextiles of different geometry were used to manufacture a polymeric matrix based composite material. First one is made of polyethylene-terephthalate (PET) fibers constituted by unidirectional yarns (woven-type) biaxially oriented in longitudinal and transversal directions. Transversal yarns are wider presenting higher amount of filaments than in longitudinal direction. Second one is a polypropylene (PP) based geotextile constituted by a diagonal (longitudinal) and transversal “tape-like” yarns having an oval shape in the intersections. It is noticeable that PP textile exhibits imperfections in the surface such a microcracks (Fig. 11.13A). In order to observe the onset and development of damage to identify the fracture progression, the textiles were placed in an epoxy matrix to manufacture a composite material. Geotextiles were mechanically characterized individually in tension mode in both longitudinal (0 degree) and transversal directions (90 degrees), in order to compare their mechanical behavior. Composite materials were tensile tested in a Universal Machine Shimadzu using 5-kN load cell at 0.1 mm/min in order to observe the crack progression along the sample.

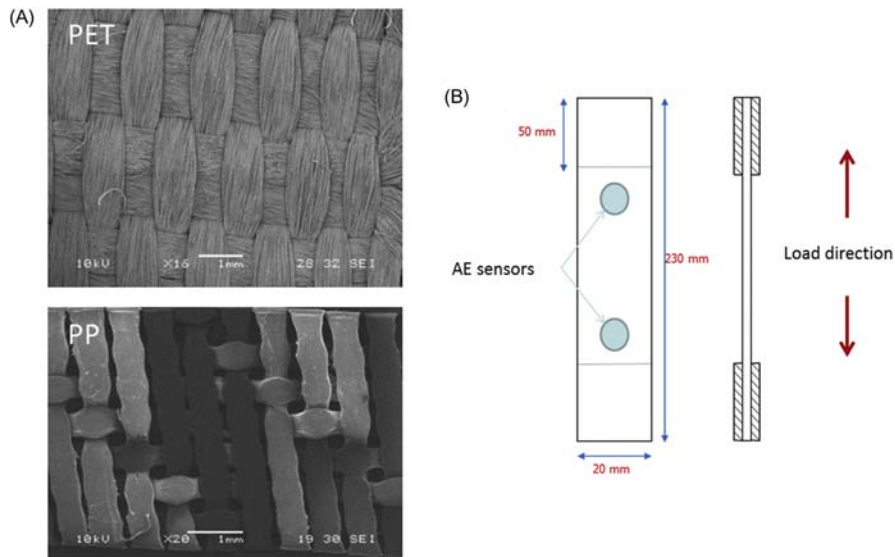


FIGURE 11.13

(A) Geotextiles architecture (PET and PP). (B) Tensile test composite sample.

AE sensors were attached to the composite samples in order to monitor damage in “real time” (Fig. 11.13B).

Results of mechanical behavior in tensile test coupled to AE analysis for epoxy/geotextile composites are shown in Fig. 11.14. Fig. 11.14A shows the stress–strain behavior of epoxy/PET geotextile composite tested in the longitudinal and transversal directions, respectively. In the longitudinal direction, first EA signals are sensed at about 1.75% strain with high amount of signals with high amplitude (50–80 dB) less disperse. Such signals can be correlated to interfacial damage and matrix crack progression. On the other hand, the stress–strain curve when epoxy/PET geotextile is tested in the transversal direction, AE signals were detected significantly sooner compared with the longitudinal sample. The stress–strain curve for epoxy/PP geotextile composite tested in the longitudinal and transverse directions are presented in Fig. 11.14B. In longitudinally tested composites, AE signals showed up at lower strains (0.6%) after linearity but with higher amplitude. Higher cracking density was found between 0.7% and 1.2% strain, which may be related to the extensive crack progression generated by the complex textile geometry. Mechanical tendency observed for epoxy/PP geotextile composites tested in the transversal direction is somehow similar to longitudinal samples; however, transversal samples showed higher density of AE signals of significant amplitude. This effect may be related to the fact that this direction presented major stress resistance; therefore it requires more load and consequently the damage generated in the composite can be larger.

Failure sequence for both PET and PP epoxy/geotextile composites are shown in Fig. 11.15. This evaluation based on the AE technique is supportive to understand and recognize, by identifying the elastic waves’ parameters to be correlated with failure mode, the maximum and minimum strain locations due to the fact that these sites indicate the probable damage initiation zones. Some authors [15,29–31] have qualitatively correlated the mechanical energy released from fiber/matrix debonding, cracking, and fiber-fracture processes with the measured AE waves energy and have shown that AE amplitude ranges are related to specific damage mechanisms. Therefore during experimental mechanical test of geotextiles-reinforced composites in the tensile mode, the sequence of damage process can be correlated to the influence of textile interweaving geometry. Initially, external load is transferred from epoxy matrix to the textile reinforcement; unidirectional yarns parallel to load direction endure the stress allowing its distribution to concentration sites. AE signals appeared at the early stage of loading exhibiting 30–58 dB amplitude correlated to matrix damage (e.g., matrix microcracking, coalescence of microcracks, matrix/matrix friction). At low strains, transversal yarns interfacial debonding generates filament separation and friction (low energy and amplitude AE signals). Then, microcracks due to fiber/matrix delamination process are initiated in load axis parallel yarns and arrested at the interface; these delamination cracks act as a shielding source and delay the formation of macrocracks. As load increases, events such as matrix plastic deformation and cracking, fiber pullout generates amplitude values observed between

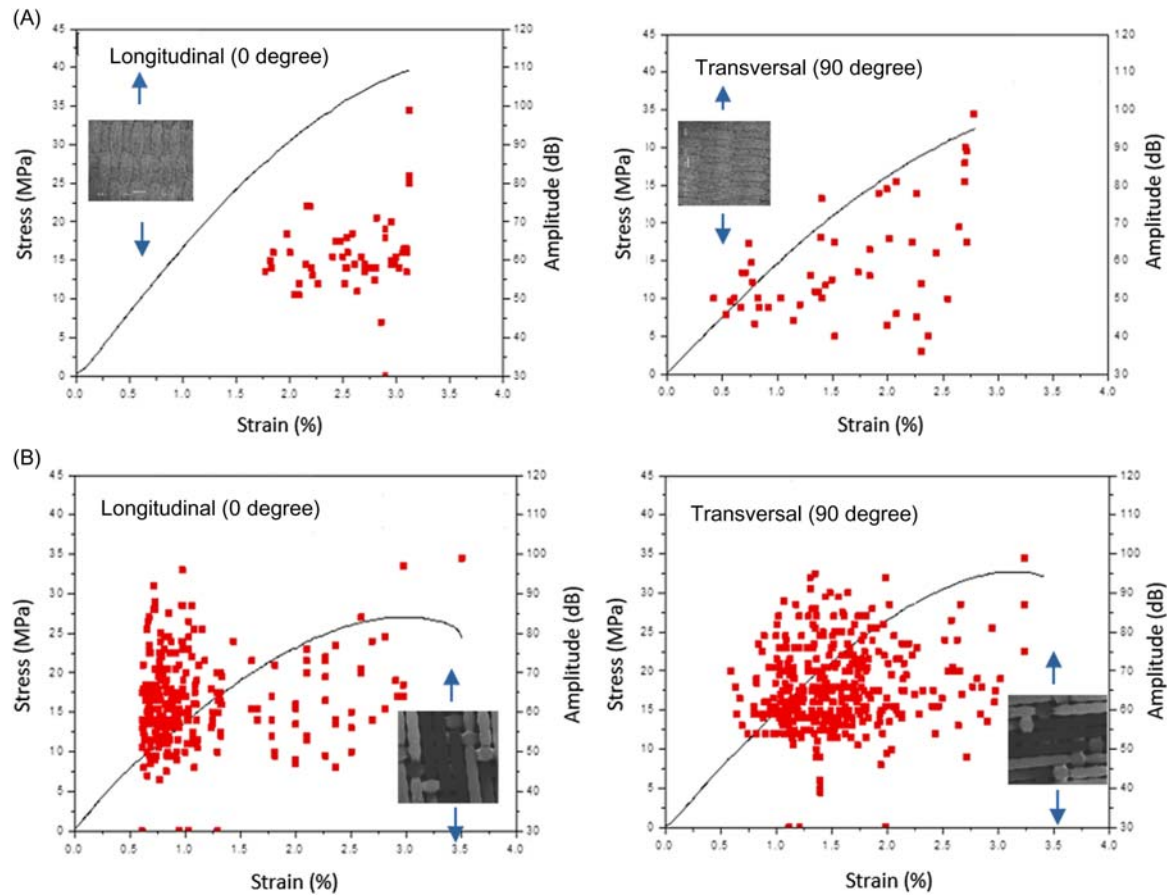


FIGURE 11.14

(A) Epoxy/PET geotextile composites. (B) Epoxy/PP geotextile composites.

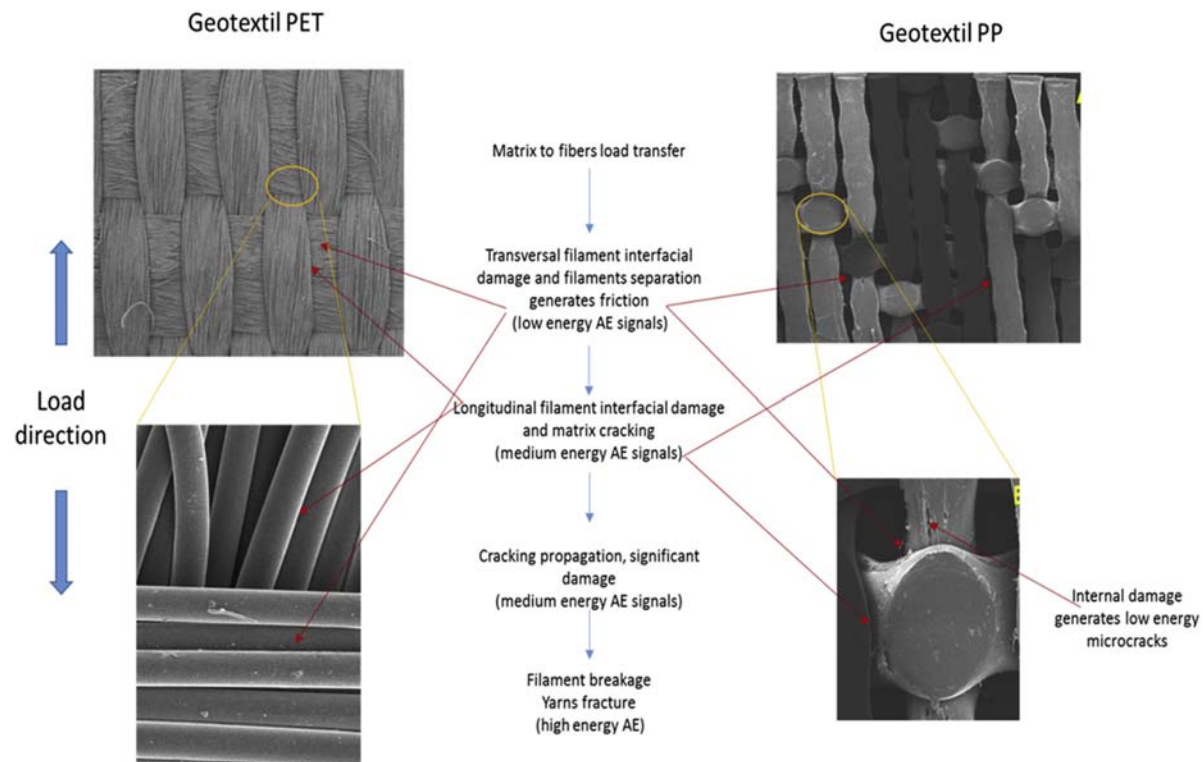


FIGURE 11.15

Failure sequence observed in PET and PP geotextiles-reinforced composites.

59 and 69 dB, which are associated with interfacial fiber/matrix damage progression. After 70 dB and higher amplitude, AE signals can be possibly connected to significant matrix cracking damage and fiber/bundle fracture.

11.2 Conclusion

In all the three cases discussed here, it has been demonstrated that architecture/geometry of the textiles are an essential factor that influences the mechanical behavior of a composite material and that it is dependent on the loading axis orientation. Damage cracking initiation, development, and progression are governed by the way in which fiber bundles are interweaved. In case 1, the influence of the knitted fabric architecture in determining the damage development in the composites was demonstrated in the results. The angle of the fabric to the loading direction determines whether damage is dominated by the head (or needle) of the loops, or by the legs of the loops. First, the observed predamage in the knitted fabric layer was identified for the first time to be damage occurring at the loop crossover points. Other authors have speculated that damage onset occurs around the head or needle of loops, but this work has shown the loop crossover points to be the sites of the first damage. Debonds at the yarn/matrix interface initially develop from these sites evincing that the critical sites for further development of damage are the fabric planes where the float stitch of courses 2 and 3 are held together by the rib stitch (course 1). This is probably because of the strain magnification here.

In case 2, damage cracking development was affected by the polyester thread (zig-zag and unidirectional). The influence of the textile stitched geometry in the initiation sites and the damage development of the composite were validated. Zig-zag polyester knit yarn was observed to govern the transversal crack development while unidirectional (0-degree) polyester thread only influenced the damage by the stitch knots and when it was transversally oriented to the load direction. In the 90-degree direction, transversal flaws were formed due to fiber/matrix debondings that coalesce at higher strain to generate the significant cracks. These visible cracks have a nearly equidistant spacing and are equally distributed in the +45- and -45-degree plies. First AE signals detected during tensile tests were identified as damage induced in the material at the fiber/matrix interphase and demonstrated how damage initiation is affected by NCF orientation.

In case 3, mechanical properties were studied in geotextiles made up of PET and PP, both textiles with different architecture/geometry of waving. AE signals in epoxy/PP geotextile composite exhibited higher crack density in comparison to epoxy/PET geotextile composite. This effect indicates that epoxy/PP geotextile composite generates more damage events during tensile tests, which are intimately related to the complexity of threads architecture/geometry that originates active concentrations sites that release energy when having internal damage.

AE technique was employed to identify the mechanisms of fracture by correlating the signals of mechanical waves produced my damage with fracture stages.

As stated by Godin et al. [32], it is not easy to evaluate the AE signals from a composite material due to its heterogeneity and complexity. However, certain mechanical events are fully understood. Additionally, the effect of textile architecture on the composite mechanical properties and damage pattern can be correlated with its damage initiation and propagation. Through a combination of experimental work and theoretical studies, the mechanisms controlling the mechanical behavior can be explained.

11.3 Future trends

By drawing on the wealth of new knowledge in advanced materials, advanced fiber technology and composite systems are entering a new phase of development. In the next 50 years, significant innovation is expected in future development of low-cost, high-performance textile-reinforced composite materials to be applied in automotive, aerospace, wind, and industrial applications. Lightweight and cost reduction are the two significant trends in various segments of materials science in order to expand the employment of complex textile structures as reinforcement or even in sole applications, such as in geotechnical engineering. Advances in the understanding of structure/property relationships of materials and related processing technologies have made it possible to tailor-make new material systems, to meet specific engineering needs better than conventional materials. Consequently, it is sure that upcoming studies will be directed on novel fiber and matrix systems, fiber architectures, and processing techniques, as well as improving the existing technologies. The successful implementation of new textiles geometries requires rigorous analysis, theoretical and experimental, on fabric capabilities to withstand external events that can deteriorate them. The use of complex 3D textiles will bring more parameters and processes to be optimized. For example, fiber–matrix interface interactions are of great significance when designing composites. Enhancing adhesion mechanisms in the fiber–matrix area by functionalizing fiber surface has been matter of profound investigations and certainly, this tendency will continue in the near future. Experimental on appropriate chemical coupling agents and interface modification techniques will be carried out to favor textiles wettability, which is determined by the balance of surface energy in the interface and depends on the chemical nature of the fiber surface and the fiber geometry. Added to this, the application of new characterization techniques, such as AE, will be optimized as a necessity to identify damage mechanisms related to the textiles architecture to estimate its life service. AE technique will be used as self-monitoring system with the purpose of sensing not only a composite structural environment but also respond to prevent structural failures. In the future, with the advance of fiber technology, it will be possible to enable development of tailored high-performance textiles providing most efficient solutions to engineering problems.

References

- [1] J.W.S. Hearle, Engineering design of textiles, *Indian J. Fibre Text. Res.* 31 (2006) 134–141.
- [2] S.V. Lomov, G. Huysmans, Y. Luo, R.S. Parnas, A. Prodromou, I. Verpoesta, et al., Textile composites: modelling strategies, *Compos. Part A: Appl. Sci. Manuf.* 32 (10) (2001) 1379–1394.
- [3] D.J. Spencer, *Knitting Technology*, second ed., Woodhead Publishing Limited, 1998.
- [4] H. Lin, X. Zeng, M. Sherburn, A.C. Long, M. Clifford, Automated geometric modelling of textile structures, *Text. Res. J.* 82 (16) (2011) 1689–1702.
- [5] C.R. Rios, S.L. Ogin, C. Lekakou, K.H. Leong, A study of damage development in a weft knitted fabric reinforced composite. Part 1: Experiments using model sandwich laminates, *Compos.: Part A* 38 (2007) 1773–1793.
- [6] C.R. Rios, S.L. Ogin, C. Lekakou, K.H. Leong, A study of damage development in a weft knitted fabric reinforced composite. Part 2: Stress–strain and early cyclic behavior of composite laminates with realistic fabric layups, *Compos.: Part A* 38 (2007) 1794–1808.
- [7] R.A. Naik, P.G. Ifju, J.E. Masters, Effect of fibre architecture parameters on deformation fields and elastic moduli of 2-D braided composites, *J. Compos. Mater.* 28 (1994) 656–681.
- [8] C.M. Pastore, Opportunities and challenges for textile reinforced composites, *Mech. Compos. Mater.* 36 (2) (2000) 97–116.
- [9] P. Potluri, A. Manan, Mechanics of non-orthogonally interlaced textile composites, *Compos. Part A: Appl. Sci. Manuf.* 38 (4) (2007) 1216–1226.
- [10] F. Stig, S. Hallstrom, Effects of crimp and textile architecture on the stiffness and strength of composites with 3D reinforcement, *Adv. Mater. Sci. Eng.* 2019 (2019) 1–8.
- [11] S.L. Ogin, Textile-reinforced composite materials, in: A.R. Horrocks, S.C. Anand (Eds.), *Handbook of Technical Textiles*, Woodhead Publishing Limited, 2000, pp. 264–281.
- [12] M.K. Bannister, Challenges for composites into the next millennium—a reinforcement perspective, *Compos. Part A: Appl. Sci. Manuf.* 32 (7) (2001) 901–910.
- [13] M.K. Bannister, Development and application of advanced textile composites, *Proc. Inst. Mech. Eng., Part L: J. Mater.: Des. Appl.* 218 (3) (2004) 253–260.
- [14] M. Giordano, A. Calabro, C. Esposito, A. D’Amore, L. Nicolais, An acoustic-emission characterization of the failure modes in polymer-composite materials, *Compos. Sci. Technol.* 58 (1998) 1923–1928.
- [15] J. Bohse, Acoustic emission characteristics of microfailure processes in polymer blends and composites, *Compos. Sci. Technol.* 60 (2000) 1213–1226.
- [16] Y.K. Lieu, W.K. Shih, B.Z. Jang, Controlled energy dissipation in fibrous composites. II: Microscopic failure mechanisms, *Polym. Compos.* 12 (1991) 57–65.
- [17] O.E. Andreikiv, V.R. Skals’kyi, O.M. Serhienko, Acoustic-emission criteria for rapid analysis of internal defects in composite materials, *Mater. Sci.* 37 (1) (2001) 106–117.
- [18] I.M. De Rosa, C. Santulli, F. Sarasini, Acoustic emission for monitoring the mechanical behavior of natural fibre composites: A literature review, *Compos.: Part A* 40 (2009) 1456–1469.

- [19] M.A. Hamsad, A review: acoustic emission, a tool for composite-materials studies, *Exp. Mech.* 26 (1) (1986) 7–13.
- [20] A.J. Brunner, Identification of damage mechanisms in fiber-reinforced polymer-matrix composites with acoustic emission and the challenge of assessing structural integrity and service-life, *Constr. Build. Mater.* 173 (2018) 629–637.
- [21] K.H. Leong, S. Ramakrishna, Z.M. Huang, G.A. Bibo, The potential of knitting for engineering composites—a review, *Compos.: Part A* 31 (2000) 197–220.
- [22] S. Ramakrishna, N.K. Coung, H. Hamada, Tensile properties of plain weft knitted glass fiber fabric reinforced epoxy composites, *J. Reinf. Plast. Compos.* 16 (10) (1997) 946–963.
- [23] X. Ruan, T.W. Chou, Failure behaviour of knitted composites, *J. Compos. Mater.* 32 (31) (1998) 198–221.
- [24] P.J. Hogg, A. Ahmadnia, F.J. Guild, The mechanical properties of non-crimped fabric-based composites, *Composites* 24 (5) (1993) 423–432.
- [25] H. Kong, A.P. Mouritz, R. Paton, Tensile extension properties and deformation mechanisms of multiaxial non-crimp fabrics, *Compos. Struct.* 66 (1–4) (2004) 249–259.
- [26] F. Edgren, D. Mattsson, L.E. Asp, J. Varna, Formation of damage and its effects on non-crimp fabric reinforced composites loaded in tension, *Compos. Sci. Technol.* 64 (5) (2004) 675–692.
- [27] W.P. Hornsey, J.T. Carley, I.R. Coghlan, R.J. Cox, Geotextile sand container shoreline protection systems: design and application, *Geotext. Geomembr.* 29 (2011) 425–439.
- [28] R.M. Koerner, *Geotextiles From Design to Applications*, first. ed., Woodhead Publishing, UK, 2016.
- [29] D. Lariviere, P. Krawczak, C. Tiberi, P. Lucas, Interfacial properties in commingled yarn thermoplastic composites. Part II: Influence on crack initiation and propagation, *Polym. Compos.* 25 (2004) 589–600.
- [30] S. Barre, M.L. Benzeggagh, On the use of acoustic emission to investigate damage mechanisms in glass-fibre-reinforced polypropylene, *Compos. Sci. Technol.* 52 (1994) 369–376.
- [31] M.N. Shahri, J. Yousefi, M. Fotouhi, M.A. Najfabadi, Damage evaluation of composite materials using acoustic emission features and Hilbert transform, *J. Compos. Mater.* 50 (2016) 1897–1907.
- [32] N. Godin, P. Reynaud, G. Fantozzi, Challenges and limitations in the identification of acoustic emission signature of damage mechanisms in composites materials, *Appl. Sci.* 8 (2018) 1267.

Further reading

Bhattacharya and Agrawal, 2017 S.S. Bhattacharya, S.A. Agrawal, Textile reinforced structure: A review, *Int. J. Eng. Res. Appl.* 7 (7 (Part 8) (2017) 84–86.

This page intentionally left blank

Treatment effect on failure mode of industrial carbon textile at elevated temperature

12

Manh Tien Tran^{1,2}, Xuan Hong Vu¹ and Emmanuel Ferrier¹

¹*University of Lyon, University Claude BERNARD Lyon 1, Laboratory of Composite Materials for Construction LMC2, Villeurbanne, France*

²*Hanoi University of Mining and Geology (HUMG), Hanoi, Vietnam*

12.1 Introduction

Over the past some decades, the industrial textiles in different nature were widely fabricated in factory as a commercial product thanks to their diverse applications in automotive engineering, civil engineering, transportation and energy, medical purposes, etc. [1–8]. The industrial textile has been considered as an alternative material to contribute in the industry development of the world. The textile in combination with polymer matrix or cement matrix gives a new material (fiber reinforced polymer [FRP] or textile reinforced concrete [TRC]) that has better characteristics (high strength and stiffness, slight, etc.) than other traditional material [4–13]. They were also successfully used in some special cases of loading or environment such as thermal shock, seismic, contact, corrosion, etc. [4–7, 14–16]. However, in case of fire or at high temperature, changes of textile properties are considered as an important factor for the application of textiles. So, it needs more experimental studies on the industrial textile behavior under or after exposure to elevated temperatures.

In the literature, some fire tests have been carried out on specimens with the presence of textile composite to find its reinforcement capacity in fire [17–20]. Several studies have been conducted on the tensile behavior of industrial textile or textile composite specimens after exposure to elevated temperatures (called residual condition). These studies aimed to identify their tensile residual behavior with elevated temperatures [21–27]. The residual behaviors of basalt textile and basalt TRC composite specimens after exposure to 1 hour at elevated temperatures from 25°C to 1000°C were presented in [25, 26]. The effect of thickness and reinforcement configuration on flexural and impact behavior of glass textile-reinforced polymer composite was investigated in [27] after exposure to four levels of elevated temperatures 60°C, 120°C, 200°C, and 300°C. Some experimental research works have been performed on industrial textile or textile

composite specimens under simultaneous actions of temperature and mechanical loadings (called thermomechanical condition) [13,28–30]. These tests aimed to identify the tensile behavior and thermomechanical properties of studied materials at different temperatures. Nguyen et al. [13] has performed experimental and analytical investigation of elevated temperature behavior on carbon textile-reinforced polymer (FRP) specimens in two thermomechanical conditions: thermomechanical test at constant temperature (TMCT) and thermomechanical test at constant load (TMCL). Homoro et al. [30] recently studied experimentally and analytically thermomechanical behavior of alkali-resistant (AR) glass textile and discontinuous short AR glass fibers-reinforced concrete (TRC) at temperatures ranging from 25°C to 600°C. Few research was performed on carbon textile reinforced mortar (TRM) in comparison with fiber reinforced polymer (FRP) as strengthening materials of structure specimen at elevated temperatures to characterize the shear, bond, and flexure behavior [31–33]. As the results obtained in the literature, a notice could be concluded that the elevated temperature behavior of industrial textile and its composite material depended on several factors such as fiber nature, matrix nature, and reinforcement ratio in composite material, the fiber treatment by different products. Among these factors, the product of textile treatment greatly influenced to the failure mode of industrial textile and its composite material specimens at different temperatures.

As regards to the failure mode of industrial textile, in the literature, there were some studies conducted to the failure mode of industrial textile under action of mechanical load and elevated temperatures [12,13,25,26]. Zhai et al. [12] have performed an experimental investigation on longitudinal tensile specimens of glass textile treated with polypropylene resin at room and different temperatures. The results showed that the temperature effect on polypropylene resin treatment influenced to the failure mode of specimens. As regards to the failure mode of the specimens tested at 23°C, 50°C, and 70°C, Zhai et al. [12] displayed the brittle failure mode with the pull-out of the monofilaments. At 90°C, the longitudinal splitting of the filament groups is caused by the weakness of interface between fibers and resin treatment product. In this research, the optical microscopy image method was used to observe the failure mode of glass textile specimens at micro scale. As results of the optical microscopy image method in [12], it showed the extensive debonding in fiber groups along the longitudinal direction of specimens.

In their study, Rambo et al. [21,25,26] have been conducted the studies on the elevated temperature behavior of basalt textile and basalt TRC composite specimens. As results obtained, basalt textile specimens after exposure to elevated temperatures provided a linear behavior with a fragile rupture mode. This failure mode was characterized by the drop in stress of “stress–strain” curves of basalt textile specimens. As regards to the effect of textile treatment, it can be seen that a coating of styrene-acrylic latex had a positive effect to improve the mechanical properties (tensile strength and Young’s modulus) of specimens in the temperatures ranging from 75°C to 150°C. After cooling, the textile preimpregnation by

coated polymer improved the uniform stress distribution between the filaments in textile yarns. In basalt TRC composite specimen, the heating—cooling regime of residual test at temperature from 75°C to 150°C positively affected to this resin treatment of basalt textile to improve the composite performances (tensile strength and Young's modulus). As regards to the failure mode of basalt textile, scanning electron microscopy method was used to observe the damage processes in the basalt textile after exposure to different temperatures. As results, the basalt textile provided a fragile rupture mode which was caused by the minor crack-bridging action of the filaments or the melting of filaments at elevated temperatures.

In the recent study of Nguyen et al. [13], carbon textile was manually treated with two-component epoxy resin as a matrix in laboratory condition. It provided a brittle failure mode under simultaneous thermal and mechanical loadings at different temperature levels. This failure mode was also outlined by a drop in stress of “stress—strain” curves at different temperatures. By observing the carbon textile specimens after thermomechanical tests, it could be divided this failure mode by four types of specimen failure. The first failure type was remarked by the damage of both carbon textile and coated epoxy for specimens at room temperature, whereas at 200°C, the coated resin melting was caused to the debonding of the filament groups (the second failure type). The complete melting and a partial decomposition of the coated resin at 400°C lead the filament breakage at different section (the third failure type), whereas the complete burning of coated resin and the oxidation of a part carbon fiber at 600°C were notable (the fourth failure type).

To the best of the authors' knowledge, no results are available concerning the thermomechanical test carried out on the specimens of continuous carbon textile, which has been industrially treated in factory by different products in nature. There are also no results regarding effect of textile treatment on the thermomechanical behavior and failure mode of carbon textile at elevated temperature. The aim of this work is to contribute in answering the question: What is effect of textile treatment on the thermomechanical behavior and failure mode of industrial carbon textile? This work also provides the scientific community with experimental data concerning the thermomechanical behavior of industrial carbon textiles.

This chapter presents an experimental study on the tensile behavior of three different industrial carbon textiles subjected to simultaneous mechanical and elevated temperature loadings. Three continuous carbon textiles, which were industrially treated with different products in nature and as commercial products, were tested at different temperature levels varying from 25°C to 600°C. The results on three carbon textiles were compared to find the effect of fiber treatment on the thermomechanical behavior and failure mode of carbon textile. In the following sections of this chapter, experimental work including the use of equipment, test specimens, and loading paths are presented in [Section 12.2](#). The experimental results are presented in [Section 12.3](#), and then analyzed and discussed in [Section 12.4](#). This chapter ends with a presentation of main conclusions and future works.

12.2 Experimental work

This section presents the equipment used and loading paths for characterization of residual and thermomechanical behavior of carbon textile.

12.2.1 Equipment used

The test machine, furnace, and laser sensor were used in this research in order to generate the simultaneous actions of elevated temperature and mechanical loading to specimen, and to measure the thermomechanical strain of carbon textile specimen.

12.2.1.1 Test machine

The test machine used in this research, TM20kN-1200°C, can generate a maximal force up to 20 kN for the direct traction test. The traction loading is controlled by the vertical displacement of traverse thanks to a control program (test expert II software) in the computer of the control system for the test setup. The increasing rate of the force depends on the type of material (ultimate strength and stiffness). During the test, the data including mechanical load and traverse movement of the test machine are recorded at least twice per second and can then be exported in the form of datasheets for results analysis. Fig. 12.1 shows the universal tensile machine with other equipment for thermomechanical test.

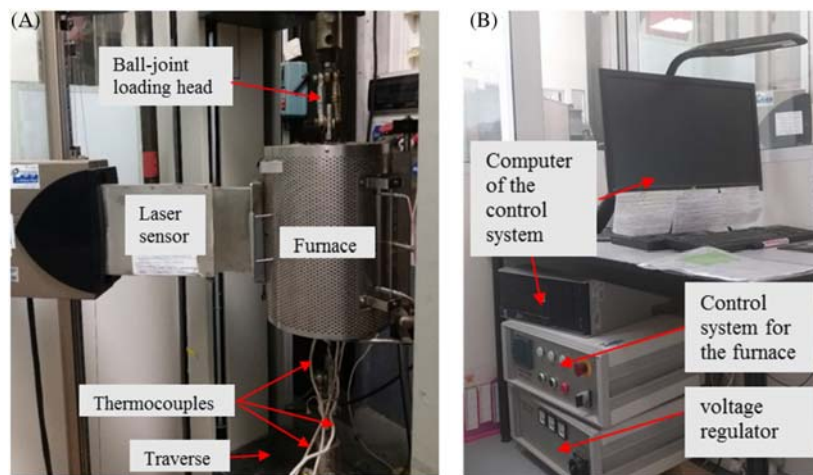


FIGURE 12.1

Universal traction machine TM20kN-1200°C. (A) Overview of the test setup and (B) control system for test setup.

12.2.1.2 Furnace

The test machine is equipped with a small cylindrical furnace with three dimensions like that: height of 40 cm, an internal diameter of 10 cm, and an external diameter of 27.5 cm. This furnace can generate, in the same time with mechanical loading during the test, temperature loadings of specimens potentially up to 1200°C with the maximum heating rate of 30°C/min. The temperatures in the furnace are determined by integrated thermocouples and also controlled by the test program. With the equipment of furnace, the test machine can make it possible to apply simultaneous tensile mechanical and elevated temperature loadings on a sample. Fig. 12.2 shows the detail of the furnace installed on the test machine.

12.2.1.3 Laser sensor

In this research, the noncontact measurement method of laser sensor was used to measure the thermomechanical strain of test specimens. The measurement principle of this method is explained by the description of the laser sensor system. The measuring system consists of two charge coupled device cameras with one lighting unit for each one and a linear motor for continuous adjustment of the spacing between both cameras. The cameras are vertically arranged and in parallel view with the specimen in the horizontal direction. By using the motorized camera setting, it can be set different reference lengths for measuring axial strain of specimen. The illumination is produced by a laser diode with a diverging lens upstream. The lens sweeps the light beam and thus creates on the test surface a lighting mark extended in the vertical direction whose dimensions roughly correspond to the visual field of the camera. The camera image is digitally recorded by

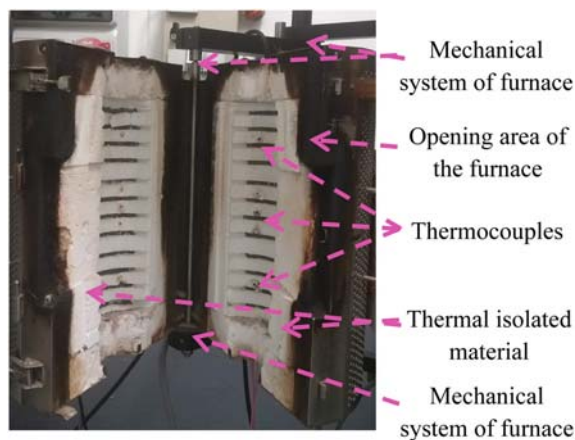
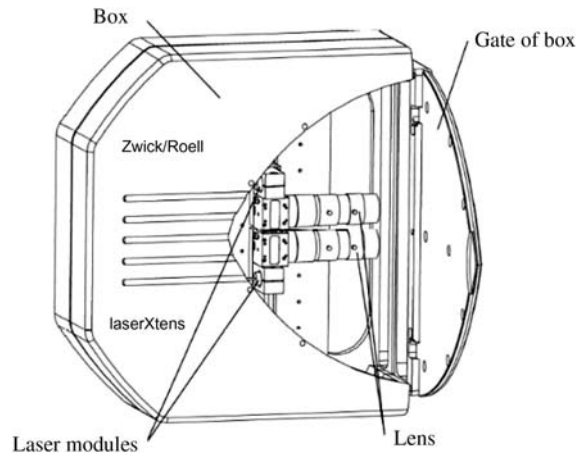


FIGURE 12.2

Detail of the furnace for thermomechanical test.

**FIGURE 12.3**

Laser sensor details for the measurement of specimen axial strain.

the control computer. The image processing software exploits the movements of the images and derives values of axial strain of specimen (see Fig. 12.3).

The existence of a small furnace to generate the temperature action around the test specimen is one of the difficulties for a method of measuring contact. Moreover, the thinness of the test specimen of carbon textile can create difficulty in the specimen axial strain measurement with contact measurement methods. In particular, the elevated temperature in the furnace greatly influences the results of other strain measurement instruments in the contact measurement method. With the noncontact measurement method by the laser sensor, it provides a relative displacement between two laser bobbins on the carbon textile specimen (Fig. 12.3). The axial strain of the carbon textile specimen is calculated as the ratio of the relative displacement of two laser bobbins and their initial distance. When the tested material is homogeneous, as carbon textile, the measurement of the laser sensor can provide the axial strain for the part of the specimen placed in the furnace during the thermomechanical test. This method of measurement has been used, developed, and validated in previous works and has given good results for different composite materials [13,28–30].

12.2.2 Specimens

The materials used in this research are three continuous carbon textiles which are called GC1, GC2, and GC3 textile. They were manufactured in a factory as industrial products for multi applications. They have good thermal and mechanical characteristics: very high tensile strength and modulus, noncorrosion, low mass per unit area, simple and flexible application, etc. Three carbon textiles were coated with different treatment products in nature. The GC1 and GC3 textiles were

preimpregnated by epoxy resin with two different preimpregnation ratios, completely preimpregnated for the GC1 textile and a very low level for GC3 textile, whereas the GC2 textile was treated with a product of amorphous silica as a coating. They were also manufactured in grid form with different grid geometries (see Fig. 12.4). The properties of the three studied carbon textiles are summarized in Table 12.1.

The test specimens in this experimental study were prepared by hand in the laboratory. First, a longitudinal yarn (the warp) of the textile was cut to obtain carbon textile samples of 750 mm in length. After that, two ends of the specimens were bonded with aluminium plates for good transmission of the mechanical force to the textile specimens. Finally, the specimens were embraced at room temperature for 7 days, then referenced and ready for the tests at different temperatures (see Fig. 12.5). There were 42 tests carried out on the specimens of three carbon textiles at different temperature levels varying from 25°C to 600°C. Table 12.2 shows the list of the specimens and the tests carried out in this research.

12.2.3 Loading paths

The textile specimen was tested in the thermomechanical condition at constant temperature (TMCT) to identify the thermomechanical behavior of three carbon textiles. The TMCT loading path consists of three phases as described in previous

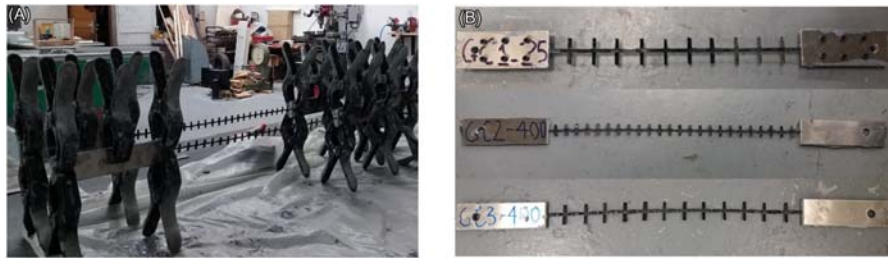


FIGURE 12.4

Images of three carbon textiles (GC1, GC2, GC3) used in the experimental study. (A) GC1 carbon textile; (B) GC2 carbon textile; and (C) GC3 carbon textile.

Table 12.1 Properties of three studied carbon textiles.

Properties	GC1	GC2	GC3
Density (g/cm ³)	3.43	1.79	1.89
Grid geometry (longitudinal × transverse spacing) (mm × mm)	46 × 41	17 × 17	33 × 33
Type of coating	Epoxy resin	Amorphous silica	Epoxy resin
Cross-sectional area of one individual strand	1.85 mm ²	1.795 mm ²	1.80 mm ²

**FIGURE 12.5**

Preparation of samples. (A) Embracing for specimens and (B) carbon textile samples ready for test.

Table 12.2 List of tests conducted on the specimens of three carbon textiles.

Designation of the specimen	Dimensions of the specimen (cross section, S [mm ²] length, l [mm])	Temperature (°C)	Exposure duration	Number of tests
GC1-T-a,b,c	S = 1.85 (mm ²); l = 425 (mm)	T = 25, 200, 400, 500, 600	1 h	14
GC2-T-a,b,c	S = 1.795 (mm ²); l = 420 (mm)	T = 25, 200, 400, 500, 600	1 h	14
GC3-T-a,b,c	S = 1.80 (mm ²); l = 425 (mm)	T = 25, 200, 400, 500, 600	1 h	14
Total of tests				42

works [13,28–30,34]. The first test phase consists of increasing the temperature around sample to the desired temperature level. The rate of temperature increase varies from 6°C/min to 20°C/min depending on the target temperature (from 200°C to 600°C) of the test. The second test phase consists of maintaining the target temperature (T_{target}) for a period of 1 hour in order to homogenize the temperature around specimen. The third test phase consists of applying the quasi-static tensile load monotonically to specimen until failure; the laser sensor is activated during this phase to measure the axial strain of sample (see Fig. 12.6).

The stress of specimen corresponding to the failure point is quantified as the ultimate stress (σ_{UTS}). The axial strain of specimen corresponding to failure point is quantified as the maximum axial thermomechanical deformation ($\varepsilon_{\text{TM,UTS}}$). Fig. 12.6 shows the thermomechanical test procedure on the test specimens of carbon textiles with three phases. After the test, all data, including temperatures, mechanical load, and specimen axial thermomechanical strain or traverse movement, are recorded at least twice per second and can then be exported in the form of datasheets for results analysis.

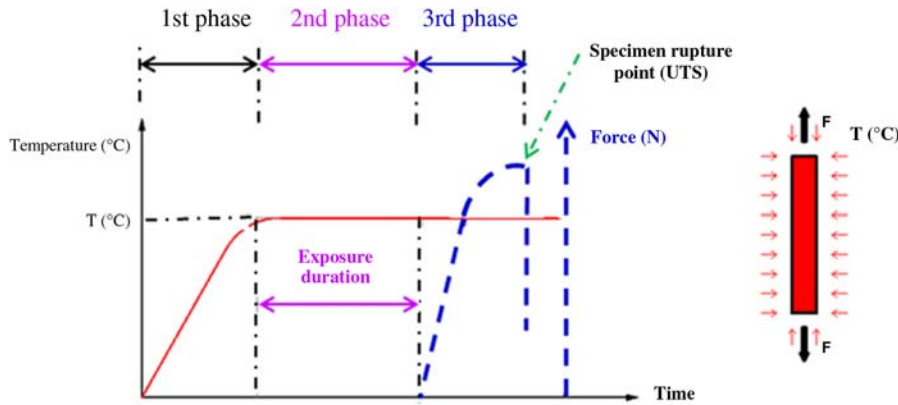


FIGURE 12.6

Loading path of thermomechanical tests carried out on specimens of three carbon textiles.

12.3 Results

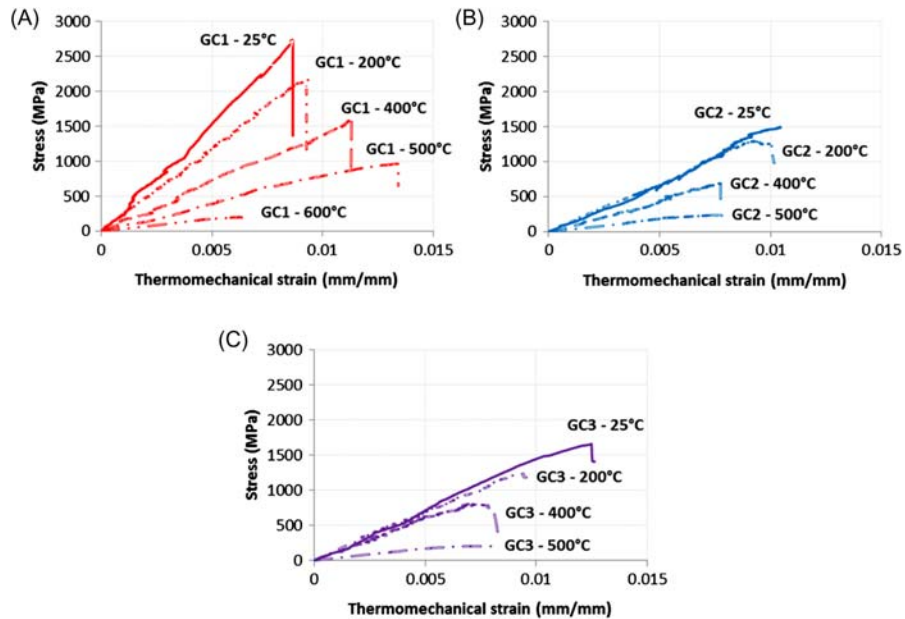
This section presents the results of thermomechanical tensile tests performed on carbon textile specimen in comparison with other results of previous works in the literature.

12.3.1 Elevated temperature behavior of industrial textiles

Fig. 12.7A–C shows the “stress–thermomechanical strain” curves of three carbon textile samples (GC1: Fig. 12.7A; GC2: Fig. 12.7B; GC3: Fig. 12.7C) at different temperatures ranging from 25°C to 600°C.

Fig. 12.7A–C show only one average “stress–strain” curve for each test temperature so that the curves can be legible for all temperature levels varying from 20°C to 500°C for each carbon textile. From Fig. 12.7, it could be seen that all carbon textile samples exposed to 1 hour at elevated temperatures gave an almost linear behavior up to the test specimen failure. The “stress–thermomechanical strain” curves of both GC2 (Fig. 12.7B) and GC3 (Fig. 12.7C) carbon textiles were similar at each temperature level, while the initial slope (stiffness) of the stress–strain curve of the GC1 carbon textile was higher about two times than that of GC2 and GC3 in the same temperature level.

The ultimate strength of three carbon textiles at 25°C is 2616, 1311, and 1443 MPa (on average), respectively for GC1, GC2, and GC3 carbon textile. With increasing of temperature, these values decrease progressively into a negligible value at 600°C. The GC1 carbon textile gave an ultimate strength of 205 MPa (on average) corresponding to a 92.8% reduction of this value at ambient temperature, whereas two other textile specimens (GC2 and GC3 textiles)

**FIGURE 12.7**

Thermomechanical behavior of three carbon textile: stress–thermomechanical strain relationship at different temperatures. (A) GC1; (B) GC2; and (C) GC3.

have been broken before the third phase of test. That's why there were not available results for thermomechanical tests of GC2 and GC3 carbon textiles. Table 12.3 presents the other average ultimate strength values of thermomechanical tests on the specimens of three carbon textiles at different temperature levels varying from 25°C to 600°C. In Table 12.3, it could be also found the standard deviation values of the mechanical properties of three carbon textiles at different temperatures.

As regard to the Young's modulus of three carbon textiles, it can be seen in Fig. 12.7 that the temperature action also influenced on the initial slope of the “stress–thermomechanical strain” curves of three carbon textiles. So, the stiffness of three carbon textiles decreases progressively when the temperature increases. Young's modulus of three carbon textiles was determined by two points corresponding to the deformation of 0.05% and 0.25% of “stress–thermomechanical strain” curve. At room temperature, Young's modulus of three carbon textiles was 256, 144, and 130 GPa (on average), respectively, for GC1, GC2, and GC3 carbon textiles. At the temperature of 600°C, the thermomechanical test gave a very low Young's modulus value of 29 GPa (on average) for GC1 carbon textile, and nonvalue for two others. Table 12.3 shows all average values of thermomechanical test concerning the Young's modulus of three carbon textiles.

Table 12.3 Results of the direct tensile tests performed on three carbon textile specimens (the standard deviation values are written in parallel).

Temperature (°C)	Heating rate (°C/min)	GC1 textile		GC2 textile		GC2 textile	
		Ultimate stress (MPa)	Young's modulus (GPa)	Ultimate stress (MPa)	Young's modulus (GPa)	Ultimate stress (MPa)	Young's modulus (GPa)
25	0	2616.6 (124.9)	256.2 (39.7)	1311.5 (158.3)	143.8 (7.2)	1442.7 (242.2)	130.2 (7.1)
200	6.67	2169.5 (175.5)	201.6 (29.0)	1152.5 (204.9)	138.6 (14.0)	1168.1 (103.0)	117.4 (25.4)
400	13.33	1652.2 (40.5)	138.8 (23.4)	708.8 (71.8)	107.1 (18.6)	845.9 (89.1)	95.9 (21.6)
500	16.67	795.7 (145.4)	77.6 (4.5)	308.1 (67.8)	39.7 (9.5)	185.7 (18.6)	38.4 (6.4)
600	200	204.9	29.5	—	—	—	—

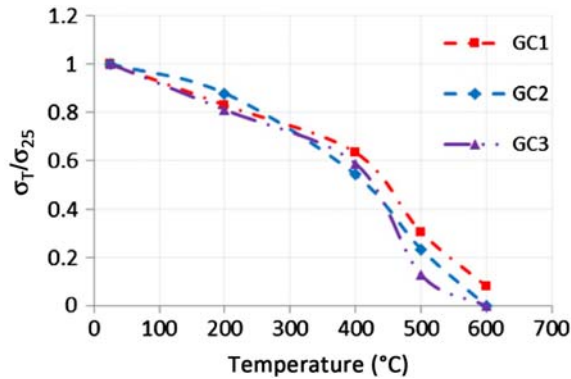
12.3.2 Evolution of the thermomechanical properties as a function of temperature

As results presented in Section 12.3.1, the mechanical properties of three carbon textile varied as a function of elevated temperatures. So, it could be defined the normalized mechanical properties (normalized ultimate stress and Young's modulus) for mechanical property evolution depending on the temperature. This value is determined by the ratio between the mechanical property at a temperature (T) and that at room temperature (σ_T/σ_{25} or E_T/E_{25}). The normalized mechanical properties of three carbon textiles are presented in Table 12.4. From Table 12.4, it can be seen that three carbon textiles gave the similar reductions of the normalized ultimate stress values when the temperature increases from 25°C to 400°C. The normalized ultimate stresses were 82.9%, 87.9% or 81.0%, respectively for three carbon textiles at 200°C, and were 63.1%, 54.0%, and 58.6% respectively for three carbon textiles at 400°C. At the temperatures above 400°C, the GC1 textile remained more mechanical capacities than the other two textiles (GC2 and GC3). This carbon textile sustained 32.5% and 7.8% of its ultimate stress value at room temperature, respectively for 500°C and 600°C, whereas the other two textiles had an ultimate stress less than 22% and 0% compared with the ambient temperature value, respectively for 500°C and 600°C. Fig. 12.8 shows the evolution of the normalized ultimate stress of three carbon textiles as a function of temperature.

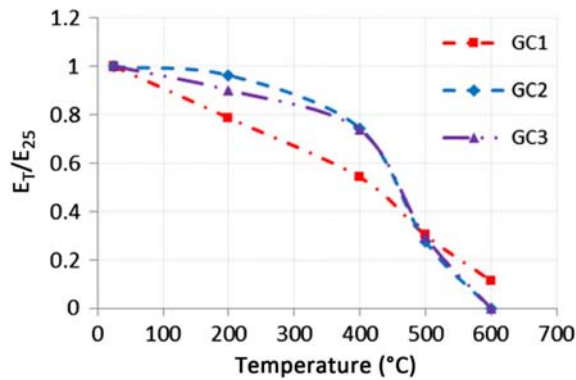
From Fig. 12.8, the ultimate stress evolution of the GC1 and GC3 carbon textiles can be divided into two zones. At temperatures ranging from 25°C to 400°C, the ultimate stress slightly decreased as a function of temperature, about 20% at 200°C and 40% at 400°C. At temperatures ranging from 400°C to 600°C, an important reduction of ultimate stress could be found. The complete decomposition of preimpregnated products by epoxy resin in carbon textiles caused this considerable reduction. Fig. 12.9 presents the evolution of the normalized Young's modulus of three carbon textiles as a function of temperature. From Fig. 12.9, it

Table 12.4 Evolution of the normalized mechanical properties of three carbon textiles as a function of temperature.

T °C	Normalized ultimate stress σ_T/σ_{25} (%)			Normalized Young's modulus E_T/E_{25} (%)		
	GC1	GC2	GC3	GC1	GC2	GC3
25	100	100	100	100	100	100
200	82.9	87.9	81.0	78.7	96.4	90.1
400	63.1	54.0	58.6	54.2	74.4	73.6
500	30.4	21.1	12.9	30.3	23.8	29.5
600	7.8	0	0	11.5	0	0

**FIGURE 12.8**

Evolution of normalized ultimate stress obtained for the studied carbon textiles.

**FIGURE 12.9**

Evolution of normalized Young's modulus obtained for the studied carbon textiles.

can be seen that GC2 and GC3 carbon textiles had almost the same Young's modulus evolution as a function of elevated temperatures. The normalized Young's modulus values were 96.4% and 90.1% at 200°C, 74.4% and 73.6% at 400°C, 23.8% and 29.5% at 500°C, respectively for GC2 and GC3 carbon textiles. From Fig. 12.9, it can be seen that the influence of temperature on Young's modulus of the GC2 and GC3 carbon textiles could be divided into two temperature intervals. The first interval, from 25°C to 400°C, was described by a slight reduction of Young's modulus (about 25% at 400°C). In the second interval, from 400°C to 600°C, Young's modulus greatly decreased and was almost negligible at 600°C. Whereas Young's modulus of GC1 carbon textile progressively decreased and retained 78.7%, 54.2%, 30.3%, and 11.5% of its value at room temperature, respectively, for 200°C, 400°C, 500°C, and 600°C.

12.3.3 Discussion

This section presents discussions to explain the effect of carbon fiber treatment products on the results obtained (ultimate strength and Young's modulus, thermo-mechanical property evolution depending on temperature, and failure modes of textile specimens), which are presented in [Section 12.3](#).

12.3.3.1 Effect of textile treatment on the elevated temperature behavior of carbon textiles

As result presented in [Section 12.3.1](#), it could be found the influence of treatment by different products on the behavior of three carbon textiles at elevated temperatures. Three studied carbon textiles gave the different values of ultimate stress and Young's modulus in the same temperature level even if they have almost equivalent cross-sections for a textile yarn.

The GC1 carbon textile has an important mechanical capacity and is better than two other carbon textiles (GC2, GC3) thanks to its very good preimpregnation with an epoxy resin product (see [Fig. 12.10A](#)). This treatment par epoxy resin gave a good load transmission between the monofilaments of textile yarn which has assured the working in common between the monofilaments. The ultimate stress of the GC1 textile was higher about two times than this value of the other two textiles at different temperatures (see [Fig. 12.10A](#)). At the temperature above 400°C, although epoxy resin treatment has begun to be partially decomposed and burned, but it still assured the load transmission between the monofilaments of GC1 textile yarn. As regards to the treatment of two other textiles, the amorphous silica for GC2 carbon textile and the epoxy resin with very low content for GC3 had the similar effect on loading capacity of textile. This little bonding between the monofilaments within GC2 (or GC3) caused less carrying load of a textile yarn of GC2 (or GC3) than that of the GC1 carbon textile. As results of Young's modulus at elevated temperatures, it could be also found the effect of the preimpregnated product on stiffness of three carbon textiles. The perfect

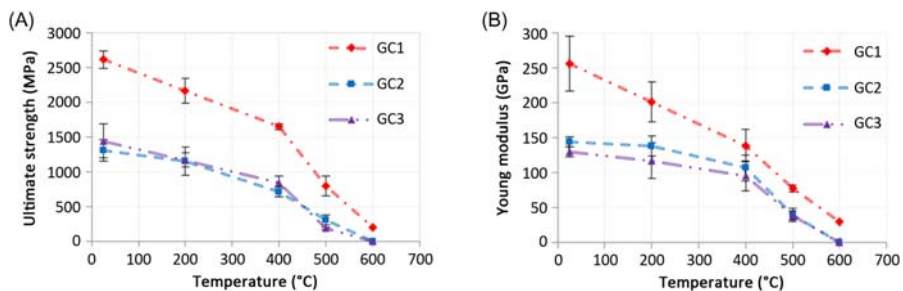


FIGURE 12.10

Evolution of mechanical properties of three carbon textiles with elevated temperatures. (A) Ultimate strength and (B) Young's modulus.

preimpregnation of an epoxy resin product for GC1 carbon textile has led to a good relationship between the monofilaments of textile yarn. So, they have worked together in the longitudinal direction to support the tensile loading. This led to a higher stiffness value of GC1 compared to those of two other textiles (GC2 and GC3). The coating treatment of the GC2 and GC3 textiles also gave the same effect on stiffness capacity at different temperatures. As experimental results, the Young's modulus of two carbon textiles (GC2 and GC3) was approximately similar while this value of GC1 carbon textile was greater about 1.5 times than that of two other carbon textiles (see Fig. 12.10B).

Concerning the mechanical property evolution as a function of temperature, the experimental results showed that the thermomechanical properties of three carbon textiles differently decrease for each studied carbon textile when the temperature increases. The evolution of the ultimate stress and/or Young's modulus depended on the treatment product and the preimpregnation ratio of the carbon textiles. As regards to the reduction of the ultimate stress, the carbon textiles treated with the resin product (GC1 and GC3) gave a typical evolution depending on the temperature. There are two intervals of this evolution: a slight reduction (approximately 40%) in the temperature ranging from 25°C to 400°C and a significant decrease at temperatures from 400°C to 600°C. In the first interval, the normalized ultimate stress of two carbon textiles (GC1 and GC3) was approximately similar as presented in Section 12.3.1. It means that significant contribution of the impregnated resin has still ensured good charge transmission between monofilaments when this treatment product has not yet started to decompose or burn at the temperature from 25°C to 400°C. In the second interval, the textile perfectly preimpregnated in the epoxy resin (GC1 textile) could remain very well its ultimate stress compared with the value at room temperature while the other one (GC3 textile) had very low value of normalized ultimate stress. It means that the impregnated ratio significantly influenced on the evolution of the ultimate stress at the temperature above 400°C. As regards to the ultimate stress evolution of the GC2 carbon textile, the treatment by an amorphous silica product has led to a curved progressive decrease with the increasing of the temperature. The elevated temperatures have not significantly influenced on this treatment product for a physical or chemical transformation in GC2 carbon textile. This treatment product aims to improve the bond between the carbon textile and the matrix in composite material.

Concerning the Young's modulus evolution of three carbon textiles as a function of the temperature, it could be found that this evolution of the GC2 and GC3 textile was similar with two intervals: the first interval of a slight reduction (approximately 25%) in the temperatures ranging from 25°C to 400°C and the second from a very significant decrease to a negligible value at 600°C. It means that the treatment products by amorphous silica for GC2 textile and epoxy resin with very low ratio for GC3 textile had the similar contribution to their stiffness at elevated temperatures. At moderate temperatures from 25°C to 400°C, there was some contribution of the impregnated epoxy resin to GC3 carbon textile stiffness. So, it has made a small difference between the normalized Young's modulus

values of two textiles (76.4% for GC2 and 73.6% for GC3). At the temperatures above 400°C, there was complete decomposition of the impregnated epoxy resin, which caused no more contribution of this treatment product to GC3 carbon textile stiffness like that of amorphous silica product in GC2 carbon textile. As regards to the stiffness of GC1 carbon textile, the impregnated resin contribution in a textile yarn caused a progressive reduction of Young's modulus as shown in Fig. 12.10B. The effect of temperature on the resin matrix significantly influenced Young's modulus of the GC1 carbon textile. At a moderate temperature (from 25°C to 400°C), the impregnated resin was gradually softened, which caused a lesser contribution to carbon textile stiffness. At the temperatures above 400°C, the GC1 carbon textile was gradually decomposed and burned until its complete decomposition, which largely influenced the working in common between carbon monofilaments.

12.3.3.2 Failure modes

The failure modes of carbon textile are presented by observing the specimens after the tests at different temperatures in this section. The results revealed two main failure modes for the three carbon textiles: a brittle failure mode marked by a drop in stress on the “stress – thermomechanical strain” curve, and a progressive failure featured by a stress progressively decreases as a function of the deformation on the “stress – thermomechanical strain” curve. This argument is consistent with the results available in the literature [35].

At room temperature, the failure mode of three carbon textiles was similar like that of carbon fiber. The specimens were broken in an abrupt way which is called fragile rupture mode when the material reached a limited state. Thanks to the textile treatment by an epoxy resin product, there was a good load transmission between the monofilaments of GC1 and GC3 carbon textile yarn (in particular GC1 textile). So, the test specimens were completely broken by the monofilaments damage in the same time on the weakest section of textile yarn. It means that the rupture energy was suddenly liberated by the breakage of all monofilaments in a textile yarn. Fig. 12.11 shows the failure modes of GC1 carbon textile (Fig. 12.11A) and of GC2 (Fig. 12.11B) under tensile loading at room temperature. It can be seen in Fig. 12.11A that GC1 textile specimens have lost a part of its corps in the destroyed section which supported a great reaction force after the specimen failure. This apparition of the reaction force could be explained by the cause from the treatment effect of GC1 carbon textile. With the perfect preimpregnation in an epoxy resin product, GC1 carbon textile has provided very good mechanical properties (ultimate stress, Young's modulus). So, the deformable elastic energy of GC1 carbon textile specimen was greatly accumulated inside its corps when the specimen was deformed under tensile loading. The deformable elastic energy at limited state before being broken was called rupture energy of specimen. The liberation of the rupture energy in abrupt way caused to the great reaction force in GC1 textile specimen.

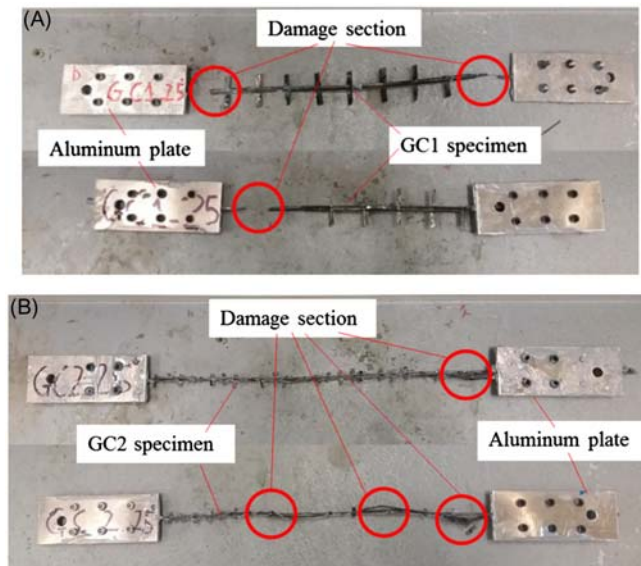


FIGURE 12.11

Failure modes of the test specimens of GC1 and GC2 carbon textiles under tensile loading at room temperature. (A) Failure mode of GC1 carbon textile under tensile loading at room temperature and (B) failure mode of GC2 carbon textile under tensile loading at room temperature.

Concerning the failure mode of GC2 carbon textile specimen under tensile loading at room temperature, it was also characterized by a drop in the stress on the “stress – strain” curve. However, this failure mode of textile yarn was different with two others textiles. This difference obtained was from the treatment of GC2 carbon textile. The monofilaments of GC2 textile were treated by an amorphous silica product which has a low contribution to link between the monofilaments for joint working themselves. The samples thus badly retained their initial form. The damage appeared in a GC2 textile yarn from each small monofilaments group that had less charge capacity than the others. It means that the liberation of rupture energy was progressively realized in these monofilaments groups. That’s why it can be seen the small drop in stress on the “stress – strain” curve of GC2 carbon textile. From Fig. 12.11B, it could be observed the separated filament groups which has broken and separated in the GC2 carbon textile specimen.

At temperature ranging from 200°C to 400°C, the temperature action has influenced on the failure mode of three carbon textile specimens observed after thermomechanical tests. GC1 carbon textile was preimpregnated in an epoxy resin product which has been softened and decomposed partially (in particular at 400°C), so it can be seen the separated filament groups with treated epoxy resin in the damage sections (see Fig. 12.12A–D). At 400°C, the number of separated

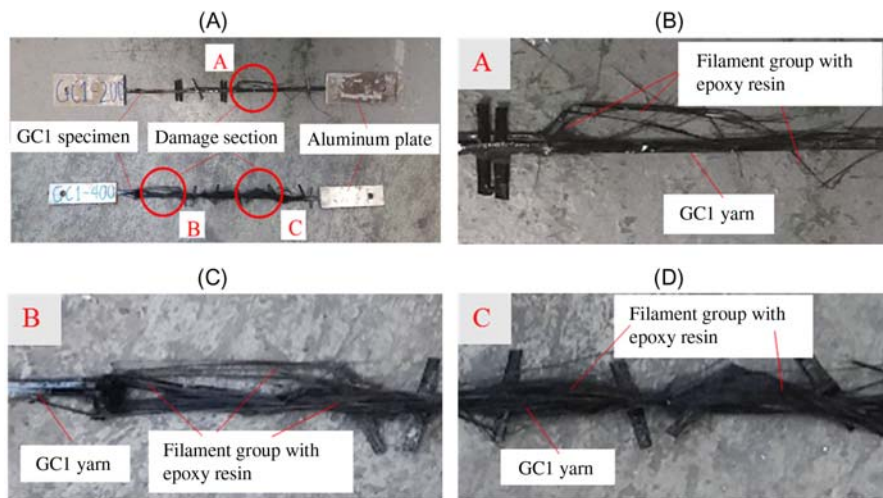


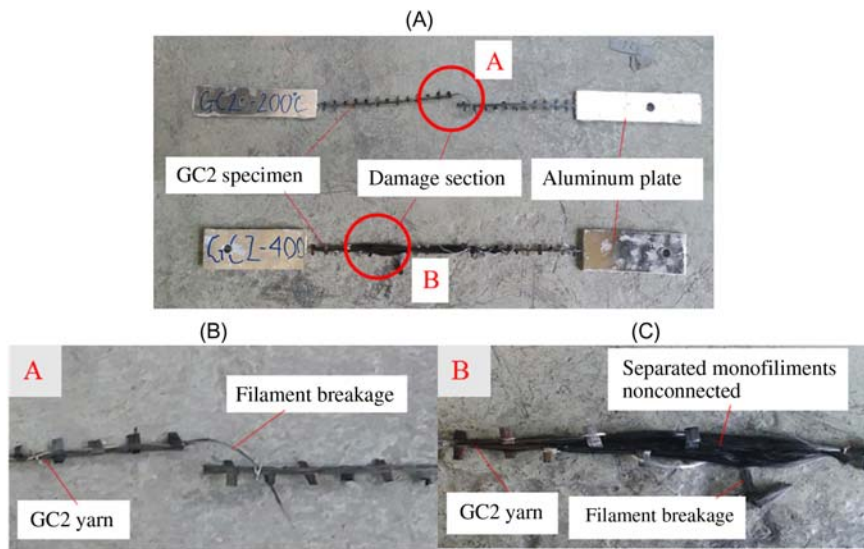
FIGURE 12.12

Failure mode of test specimens of GC1 carbon textile at temperature from 200°C to 400°C. (A) GC1: failure mode 200°C–400°C; (B) GC1: zoom of the damage section A; (C) GC1: zoom of the damage section B; and (D) GC1: zoom of the damage section C.

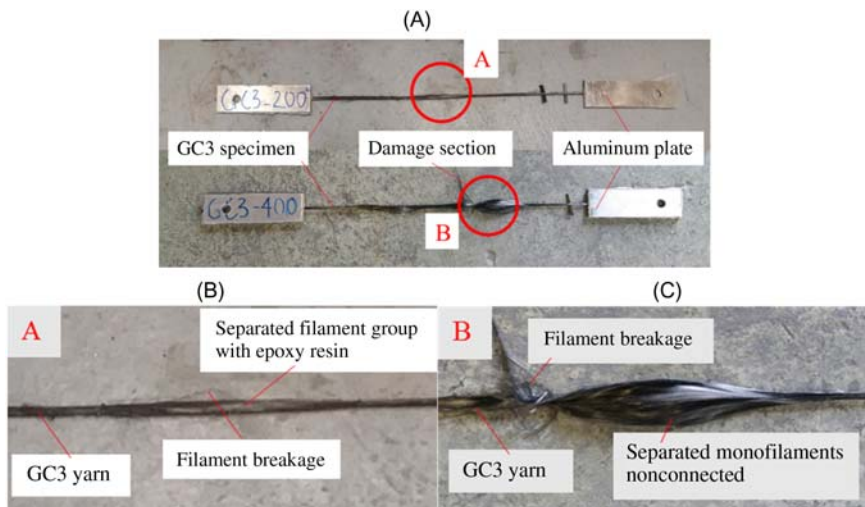
filament groups in damage section of GC1 specimen has increased in comparison with that at 200°C (see Fig. 12.12). It could be explained this result that the softening and melting of treated epoxy resin led the reduction of this product contribution for joint working of the monofilaments in textile yarn. Fig. 12.12A–D shows the failure mode of GC1 carbon textile in the range of temperature from 200°C to 400°C.

As regards to the failure mode of GC2 carbon textile in temperature from 200°C to 400°C, it could be observed that test specimens have progressively damaged on each small group of monofilaments that had less charge capacity than the others. It was similar like that at room temperature because the coating with amorphous silica was not greatly influenced by the action of the elevated temperatures. From Fig. 12.13A–C, it could be seen that the monofilaments were separated in the damage section of GC2 textile specimens at 400°C, and there was no-connection between themselves.

Fig. 12.14A–C shows the failure mode of GC3 textile specimens at temperatures from 200°C to 400°C. With a treatment by an epoxy resin product, GC3 carbon textile gave a failure mode similar like that of GC1 textile. At 200°C, the filament breakage appeared on separated filament groups with epoxy resin in the damage section of GC3 specimens. However, with low content of treatment, there was not any more epoxy resin treatment in the damage section of GC3 textile specimens at 400°C. The monofilaments of GC3 textile yarn were separated like as the failure mode of GC2 carbon textile specimen at 400°C. The contribution of

**FIGURE 12.13**

Failure mode of the test specimens of GC2 carbon textile at temperature from 200°C to 400°C. (A) GC2: failure mode at temperature from 200°C to 400°C; (B) GC2: zoom of the damage section A; and (C) GC2: zoom of the damage section B.

**FIGURE 12.14**

Failure mode of the test specimens of GC3 carbon textile at temperature from 200°C to 400°C. (A) GC3: failure mode at temperature from 200°C to 400°C; (B) zoom of the damage section A; and (C) zoom of the damage section B.

epoxy resin treatment in GC3 textile yarn was similar with that of amorphous silica product in GC2 textile yarn. It could be said that the failure mode of GC3 textile was between that of GC1 and GC2 carbon textiles.

Failure modes of three carbon textiles at temperature above 400°C are shown in Fig. 12.15A–C for GC1, Fig. 12.16A–C for GC2 and Fig. 12.17A–C for GC3. At the temperature above 400°C, three carbon textiles gave the same failure mode which was characterized by the progressive rupture of test specimens. For the carbon textiles treated with an epoxy resin product [GC1, Fig. 12.15A–C], and GC3 [Fig. 12.17A–C], the complete decomposition of preimpregnated resin caused to the separation of the monofilaments of textile yarn. There was no-connection between them for a coworking. So, the rupture energy was progressively liberated by the breakage of each monofilament in a textile yarn. The deformation of the sample increased whereas the stress decreased progressively after reaching the maximum stress value. From Figs. 12.15 and 12.17, it could be observed the fiber breakage on the damage section of carbon textile specimens after the thermomechanical tests. For the carbon textile treated with an amorphous silica product [GC2, Fig. 12.16A–C], it could be explained the failure mode of GC2 in the same way as that of GC1 and GC3 at 400°C. At 500°C and 600°C, GC2 carbon textile was partially oxidized and the test specimens are grounded by the fiber stretching. The progressive failure of each monofilament in the textile is caused mainly by this failure mode.

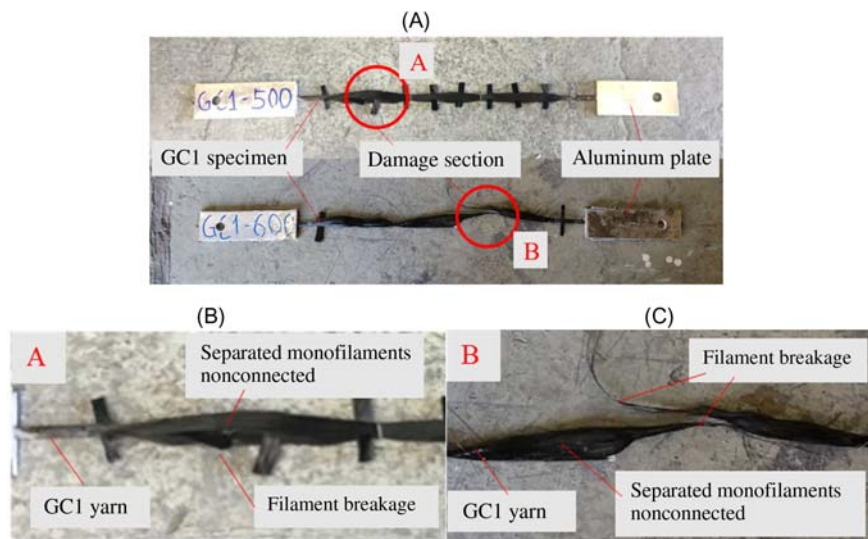
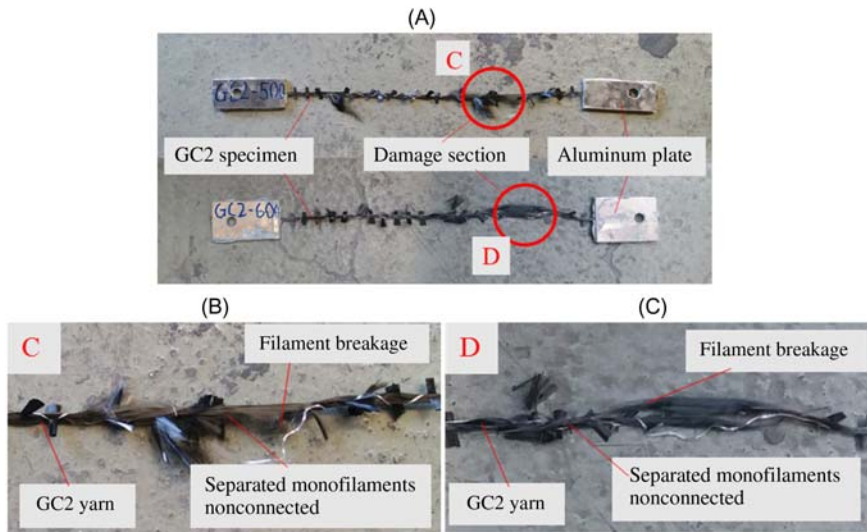
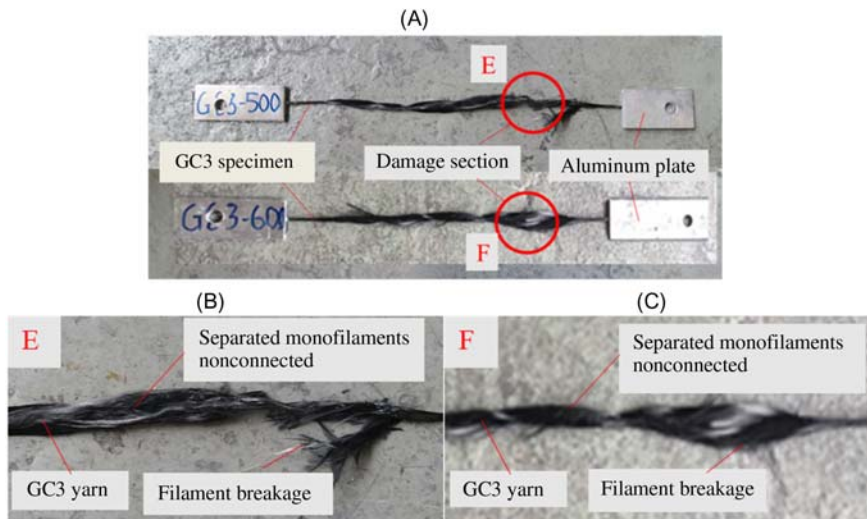


FIGURE 12.15

Failure mode of the test specimens of GC1 carbon textile at temperature above 400°C. (A) GC1: failure mode at temperature above 400°C; (B) GC1: zoom of the damage section A; and (C) GC1: zoom of the damage section B.

**FIGURE 12.16**

Failure mode of the test specimens of GC2 carbon textile at temperature above 400°C. (A) GC2: failure mode at temperature above 400°C; (B) GC2: zoom of the damage section C; and (C) GC2: zoom of the damage section D.

**FIGURE 12.17**

Failure mode of the test specimens of GC3 carbon textile at temperature above 400°C. (A) GC3: failure mode at temperature above 400°C; (B) GC3: zoom of the damage section E; and (C) GC3: zoom of the damage section F.

12.4 Conclusions

This book chapter presents the experimental works for the characterization of the thermomechanical behavior of carbon textiles subjected to mechanical and thermal loads. The objective of this work is to characterize the effect of textile treatment on thermomechanical behavior and failure mode of three carbon textiles by comparing the results obtained from tests carried out on carbon textile specimens. Three carbon textiles (GC1, GC2, and GC3) have been tested under thermomechanical loading at five temperature levels (25°C, 200°C, 400°C, 500°C, and 600°C). All thermomechanical properties obtained such as ultimate stress, Young's modulus, and thermomechanical axial strain were identified and presented. As results obtained, some conclusions could be drawn for this work:

1. The textile treatment by different products in nature influenced on the thermomechanical behavior of carbon textile. Thanks to the complete preimpregnation by the epoxy resin, the GC1 carbon textile possessed better ultimate stress and Young's modulus in comparison with those of the other textiles: the ratios between mechanical properties of GC1 textile and those of two other textiles were approximately 2 times for ultimate stress and 1.5 times for Young's modulus.
2. The treated product also influenced on the evolution of the textile mechanical properties as a function of temperature. The treatment with an amorphous silica product for GC2 textile and with very low content of epoxy resin for GC3, gave a reduction of Young's modulus with two intervals: the first one with a slight reduction (approximately 20%) for the temperature levels varying from 25°C to 400°C, and the second with a very significant decrease for the temperature levels above 400°C, whereas the GC1 and GC3 carbon textiles (treated with an epoxy resin product) exhibited the similar reduction of ultimate stress with the increasing temperature.
3. The textile treatment also gave different failure modes of carbon textile specimens. A fragile failure mode was observed on carbon textile specimens which were treated with an epoxy resin product at moderate temperature (GC1 and GC3), whereas the carbon textile GC2 (treated with an amorphous silica product) exhibited a progressive failure of the specimens.

12.5 Future trends

Recently, for an application in the electronic industries, the carbon-based conductive threads have been proposed to be used as an alternative conductive thread having a flexibility and reliability because the carbon filament has naturally very low electro-resistance. With a treatment of the polymer product, the conductive polymer-coated threads could increase its electro-resistance because of an action of the elevated temperature [36,37] and also have the failure modes as shown in

this work. Based on the results of experimental analysis concerning the failure modes of industrial carbon textiles at elevated temperature, many solutions could be suggested in order to avoid failure for these textiles. It's possible to combine the carbon textile with a type of matrix which has good thermal capacity to give a composite material with better thermal property, or use a thermal isolation layer to thermally protect the carbon filaments and polymer treatment.

References

- [1] V. Koncar, Introduction to smart textiles and their applications, in: *Smart Textiles and Their Applications*, Woodhead Publishing Series in Textiles, Elsevier, 2016, pp. 1–8.
- [2] E. Stammen, K. Dilger, Part II: Adhesive bonding of textiles: applications, in: *Joining Textiles: Principles and Applications*, Woodhead Publishing Series in Textiles, Elsevier, 2013, pp. 275–308.
- [3] T. Gries, M. Raina, T. Quadflieg, O. Stolyarov, Manufacturing of textiles for civil engineering applications, in: *Textile Fibre Composites in Civil Engineering*, Woodhead Publishing Series in Civil and Structural Engineering, Elsevier, 2016, pp. 3–24. Available from: <https://doi.org/10.1016/B978-1-78242-446-8.00002-1>
- [4] Q.T. Shubhra, A. Alam, M. Quaiyyum, Mechanical properties of polypropylene composites: a review, *J. Thermoplast. Compos. Mater.* 26 (2011) 362–391.
- [5] K. Van de Velde, P. Kiekens, Thermoplastic polymers: overview of several properties and their consequences in Flax fibre reinforced composites, *Polym. Test.* 20 (2001) 885–893.
- [6] J.L. Thomason, The influence of fibre length and concentration on the properties of glass fibre reinforced polypropylene: 7. Interface strength and fibre strain in injection moulded long fibre PP at high fibre content, *Composites Part A.* 38 (2007) 210–216.
- [7] V.S. Balakrishnan, H. Seidlitz, Potential repair techniques for automotive composites: a review, *Compos. Part B: Eng.* 145 (2018) 28–38.
- [8] P. Abel, C. Lauter, T. Gries, T. Troester, Textile composites in the automotive industry, in: *Fatigue of Textile Composites*, Woodhead Publishing Series in Composites Science and Engineering, Elsevier, 2015, pp. 383–401.
- [9] M. Butler, M. Lieboldt, V. Mechtcherine, Application of textile-reinforced concrete (TRC) for structural strengthening and in prefabrication, in: G. van Zijl, W.P. Boshoff (Eds.), *Advances in Cement-Based Materials*, Taylor & Francis Group, London, 2010, pp. 127–136.
- [10] W. Bramshuber, Textile Reinforced Concrete. RILEM Report 36. State-of-the-Art Report of RILEM Technical Committee, TC 201-TRC.T, 2006.
- [11] V. Mechtcherine, Novel cement-based composites for the strengthening and repair of concrete structures. Review article, *Constr. Build. Mater.* 41 (2013) 365–373.
- [12] Z. Zhai, C. Groschel, D. Drummer, Tensile behavior of quasi-unidirectional glass fiber/polypropylene composites at room and elevated temperatures, *Polym. Test.* 54 (2016) 126–133.
- [13] P.L. Nguyen, X.H. Vu, E. Ferrier, Elevated temperature behaviour of carbon fibre-reinforced polymer applied by hand lay-up (M-CFRP) under simultaneous thermal and mechanical loadings: experimental and analytical investigation, *Fire Saf. J.* 100 (2018) 103–117.

- [14] I.E. Özlenen, P. Roshan, Composite textiles in high-performance apparel, in: *High-Performance Apparel: Materials, Development, and Applications*, Woodhead Publishing Series in Textiles, Elsevier, 2018, pp. 377–420. Available from: <https://doi.org/10.1016/B978-0-08-100904-8.00019-5>
- [15] Composites help strengthen earthquake resistance, *Reinforced Plastics*, 61 (5) (2017) 265.
- [16] M.D. Zoppo, M. Di Ludovico, A. Balsamo, A. Prota, G. Manfredi, FRP for seismic strengthening of shear controlled RC columns: experience from earthquakes and experimental analysis, *Compos. Part B: Eng.* 129 (2017) 47–57.
- [17] T. Hulin, D.H. Lauridsen, K. Hodicky, J.W. Schmidt, H. Stang, Influence of basalt FRP mesh reinforcement on high-performance concrete thin plates at high temperatures, *J. Compos. Constr.* 20 (1) (2015).
- [18] D. Ehlig, F. Jesse, M. Curbach, High temperature tests on textile reinforced concrete (TRC) strain specimens, in: W. Brameshuber (Eds.), 2nd ICTRC, Textile Reinforced Concrete. Proceeding of the Inter. RILEM Conf. on Material Science (MatSci) v.I (PRO 75), RILEM. Publications SARL, Aachen, 2010, pp. 141–151.
- [19] T. Büttner, J. Orlowsky, M. Raupach, Fire resistance tests of textile reinforced concrete under static loading—results and future developments, in: *Proceeding of the 5th Inter. RILEM Workshop on High Performance Fiber Reinforced Cement Composites (HPFRCC5)*, 2014, pp. 361–370.
- [20] H.W. Reinhardt, M. Krüger, M. Raupach, *Behavior of Textile-Reinforced Concrete in Fire*, 250, ACI Special Publication, 2008, pp. 99–110.
- [21] D.A.S. Rambo, Y. Yao, F.A. Silva, R.D.T. Filho, B. Mobasher, Experimental investigation and modelling of the temperature effects on the tensile behavior of textile reinforced refractory concretes, *Cement Concrete Compos.* 75 (2017) 51–61.
- [22] S. Xu, L. Shen, J. Wang, The high-temperature resistance performance of TRC thin-plates with different cementitious materials: experimental study, *Constr. Build. Mater.* 115 (2016) 506–519.
- [23] I. Colombo, M. Colombo, A. Magri, G. Zani, M. di Prisco, *Textile reinforced mortar at high temperatures*, Applied Mechanics and Materials, Trans Tech Publications, 2011, pp. 202–207.
- [24] J. Donnini, F.D.C. Basalo, V. Corinaldesi, G. Lancioni, A. Nanni, Fabric-reinforced cementitious matrix behavior at high-temperature: experimental and numerical results, *Compos. Part B: Eng.* 108 (2017) 108–121.
- [25] D.A.S. Rambo, F.A. Silva, R.D.T. Filho, N. Ukrainczyk, E. Koenders, Tensile strength of a calcium-aluminate cementitious composite reinforced with basalt textile in a high-temperature environment, *Cement Concrete Compos.* 70 (2016) 183–193.
- [26] D.A.S. Rambo, F.A. Silva, R.D.T. Filho, O.F.M. Gomes, Effect of elevated temperatures on the mechanical behavior of basalt textile reinforced refractory concrete, *Mater. Design* 65 (2015) 24–33.
- [27] M. Bazli, H. Ashrafi, A. Jafari, X.L. Zhao, H. Gholipour, A.V. Oskoue, Effect of thickness and reinforcement configuration on flexural and impact behaviour of GFRP laminates after exposure to elevated temperatures, *Compos. Part B: Eng.* 157 (2019) 76–99.
- [28] T.H. Nguyen, X.H. Vu, A.S. Larbi, E. Ferrier, Experimental study of the effect of simultaneous mechanical and high-temperature loadings on the behaviour of textile-reinforced concrete (TRC), *Constr. Build. Mater.* 125 (2016) 253–270.

- [29] T. Tlajji, X.H. Vu, E. Ferrier, A.S. Larbi, Thermomechanical behaviour and residual properties of textile reinforced concrete (TRC) subjected to elevated and high temperature loading: Experimental and comparative study, *Compos. Part B: Eng.* 144 (2018) 99–110.
- [30] O. Homoro, X.H. Vu, E. Ferrier, Experimental and analytical study of the thermo-mechanical behaviour of textile-reinforced concrete (TRC) at elevated temperatures: role of discontinuous short glass fibres, *Constr. Build. Mater.* 190C (2018) 645–663.
- [31] Z.C. Tetta, D.A. Bournas, TRM vs FRP jacketing in shear strengthening of concrete members subjected to high temperatures, *Compos. Part B: Eng.* 106 (2016) 190–205.
- [32] S.M. Raoof, D.A. Bournas, TRM versus FRP in flexural strengthening of RC beams: behaviour at high temperatures, *Constr. Build. Mater.* 154 (2017) 424–437.
- [33] S.M. Raoof, D.A. Bournas, Bond between TRM versus FRP composites concrete at high temperatures, *Compos. Part B: Eng.* 127 (2017) 150–165.
- [34] P.L. Nguyen, X.H. Vu, E. Ferrier, Characterization of pultruded carbon fibre reinforced polymer (P-CFRP) under two elevated temperature-mechanical load cases: residual and thermo-mechanical regimes, *Constr. Build. Mater.* 165 (2018) 395–412.
- [35] A. Mouritz, A. Gibson, *Fire Properties of Polymer Composite Materials*, Springer, 2006.
- [36] S. Tsukada, H. Nakashima, K. Torimitsu, Conductive polymer combined silk fiber bundle for bioelectrical signal recording, *PLoS One* 7 (2012) e33689.
- [37] C.M. Choi, S.N. Kwon, S.I. Na, Conductive PEDOT: PSS-coated poly-paraphenylene terephthalamide thread for highly durable electronic textiles, *J. Ind. Eng. Chem.* 50 (2017) 155–161.

This page intentionally left blank

Index

Note: Page numbers followed by “*f*” and “*t*” refer to figures and tables, respectively.

A

ABAQUS, 294
 Accelerated laboratory test, 89
 Accelerated life testing (ALT), 226–229
 design for supercapacitors, 247–248
 design of experiments, 247
 test setup, 247–248
 supercapacitors performance parameters and failure criteria, 248–252
 physics of failure models, 253–258
 Accelerated temperature cycling (ATC), 4, 108
 reliability data, 139*t*
 Accelerated temperature cycling profile effect, 119–121, 131
 Accelerated thermal cycling. *See* Accelerated temperature cycling (ATC)
 Acceleration
 vibration reliability tests, 9
 voltage effects, 30–31
 Acceleration factor (AF), 228–229, 256–258
 Acoustic emission (AE) technique, 303–304, 307–309
 damage source location, 309*f*
 Acoustic signals, 308
 parameters, 308*f*
 Activation energy, 256–258
 Active soldering fluxes, 176
 AE technique. *See* Acoustic emission (AE) technique
 AF. *See* Acceleration factor (AF)
 Ag-PDMS. *See* Silver nanoparticles in combination with PDMS rubber (Ag-PDMS)
 Aging electrical systems, 260–263
 Air-jet spun yarns, 277, 280*f*
 Airborne particulate contaminants, 96
 Airborne salinity, 88
 Alkali-resistant (AR) glass textile, 329–330
 ALT. *See* Accelerated life testing (ALT)
 Aluminum electrolytic capacitors failure, 241
 American Standard Testing Machine D412 (ASTM D412), 207
 Amorphized Co₃Fe thin film, electron radiation–induced recrystallization in, 60–65
 ANSYS Release 17.0, 180–181
 AR glass textile. *See* Alkali-resistant (AR) glass textile

Arrhenius model, 228–229, 242–243, 258
 Arrhenius reaction rate equation, 256
 Artificial intelligence, 287
 Artificial neural networks, 287–288
 ASTM D412. *See* American Standard Testing Machine D412 (ASTM D412)
 ATC. *See* Accelerated temperature cycling (ATC)
 Atmospheric corrosivity, 87
 Avrami exponent, 63–64
 Axial strain of specimen, 336

B

Back propagation artificial neuro network, 288
 Backscattered electrons (BSE), 23
 Backward compatible mixing, 113, 115–126
 accelerated temperature cycling profile, effect of, 119–121
 Ag content, effect of, 121–126
 full and partial mix, 115–118
 Baja California, 87
 Ball grid array (BGA), 3, 4*f*, 113
 effect, 130
 Band frequency filtering, 265–266
 Basalt textile specimens, 330–331
 “Bathtub” curve, 232
 Battery-powered devices, 245
 Beach ball structure, 133
 Beam-spring-mass model, 179
 BGA. *See* Ball grid array (BGA)
 Bi-bearing alloy. *See* Innolot
 Bill of materials (BOM), 226, 239
 Brittle fibers failure, 294–295
 Brittle high-performance fibers, yarn failure from, 293–294
 Brown & Srawley analytical model, FE modeling on, 72–73, 73*f*, 75*f*
 BSE. *See* Backscattered electrons (BSE)

C

Capacitance (C), 241
 Carbon (C), 162, 294
 fibers, 290
 woven macrostructure, 292*f*
 Carbon nanotubes (CNTs), 291–292
 yarn failure, 295–296
 Carbon textiles, 331, 335*f*
 equipment used, 332–334

- Carbon textiles (*Continued*)
 - furnace, 333
 - laser sensor, 333–334
 - test machine, 332
 - experimental work, 332–336
 - failure modes, 344–349, 345*f*
 - loading paths, 335–336, 337*f*
 - mechanical properties, 342*f*
 - preparation of samples, 336*f*
 - properties, 335*t*
 - results, 337–349, 339*t*
 - elevated temperature behavior of industrial textiles, 337–339
 - thermomechanical behavior, 338*f*
 - thermomechanical properties as function of temperature, 340–341
 - specimens, 334–335
 - tests, 336*t*
 - effect of textile treatment on elevated temperature behavior, 342–344
 - Caribbean zone, 87
 - Castin, 136–137
 - CCA. *See* Circuit Card Assembly (CCA)
 - Ceramic failure model, 231, 232*f*
 - Chemical FA, 19
 - Chemical mapping, 93–94
 - Chemical mechanical polishing (CMP), 36
 - ChipArray Ball Grid Array (192CABGA), 130–131, 130*f*
 - ChipArray Thin Core Ball Grid Array (84CTBGA), 130–131, 130*f*
 - Chlorine, 91, 96
 - Chromium, 93
 - Circuit Card Assembly (CCA), 171–172
 - CL. *See* Condenser lens (CL)
 - CMP. *See* Chemical mechanical polishing (CMP)
 - CNTs. *See* Carbon nanotubes (CNTs)
 - Co₃Fe thin film, electron radiation–induced unilateral amorphization of, 57–59
 - Coalescence of cracks, 320
 - Cocoyoc zone, 96
 - Coefficient of thermal expansion (CTE), 4–5, 119
 - Coefficients matrix, 185
 - CoFeB material
 - boron diffusion and segregation induced phase and microstructure changes in, 55–65
 - CoFeB/SiO₂ thin film stack, 65
 - Component's qualification, 239
 - Composite materials reinforced with textiles, 306
 - Composite structures, yarn failure in, 290–293
 - Condenser lens (CL), 31
 - Conductive ink, 207
 - stress–strain analysis of substrate and, 208–209
 - Conductive yarn failures, 296–297
 - Connection defects detection
 - algorithm based on band frequency filtering, 265–266
 - and localization, 263–265
 - Contaminants, 153
 - Contamination, in electronics failure, 153
 - active soldering fluxes, 176
 - contamination of electronic circuits, 175–176
 - contamination as primary cause of motor failures, 154–165
 - chemical characterizations, 158–165
 - control stock motor, 155–156
 - electrical characterization, 155
 - failed motor, 156–158
 - motor operation investigation, 154–155
 - electrolyte contamination, 165–175
 - Coplanar cracks, 74–82, 76*f*
 - effect of horizontal distance between, 78–82
 - Corner solder joint deflection
 - impact loading, 195
 - validation in harmonic loading, 190–191
 - Corrosion products, 90
 - Crack density, 311–312
 - Critical solder joint stress analysis
 - in harmonic loading, 191–195
 - printed circuit board stiffness, 192–194
 - solder joint geometry, 195
 - impact loading, 196
 - printed circuit board stiffness, 196
 - solder joint geometry, 196
 - Cryo-TEM imaging, 41–43
 - CTE. *See* Coefficient of thermal expansion (CTE)
 - Curve fitting technique, 209
- ## D
- Damage analysis, 90–97
 - in knitted fabric, 309–315
 - Damage charts, 91–93, 92*f*
 - Daniel's statistical model, 287–288
 - Data averaging, 233–234
 - Deformation behavior of SEC, 216–219
 - Deformation mechanics of interconnect, 11, 11*f*
 - Degradation analysis
 - based on data collected from economical setup, 254–255
 - based on data collected from research setup, 253–258
 - Deionized water (DI water), 175
 - Dew, 87–88
 - DI water. *See* Deionized water (DI water)
 - Displacement amplitude (*Z*), 185
 - 6DoF pneumatic hammer, 234–235
 - Domain reflectometry (TDR), 260–263

Drop and vibration failure, 12*f*

Drop test experiment, 8, 8*f*

Dual-beam FIB, 38–39

Ductile failures, 11–12

Dwell time effect, 131

E

EBSD. *See* Electron backscattered diffraction (EBSD)

EDS. *See* Energy-dispersive X-ray spectroscopy (EDS)

EELS. *See* Electron energy loss spectroscopy (EELS)

EFA. *See* Electrical failure analysis (EFA)

Elastically coupled two-parallel-plate system, 179

Elastomers, 205

Electrical characterization of motor, 155

Electrical failure analysis (EFA), 19

Electroless nickel immersion gold (ENIG), 137

Electrolyte

contamination, 165–176

leaking capacitor, 166–173

types of electrolyte contamination damage, 173–175

contamination damage, 173–175

corrosion of copper wire, 174*f*

electromigration of metals, 175*f*

electrolytic capacitors reliability analysis, 239–244

physical stresses factors for aluminum capacitors, 241*t*

Electron backscattered diffraction (EBSD), 108–109

Electron beam radiation, 20–21

damage, 21–22, 21*f*

during electron beam coating before FIB milling, 37–38

during electron beam survey before FIB milling, 36–37

during FIB milling for TEM sample preparation, 38–40

to low-*k* and ultralow-*k* dielectric materials, 25–35

during SEM failure analysis, 22–35

during TEM analysis, 40–45

during TEM failure analysis, 45–65

during FIB and TEM failure analysis, 35–45

structure deformation, 25*f*

Electron beam–induced knock-on damage, 26–28

Electron beam–induced radiolysis damage, 30

Electron beam–induced thermal damage, 28–30

Electron bombardment–induced sputtering, 21–22

Electron energy loss spectroscopy (EELS), 20

Electron radiation–induced recrystallization in amorphized Co₃Fe thin film, 60–65

Electron radiation–induced unilateral amorphization of Co₃Fe thin film, 57–59

Electronic assemblies, analytical solutions of, 179, 239

experimental modal analysis, 180

FE modeling, 180–182

forced vibration, 185–189

free vibration, 182–185

results

forced vibration, 190–200

free vibration, 190

test assembly details, 180

Electronic circuits, contamination of, 175–176

Electronic devices, failure of

shock and vibration, 8–12

thermal cycling, 4–8

Electronic package, 3, 3*f*

Electronic products, 107, 179

Electronic systems

application examples, 239–258

combined methodology on FLM, 244–245

electrolytic capacitors reliability analysis, 239–244

supercapacitors reliability analysis, 245–258

[HALT + ALT] rather than ALT, 234–236, 236*f*

improved reliability assessment method, 226–238

improved reliability estimation methods, 229

intelligent life data analysis, 232–234

new trends to improving reliability analysis, 259–266

mission profile, 259–260

online condition monitoring, 260–266

prediction handbooks, 229–231

reliability block diagram tools and fault tree analysis for complex systems, 236–238

Electronic textiles, yarn failure in, 296–297

Electronics industry, failure detection methods in, 12–14

Elevated temperature behavior of industrial textiles, 337–339

Energy-dispersive X-ray spectroscopy (EDS), 20, 89–90, 161–162

Enforced motion method, 182

Engineering asset, 225–226

ENIG. *See* Electroless nickel immersion gold (ENIG)

Equivalent series resistance (ESR), 241

Equivalent stress analysis of thermal sensor circuit design, 219

ESR. *See* Equivalent series resistance (ESR)

Etch process, 31–32
 Eutectic SAC387, 128–129
 Eutectic SnPb, 136–137
 Ex situ photoresist spin coating, 37

F

FA. *See* Failure analysis (FA)
 Fabric-reinforced “textile” composites, 307
 Fabrics, yarn failure in, 288–290
 Failed motor, 156–158
 commutators with surface deposits, 160*f*
 oscilloscope capture displaying current waveform, 158*f*
 Failure analysis (FA), 19
 correlation, 20*f*
 Failure mode and effect analysis (FMEA), 250
 Fatigue life assessment of electronic assemblies, 179
 Fault tree analysis (FTA), 225–226
 for complex systems, 236–238, 237*f*
 FE modeling. *See* Finite element (FE) modeling
 FEA. *See* Finite element analysis (FEA)
 FIB. *See* Focus ion beam (FIB)
 Fiber reinforced polymer (FRP), 329
 Fiberglass, 290, 291*f*
 woven macrostructure, 291*f*
 FIDES prediction handbook, 229–231
 Finite element analysis (FEA), 182, 206, 209–215
 boundary conditions
 analysis using simulation process, 214–215
 printing circuits, 212–213
 thermal sensor circuit, 213–214
 modeling and meshing of different printing shapes models, 210–212
 Finite element (FE) modeling, 72–77, 180–182, 181*f*
 based on Brown & Srawley analytical model, 72–73, 73*f*, 75*f*
 linear elastic material properties used in, 182*t*
 multiple cracks analysis on solder joint behavior, 74–77
 solder joint modeling, 73–74
 Fire tests, 329–330
 First generation Sn–Ag–Cu solder alloys, 128–135
 FLM. *See* Front light module (FLM)
 FMEA. *See* Failure mode and effect analysis (FMEA)
 Focus ion beam (FIB), 22
 electron beam radiation damage
 during electron beam coating, 37–38
 during electron beam survey, 36–37
 electron beam radiation during, 35–45

 slice, 38–39
 for TEM sample preparation, 38–40
 Forced vibration
 harmonic loading, 185–187, 190–195
 impact loading, 195–200
 corner solder joint deflection, 195
 critical solder joint stress analysis, 196
 shock loading, 187–189
 solder stress response spectrum, 197–200, 198*f*, 200*f*
 Forward back propagation model, 288
 Forward compatible mixing, 113–115
 Fourier-transform infrared (FTIR) spectroscopy
 method, 162–164, 171–172
 Fractography, 320
 Fracture
 analysis, 82
 behavior, 71, 82
 Free vibration, 182–185, 190
 geometric and material parameters of two elastically coupled plates problem, 183*r*
 SDOF system, 184*f*
 Frequency domain reflectometry, 260–263
 Friction spun yarns, 277
 Front light module (FLM), 239
 circuit diagram, 239
 combined methodology on, 244–245
 FRP. *See* Fiber reinforced polymer (FRP)
 FTA. *See* Fault tree analysis (FTA)
 FTIR spectroscopy method. *See* Fourier-transform infrared (FTIR) spectroscopy method
 Furnace, 333

G

Galvanic corrosion, 93, 96
 Gas chromatography/mass spectrometry (GC/MS), 162
 Gauge length, yarn failure depending on, 285–286
 GC/MS. *See* Gas chromatography/mass spectrometry (GC/MS)
 GC1 textile, 334–335, 342–343
 GC2 textile, 334–335, 342–343
 GC3 textile, 334–335, 342–343
 Geotextiles, mechanical properties of, 318–324, 320*f*
 Glass fibers, 290, 294
 Global mesh function, 211
 Gold plating, 96
 Gravimetric method, 88
 Gravity accelerations, 8
 Grey Wolf Optimizer, 288
 Gun lens, 31

H

- HALT. *See* Highly accelerated life testing (HALT)
- Harmonic loading, 185–187
 - corner solder joint deflection validation, 190–191
 - critical solder joint stress analysis, 191–195
- Harmonic vibration, 10–11, 10*f*
- High-dose electron radiation, 54–55
- High-performance solders, 135–141. *See also*
 - Low-temperature solders
 - commercialized alloys, 136–141
 - micro-alloying effect on Sn–Ag–Cu, 136
- High-resolution TEM (HRTEM), 55
- High-strength yarn failure, 293–294
- Highly accelerated life testing (HALT), 234
- Hollow-spindle spinning method, 277
- Hooke's law equation, 196
- HRTEM. *See* High-resolution TEM (HRTEM)
- Humidity detector, 166
- Hybrid yarn manufacturing, 295

I

- IC. *See* Integrated circuit (IC)
- IEC TR 62380, 229–230
- IMCs. *See* Intermetallic compounds (IMCs)
- IMD. *See* Intermetal dielectrics (IMD)
- IML. *See* Intermetallic layer (IML)
- Impact loading, 8, 195–200
 - corner solder joint deflection, 195
 - critical solder joint stress analysis, 196
- Impedance measurement technique, 260–263, 264*f*
- In situ electron beam coating in FIB tool, 37
- Indoor corrosion, 87
 - damage analysis, 90–97
 - damage levels, 91*t*
 - methods, 89–90
- Inductance, capacitance, and resistance (LCR)
 - measurements, 260–263
- Industrial carbon textile, 331
- Industrial textiles, 329
 - elevated temperature behavior, 337–339
- Inelastic scattering–induced ionization effect, 28
- Infrared spectrum, 164, 164*f*
- Innolot, 136–137
- Integrated circuit (IC), 3, 19, 90–91
 - component, 179
 - piezoelectric impact hammer, 180
- Intelligent life data analysis, 232–234
- Interdendritic regions, 132–133
- Intermetal dielectrics (IMD), 24
- Intermetallic compounds (IMCs), 71, 115
- Intermetallic layer (IML), 126

- Interweave geometry effect, 318–324
- “Ionic” contamination, 165–166
- IPC-9701 standard, 14
- IPC/JEDEC-9702 standard, 14
- IPC-SM-785 standard, 13

J

- Japanese Electronics Industry Association (JEITA), 128–129
- JESD22-B111 failure criteria, 14
- Johnson–Mehl–Avrami model (JMA model), 63–64
- Joint Electron Device Engineering Council (JEDEC), 8

K

- Kevlar yarns, 293
- Kinetic isothermal grain growth, 62–63
- Knitted fabrics, 305–306, 305*f*
 - damage analysis in, 309–315
 - composite, 313*f*
 - crack development for samples, 312*f*
 - microdebonding, 314*f*
 - stress–strain AE events for textile composite, 314*f*
- Knitted loops, 310
- Knock-on damage, 21–22

L

- Land grid array (LGA), 133
- Laser sensor, 333–334
- LCR measurements. *See* Inductance, capacitance, and resistance (LCR) measurements
- LDA. *See* Life data analysis (LDA)
- Lead free solder alloys, 107, 109–112
 - failure and microstructure, 108–109
 - first-and second-generation Sn–Ag–Cu solder alloys, 128–135
 - high-performance solders, 135–141
 - low-temperature solders, 141–145
- Pb-doped solder alloys, 113–128
 - backward compatible mixing, 113, 115–126
 - forward compatible mixing, 113–115
 - lessons learning, 126–128
 - trade names of alloys and nominal compositions, 108*t*
- LEADFREE project, 141–142
- Leaking capacitor, 166–173
 - capacitor on commercial off-the-shelf humidity detector circuit card, 171*f*
 - elemental spectrum collected of humidity detector residue, 168*f*

Leaking capacitor (*Continued*)
 humidity detector with residues on surface, 167*f*
 infrared spectral database matching result of
 connector residue spectrum, 170*f*
 residues on connector frame, 167*f*
 LGA. *See* Land grid array (LGA)
 Life data analysis (LDA), 226, 228
 Life-stress models, 241
 Liquid electrolytes, 166
 LK dielectrics. *See* Low-*k* dielectrics (LK
 dielectrics)
 Loading paths of carbon textiles, 335–336, 337*f*
 Localization, connection defects detection and,
 263–265
 Low dose
 in FEI TEMs, 41
 TEM technique, 41, 43
 Low-*k* dielectrics (LK dielectrics), 20–25
 control of electron beam radiation damage to,
 30–35
 electron beam radiation damage to, 25–30
 radiation damage to, 35–45
 SiCOH materials, 25, 33–34
 Low-temperature solders, 141–145. *See also*
 High-performance solders
 lessons learning, 145
 micro-alloying effect, 142–144
 effect of substrate, 142

M

MAC number. *See* Modal assurance criterion
 number (MAC number)
 Magnetic random access memory (MRAM), 22
 Magnetic tunneling junction (MTJ), 55
 Maximum axial thermomechanical deformation,
 336
 Maximum likelihood estimator (MLE), 234
 Mean time between failures (MTBF), 228
 Mechanical bending of PCB, 11
 Mechanical vibrations, 9
 Median rank (MR), 234
 Meshing, 211
 Micro-alloying, 136–137
 effect on Sn–Ag–Cu, 136
 Microscale modeling, 295
 Microstructural evolution, 5–8, 108, 131–135
 Microstructural failure mechanisms, 307
 Milano weft-knitted fabric structure, 310, 311*f*
 Mind evolutionary neural network, 288
 Mission profile, 259–260
 MLE. *See* Maximum likelihood estimator (MLE)
 Modal assurance criterion number (MAC number),
 190

Modern electronics technologies, 227–228
 Moiré fringes, 62
 MR. *See* Median rank (MR)
 MRAM. *See* Magnetic random access memory
 (MRAM)
 MTBF. *See* Mean time between
 failures (MTBF)
 MTJ. *See* Magnetic tunneling junction (MTJ)
 Multiaxial E-glass textile, 315
 Multiaxial noncrimp fabrics
 damage sequence in, 315–317
 technical specification of textile, 316*t*
 Z-pinned multiaxial textile architecture/
 geometry, 317*f*
 Multilinear plastic model, 208–209
 Multiple cracks analysis on solder joint behavior,
 74–77
 Multiple linear regression technique, 288

N

Nanofibers, 289–290
 National Instruments data acquisition system
 model 4413 (NIDAQ-4413), 180
 NCFs. *See* Noncrimp fabrics (NCFs)
 Neo-Hookean model, 206–209
 Neuro-fuzzy models, 287
 Newtons (N), 277
 Nickel, 93–94
 Ni-doped SAC305, 136
 NIDAQ-4413. *See* National Instruments data
 acquisition system model 4413
 (NIDAQ-4413)
 Nitride thinning-down, 51–53
 Nitrogen oxides (NO_x), 88, 98–99
 Noncrimp fabrics (NCFs), 315
 Nondestructive techniques, 307
 Nonsilicone contamination source, 165
 Nonstitched materials, 305, 305*f*
 Nonvolatile memory (NVM), 45–46
 Nonwoven fabrics, 305, 305*f*
 Normalized mechanical properties of carbon
 textiles, 340, 340*t*
 Normalized ultimate stress, 340, 341*f*
 Normalized Young's modulus, 340–341, 341*f*
 NVM. *See* Nonvolatile memory (NVM)

O

ODE. *See* Ordinary differential equation (ODE)
 Online condition monitoring, 260–266
 concepts for connection defects detection,
 prognostics, and localization, 263–266
 algorithm based on band frequency filtering,
 265–266

method for connection defects detection and localization, 263–265

ONO. *See* Oxide–nitride–oxide (ONO)

Ordinary differential equation (ODE), 188

Organic acids, 165–166

Organic solderability preservative (OSP), 142

Outgassing, 153

Oxide–nitride–oxide (ONO), 46–50

Oxygen (O), 162, 167–168

Ozone, 98–99

P

pA. *See* Pico-ampere (pA)

Parallel edge cracks, 74

effect of distance between, 77–78

Parametric stochastic model, 232

PCBs. *See* Printed circuit boards (PCBs)

PDMS. *See* Polydimethylsiloxane (PDMS)

PES. *See* Polyester (PES)

PET. *See* Polyethylene-terephthalate (PET)

PFA. *See* Physical failure analysis (PFA)

Photoresist (PR), 37

Physical failure analysis (PFA), 19, 22–25

Physics of failure model (PoF model), 225–226, 246, 253–258

product development cycle, 239, 240f

prognostics model, 231, 233f

Pico-ampere (pA), 28

Piezoelectric sensors, 307–308

PLM. *See* Polarized light microscopy (PLM)

PoF model. *See* Physics of failure model (PoF model)

Poisson's ratio, 209

Polarized light microscopy (PLM), 108–109

bright-field and PLM images of SAC–Mn solder joints, 110f

image and EBSD map of SAC–Mn solder joint, 111f

Pollutants, 88, 90

Polydimethylsiloxane (PDMS), 205–207

Polyester (PES), 315

Polyethylene-terephthalate (PET), 320–321, 322f, 323f

Polynomial fit (polyfit), 234

Polypropylene (PP), 320–321, 322f, 323f

Polyvinyl chloride (PVC), 93

Pore-like defects, 53–54

Potassium carbonate, 165–166

PP. *See* Polypropylene (PP)

PR. *See* Photoresist (PR)

Prediction handbooks, 226–231

Printed circuit boards (PCBs), 3, 89–91, 107, 179, 182–184

bending, 11

stiffness

in harmonic loading, 192–194

in impact loading, 196

Printing circuits, 212–213

Printing process of circuits, 207

Probe current effects, 31–32

Pt anticharging coat layer effects, 32–35

PVC. *See* Polyvinyl chloride (PVC)

R

Radiation damage, 21–22

to low-*k* and ultralow-*k* dielectrics, 35–45

to silicon nitride, 45–55

Radiation-enhanced diffusion effects (RED effects), 51–53, 64–65

Radiation-induced heating effect, 29–30

Radiation-induced ionization effects, 21–22

Radiolysis damage, 30

Random vibrations, 10–11, 10f

RBD. *See* Reliability block diagram (RBD)

RED effects. *See* Radiation-enhanced diffusion effects (RED effects)

Reliability, 179

improved reliability assessment method, 226–238

ALT, 228–229

LDA, 228

prediction handbooks, 227–228

improved reliability estimation method, 229

Reliability block diagram (RBD), 225–226

decomposition of system, 238f

tools, 236–238

Residual condition, 329–330

Restriction on hazardous substances (RoHS), 107, 128–129

Ring spun yarns, 282

Ritz method, 182–184

RoHS. *See* Restriction on hazardous substances (RoHS)

Rotor spun yarns, 277

S

SAC alloys. *See* Sn–Ag–Cu alloys (SAC alloys)

SAC305 alloy, 128–129, 141–142

BGAs, 113

SAC387 as conventional Pb-free alloy, 136–137

SAC396 alloy, 128–129

Sb-doped SAC alloy. *See* Castin

Scanning electron microscope (SEM), 20, 89–90, 161. *See also* Transmission electron microscope (TEM)

- Scanning electron microscope (SEM) (*Continued*)
 - electron beam radiation damage during SEM failure analysis, 22–35
 - physical FA and low- k /ultralow- k dielectrics, 22–25
- Scanning TEM (STEM), 53–54
- Screen mesh fabrication, 207
- SDOF system. *See* Single-degree-of-freedom system (SDOF system)
- SE. *See* Secondary electrons (SE)
- SEC. *See* Stretchable electronic circuit (SEC)
- Second-generation Sn–Ag–Cu solder alloys, 128–135
- Secondary electrons (SE), 23
- SEM. *See* Scanning electron microscope (SEM)
- Semiconductor industry, 20
- SerialEM in JEOL TEMs, 41
- Shielding effect, 77
- Shimadzu universal machine, 316
- Shock, 8–12
 - failure, 11–12
 - loading, 187–189
- Shock response spectrum (SRS), 197–199
- SIFs. *See* Stress intensity factors (SIFs)
- Silicon (Si), 162
- Silicon nitride, radiation damage to, 45–55
- Silicone rubber, 206–207
- Silver (Ag), 99, 206–207
 - Ag₃Sn IMC particles, 119–121
 - content effect, 121–126, 130–131
- Silver nanoparticles in combination with PDMS rubber (Ag-PDMS), 206–207
- Single-beam FIB, 38–39
- Single-degree-of-freedom system (SDOF system), 184, 184*f*
- Sinusoidal signal, 10–11
- Sn–9Zn solder joint, 142
- Sn–Ag–Cu alloys (SAC alloys), 73, 107
 - first-and second-generation, 128–135
 - effect of accelerated temperature cycling profile, 131
 - Ag content effect, 130–131
 - effect of BGA component, 130
 - dwell time effect, 131
 - lessons learning, 135
 - microstructural evolution and failure mechanisms, 131–135
 - micro-alloying effect on, 136
- Sodium chloride, 165–166
- Solder failure, 14
- Solder joints, 3, 5–8, 107
 - axial deflection, 189
 - failure, 71
 - geometry
 - in harmonic loading, 195
 - in impact loading, 196
 - modeling
 - FE, 73–74
 - material properties for, 74*t*
- Solder Product Value Council (SPVC), 128–129
- Solder stress response spectrum, 197–200
- Solidification sequence, 132–133
- Spectra polyethylene fibers, 293–294
- Spinning methods, 277
 - staple yarn failure depending on, 281–285
- Spun yarn failure, 277
 - air-jet spun yarn, 280*f*
 - helical structure of ring spun yarn, 278*f*
 - Wrap spun yarn, 281*f*
- SPVC. *See* Solder Product Value Council (SPVC)
- SRS. *See* Shock response spectrum (SRS)
- Standard field tests, 89
- Staple yarn failure, 281–285
 - broken end of staple fiber yarn, 282*f*
 - stress–strain curve of staple fiber yarn, 281*f*
- STAR Modal Version 7.0, 180
- Static loading, 73, 179
- Statistical learning theory, 288
- STEM. *See* Scanning TEM (STEM)
- Stitched fabrics, 305, 305*f*
- Stochastic model, 229
- Stock motor control, 155–156
 - oscilloscope capture displaying commutation current, 157*f*
 - oscilloscope capture displaying current waveform, 156*f*
- Strain rate, yarn failure depending on, 286–287
- Stress analysis. *See also* Stretchable conductive polymer, stress analysis of
 - equivalent, 219
 - critical solder joint, 191–196
- Stress intensity factors (SIFs), 71–74
 - at A1 and A2 crack tips, 77*f*, 78*f*, 79*f*
 - against x -distance, 80*f*, 81*f*
- Stress–strain analysis of substrate and conductive ink, 208–209
 - multilinear plastic model, 209
 - Neo-Hookean model, 209
- Stress–strain behavior, 206–207
- Stress–strain curve, 345
- “Stress–thermomechanical strain” curve, 344
 - of carbon textile samples, 337
- Stretchability, 206
- Stretchable conductive polymer, stress analysis of
 - equivalent stress analysis of thermal sensor circuit design, 219
 - equivalent stress limitation, 221
 - experimental procedure

printing process of circuits, 207
 sample preparation, 206–207
 universal tensile testing, 207
 FEA, 209–215
 future recommendations, 222
 stress–strain analysis of substrate and
 conductive ink, 208–209
 width effect in reducing equivalent stress in
 thermal sensor circuit, 220–221
 Stretchable electronic circuit (SEC), 205
 deformation behavior of, 216–219
 material properties of material, 215–216
 Sulfur (S), 162, 167–168
 Sulfuric acid, 171
 Supercapacitors
 accelerated life test design for, 247–248
 failure modes, 246–247
 reliability analysis, 245–258
 Surface crystallization, 64

T

Taguchi design of experiment, 288
 Tampico, 89, 99
 TDR. *See* Domain reflectometry (TDR)
 TEM. *See* Transmission electron microscope
 (TEM)
 Test machine, 332
 Textile
 architectures, 304–306, 305*f*
 raw materials for textiles manufacturing, 304*f*
 mechanical and damage characterization,
 309–324
 damage analysis in knitted fabric, 309–315
 damage sequence in multiaxial noncrimp
 fabrics, 315–317
 mechanical properties of geotextiles,
 318–324
 mechanical properties, 306
 textiles-reinforced composites, 306–307
 treatment effect on elevated temperature
 behavior, 342–344
 Textile reinforced concrete (TRC), 329
 Thermal cycling, 4–8
 deformation of solder joint, 6*f*
 failure, 7*f*
 one thermal cycle profile, 5*f*
 Thermal fatigue, 128–129
 Thermal sensor circuit
 equivalent stress analysis, 219
 width effect in reducing equivalent stress,
 220–221
 Thermal sensor circuit, 213–214
 Thermomechanical condition, 329–330

Thermomechanical test at constant load (TMCL),
 329–330
 Thermomechanical test at constant temperature
 (TMCT), 329–330, 335–336
 Thermoplastic polyurethane (TPU), 205
 Through-the-coating corrosion, 96
 Time to failure (TTF), 233–234, 242
 Tin (Sn), 91, 108–109
 Tin–lead (SnPb)
 BGAs, 113
 manufacturing processes, 113
 Tin–silver–copper alloys. *See* Sn–Ag–Cu alloys
 (SAC alloys)
 TMCL. *See* Thermomechanical test at constant
 load (TMCL)
 TMCT. *See* Thermomechanical test at constant
 temperature (TMCT)
 TPU. *See* Thermoplastic polyurethane (TPU)
 Transient elastic waves, 309
 Transmission electron microscope (TEM), 20.
 See also Scanning electron microscope
 (SEM)
 electron beam radiation damage, 40–45
 during FIB milling for, 38–40
 electron beam radiation during, 35–45
 Transversal yarns, 320–321
 TRC. *See* Textile reinforced concrete (TRC)
 TTF. *See* Time to failure (TTF)
 Two-dimensional triaxial braided composite
 material, 292

U

Ultimate stress, 336
 Ultralow- k (ULK) dielectrics, 20–21
 control of electron beam radiation damage to,
 30–35
 electron beam radiation damage to, 25–30
 radiation damage to, 35–45
 in semiconductor industry, 22–25
 shrinkage, 41*t*
 SiCOH materials, 25
 Uniaxial tensile test, 215
 Universal tensile testing, 207, 208*f*

V

Vector machine models, 288
 Vibration, 8–12
 test setup, 9*f*
 Volatile organic components (VOCs), 98–99

W

Wale direction, 310

Walopur TPU, 205
 Weibull analysis, 226
 Weibull model, 229, 250
 Weibull plots, 108, 113, 124*f*
 of forward compatible mixed and SnPb
 assembly, 114*f*
 of fully mixed, partially mixed, SAC, and SnPb
 assemblies, 116*f*
 of fully mixed and partially mixed assembly,
 117*f*
 of Sn–9Zn with ENIG, IAg, and OSP surface
 finish, 143*f*
 Weighted failure rate, 227–228
 Woven fabrics, 305, 305*f*
 Wrap yarns, 277, 281*f*, 283–285

Y

Yarn failure, 281
 from brittle high-performance fibers, 293–294

CNTs yarn failure, 295–296
 in composite structures, 290–293
 depending on gauge length, 285–286
 depending on strain rate, 286–287
 in electronic textiles, 296–297
 in fabrics, 288–290
 high-strength yarn failure, 294–295
 modeling, 287–288
 spun yarn failure, 277
 staple yarn failure depending on spinning
 method, 281–285
 Young's modulus of carbon textiles, 338,
 343–344

Z

Zeiss Ultra55 SEM probe current, 31, 32*f*
 Zinc, 93, 96
 Z-pinned multiaxial textile, 315–316, 317*f*
 Zylon yarns, 294

Handbook of Materials Failure Analysis

With Case Studies from the Electronic and Textile Industries

Edited by

Abdel Salam Hamdy Makhlouf and Mahmood Aliofkhazraei

Case studies in failure analysis for engineers and researchers working to mitigate or eliminate costly materials-related failures within the electronics and textiles industries.

Handbook of Materials Failure Analysis: With Case Studies from the Electronics and Textiles Industries examines the reasons why materials fail in certain situations, including material defects and mechanical failures as a result of a variety of causes. The book is organized into two parts. Part 1 covers the challenges of materials failure analysis with case studies from electronic industries. Part 2 covers materials failure analysis with challenges common to the textile industries. Chapters provide an overview of materials failure analysis and its impacts in electronic and textile industries, and then proceeds logically from a discussion of the failure analysis process, specific tools and techniques, to an analysis of types and causes of failure analysis in these industries. The book covers the most common types of materials failure analysis along with possible solutions.

Failure can occur for several reasons: materials defect-related failure; materials design-related failure; or corrosion-related failures. The suitability of the materials to work in a definite environment is an important issue. This book is essential reading for materials scientists, engineers, chemists, managers, directors and researchers working in materials performance, failure analysis, materials selection and durability.

- Provides the most up-to-date and balanced coverage of failure analysis, combining foundational knowledge and current research on the latest developments and innovations in the field
- Offers an ideal accompaniment for those interested in materials forensic investigation, failure of materials, static failure analysis, dynamic failure analysis, and fatigue life prediction
- Presents compelling new case studies from key industries to demonstrate concepts

Abdel Salam Hamdy Makhlouf, Vice President & Engineering Consultant, Integrated Mechanical Material Corrosion Consulting (IM2C), Texas, USA, Professor, Central Metallurgical Research and Development Institute, Cairo, Egypt

Mahmood Aliofkhazraei, Assistant Professor, Department of Materials Science, Tarbiat Modares University, Tehran, Iran



Butterworth-Heinemann

An imprint of Elsevier

elsevier.com/books-and-journals

TECHNOLOGY AND ENGINEERING /
MATERIALS SCIENCE

ISBN 978-0-08-101937-5



9 780081 019375