



Failure Analysis in Biocomposites, Fibre- Reinforced Composites and Hybrid Composites

Edited by Mohammad Jawaid, Mohamed Thariq
and Naheed Saba

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Woodhead Publishing Series in Composites
Science and Engineering

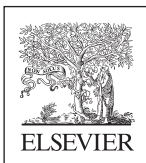
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WOODHEAD
PUBLISHING

An imprint of Elsevier

Woodhead Publishing is an imprint of Elsevier
The Officers' Mess Business Centre, Royston Road, Duxford, CB22 4QH, United Kingdom
50 Hampshire Street, 5th Floor, Cambridge, MA 02139, United States
The Boulevard, Langford Lane, Kidlington, OX5 1GB, United Kingdom

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Library of Congress Cataloging-in-Publication Data

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

British Library Cataloguing-in-Publication Data

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

ISBN: 978-0-08-102293-1

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



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Publisher: Matthew Deans

Acquisition Editor: Gwen Jones

Editorial Project Manager: Thomas Van Der Ploeg

Production Project Manager: Poulouse Joseph

Designer: Mark Rogers

Typeset by TNQ Technologies

Dedicated to

Dr. Asif Saba

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Contents

List of contributors	xi
About the editors	xv
Preface	xvii
1. Fatigue delamination of carbon fiber-reinforced polymer-matrix composites	1
<i>Tanveer Ahmed Khan, Hoon Kim and Hyun-Joong Kim</i>	
1.1 Introduction	1
1.2 Processing of polymer-matrix composite	3
1.3 Load control delamination fatigue	11
1.4 Classification of the methods for prediction delamination growth	13
1.5 Conclusion	20
References	21
Further reading	28
2. Investigation of the deformation behavior of epoxy-based composite materials	29
<i>Hind Abdellaoui, Marya Raji, Rachid Bouhfid and Abou el kacem Qaiss</i>	
2.1 Introduction	29
2.2 Thermosetting matrix: epoxy	30
2.3 Mechanisms of rupture and damage of composites based on epoxy	31
2.4 Epoxy reinforced composites	37
2.5 Rupture criteria	42
2.6 Conclusion	48
Acknowledgements	48
References	48
3. Effects of strain rate on failure mechanisms and energy absorption in polymer composites	51
<i>M.R. Nurul Fazita, H.P.S. Abdul Khalil, A. Nor Amira Izzati and Samsul Rizal</i>	
3.1 Introduction	51
3.2 Energy absorption capability	52
3.3 Failure mechanisms	52
3.4 Strain rate	53
3.5 The effect of strain rate on the failure mechanisms and energy absorption	58
3.6 Conclusions	76
References	76

4. Bolted joint behavior of hybrid composites	79
<i>Ng Lin Feng, Sivakumar Dhar Malingam and Siva Irulappasamy</i>	
4.1 Introduction	79
4.2 Overview of hybrid composite materials	80
4.3 Structural applications of bolted joint	82
4.4 Factors influencing the bearing strength of hybrid composites	83
4.5 Failure analysis of hybrid composite due to bolted joint	87
4.6 Conclusions	92
References	93
5. Failure analysis and the optimal toughness design of sheep—wool reinforced epoxy composites	97
<i>K.N. Bharath, G.B. Manjunatha and K. Santhosh</i>	
5.1 Introduction	97
5.2 Experimental details	98
5.3 Results and discussion	100
5.4 Linear regressions	105
5.5 Conclusion	106
References	106
6. Edge crack effect on tensile behavior of diversified wood particulate composites	109
<i>Nadendla Srinivasababu</i>	
6.1 Introduction	109
6.2 Discarded particulate natural fiber materials	111
6.3 Preparation of particulate composites and testing	112
6.4 Results and discussion	112
6.5 Conclusions	129
References	130
7. Investigation of mechanical testing on hybrid composite materials	133
<i>Mohd Hafizal Hamidon, M.T.H. Sultan and Ahmad Hamdan Ariffin</i>	
7.1 Introduction	133
7.2 Methodology	140
7.3 Failure analysis on tensile and compression	149
7.4 Conclusions	153
References	154
8. Cracks, microcracks, and fracture toughness of polymer composites: formation, testing method, nondestructive detection, and modifications	157
<i>M.A. Azmah Hanim, Dermot Brabazon and M.S.J. Hashmi</i>	
8.1 Introduction	157
8.2 Formation of cracks and microcracks in polymeric composites	158

8.3	Fracture mechanics in polymeric composites	159
8.4	Detection of cracks and microcracks using the nondestructive testing method in a polymeric composite	166
8.5	Current modifications to improve fracture toughness for polymeric composites	172
8.6	Conclusion	178
	References	179
9.	Shape memory polymer and its composites as morphing materials	181
	<i>M.H. Mat Yazik and M.T.H. Sultan</i>	
9.1	Introduction	181
9.2	Concept of shape memory polymer	182
9.3	History of shape memory polymer	183
9.4	Reinforcement of shape memory polymer (SMP)	184
9.5	Applications of shape memory polymer (SMP)/shape memory polymer composites (SMPC)	189
9.6	Conclusion	194
	Acknowledgments	195
	References	195
10.	Fabric-Reinforced Cementitious Matrix (FRCM) composites: mechanical behavior and application to masonry walls	199
	<i>Fulvio Parisi, Costantino Menna and Andrea Prota</i>	
10.1	Introduction	199
10.2	Mechanical behavior of FRCM composites	201
10.3	Mechanical behavior of masonry walls with FRCM strengthening	208
10.4	Conclusions	222
	References	224
11.	Failure analysis in hybrid composites prepared using industrial wastes	229
	<i>V. Arumugaprabu, Tae Jo Ko, M. Uthayakumar and R. Deepak Joel Johnson</i>	
11.1	Introduction	229
11.2	Materials and methods	230
11.3	Mechanical performance studies on the hybrid composites	236
11.4	Effect of industrial waste-filled hybrid composites on the mechanical performance	239
11.5	Failure analysis of the hybrid composites with industrial wastes	241
11.6	Conclusions	243
	References	243
	Index	245

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Preface

The advanced characterization techniques used for analysis of composite materials fabricated by incorporating natural fibers/biomass, synthetic fibers, and combination of these materials as fillers and reinforcements to enhance end materials' performance toward utilization in automotive, aerospace, construction, and building components. This book remarkably fills the gap in the published literature involving failure analysis of biocomposites, fiber-reinforced composites, and hybrid composites and provides a reference material for future research in natural and hybrid composite materials, which are currently in great demand due to their sustainable, recyclable, and eco-friendly nature as required in different applications.

This book is focused on failure analysis in composite materials. It covers every aspect of fracture and failure in natural/synthetic fiber-reinforced polymer-based composite materials, comprising the latest data. From crack propagation to crack growth, from notch-size effect to damage-tolerant design, it describes a range of techniques and strategies for compositional and failure analysis of polymeric materials and products. It also illustrates the application of analytical methods for solving commonly encountered problems in the composite-based industries.

This book covers topics including: fatigue delamination of carbon fiber-reinforced polymer-matrix composites; investigation of the deformation behavior of epoxy-based composite materials; effect of strain rate on the failure mechanisms and energy absorption in polymer composites; failure behavior of bolted joint glass/Kenaf fiber-reinforced polypropylene hybrid composites; edge-crack effect on tensile behavior of diversified wood particulate composites; investigation of mechanical testing on hybrid composite materials; cracks, microcracks, and fracture in composite structure: formation, detection, autonomic repair; development of morphing wing skin using reinforced shape-memory polymer; fabric-reinforced cementitious matrix (FRCM) composites: mechanical behavior and application to masonry walls; and failure analysis in hybrid composites using industrial wastes.

We are extremely thankful to all authors from different part of worlds who have contributed chapters in this edited book and support it by providing valuable ideas and knowledge. We appreciate their knowledge from such diverse fields as failure analysis in biocomposites, fiber-reinforced composites, and hybrid composites and

in accepting editorial suggestions to finally produce this venture that we hope will be a success. We are also grateful to the Elsevier, UK, support team, especially Gwen Jones, Thomas Van Der Ploeg, Poulouse Joseph and Sandhya Narayanan helping us to finalize this book.

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Fatigue delamination of carbon fiber-reinforced polymer-matrix composites

1

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1.1 Introduction

The composite materials with precise quality and rigidity have been overwhelmingly significant in their use in structures where weight is vital and flexible. Composites made of laminates demonstrate a perturbing inclination to the frame and development of cracks between the layers. This phenomenon, commonly known as delamination, implies a standout among the peak life-constraining failure modes of laminated composites.

Amid the assembly procedure, the delamination process can be activated either by defects acquired or made by auxiliary components over the span of the service life of the material due to interlaminar stresses that can prompt the start of delamination. Several decades have passed since affirming the significance of the interlaminar disappointment [1,2], regardless it remains a deciding component controlling the auxiliary components made of covered composites [3].

In recent years the carbon fiber-strengthened polymer composites are generally used as a part of aviation, marine, car, and propelled designing applications because their excellent mechanical properties. Then again, these structures experience cyclic exhaustion loadings amid benefit life each time, for example, flying machine wings, helicopter sharp edges, wind turbine edges, etc. [4]. The one fundamental worry all through the plan of these composite structures is the weariness harm appraisal: the quality and durability of the basic composite parts must consider the regular harm emerging under in-benefit stacking. In the interim, it brings plan and examination challenges caused by the employed by-handle detailing of composites, which is very surprising to come from the customary metal structures [5].

Delamination is one of the vital factors for composites from loss introduction to ending failures. The delamination development actions have extended significant attention in the research communities in the past [6–11], although the delamination performance of composites has not been fully understood in difficult situations, for example, with multidirectional interfaces, fatigue loading, and fiber linking case [12].

Direct flexible break mechanics is regularly used for the interlaminar crack of composites. Strain energy release rate (SERR) is acknowledged as the crack representing parameters to assess interlaminar break durability for composites as opposed to the stress intensity factor (SIF) for metals because of the straightforwardness of the estimation. Trial studies and test techniques for delamination resistance have been assessed by Davies et al. [13] and Brunner et al. [14], and individual numerical examinations were explored by Tay [15].

Multidirectional interface and fiber connecting are two critical factors in actual engineering applications, which bring huge impact on the interlaminar crack of composite structures. As a compressed outcome from reports in script [10,16–18], multidirectional laminates dependably show higher interlaminar break strength, which is thought to be caused by outward toughening instruments, for example, blunted split tips or deviation of the break from the primary split plane to the adjoining layers and some in-utilize vitality assimilation [19]. Fiber connecting could, likewise, convey impressive delamination resistance because of the vitality retained in the crossing over the zone behind the split tip. For certain composite material frameworks, fiber crossing over was discovered unavoidable and can be upgraded by multidirectional utilize introductions [20]. A few laws [6,21,22] have been created in light of connecting zone model to assess the connection between the fiber crossing over anxiety and the crack tip opening displacement (CTOD). Considering the fiber-spanning impact, the finite element methods (FEM)-durable component was produced in light of the connecting zone laws [23,24].

For fatigue-delamination engendering composites, a Paris Law undifferentiated from direct log–log connection between the weariness split development rate and the SERR has been set up by some noteworthy crucial works [25–28]. Weakness debasement laws [29–32] in view of firm interface components and the Paris Law are produced to play out a numerical report and anticipate the exhaustion split development by FEM programs. Nonetheless, the Paris Law will end up plainly unacceptable for fiber crossing over cases because the weakness break development rate and limit is essentially influenced by the extra delamination resistance. Momentous R-bend impacts on the exhaustion delamination have been watched and broke down by Hojo et al. [9] for Zanchor-strengthened covers, Argüilles et al. [33] for unidirectional overlays with fiber connecting and Shivakumar et al. [34] for woven/twisted fiber composites. A spanning model was produced uniquely for weakness delamination by Gregory and Spearing [35], finding that the diffuse of split engendering information was fundamentally decreased by applying the model.

An innovative fatigue-delamination resistance parameter was presented by Peng et al. [12,36,37] to quantitatively assess the impact of R-curves on the fatigue-delamination development. The standardized fatigue split development rate and limit demonstrates taking the proportion of SERR to the fatigue-delamination resistance, as the representing crack parameter were consequently created. Exceptional arrangement with tests was acquired by applying the models on test information from carbon/bismaleimide composite laminates.

1.2 Processing of polymer-matrix composite

Amid the previous couple of years, the nanoparticles have been integrated into the polymer network to build up a polymer nanocomposite. Despite handling procedures of the nanocomposite being intrinsically extraordinary, each one of them need to discourse the additional issues that influence the properties of the nanocomposite: scattering, arrangement, and functions. The normal handling strategies of polymer nanocomposites comprise are discussed in the next sections.

1.2.1 Solution processing

The nanoparticles are primarily scattered in a dissolvable or polymer arrangement. Fig. 1.1 demonstrates the schematic of arrangement handling.

The nanoparticle/polymer/solvent solution is blended by energetic fomentation, for example, attractive mixing, high shear blending, reflux, or sonication. For sonication, there are two sorts of hardware: gentle sonication in a liquid shower and high-control sonication utilizing a horn or tip. The nanoparticles in general cannot be scattered well in the greater part of solvents because of their exclusively high viewpoint proportions. The nature of the scattering can be enhanced by treating the nanoparticles by a few surfactants [38]. Once the nanoparticles are evenly scattered, the polymer arrangement

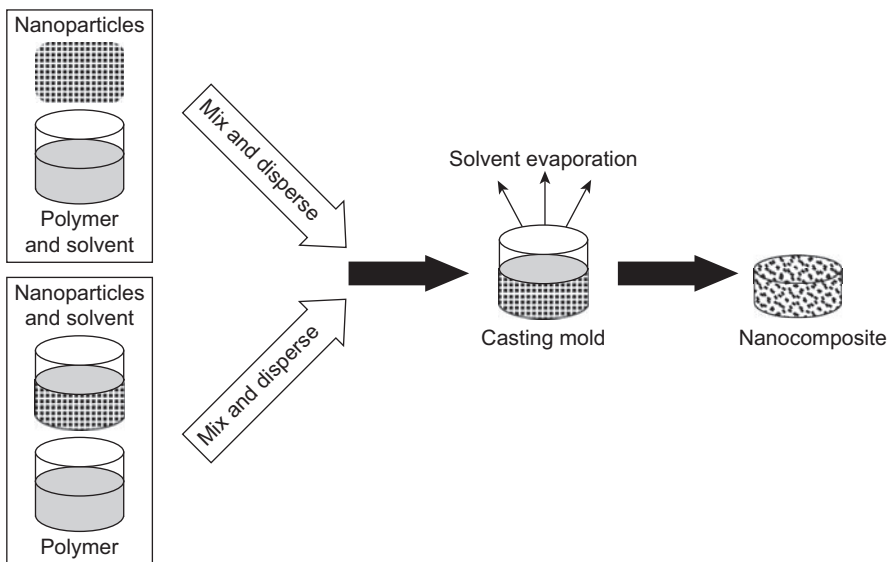


Figure 1.1 Schematic of solution processing.

From Gou J, Zhuge J, Liang F, Processing of polymer nanocomposites. In: Suresh GA, Kuang TS, editors. Manufacturing techniques for polymer matrix composites (PMCs). Woodhead Publishing; 2012. pp. 97–102.

is filled a shape. The dissolvable is at that time disappeared, leaving the composite film or sheet upon the consummation of dissipation. Arrangement handling is usually used to set up the nanocomposites by a sheet or film like arrangement [39]. The determination of solvents depends on a dissolvability of polymers.

Table 1.1 demonstrates the properties of various kinds of solvents. Jin et al. directed the examinations to scatter high convergence of multiwalled carbon nanotubes (MWCNTs) into the polymer through arrangement handling [40]. In their investigation, the MWCNTs produced by a bend release were scattered in chloroform by sonication. The polymer network, poly hydroxyl amino ether (PHAE) was broken up into the MWCNT–chloroform arrangement amid the sonication procedure. The greatly scattered suspension was filled in the Teflon form and permitted to dry in a smoke cover. The arrangement handling empowered them to set up the nanocomposite containing 50 wt% of MWCNTs with great quality. A new examination gathering thought about the nature of mechanical properties and scattering of the nanocomposites arranged by various sorts of preparing techniques [41]. Among the immediate blending, the extended graphite (EG) was scattered in the hardener of epoxy pitch (DGEBA) by means of an attractive stirrer. The epoxy resin was then incorporated into the EG/hardener arrangement and was blended on a hot plate. Accelerator agent was added to the mixture and blended with moderate unsettling taken after via degassing. The nanocomposite was prepared as a result of throwing the last arrangement into a Teflon form. Amid the arrangement handling, the EG was initially scattered into $(\text{CH}_3)_2\text{CO}$ via sonication and the DGEBA epoxy resin was brought into the suspension and sonicated. The arrangement was mixed on a hot plate until the point that $(\text{CH}_3)_2\text{CO}$ was fully dissipated. The hardener and quickening agent were then included, trailed via

Table 1.1 Properties of typical solvents

Solvent	Boiling point (°C)	Dielectric constant	Density (g/ml)
Non-polar solvents			
Pentane	36	1.84	0.626
Toluene	111	2.38	0.867
Benzene	80	2.3	0.879
Polar aprotic solvents			
Acetone	56	21	0.786
Dimethylformamide	153	38	0.944
Dichloromethane	40	9.1	1.3266
Polar protic solvents			
Water	100	80	1
Methanol	65	33	0.791
Ethanol	79	24.55	0.789

degassing, throwing as well as curing process. The nanocomposite samples arranged through these two unique procedures were analyzed via optical microscopy. The test showed that the nature of the nanocomposite arranged by the arrangement handling was vastly improved compared to the nanocomposite arranged by the immediate blending.

1.2.2 *In situ* polymerization process

Fig. 1.2 demonstrates the schematic of in situ polymerization process. Amid the in situ polymerization method, nanoparticles, e.g., the carbon nanotubes (CNTs) and layered silicates are primarily scattered in a fluid monomer. The polymerization response is started either by radiation or heat, through the dissemination of a reasonable initiator, through a natural initiator, or via impetus settled on the surface of nanoparticles [38]. Upon the completion of polymerization, the polymer particles are either wrapped covalently or around and connected to the nanoparticles depending up on the polymer being framed and the surface convenience of nanoparticles [39]. To prepare the nanocomposites comprising thermally unstable and insoluble polymer, the in situ polymerization can be used, which cannot be treated by melting or solution procedure. Depending on the prerequisite molecular-weight and molecular-weight dispersion of the polymers, the radical, ring opening, anionic, chain transfer metathesis polymerizations are used. Empowering the grafting of polymer macromolecules on to the surface of nanoparticles is a major benefit of the in situ polymerization procedure, which allows the preparation of the nanocomposite with a high-stacking level of nanoparticles and decent miscibility through the polymer [42]. The Toyota Motor Corp. in the mid-1990s was the first to develop the commercial application of nanocomposites (clay/polyamide-6) [43]. Zeng and Lee settled poly (methyl methacrylate) and polystyrene/clay nanocomposites by in situ polymerization [44]. They found that the compatibility of the initiator and monomer with the surface of the clay was a critical factor to affect the dispersion of clay. They discovered similarity of monomer and initiator through the surface of clay which is the critical factor to affect the dispersion of clay. They stated that to achieve great value of dispersion a mixture of more polar,

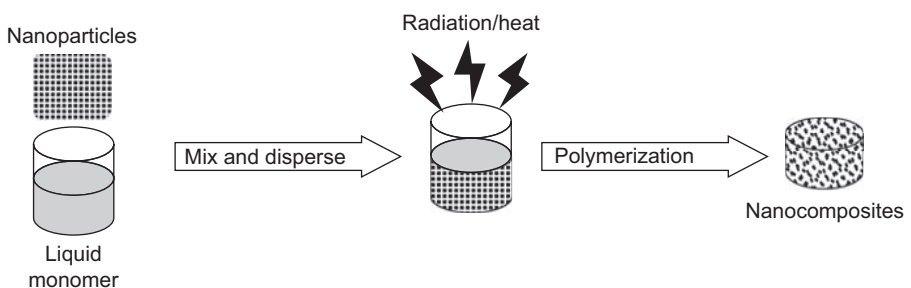


Figure 1.2 Schematic of in situ polymerization process.

From Gou J, Zhuge J, Liang F, Processing of polymer nanocomposites. In: Suresh GA, Kuang TS, editors. Manufacturing techniques for polymer matrix composites (PMCs). Woodhead Publishing; 2012. pp. 97–102.

less hydrophobic monomer and initiator was favored. Furthermore, the dispersion value was additionally enhanced by presenting polymerizable groups on the surface of the clay. The peeled PMMA and PS/clay nanocomposites were effectively produced with a 5 wt% concentration of clay. Kim et al. used this procedure to formulate MWCNT/high density polyethylene (HDPE) nanocomposites [45]. They primarily appended a metallocene catalyst composite on the edge of the nanotubes and on the edges by the surface prompted polymerization the polymer divisions were then produced and the nanotubes were evenly distributed and covered by PE molecules by via scanning electron microscopy (SEM) and transmission electron microscopy (TEM) investigation. The dispersal of silica into polyamide 6 via in situ polymerization is studied by Yang et al. [46]. They reformed the silica nanoparticles with amino butyric acid by spread in ϵ -caproamide using stirring. In a nitrogen atmospheric conditions, the mixture was polymerized at a high temperature and the result indicates that the reformed silica was spread more similarly, than the original silica. By preparing an inorganic/organic nanocomposite, which can evade the agglomeration of inorganic particles and recuperate the interfacial interfaces between the polymer and inorganic components, this procedure remained beneficial.

1.2.3 Melting process

A physical procedure that costs the phase transition of a substance from a solid to a liquid is known as melting process. It is an alternate procedure to pact with the polymer matrix, which is insoluble. It is especially valuable in setting up the nanocomposites together by thermoplastic polymers, such as polyamide-6, polycarbonate, HDPE, polystyrene, polypropylene, and so on. This procedure enables a big volume of nanoparticles to be blended into polymer network. After being heated up, it is found that thermoplastic polymers turn into soft and once cooled down the properties of polymers withstand the unchanged. As such, the semicrystalline polymers and amorphous polymers can be treated beyond their melting temperature and glass transition temperature, respectively. The melting process diagram is shown in Fig. 1.3, and polymer pellets are

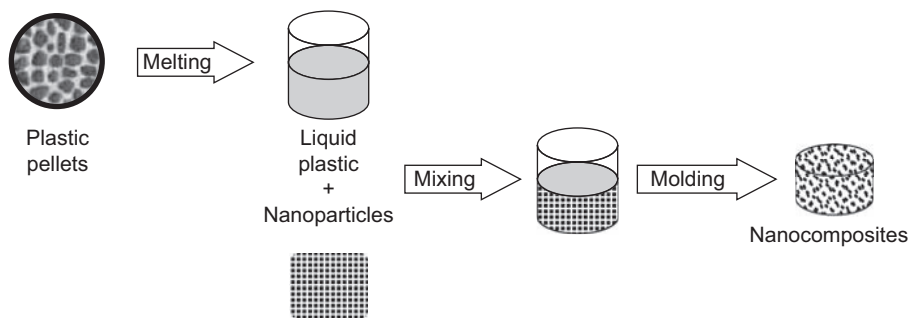


Figure 1.3 Schematic of melting processing.

From reference Gou J, Zhuge J, Liang F, Processing of polymer nanocomposites. In: Suresh GA, Kuang TS, editors. Manufacturing techniques for polymer matrix composites (PMCs). Woodhead Publishing; 2012. pp. 97–102.

melted to form a viscid fluid during the procedure. Through high shear blender or an extruder, the nanoparticles are then mixed in to fluid-polymer and the ultimate bulk nanocomposite samples can be acquired via compression molding, extrusion or injection molding. The introduction of nanoparticles into the melting polymer will impact its viscosity, which perhaps run to the unanticipated polymer breakdown beneath high shear circumstance. Hence, operational situations would stay optimized for the entire series of polymer/nanoparticle mixtures [38]. Owing to its easiness and rapidity, this procedure has been broadly useful for full scale-industry manufacturing.

1.2.4 Carbon nanotube (CNT) paper-based nanocomposites

The CNT paper has been manufactured by numerous techniques, and it can be either a one-step or two-step process. In the two-step process the CNT are first synthesized and then paper is made by nanotubes. The CNT paper is grown-up directly in the one-step process. The main process to make the CNT paper is the two-step process which includes spin-coating, vacuum/pressure purification, and the domino-pushing technique.

1.2.4.1 Vacuum/pressure filtration

The suspension of CNT is filtrated through the filter-paper to make a detached nonwoven mat, known as the filtration process [47]. The filtration process is separated in to four steps: (1) the purification and preparation of CNTs; (2) the uniform dispersal of CNT into solvent to form a steady suspension; (3) the purification of suspension of CNT beneath pressure or vacuum; and (4) parching the nanotube paper in a vacuum oven. The advantages of filtration techniques include its simple-manufacturing process and also its low cost. The easy accumulation of nanotubes and the high-level of curviness may result in the brittleness of nanotube paper.

1.2.4.2 Spin-coating method

Through spin-coating from an exceptionally concentrated nanotube dispersal, an even CNT paper is formed, which is steadied by chemical oxidation. The solution of filtered CNTs is sprayed in this procedure into a high-speed rotating glass panel. By varying the concentration of the nanotubes and the speed, rotating the thickness, the nanotube paper can be managed. By evening the surface quality and diminished thickness the high grade nanotube paper can be acquired, which leads to promising applications in the area of solar batteries. The aligned-MWCNTs are fabricated and an aligned CNT-centered membrane electrode is used as the anode material in a rechargeable lithium ion battery. The process of the spin-coating technique is shown in Fig. 1.4.

1.2.4.3 Domino pushing method

The domino pushing is an easy but efficient macroscopic operation of CNT ranges which has been established to build denser aligned-CNT papers [48].

Fig. 1.5 shows the schematic of the domino-pushing process. At first, with a portion of microporous membrane the nanotube range is concealed and the nanotubes in the

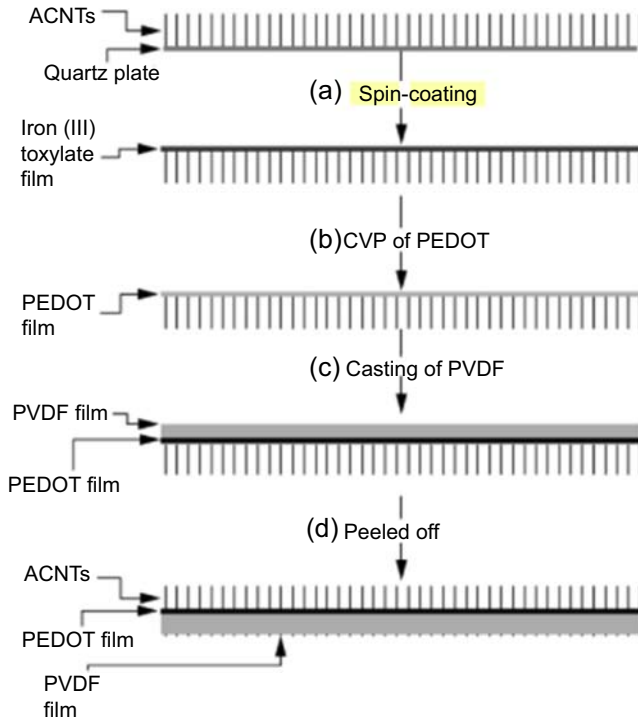


Figure 1.4 Schematic of spin-coating process.

From Ago H, Petritsch K, Shaffer MSP. Composites of carbon nanotubes and conjugated polymers for photovoltaic devices. *Adv Mater* 1999;11:1281–85.

range are then enforced down to one-direction via a pushing cylinder, which is located on nanotube range with a continual pressure. Owing to robust van der Waals forces and form an aligned-CNT paper, all the nanotubes in the range are enticed together. Through the membrane, the aligned-CNT paper is then peeled off of the silicon substrate. The ethanol is extended on the microporous membrane and pervades over the membrane, and after this, the aligned-CNT paper can be peeled off from the membrane. The curviness and accumulation of the nanotubes can be evaded by this process; but any contaminations remain through the nanotubes, and the domino-pushing method is more difficult compared to the filtration and spin-coating processes.

1.2.4.4 Direct synthesis

In this process, the synthesis of CNT paper in situ via trichloro benzene (TCB) by means of a precursor. There are some reports that CNT paper is developed by the chemical vapor deposition (CVD) technique by a one-step process. Without use of any surfactant or acid treatment the ferro-magnetic CNT paper filled with Fe nanowires (Fe-CNT) was synthesized in situ [49]. The two-step process is more complicated than the one-step process because the organic solvent can be evaded in the one-step method.

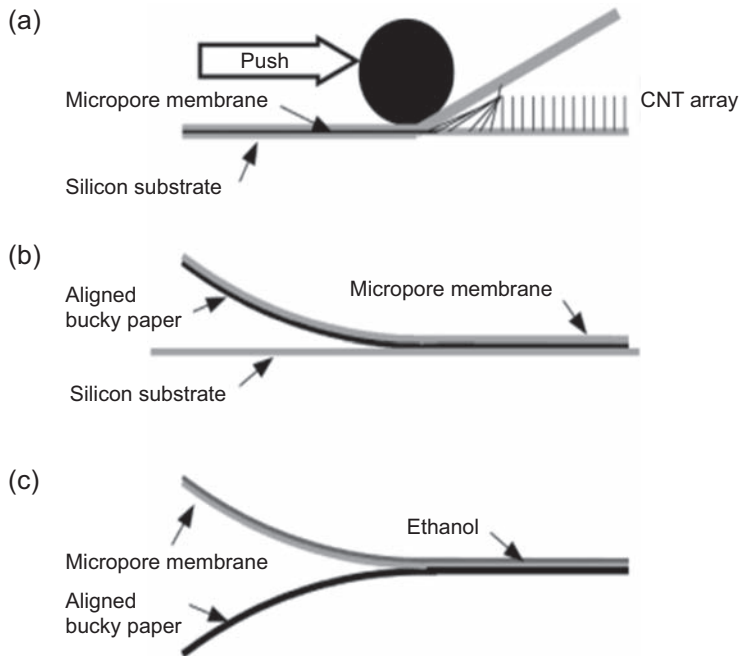


Figure 1.5 Schematic of domino-pushing method (a) Forming aligned buckypaper (b) Peel the buckypaper off from the silicon substrate (c) Peel the buckypaper off from the microporous membrane.

From Li YH, Zhao YM, Roe M. In-plane large single walled carbon nanotube films: in situ synthesis and field emission properties. *Small* 2006;2:1026–30; Wang D, Song P, Liu C, Wu W, Fan S. Highly oriented carbon nanotube papers made of aligned carbon nanotubes. *Nanotechnology* 2008; 19(7), p. 075609.

Although, the good mechanical properties are acquired. In the one-step process the high amount of impurity content bounds the use of CNT paper.

1.2.5 Graphene paper-based nanocomposites

Graphene is a form of carbon particles comprising of a single layer arranged into a two-dimensional honey-comb lattice. Many research papers about graphene have been published in various journals since its discovery. Graphene is acknowledged as very tough and stronger than diamond. Graphene efficiently conducts electrons exceptionally well.

1.2.5.1 Vacuum filtration

The peeling of graphite is the primary step for the fabrication of graphene paper, and the micromechanical cleavage and chemical peeling of the graphene-oxide (GO) are the key technique to peeling the graphite. The mechanical peeling of graphite to get

the graphene was first worked by Andre Geim et al. [50]. To make the graphite-crystals in to a lesser layer graphite the cohesive tape was used over and over. In acetone, the fastened, optically transparent lamina tape was dissolved, and after some steps the lamina included the mono-layer sediment on a silicon lamina. Single atomic-planes were detected in an optical microscope. The solution-centered technique connecting the chemical-oxidation of graphite to hydrophilic graphite-oxide, which can be freely peeled as single GO sheets by ultra-sonication in water, is established by Ruoff et al. [51]. From the GO colloids the graphene paper is manufactured in the next stage and with the help of vacuum the GO colloids can be directly filtrated. Across the vacuum purification by an ANODISC membrane, Dr. Zal et al. fabricated the graphene paper [52]. To reduce the disarray layering arrangement triggered by water-flow, a precise multipurification course (5 mL at a time) was used. Prior to detaching the graphene paper off the membrane, the graphene paper was pressure dried and also dried in a vacuum oven aimed at overnight. By using a furnace, the unattached paper was thermally strengthened and then cold-pressed at room temperature. The strengthening procedure advances the thermal conductivity by disintegrating the PEI molecule that is adsorbed on the graphene particles although still preserving the penetrability of the graphene paper. The mechanically compacting sample efficiently increases the interaction range among separate sheets and decreases the pore-volume of the graphene paper. Owing to the solid influence of arrangement and larger interaction range of the strengthened graphene paper, the in-plane thermal conductivity is escalating at a result of 80% and the through-plane conductivity is reduced by 10%.

1.2.5.2 Electrophoretic deposition

To manufacture the graphene paper, an electrophoretic deposition (EPD) method has been established [53]. The film is comprised of overlaid and heaped platelets of lower GO (G-O).

The experimental set up of EPD-GO film with GO scattered in liquid and sonicated at room temperature is shown in Fig. 1.6. Next the arrangement of colloidal suspensions of separate GO platelets in distilled water, the EPD-GO was placed on stainless-steel and several additional electrically conductive substrates. The applied DC (direct current) voltage and standard concentration of GO were 10V and 1.5 mg/mL respectively; and the standard concentrations of GO and applied direct current (DC) voltage were 1.5 mg/mL and 10 V, respectively; the installation time remained in the scale of 1–10 min. When a DC voltage was connected the GO platelets traveled to the positive electrode. The concentration of the GO suspension, the applied DC voltage, and the conductivity of the substrate are the numerous aspects where the installation rate depended. A flat film was placed on the stainless-steel positive electrode in a brief period of time with 10 V of applied voltage. Next to the dispositions, the samples were dried at room temperature. By changing the time and current, the films, with a thickness range from a few hundred nanometers to tens of micrometers, could be placed.

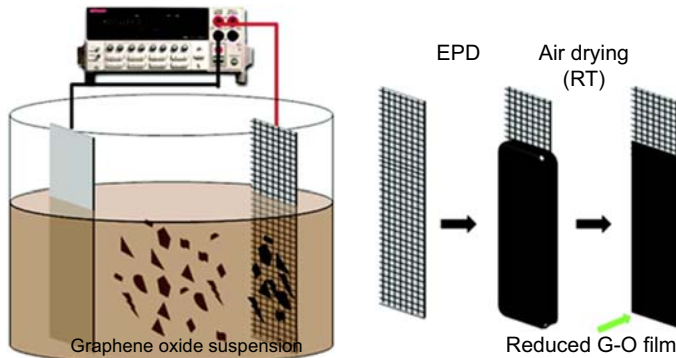


Figure 1.6 Schematic of electrophoretic deposition process.

From Sung JA, Zhu YW, Lee SH, Stoller MD, Emilsson T, Park SJ, et al. Thin film fabrication and simultaneous anodic reduction of deposited graphene oxide platelets by electrophoretic deposition. *J Phys Chem Lett* 2010;1(8):1259–63.

1.3 Load control delamination fatigue

The fraction of composite structures where the weight is critical are growing in entirely all applications. A main exertion of the composite industry in particular small delamination resistance has concentrated on removing the intrinsic infirmity of composite resources. Solutions to advance interlaminar stiffness comprise resin interleaving, strengthening of the resin, and further, the width strengthening comprising 3D interlacing, Z-fiber pinning, sewing, and clumping.

The lone procedure appropriate for the strengthening of structures fabricated by means of the prepregged/autoclave direction is the Z-fiber pinioning (afterward denoted as Z-pinning). A study about the influence of Z-pins on the composite laminates was done by Mouritz [54]. To escalate the delamination resistance, the efficiency of Z-pins was studied by researchers [55–62], as was the effect of loss toleration [63,64], and joint strength [65–67] has been established openly by means of both modeling and experimental methods. Though not all Z-pinning has a positive impact, through the decrease of the in-plane elastic properties such as strength, modulus, and fatigue execution, the progress of interlaminar properties arises [68–72]. For instance, because of an approximate 30% drop of compressive strength reported by Steeves and Fleck [72] due to this reason, the role of Z-pins is restricted to reinforcement of structures possible to fail through delamination. To date the practical application of Z-pinning is restricted to military airspace [73] and Formula 1 racing [74]. Mouritz reviewed the number of concerns (like low-cost production, physical performance, and durability) to be addressed before using the Z-pin to its full capacity in civil airspace uses [54]. For the growth of a mechanical model for delamination growth prediction the comprehension of procedure of delamination growth is essential and once examining the procedure, the involvement of individualized procedure is able to

adamant and mechanical model can be technologically advanced for the delamination growth. There are two types of load control delamination fatigues i.e., Mode I and Mode II fatigue loading are described below.

1.3.1 Mode I

The composites of high performing fiber-reinforced polymer (FRP) are operated normally in key aerospace engineering to decrease its weight and increase its stiffness and strength. Due to the impact of low velocity, the delamination of composites structure triggered and it is one of the main difficulties enticing mostly concerned from the safety perspective. The damages caused due to growth of such delamination results in decreases of strength, toughness, and fatigue-life [8]. There are various studies concentrated mostly on the appraisal of fatigue-delamination growth of composites laminate and interlaminar fracture stiffness. Davies et al. and Tay reviewed the importance of the experimental study on the interlaminar fractural benefits of FRP composites [13,15].

Through double-cantilever beam tests the Mode I interlaminar stiffness is generally measured and also has been studied broadly. To regulate the crucial strain energy release rate (SERR) in the Mode-I a typical technique has been recognized which has been centered on the idea of linear-elastic fractured mechanics [75,76], though it is restricted to unidirectional laminate and quasi-static loadings. The minute SERR is under vital importance, the delamination characteristic in fatigue loadings have been studied extensively for unidirectional laminates. Some major work has been accomplished by some researchers [9,77,78]. The elaborated mode-I, mode-II, and mixture mode delamination growth of carbon/epoxy composite in fatigue-loadings through an improved Paris-Law is studied by Asp et al. [26]. The onset significance of SERR, G_{Ith} , under which no fatigue crack growth arises was brought into being for the assessment of delaminated characteristics below cyclic-loading [79]. There is an absence of research regarding multidirection lay-up laminate, in comparison with the success for the unidirectional composite, though lay-ups by means of dissimilar fibers orientation of the laminates are chosen in maximum uses [14]. By using double-cantilever beam, the delamination resistance beneath static loadings have been tried by various researchers [20,80,81]. The results reveal that there is no significant reliance of delamination stiffness on the fractures interface (fiber-orientations) aimed at crack instigation [81]. Though compared to the equivalent unidirectional laminate, the growing crack-length, ample sharper resistance-curves (R-curves) for cross-ply composites were discovered [14]. Unfortunately, the science behind fatigue-crack growth of multidirectional layup laminates is not clearly understood due to lack of research. Owing to lack of research on delamination properties beneath cycling load, the use of composite laminates leftover lower capability.

1.3.2 Mode II

Exploration taking place on delamination resistances tests beneath fatigue mode II (in-plane shear) stacking of FRP matrix composites initiated during the late 1980s until

today and many research has been done to date [82–95]. Largely this research deals with carbon-fiber reinforced polymer (CFRP), precisely various kinds of epoxy-resins [82,84,86–90,93,94], dualistic by thermoplastic polyether ether ketone (PEEK) [3,4] and a small number of glass fiber-reinforced polymer (GFRP), once again using epoxy-resin by means of matrixes [83,92,94]. The most commonly used test set up is a three-point bending end notch flexures (ENF) test [82–86,90–93]. The other test set ups, like four-point bending end-notched flexure (4-ENF) [94], end load split (ELS) [88], related end notch cantilever beams (ENCB) [87], or the central-cut ply's (CCP) sampling [95]. Besides the exemption, that examines the development and proliferation of mode-II fatigue delaminations via acoustic-emission destruction observing [83] and fractographic on impact-damage laminates afterward cyclical compression to compression stacking [89], rest all assess the de-lamination rate vs. applied load (GII_{max} or ΔGII) and presents the equivalent Paris-plots. In addition, to experimental examinations, several researcher groups also have aimed to build up the models meant for explaining mode-II fatigue delamination propagations and compared with previous data [91,95].

In this investigation delivers a base aimed to know the complexities and difficulties of mode II fatigue delamination crack propagations that will be used for the improvement of a standardized test technique. Some researchers recently reviewed the delamination resistance assessment technique improvement [14], which has generated one-standard on quasi-static mode II testing [16], two draft standards for quasi-static mode II [96,97], and preparations aimed at a combined transnational round-robin on mode II fatigues which, nevertheless, stayed by no means completed [14]. To draft a systematized test technique needs founding a process, important scopes and goals and data analysis which is relevant in an industrialized test environs. If essential, the drafting process is then reconnoitered in round-robin test by an array of FRP matrices laminated as well as improved or advanced. Goals for round-robin tests are defining interresearch laboratory replication, describing appliances and limits, and dispersals, as well as authenticating the data-exploration. From the time when the published mode II fatigue tests data [82–95] were found commencing several kinds of laminate, and beneath numerous test circumstances, they do not deliver info on interresearch laboratories replication and scatterings. The current involvement testifies to an initial attempt at emerging a uniform process for mode II fatigue delamination resistances. This one mostly focuses on the difficulties that come across hitherto and in closing summaries additional stages that remain judged vital intended for attaining a test process that can be presented in place of standardizations.

1.4 Classification of the methods for prediction delamination growth

Over the span of recent years, the journey to convey ever lighter structures has provoked an extremely extended use of both composite materials and adhesive bonding's. To ensure the prosperity of the resulting structures, it is essential to understand their

fatigue behavior. In this way, the growth of the utilization of composite as well as adhesive bonding's goes together by means of progress of fatigue-models. The De-grieck and Van-Paepegem [78] classified these models in to the 3 main groups: phenomenological-model, progressive-damage model also fatigue-life model. Among these three models, the fatigue-life model is not associated with the concrete degradation mechanism, but it uses an SN (Stress vs. Number of Cycles to Failure) curve or similar process to predict the entire fatigue-life. The phenomenological-model describes the slow decline of macroscopic visible property, mostly (remaining) power or toughness. The progressive-damage model predicts the development of real damages structure, for example matrix-cracks or delaminations. To accomplish a harmless living valuation of an arrangement, a fatigue-life and phenomenological-research model can be utilized. The new cause of fault investigation or related impairment continued through use can't be utilized to assess the outcome. Moreover, involvement around the metal structures demonstrates a "slow-growth" theory that permits a plan of simpler arrangements. A certain outgrowth of loss owing to fatigue-load is approved in a slow-growth point of view. An average examination design is used to ensure advantageous repair and in this way to safeguard the prosperity of the structure. Advanced capacity stages are tolerable inside the arrangement, and a simpler weightiness plan can be accomplished by allowing damage growth. By allowing damage advancement, higher loading levels are acceptable inside the structure and a lighter weight arrangement can be consummate. By means of a significance, a slow-growth viewpoint needs a prototypical commencing of the group of advanced damage models. A summary of the existing advanced damage models aimed at a single precise type of destruction delamination is the purpose of this analysis. The delamination model is the best among most broadly observed models in composite materials, and it is usual to both adhesive bonding as well as composite materials. In adhesives bonds the delamination is repeatedly mentioned to as dis-bonding. Though, it is sure that dis-bonding is a type of delamination, if any reflects a bonded-joints to be a layered-structures. So remain the prediction-method advanced to dole out by them, certainly, the procedures drawn in together dis-bonding as well as delamination remain very much alike. A difficulty confronted by the research is that the delaminations can raise together owing to quasi-static loads as well as owing to cyclic-loads while investigating information upon delamination development. The development owing cyclic-loads usually happens on maximum-load level which remain lesser to vital toward reason of development below quasi-static loads. Despite the fact that there are some fractography evidences where the frameworks included are equivalent for weak lattice materials (e.g., epoxy-resins) [77,98,99], models for static delamination advancement are all things considered not fit for envisioning improvement due to fatigue loading. The other way around is the fatigue-growth models remain usually nix qualified of predicting-static delamination development. It must remain noticed that on behalf of strong matrixes material (for example thermoplastic-resins like polyether ether ketone) there is proof that the instruments participated in static-growth not remain similar by means of those affecting fatigue-delamination development [3]. Sadly, numerous models in the literature remain merely branded by means of delamination

developmental model, deprived of recognizing if those remain valid to static-growth, fatigue-growth or else for together.

For the predictions of delamination development there are several models and techniques, and these are generally classified into four types:

1. Stress/strain-based techniques. The stress/strain techniques are related the delamination development to the stress or strain in the materials.
2. Fracture mechanics—based techniques. These techniques associate the delamination development to fracture-mechanic assets: for instance, stress-intensity-factor (SIF) or strain energy release rate (SERR). Generally, these techniques are construct particularly with respect to the sub-space of linear elastic fracture mechanics (LEFM).
3. Cohesive zone models (CZMs). The CZMs are FEM-centered models around which the crossing point connecting the two layers have been demonstrated by cohesive zone elements. Instead of incorporating a traction-displacement relation, these components do not develop continuous toughness. A damage-parameter is utilized to reduce progressively the toughness of the component and simulating the development of damage.
4. Extended finite element method (XFEM) model. The extended finite-element method model is a method that agree to incoherence exists surrounded by a finite-elements, somewhat merely on the peripheries, as a result of spending alleged improvement functioning.

The stress/strain-based techniques developed by Volkersen in the late 1930s is one of the first and oldest model [100]. During the 1970s the fracture-mechanics techniques have been recommended for delamination problems [101,102] resulting in their effective opening to dole out through fatigue-crack development in metals a decade prior. CZMs are new and the first one have been utilized for delamination difficulties in the late 1980s [103]. The XFEMs are have been developed in recent years.

1.4.1 Stress/strain-based methods

The stress/strain based methods have for a long while been used to choose the nature of adhesive bonds. Gleich [104] has given a wide-range review of the most vital techniques created throughout the 20th century. According to these investigations the fundamental issue studies are stress and strain spreading inside an adhesive bond as well as the meaning of appropriate quasi-static-failure standards. The most important applications of stress/strain techniques are usually static delamination difficulties, although in the literature there are some uses of stress/strain techniques in the fatigue-investigations. Normally, stress/strain techniques remain merely utilized to discover the fatigue life and no means of predicting delamination development. It is similar to the action of fatigue-crack development around the metals, whereas the stress-amplitudes can remain utilized to find out the fatigue-life of the sample. Fatigue-crack development is anticipated by the SIF [105]. The researchers Renton and Vinson used the stress/strain technique [106] but then measured what the highest stress aimed at affixed fatigue-life will achieve. The other researchers Liniecki et al. [107], Imanaka and Iwata [108,109] and Ishii et al. [110] use-up the stress or stress-amplitude toward predicts the fatigue-life of the sample. These type of models doesn't deliver at all knowledge upon the delamination development value in addition, to can't remain utilized to predicts the influence of faults although this information can be

useful. Furthermore, they can't remain utilized to assist a slow-growth plan viewpoint, used for realizing the delamination development is vital. Merely dualistic models can remain set up in the literature where the stress remains utilized to predicts delamination development. The one is researcher Ratwani and Kan [111] and the another is Poursartip and Chinatambi [112]. Ratwani and Kan show that delamination development may perhaps be defined via the equation

$$da/dN = C(\tau_{\max} - \tau_{\min} - \tau_{th})^n am \quad (1.1)$$

where da/dN is the delamination development rate, τ is the shear stress, and τ_{th} is the threshold-shear stress, underneath which delamination does not happen. This condition was effectively used to anticipate S–N bends for various examples. In spite of the fact that condition 1 is composed as far as stress, take note of that $K = \sigma/\pi a$. Accordingly, condition 1 is equal to using the anxiety power factor to relate with delamination development rate when $m = 0.5n$. Ratwani and Kan states that “Expecting the delamination in the composite to carry on like a split, the power m in Eq. (1.1) might be taken as $0.5n$ [110]”. Poursartip and Chinatambi [113] examined the development of delaminations after gaps in carbon-fiber-epoxy laminates and suggested the accompanying, truly stress centered equation for the delamination development rate:

$$da/dN = C(1 + R/1 - R)^m (\Delta\sigma)^n \quad (1.2)$$

whereas R is the stress-ratio, $\Delta\sigma$ is the stress-amplitude, and C , m , and n are fitting-parameters that are presumed to remain material acreages. On the other hand, this equation can be written as:

$$da/dN = 2nC(1 + R/1 - R)^m - n\sigma_n \text{ mean} \quad (1.3)$$

this equation displays the drawing-up of Poursartip and Chinatambi, which comprises together the stress-range and mean-stress as data on the stress cycle. In fact, the purpose behind picking the type of Eq. (1.2) remained to comprise the mean-stress in the delamination development connection.

The Poursartip and Chinatambi similarly, linked the delamination development rate through the SERR to make use of connection [114]:

$$G = \frac{P^2}{2B} \frac{dC}{da} \quad (1.4)$$

where G is SERR, P is load, B is specimen-width, and C is the acquiescence. This allows Eq. (1.2) to be re-written in terms of ΔG , yielding:

$$\frac{da}{dN} = C (1 + R/1 - R)^m (\Delta G)^n \quad (1.5)$$

Note that despite the fact that this equation appears like Eq. (1.2), the estimations of C , m , and n in Eq. (1.5) will be changed. The Poursartip and Chinatambi analysis did not see much difference in virtuousness of fitting amid with $\Delta\sigma$ or ΔG . As they take note of, this is on the grounds that in the sample they utilized, $dC = da$ is consistent and free of the delamination length. All things considered utilization of the SERR or the stress is for sure identical, therefore, this condition doesn't grip for all geometries.

To genuinely assess whether this remains extra suitable to use SERR or stress to predict delamination development, one needs to lead the correlation on a sample where the consistence change is a capacity of delamination length. No such similar investigations could be found in the literature. Yet, in view of the involvement with fatigue-crack development in metals [105], one expects the stress without anything else is not an adequate closeness parameter to portray delamination development.

1.4.2 Fracture-mechanics based methods

The fracture mechanics based methods interface delamination development to the crack mechanics ideas of stress intensity factor (SIF) and strain energy release rate (SERR). Note that these two parameters are identical and that utilizing one as opposed to alternate does not give distinctive data. The problems met in computation of the SIF in homogeneous-layered material, for instance fiber reinforced polymer (FRP), have turned into the SERR the favorite stricture used for demonstrating of delamination development. The SERR can be computed analytical way or else finite-element-analysis (FEA) be utilized contingent on the sample alignment. The widely recognized finite-element technique to discover SERR remains the virtual-crack-closure-technique (VCCT) [115,116]. Throughout experimentations the SERR can be moderately attained via assessing the variation of observance by means of crack length (dC/da). This may be utilized to calculate the SERR by Eq. (1.4). The other reason it is preferred as the fracture-mechanic parameters because of the relative ease of calculating the SERR throughout experimentation. The use of fracture-mechanic for delamination difficulties originates after the accomplishment of Paris as well as coworkers to apply the fracture-mechanic in order to learn the fatigue-crack development in metals [117–119]. The Jones et al. recently provides a brief summary of these primary growths [112]. Essentially, most fracture-mechanic techniques used for delamination development remain built on the Paris-relation, printed by means of the SERR as well as changed to a larger or smaller amount. The simple outline of this equation exists [117–119]:

$$da dN = C \Delta K n \quad (1.6)$$

where C and n are fitting-parameters.

The SERR as well as SIF are the same as observed in earlier discussion in this chapter and also revealed by Irwin [120] and Sih et al. [121]. In this way the delamination development prediction in Eq. (1.6) is generally rewritten in-terms of SERR. So far there looks to be no consensus on whether to use ΔG (in similarity toward ΔK in

the primary Eq. 1.1) or $G_{\max}$, as the base of equation. Hence, the overall shape of the Paris-equation lies behind the fracture mechanic—based delamination development model:

$$da \, dN = Cf(G)n \quad (1.7)$$

Eq. (1.7) defines log linear action, and therefore only takes a portion of delamination development action.

1.4.3 Cohesive zone models (CZMs)

The CZM tactic is a finite-element technique. Using the VCCT in a predictive-model, remeshing is vital as the cracks advance. With the CZM tactic it is prevented by displaying the edges beside which delaminations remain predictable to grow-up by means of cohesive-zone components. The components are not linear-elastic, however, and obey an agreed upon grip-shift connection. Frequently some sort of damage-parameter is used to gradually decrease the toughness, simulating damage development in the components.

CZMs have appreciated a few successes in predictions of static as well as fatigue delamination development. An outline of the progress of the CZMs is given in this division.

1.4.3.1 Early cohesive zone model (CZM) approaches

The starting point for CZMs were the cohesive zone designs advanced by Dugdale [122] and Barenblatt [123]. In view of their ideas, Needleman [103,124] examined various principal decohesion issues using the CZMs. Different functions go along with this a decade later. A brief outline of this research has been written by Hutchinson and Evans [125]. An analysis of the primary developmental CZMs is also provided by Camanho et al. [126]. Moving toward model-fatigue delamination development, an irreparable toughness decrease should remain affixed to the CZMs design [127]. Needleman was one of the first to propose a design [128] followed by Camacho and Ortiz [129]. Various researchers then developed these ideas into models for fatigue development by Foulk et al. [130], de-Andres et al. [131], Nguyen et al. [132], Yang et al. [133], and Roe and Siegmund [127]. Foulk et al. [130] incorporated unloading as well as reloading behavior in their constitutive-model. It results about an irreparable behavior of the traction separation connection and last Andrés et al. [131] adopted a comparative strategy, additionally, proposed a damage-parameter described as:

$$D = \varphi(\delta_{\max}) Gc \quad (1.8)$$

where φ is the potential related with the footing removal connection and $\delta_{\max}$ is the greatest uprooting come to amid the stacking segment of a load-cycle. As a result of a one-time Taylor chain growth, the rate of the damage-parameter is inferred and is utilized for fast forward loading/unloading behavior of traction separation law.

This permits the damage-length to be assessed at a some degree set of cycle-numbers, with no requirement to get the intermediary behavior by means of cycle by cycle investigation. In order to ease the toughness as well as power by means of role of the amount of cycles the Nguyen et al. utilized an exponential-decay aspect [132]. The researcher Yang et al. [133] framed a damage-parameter with the end goal that damage was too accumulated for the period of the unloading portions of the load-cycle. This plan enables both start and development to be displayed with a similar law, however, it requires isolate break propel principles. The Roe and Siegmund [127] planned a damage-parameter which remains connected to the traction separation behavior and manages the unloading as well as reloading behavior.

1.4.3.2 Further developments

Robinson et al. [134] planned a damage parameter that was divided into two portions: the first meant for the static portion of delamination development and the second aimed at the fatigue-portion. The progression of this damage parameter took after the connection suggested by Peerlings et al. [135] in addition to the interface elements as well as cohesive laws depended and taking place based on Alfano and Crisfield [136].

1.4.4 Extended finite element method

Enhancement capacities are added to specific hubs, which enables the split to become discretionary through the component as opposed to simply along the edge. Along these lines the development can be reproduced without the need to predefine a split way or break plane. To allow the cracks to develop randomly over the component instead of alongside the periphery, the enhancement roles instead remain affixed to specific nodules. Therefore, crack development should be replicated with no necessity to predefine a crack way or crack level.

Belytschko and Black [137] were the first to propose the term extended finite element method (XFEM), and based on the idea of dividing of unity were formulated by Melenk and Babuška [138], and Duarte and Oden [139]. The Belytschko and Black presented the capability of their technique by means of mathematically replicating a rotating crack; however, no experimental validation was put forward. Moes et al. [140] advanced the technique defined in reference [137] to manage long or potentially 3D cracks in addition to offering a number of numerical outcomes. Wells and Sluys [141] provide parallel demonstrations, but once again with no evaluation of experimental facts. Likewise, when displaying cohesive cracking it is conceivable to incorporate a cohesive law leading the traction separation behavior of the crack opening into the XFEM design, as initially projected by Moes and Belytschko [142]. The XFEM have been planned above improvement projections from the first decade of 21st century. These growths have been summarized by Huynh and Belytschko [143].

Over the recent years a number of studies have been performed more on practical problems. Iarve et al. [144] demonstrated the association amongst matrix cracking as well as delamination in the composite; Campilho et al. [145] explored cracking in

adhesive-joints; Curiel Sosa and Karapurath [146] explored delamination in FML. All these research journals revealed and provide a link through experimental outcomes of rational and decent connections in the model prediction as well as the experimental outcomes. Though Campilho et al. [145] in his research reveals that validation is the repetitive, as the experimental data has been utilized to produce the model inputs. The other two research journals do not deliver sufficient information on the basis of the model responses to fix if the validation was completed appropriately for this situation.

Ling et al. [147] have shown that CZMs may remain incorporated into an augmented finite-element-method (A-FEM). A-FEM is a technique initially recommended by Hansbo and Hansbo [148,149], though it utilizes an alternate design and is equal to XFEM [13]. To date almost all the XFEM models that have been reported in literature have only considered quasi static delamination growth. Various XFEM tactics are incorporated using a cohesive-law design [141,143,144]. By consolidating these details with an appropriate damage parameters, equally acted in reliable CZM tactics, it must validate fatigue delamination development utilizing the XFEM. These attempts have not been accounted in the literature review. Bhattacharya et al. established the XFEM tactic for fatigue-crack development alongside the edges in some bilayer materials [150]. These tactics depends on utilization of the Paris-connection in the primary shape (viz. in light of SIF, Eq. 1.6). Because of the complexity of calculating the SIF in composite materials, such a method may be less appropriate for delamination growth in a composite. On the other hand, Bacarreza and Aliabadi recommended a technique in which XFEM is used to discover the delamination development path, and VCCT is used to discover the SERR principles [151]. Finally, whole XFEM-centered delamination development techniques recommended to date along with promising prospect techniques centered on the CZM as discussed above, depend upon fracture-mechanic models, linking SIF or SERR by the delamination development rate.

1.5 Conclusion

This chapter has delivered an efficient theoretical context for understanding the fatigue delamination of carbon fiber-reinforced polymer-matrix composite. No effort has been made to provide a broad description of the enormous literature on the subject. An importance has been sited on indulgent of the processing of polymer-matrix composites, load control delamination fatigue, and, classification of methods for prediction delamination growth and incorporating this in a systematic way in the representation of delamination development. The present report covers a wide span of disciplines and is therefore far from comprehensive. The goal has been to review some of the most important issues concerning the fatigue delamination of carbon fiber-reinforced polymer-matrix composite. Particular emphasis has been placed on numerous techniques and models that have been proposed for the expectation of delamination development.

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Further reading

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Investigation of the deformation behavior of epoxy-based composite materials

2

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2.1 Introduction

In aerospace, automotive, or other fields of predilection, epoxy matrix composite materials are expanding in use due to their large specific properties [1–3]. However, in these areas, it is not enough just to use these materials but rather to ensure that their use is beneficial in terms of reliability and durability regardless of the stresses or environmental conditions. These considerations lead us to draw more attention to the phenomenon of deformation of thermosetting composites while leaning toward the understanding of the process of rupture and damage of both the matrix and the composites [2,4].

Generally, in composite materials with a thermosetting matrix, the fracture is produced by breaking the fibers, transverse rupture of the matrix, and longitudinal rupture of the matrix or by the fiber-matrix interface decohesion. The presence of a mechanism mechanically generates the presence of another which follows through propagation until rupture if the applied load increases continuously. These mechanisms of rupture remain the same whether unidirectional composites or laminates. Only to the latter, there is added the phenomenon of delaminating which is characterized by decohesion of the layers, generated by the implementation conditions [2].

The deformation of the composites can be evaluated through the usual static or dynamic stresses (tensile, flexion, torsion, etc.), of which the study of how to reduce or delay the deformation of these composites can also be a tempting research subject, starting from deformation of epoxy, loaded epoxy, and fiber reinforced, loaded epoxy [1,5]. By treating the deformation of composites, it cannot go unnoticed without discussing the criteria of rupture by making sure that the structure is in good health or it reaches an alarming state by exceeding the elastic limit and generating a permanent deformation.

Architecturally, the work of this chapter is structured in such a way as to first cite the advantages and disadvantages of the epoxy matrix for use in composites and then to explain the different mechanisms of damage and rupture of composites based on this type of matrix may be used for unidirectional composites or

laminates. A passage in the literature is needed, focusing on the work carried out in our laboratories concerning the deformation of epoxy matrix composites. Ultimately, this work will be completed by the different criteria of rupture to be considered in composite materials.

2.2 Thermosetting matrix: epoxy

2.2.1 Chemical composition

Epoxy resin belongs to the family of thermosetting polymers, which consists of a three-dimensional covalent chain of repeating molecular units [6]. This reaction is triggered by hardeners and by the temperature. Epoxy resins are composed of an epoxy part and a hardening part. This latter contains termini capable of being added to the epoxy rings of the resin during processing or catalytic functions which remove the copolymerization of the epoxy system (Fig. 2.1) [7,8].

Under the action of temperature, these resins retain a solid behavior, the macromolecular chains being connected together by cross-linking bonds. At higher temperatures, they degrade without melting [8].

2.2.2 Advantages and disadvantages of epoxy

Due to their good mechanical characteristics, epoxy resins generally used without fillers are matrices of high-performance composites, particularly in aeronautics, missile space, etc. For this reason, they represent only 5% of the composite market because of their high price [1]. Epoxy resins possess many advantages over another kind of resins; nevertheless, they have certain drawbacks which hinder their wide use [1]. Table 2.1 summarizes some of their advantages and disadvantages:

2.2.3 Epoxy characteristics

An epoxy resin's major role is to transfer the mechanical stresses to the fibers and to protect the structure of the external environment. The epoxy has certain deformability and a good compatibility with the fibers. These essential epoxy characteristics are summarized in Table 2.2.

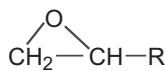


Figure 2.1 Epoxy chains.

Table 2.1 Advantages and disadvantages of epoxy resins

Advantages	Disadvantages
They have good mechanical properties (tensile strength, flexion, compression, resilience, etc.) which are superior to those of large diffusion resins such as polyester	Require a long polymerization time
They have good resistance to high temperatures, coming up to 150–190°C continuously	Susceptible to cracking
They have good physical properties, especially in a moist environment, as well as excellent chemical resistance	Higher price
Very good adhesion on a wide variety of materials	Very strict conditions of use
Low shrinkage (about 1% vs. 6% for polyester)	Aggressive product for skin
	Very harmful sanding dusts

Table 2.2 Mechanical characteristics of epoxy matrix [9]

Epoxy intrinsic characteristics	
Density	1100 à 1500 kg/m ³
Modulus of elasticity in traction	30 to 5 GPa
Breaking stress	60–80 MPa
Bending failure stress	100–150 MPa
Elongation at break	2 à 5%
Shear strength	30–50 MPa
Deflection temperature under loads	290°C

2.3 Mechanisms of rupture and damage of composites based on epoxy

Usually, there is a mechanism of rupture that explains the process that leads to a local discontinuity of matter. This discontinuity may be presented as a crack or cavity internally located in the material and leading to an initiation and propagation of the rupture [10,11]. The initiation of the rupture generates a microcracking, which is essentially due to a defect [12,13]. As to the propagation of the rupture, it results from the creation of new fracture surfaces (on a macroscopic scale), starting from the microcracks previously existing in the material [14].

The rupture in a composite based on epoxy or generally on the thermosetting matrix depends additionally on the reinforcement architecture: unidirectional or bidirectional

(canvas, satin, taffeta, etc.); this work will further dig into the rupture of unidirectional composites [15].

At the level of a unidirectional composite, fracture [16] occurs because of

- Fracture of fibers,
- Transverse fracture of the matrix,
- Longitudinal fracture of the matrix,
- Failure of the fiber-matrix interface.

The existence of a mechanism mechanically engenders the existence of another and develops according to the nature of the material and the conditions of the imposed mechanical stresses.

2.3.1 Mechanisms of rupture and damage of unidirectional composites

The rupture in a unidirectional composite is caused by the rupture of the fibers or the cracking of the matrix [17]. The latter is obviously caused either by:

- Transverse cracking if the tensile stress of the matrix (σ_m) reaches the breaking stress (σ_{mu}) of the matrix (Fig. 2.2) [18].
- Longitudinal cracking when the shear stress (τ_m) in the matrix reaches the breaking shear stress (τ_{mu}), generally in the vicinity of a fiber (Fig. 2.3) [9].

Concerning the rupture of the fibers, it intervenes when the tensile stress σ_f in a fiber reaches the stress at break of the fiber σ_{fu} (Fig. 2.4) [19].

The rupture of the fibers generates a stress concentration in the vicinity of the rupture. Therefore, the resulting rupture process depends mainly on the breaking stress of the fibers and the ability of the matrix to absorb the released energy [20,21]. Fig. 2.5 shows the different fracture processes of the matrix associated with the rupture of a fiber.

The rupture in the unidirectional composite depends essentially on the properties of the fibers, the matrix, the fiber-matrix interface, the volume fraction of the fibers, and the conditions of the imposed mechanical stresses [22,23].

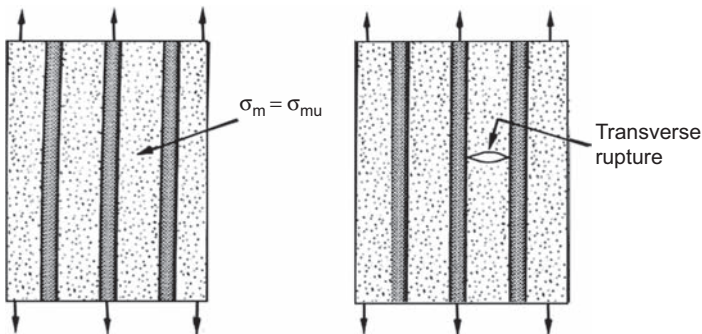


Figure 2.2 Transverse fracture of the matrix.

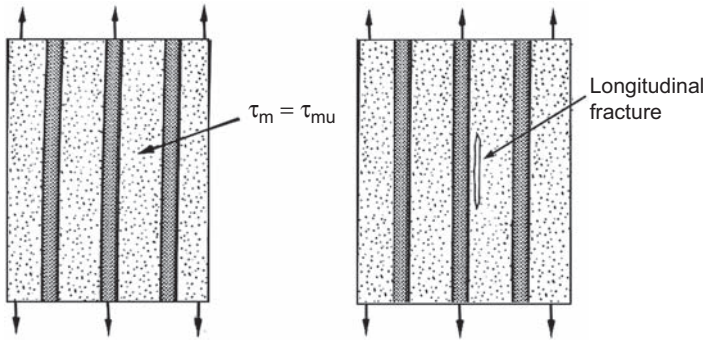


Figure 2.3 Longitudinal fracture of the matrix.

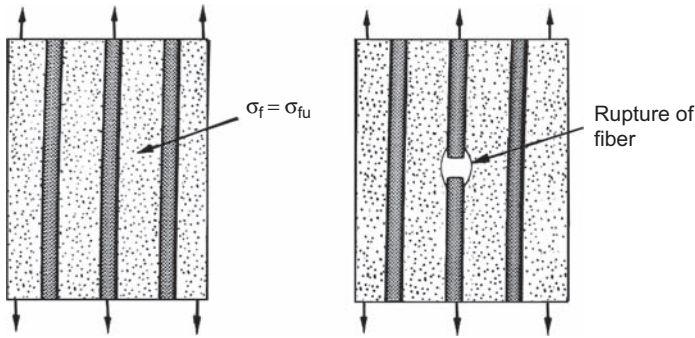


Figure 2.4 Failure of fiber.

2.3.2 Unidirectional composite submitted to longitudinal tensile

In unidirectional composites, the load is supported by the fibers when its direction is collinear with those of the fibers. In this case the initiation of the rupture is caused either by:

- Fiber rupture: occurs when the fracture at break of the fibers is lower than those of the matrix ($\epsilon_{fu} < \epsilon_{mu}$) as shown in Fig. 2.6
- Transverse rupture of the matrix if ($\epsilon_{mu} < \epsilon_{fu}$).

In addition to this, in industrial use, the thermosetting resins are generously doped while leading to a considerable reduction in the deformation at break of the matrix.

The rupture performance of thermosetting composites intended for industry and reinforced by glass fibers can be limited by a too low deformability of the matrix. Therefore, an optimization of the performance at break of this type of composite proves necessary by adapting the properties of the matrix to those of the fibers. The high-performance composites (carbon fiber composites) are managed by the properties to fiber breakage [24].

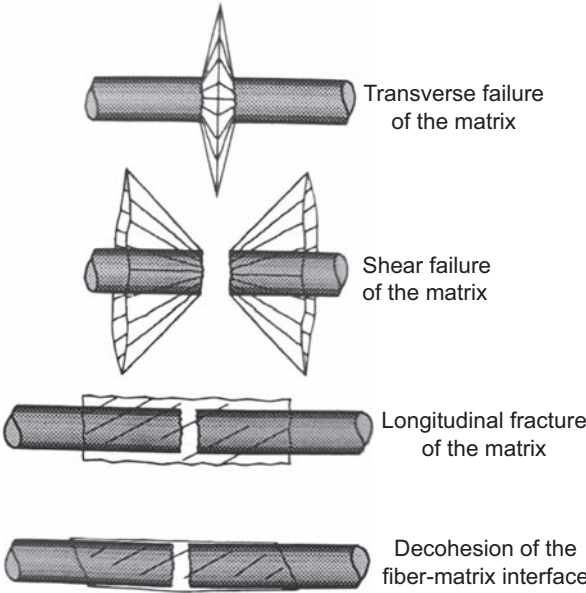


Figure 2.5 Different modes of rupture of the matrix associated the rupture of a fiber.

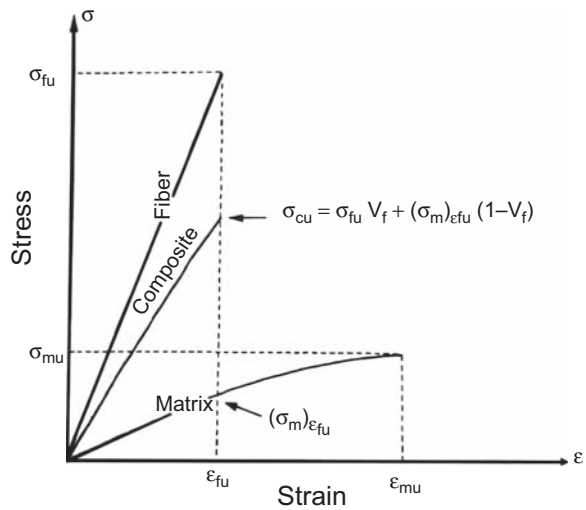
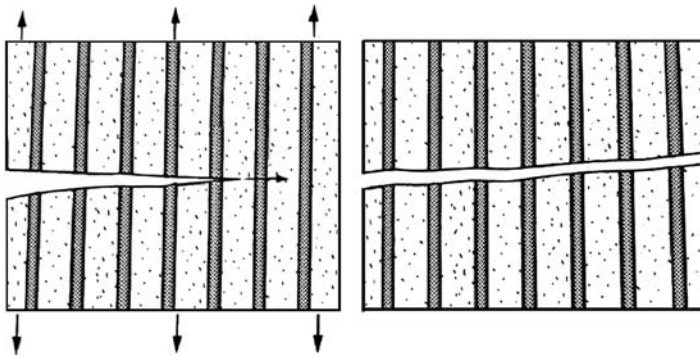


Figure 2.6 Stress-strain curves in the case of unidirectional composite.

Table 2.3 Mechanical characteristics of some synthetic fibers used in composite materials [25]

	Young modulus E_f (GPa)	Stress at break of fiber σ_{fu} (MPa)	Strain at break of fiber ϵ_{fu} (%)
Carbon fiber (high strength)	220	3000 at 4000	1.4 at 1.8
Carbon fiber (high modulus)	400	2200	0.5
Glass fiber (E)	70	2400 at 3400	3.4 at 4.8

**Figure 2.7** Propagation of the rupture in the case of high adhesion.

The mechanical characteristics for conventional fibers are grouped in the following Table 2.3.

After initiation, the propagation of the rupture depends on the fiber-matrix interface. At a high fiber-matrix interface, the rupture is initiated either by a rupture of the fibers or by rupture of the matrix, leading to a propagation of the rupture successively in the fibers and the matrix. The result is a fragile fracture (Fig. 2.7) [26].

At a weak fiber-matrix adhesion, the propagation of the rupture is of the transverse type, which will be described by [27]:

- At the head of the crack: the rupture of the matrix passes through the fibers, without breaking the fibers, but with decohesion of the fiber-matrix interface as illustrated in Fig. 2.8.

At the back of the crack: the opening of the crack induces high concentrations in the fibers carrying the crack. These stresses causing the fibers rupture at a distance more or less close to the surface of the rupture.

2.3.3 Unidirectional composite submitted to transverse tensile

In the case of a unidirectional composite subjected to a transverse tension, the rupture occurs either by rupture of the matrix or by decohesion of the fiber-matrix interface. The rupture of the matrix occurs when the tensile stress σ_m in the matrix reaches the stress at break σ_{mu} of the matrix and this can only occur if the stress at

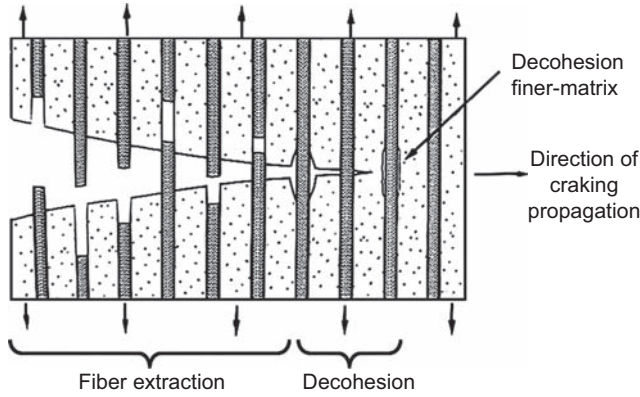


Figure 2.8 Failure propagation in the case of weak adherence fiber-matrix.

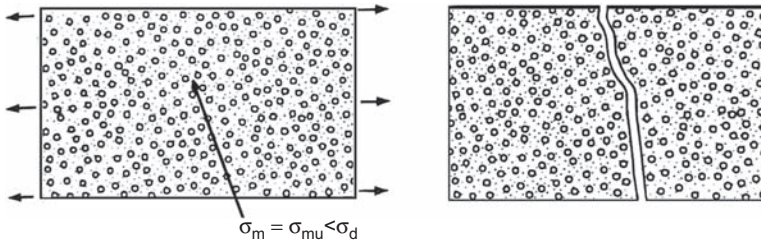


Figure 2.9 Failure of unidirectional composite under transverse tension.

break σ_{mu} of the matrix is less than the decohesion stress σ_d of the fiber-matrix interface as expressed in Fig. 2.9 [28].

2.3.4 Rupture of laminated composites

In the case of laminates, other mechanisms are added to those mentioned earlier (decohesion of the fiber-matrix interface, longitudinal and transverse rupture of the matrix, as well as the rupture of the fibers). The mechanisms that appear in laminates include the rupture between the layers, which is known under the delaminating phenomenon. These mechanisms of rupture depend on the nature of the basic constituents, the architecture of the folds, and the mode of mechanical stresses imposed [29]. The different mechanisms are summarized in Fig. 2.10.

Analyzing the breakage of certain types of laminates under a tensile stress [5]:

- In the case of the crossed laminates (0 degree/90 degrees), it is observed:
- A longitudinal rupture of the matrix and/or the rupture of the fiber-matrix interface in the layers oriented at 90 degrees.
 - The rupture of the fibers in the layers at 0 degree.
 - Transverse rupture of the matrix in layers at 0 degree.
- In the case of cross laminates (+45/−45), a longitudinal rupture in the layers at ±45, followed by delaminating between the layers is noted.

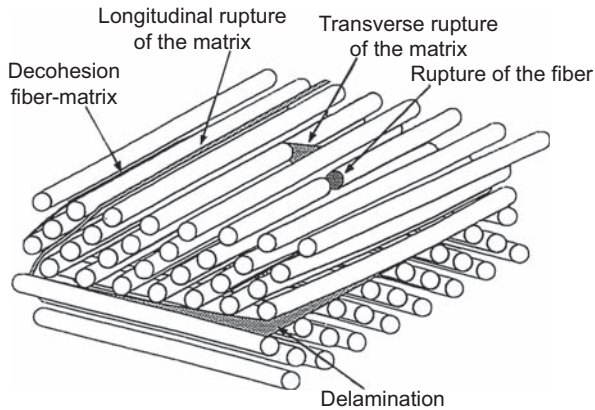


Figure 2.10 mechanisms of failure in a laminate.

Several phases of cracking are noted in these cases:

- In a first phase, a longitudinal cracking of the matrix occurs in the layers at 90 degrees.
- In a second phase, an initiation to cracking in layers at ± 45 is reported, starting from the cracks propagated in the layers at 90 degrees, with a limited propagation of these cracks at ± 45 degrees.
- The third phase is marked by the appearance of longitudinal cracks, starting from the hole in the layers at 0 degree. The cracks also generate secondary cracks at ± 45 degrees.
- In the last phase, the 0 degree cracks produce delaminating of the layers, followed by the rupture of the layers at 90 degrees, then layers at $+ 45$ degrees, and finally a rupture of fibers in the 0 degree layers leading to the final rupture of the plate.

2.4 Epoxy reinforced composites

Our focus is dedicated to the study of composites based on epoxy matrix reinforced by fibers of synthetic and natural origin, which the objective is to reveal how the deformation of manipulated composites occurs at different solicitations (tensile, torsion, etc.), as well as the various operating conditions, starting with the case epoxy doped only with fillers, epoxy reinforced with fibers, epoxy charged and reinforced at the same time, and finally the treatment of the case epoxy, charged and doubly reinforced.

2.4.1 Composite based on filled epoxy

Doping the epoxy matrix is obviously to improve its properties, but at which level does this load affect the epoxy deformation?

In a study by Abdellaoui et al. [11], they loaded the epoxy matrix with different content of an inorganic filler (illite), which is the gradually evolved (0, 5, 10, 15, 20) wt.%, to expose the effect of charge amount on the deformation of the composite (epoxy-clay). Their study reveals that the epoxy deformation is affected by the degree

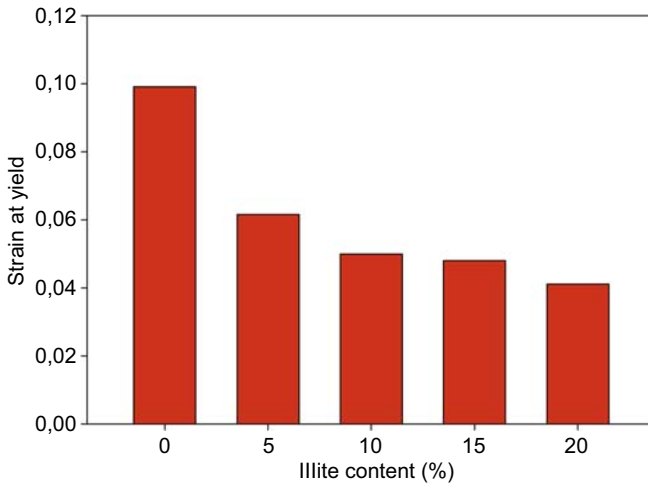


Figure 2.11 Deformation of epoxy filled with clay particles.

of dispersion and distribution of the clay particles along the epoxy polymer chain (Fig. 2.11). At low content, the composite (epoxy-clay) appears unstable attributed to the poor dispersion of the clay particles, which produces a break in the chain of the epoxy macromolecules.

With the composite subjected to tensile stress, the deformation can essentially engage in the areas covered by the clay particles with a low dispersion and low interfacial adhesion between the inorganic particles and the epoxy. By increasing clay content, the composite is more resistant to breakage by forming a bridge of clay particles along polymeric epoxy chains (10%–15%). However, above this content, 20% essentially, they have observed the formation of agglomerates of epoxy-free clay where the deformation is triggered from these zones with a high concentration (agglomeration). They concluded overall that the addition of clay weakens the epoxy ductility due to the rigid character of the clay which limits the plastic behavior of epoxy.

2.4.2 Fiber reinforced epoxy composites

In research conducted by the authors studied the strain-sensing behavior of epoxy composites with carbon nanotubes, subjected to a tensile stress [14]. Specially, they have studied the deformation behavior at different speeds of the typical stress-strain curves as illustrated in Fig. 2.12. The authors found that the tensile strength increases, while the relative deformation decreases. By increasing the load at break, the reactivity $\frac{\Delta R}{R_0}$ (R_0 is the initial resistance, ΔR is the instantaneous variation of the resistance) rose linearly, then it began to decrease to a critical deformation ϵ_c , which is attributed to:

- The reduction of the contact points of the nanotubes,
- The increase of gaps before the critical deformation,
- The reorientation and the alignment of the nanotubes in the zones of high deformation (after the critical deformation).

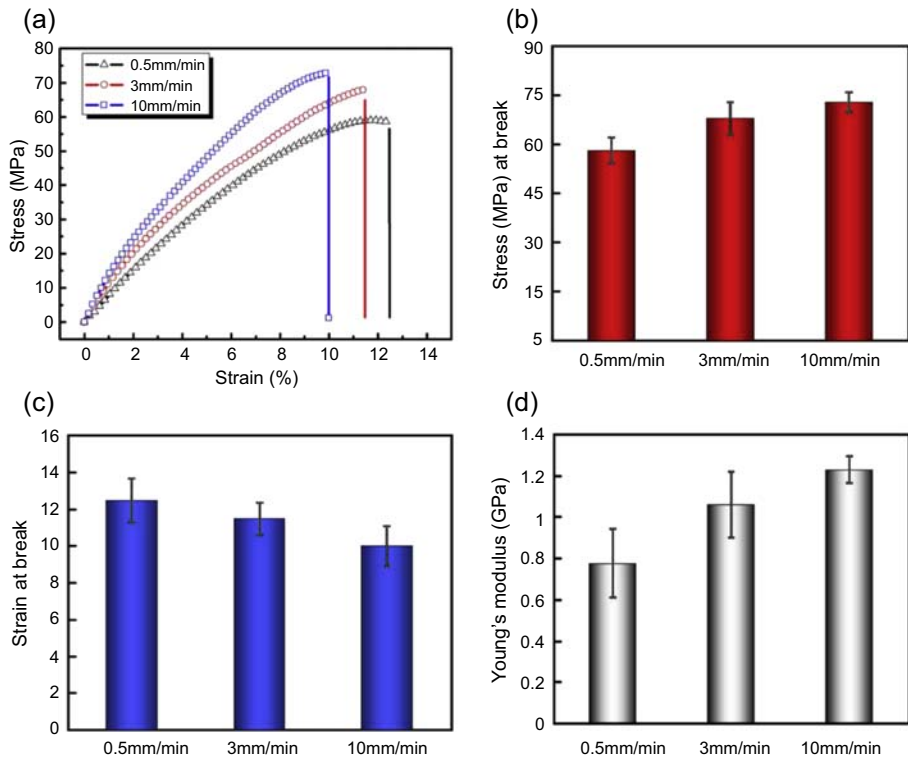


Figure 2.12 (a) Stress-strain curves and representative bar diagrams of (b) tensile strength, (c) strain and (d) Young's modulus of composites under different tensile speeds [5].

In addition, the polymeric chains do not have enough time to relax when the traction speed is fast.

Even more, in another work, where the epoxy is reinforced with natural fibers, we cite the work of Abdellaoui et al. [4], where the authors studied the deformation of laminates based on epoxy and jute fibers made according to different number of layers (1, 3, 5, and 7) and different orientations of fibers (0 and 45 degrees) to reduce the anisotropy character. The deformation of various laminates is studied by stressing the structures by tensile and torsion tests as illustrated in Figs. 2.13 and 2.14. It is found that the more the number of layers increases, the more easily the laminates become deformed.

As for the deformation generated by the torsion under a frequency interval (0.1–40 Hz), Fig. 2.14 shows the torsion modulus evolution as a function of the frequency and number of layers.

The more the frequency is increased, the complex modulus G^* becomes important. Because, at high frequencies, the deformation accelerates since neither the fibers nor the matrix has time to rearrange; Therefore, they express less and less the resistance to deform. So, during the deformation, the fibers can rotate sufficiently as to be carrying part of the load.

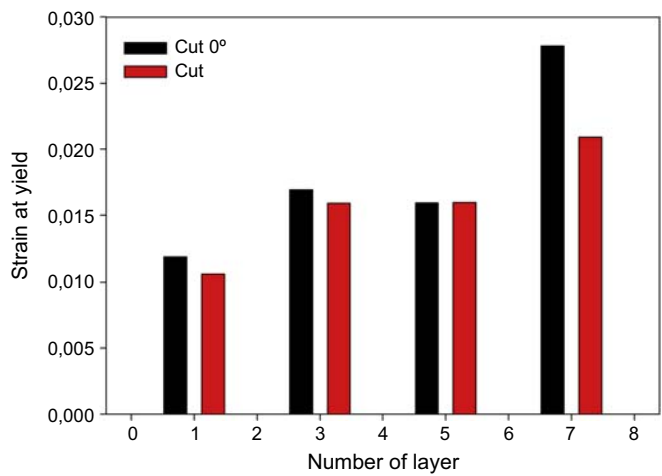


Figure 2.13 Strain versus clay content of different composites [1].

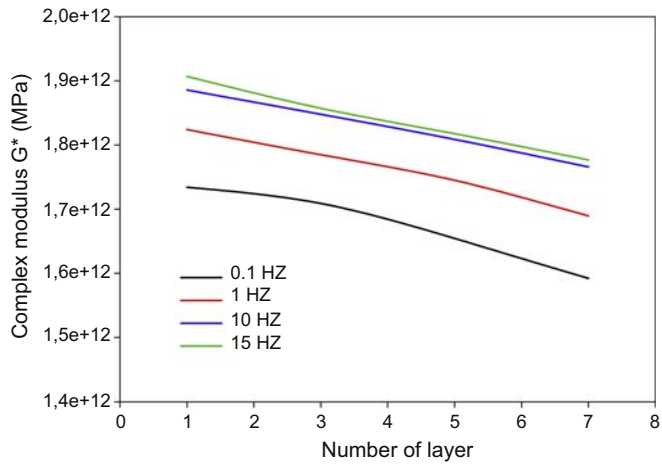


Figure 2.14 Complex modulus versus different frequencies [1].

This is expressed only by a high presence of microvacuum, which can be a source of stress concentration while increasing the applied load. The rupture will be caused at these zones because of the interfacial decohesion of the matrix-fiber.

2.4.3 Composites based on filled and reinforced epoxy

Still in the context of evaluating the epoxy deformation, we examine the deformation of epoxy both loaded and reinforced by fibers where the objective is to improve its strength. In this regard, we cite the work of Abdellaoui et al. [5], Who studied

deformation of composites based on epoxy loaded with clay and reinforced with treated and untreated jute fibers. The composites produced by compression method are subjected to tensile and torsion tests.

The results of their study reveal that untreated fiber composites deform all at the same level regardless of clay content in the epoxy (Fig. 2.15). Concerning the case of treated fiber composites, the results indicate a decrease in ductility by increasing the clay content. This decrease is obviously linked to the increase in the stiffness of the composite resulting from the alkaline treatment which heats the morphology of the fibers by promoting their adhesion to the matrix.

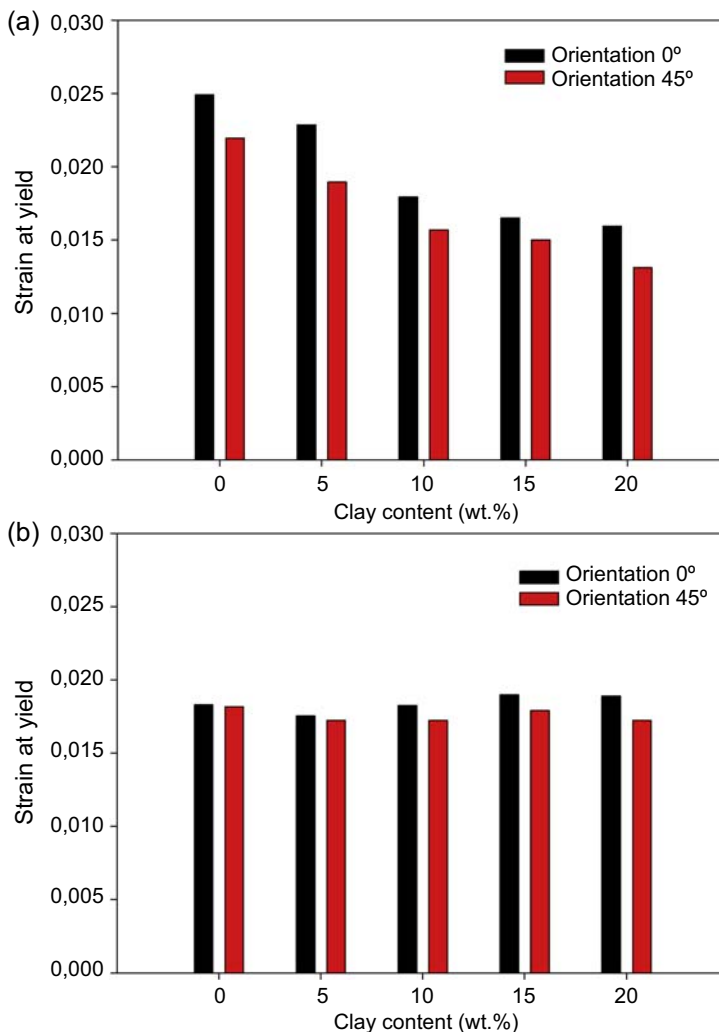


Figure 2.15 Strain of different composites with (a) composites with treated fibres; (b) composite with untreated fibres [5].

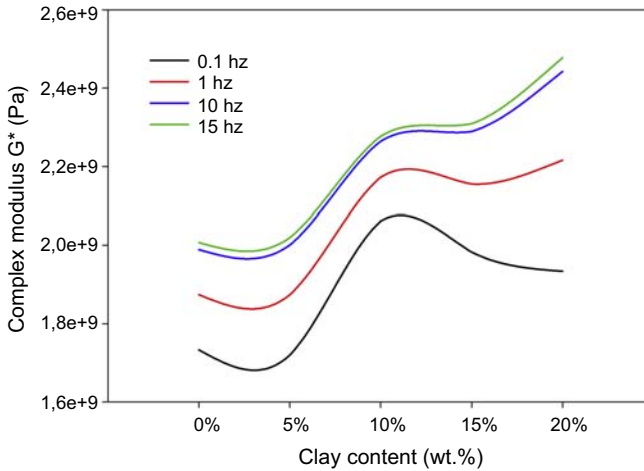


Figure 2.16 Complex modulus G^* of composites with different clay content [5].

In terms of torsion which carried out in the same conditions as quoted earlier, it was found that the torsion modulus G^* increases with the increase in the clay content for the treated and untreated fiber composites (Fig. 2.16). They have deduced that the wettability of the fibers by the matrix can be a constraint limiting the transfer of stress, as well as the agglomerations formed at high clay content can also create defect.

2.5 Rupture criteria

The criteria for rupture of a composite material generally make it possible to check the resistance conditions of the composites, especially of the laminated type based on a thermosetting matrix. For composite materials, the boundary of the elastic domain is often correlated with the appearance of microcracking (fiber breakdown, fiber-matrix decohesion, and matrix microrupture) [12]. The rupture criteria established for a single layer and then generalized throughout the laminate can be classified as follows:

- Criteria of the maximum stress
- Criteria of maximum deformation
- Interactive criteria or energy criteria

This section aims to cite briefly the criteria of the maximum stress, the maximum deformation, and some criteria considered in the case of the interactive criteria. The study of the rupture criteria in composite materials requires taking into account the base or the direction of the axes if they are in the principal base or outside this base. In this part, the criteria are considered in the case of the principal axes and outside these axes.

2.5.1 Criteria of maximum deformation

2.5.1.1 Criteria in the principal axes

To understand the notion of rupture criteria, we consider the case of a single layer of composite layered under a plane deformation state. The plane deformation state is considered when the variation according to the thickness is neglected because of its infinitesimal dimension, which makes it possible to determine only the ε_L , ε_T and ε_{LT} , where:

- ε_L : Linear deformation in the longitudinal direction,
- ε_T : Linear deformation in the transverse direction,
- ε_{LT} : Angular deformation in the plane of the layer.

Therefore, the mechanical strength conditions stipulate that the principal deformations in the layer must not reach the corresponding breaking strain. The maximum deformation criterion can be written as:

$$\begin{aligned} -X_{ec} < \varepsilon_L < X_{et} \\ -Y_{ec} < \varepsilon_T < Y_{et} \\ -S_e < \varepsilon_{LT} < S_e \end{aligned} \quad (2.1)$$

where:

- X_{ec} (X_{et}): Deformation at rupture in compression (or tension) along the longitudinal axis,
- Y_{ec} (Y_{et}): Deformation at rupture in compression (or tension) along the transverse axis,
- S_e : Shear deformation at rupture in the plane of the layer.

2.5.1.2 Deformation criteria outside the principal axes (initial base)

If the mechanical stresses (tensile or compression) are applied outside the principal axes as explained in Fig. 2.17, the obtained deformations are transformed to principal

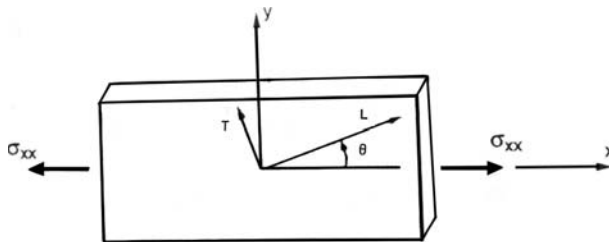


Figure 2.17 Tensile testing outside the principal axes.

deformations using a passage matrix [5] (inverse behavior law outside principal axes) which gives equations of the following form:

$$\begin{aligned}\varepsilon_L &= (S_{11} \cos^2 \theta + S_{12} \sin^2 \theta) \sigma_{xx} \\ \varepsilon_T &= (S_{12} \sin^2 \theta + S_{22} \cos^2 \theta) \sigma_{xx} \\ \varepsilon_{LT} &= (S_{66} \sin \theta \cos \theta) \sigma_{xx}\end{aligned}\tag{2.2}$$

Where the S_{ij} are flexibility constants expressed as a function of engineer modules (E_L , E_T , ν_{LT} , ν_{TL} , G_{LT}) experimentally determined in the main axes, and σ_{xx} is the longitudinal stress in the initial basis (x, y, z) (outside the principal axes).

Taking the case of a unidirectional or orthotropic composite, the relations are:

$$\begin{aligned}\varepsilon_L &= \frac{1}{E_L} (\cos^2 \theta - \nu_{LT} \sin^2 \theta) \sigma_{xx} \\ \varepsilon_T &= \frac{1}{E_T} (\sin^2 \theta - \nu_{TL} \cos^2 \theta) \sigma_{xx} \\ \gamma_{LT} &= \frac{1}{G_{LT}} (\sin \theta \cos \theta) \sigma_{xx}\end{aligned}\tag{2.3}$$

By determining the deformations in the plane and outside the principal axes, the maximum deformation criteria will be given by:

$$\begin{aligned}\frac{-X_C}{\cos^2 \theta - \nu_{LT} \sin^2 \theta} &< \sigma_{xx} < \frac{X_T}{\cos^2 \theta - \nu_{LT} \sin^2 \theta} \\ \frac{-Y_C}{\cos^2 \theta - \nu_{LT} \sin^2 \theta} &< \sigma_{xx} < \frac{Y_T}{\cos^2 \theta - \nu_{LT} \sin^2 \theta} \\ -S &< \sigma_{xx} \sin \theta \cos \theta < S\end{aligned}\tag{2.4}$$

2.5.2 Criteria of the maximum stress

Concerning the criteria relating to the maximum stress, the same procedure is used, distinguishing criteria in the main axes and criteria outside these axes.

2.5.2.1 Criteria in the principal axes

As for deformation, in the case of a layer subjected to a state of plane stresses (σ_L , σ_T , σ_{LT}), in its principal axes, the criterion of the maximum stress stipulates that the mechanical strength of the layer is reached when one of the three stresses to which the layer is subjected reaches the value of the corresponding breaking stress [5].

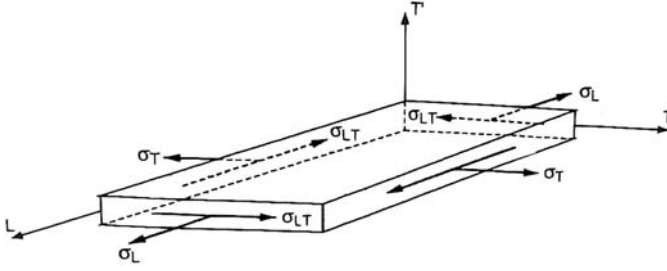


Figure 2.18 stresses in the principal axes.

The criterion of the maximum stress is written:

$$\begin{aligned}
 -X_c &< \sigma_L < X_t \\
 -Y_c &< \sigma_T < Y_t \\
 -S &< \sigma_{LT} < S
 \end{aligned}
 \tag{2.5}$$

Where X_C , X_T , Y_C , Y_T , and S are the stresses at break along the longitudinal axis, respectively, in compression and in tension, the stresses at break along the transverse axis, respectively, in compression and in tension, and the shear stress in the plane of the layer, where the longitudinal and transverse axes are the principal axes of the layer as explained in Fig. 2.18.

The stress state is not reached if the inequalities are verified and validated.

2.5.2.2 Maximum stress criteria outside the principal axes

In the case where the stress state is expressed in the initial axes (x , y , z) as mentioned in Fig. 2.19, it is necessary to reduce the stresses to the main base by an angle rotation θ . Therefore, the stresses expressed in the principal axes [5] are written according to Eq. (2.6):

$$\begin{aligned}
 \sigma_L &= \sigma_{xx} \cos^2 \theta + \sigma_{yy} \sin^2 \theta + 2\sigma_{xy} \sin \theta \cos \theta \\
 \sigma_T &= \sigma_{xx} \sin^2 \theta + \sigma_{yy} \cos^2 \theta - 2\sigma_{xy} \sin \theta \cos \theta \\
 \sigma_{LT} &= (\sigma_{xx} - \sigma_{yy}) \sin \theta \cos \theta + \sigma_{xy} (\cos^2 \theta + \sin^2 \theta)
 \end{aligned}
 \tag{2.6}$$

Then, the criterion of rupture is written in the form:

$$\begin{aligned}
 -X_c &< \sigma_L < X_t \\
 -Y_c &< \sigma_T < Y_t \\
 -S &< \sigma_{LT} < S
 \end{aligned}
 \tag{2.7}$$

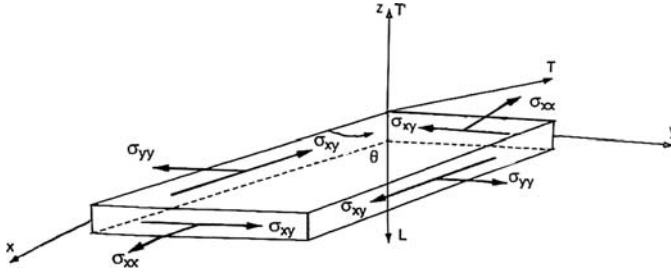


Figure 2.19 Composite layer relative to initial reference axes.

2.5.3 Interactive criteria

The criteria of maximum deformation and maximum stress are no longer sufficient, on the one hand, because they don't take into account all the experimental results and, on the other hand, they exclude the interactions that bind the stresses to the principal deformations.

The interactive criteria were able to fill this gap by extending the von Mises criteria to composite materials. The latter is connected to the energy of the deformed material stored per unit volume of the deformed material. Interactive criteria include [5]:

2.5.3.1 Hill criteria

It is considered among the interactive criteria of rupture applied to anisotropic materials. This criterion is formulated by stating that the stress limit state of a composite material is not reached as long as the following inequality is satisfied:

$$F(\sigma_T - \sigma_{T'})^2 + G(\sigma_{T'} - \sigma_L)^2 + H(\sigma_L - \sigma_T)^2 + 2L\sigma_{TT'}^2 + 2M\sigma_{LT'}^2 + 2N\sigma_{LT}^2 < 1 \quad (2.8)$$

The parameters F, G, H, L, M, and N are characteristic parameters of the studied material, which are connected to the rupture stresses X, Y, and S of the material. This inequality confirms the Hill criteria, related to the principal axes (L, T, T').

2.5.3.2 Tsai-Hill criteria

This criterion is given in the form:

$$\left(\frac{\sigma_L}{X}\right)^2 + \left(\frac{\sigma_T}{Y}\right)^2 - \frac{\sigma_L \sigma_T}{X^2} + \left(\frac{\sigma_{LT}}{S_{LT}}\right)^2 = 1 \quad (2.9)$$

Where X, Y, and S, are the rupture stresses of the considered material.

2.5.3.3 Hoffman criteria

Contrary to the Hill criterion, the Hoffman criterion takes into account the difference in the behavior of the material in tension and in compression. This criterion admits that the rupture of the material occurs when the following equality is verified:

$$C_1(\sigma_T - \sigma_T)^2 + C_2(\sigma_T - \sigma_L)^2 + C_3(\sigma_L - \sigma_T)^2 + C_4\sigma_L + C_5\sigma_T + C_6\sigma_T + C_7\sigma_{TT}^2 + C_8\sigma_{LT}^2 + C_9\sigma_{LT}^2 = 1 \quad (2.10)$$

With:

$$\begin{aligned} C_1 &= \frac{1}{2} \left[\frac{1}{Y_t Y_c} + \frac{1}{Z_t Z_c} - \frac{1}{X_t X_c} \right] \\ C_2 &= \frac{1}{2} \left[\frac{1}{Z_t Z_c} + \frac{1}{X_t X_c} - \frac{1}{Y_t Y_c} \right] \\ C_3 &= \frac{1}{2} \left[\frac{1}{X_t X_c} + \frac{1}{Y_t Y_c} - \frac{1}{Z_t Z_c} \right] \\ C_4 &= \left[\frac{1}{X_t} - \frac{1}{X_c} \right] \\ C_5 &= \left[\frac{1}{Y_t} - \frac{1}{Y_c} \right] \\ C_6 &= \left[\frac{1}{Z_t} - \frac{1}{Z_c} \right] \\ C_7 &= \frac{1}{S_{TT}^2} \\ C_8 &= \frac{1}{S_{LT}^2} \\ C_9 &= \frac{1}{S_{TT}^2} \end{aligned} \quad (2.11)$$

Where the constants $C_1, C_2, C_3, C_4, C_5, C_6, C_7, C_8, C_9$ are characteristics of the material and related to the stresses at break of the material.

2.6 Conclusion

The epoxy matrix can be the most used in the thermoset composites, especially in the high-performance materials. Therefore, studying the deformation of composites based on this matrix is colossally recommended to improve their resistance and also to predict the cause of deformation. For this reason, developing some realistic mathematical models and calculation algorithms will draw tremendous attention from researchers. Such models aim to simulate the deformation behavior of epoxy composite materials influenced by composite build up, the constituent properties as well as by interactions among the constituents, and also to optimize the forming process for manufacturing epoxy composite parts with minimum of defects.

Acknowledgements

This work was supported by MAScIR; Moroccan Foundation for Advanced Science, Innovation and Research, MESRSFC and CNRST, Morocco grant no. 1970/15.

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Effects of strain rate on failure mechanisms and energy absorption in polymer composites

3

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3.1 Introduction

Since 1980s to 1990s, polymer composites have been utilized in various aerospace, naval, and automotive structural parts due to their lightweight structures and critical performance. Due to the high demand for polymer composites in aerospace and automotive industries, it leads to great interest in using composite materials for crashworthy structures. Thus, they are now mainly used as primary load-bearing structures in high technology engineering applications compared to conventional metals [1]. This is because polymer composites can be tailored to offer superior impact energy absorption capabilities compared to metals in its basis weight [2]. The deformation response of polymers depends on a strain rate as reported by Goldberg et al. [3]. Therefore, it is vital to understand the effects of strain rate on the deformation and energy absorption capability of the polymer composites.

The understanding of polymer composite energy absorption is essential to (1) predict the impairment of structures caused by a crash, (2) evaluate the residual strength of structures after initial damage, and (3) design packaging to protect its contents during the impact. The energy absorption capability of polymer composites is also important to upgrade the human safety especially for automobile and airplane applications [4]. For example, two categories of energy absorbers are commonly being adopted in automobiles depending on the impact-loading conditions. For low-speed impacts, components such as bumpers are used to absorb energy and reduce collision severity. For high-speed impacts, components such as side beams are used to absorb energy during a crash. The key function of the crash absorber is to absorb impact energy in a controlled manner to ensure the well-being of all passengers [5]. Thus, many experimental studies have been conducted to investigate the effects of strain rate on the failure mechanisms and energy absorption in polymer composites. However, the advancement of new test methods should be considered to measure and understand the effects of strain rate on energy absorption and failure mechanism of polymer composite structures [1].

Quasi-static and impacts tests are the common methods performed in the studies on the energy-absorbing capacity. Studies on the quasi-static behavior of the polymer composite materials have focused on how the materials are deformed and distorted in response to loads applied at various orientations. Meanwhile, investigations of dynamic behavior have discovered that the strain rate and nonlocal effects substantially changed the dynamic deformation mechanisms and energy-absorbing capacity. The results of the extensive research work carried out in the quasi-static and impact testing have revealed that there are various factors which control the energy absorption capability of the composites materials, such as fiber and matrix properties, fiber architecture (stacking sequences), and strain rate [2,4,6].

3.2 Energy absorption capability

3.2.1 Definition

Energy absorption is defined as the surface below the load-displacement curve. The average load is identified as one of the determination parameters of absorbed energy capability based on the definition of energy absorption and peak load value, which was attained in the first step of a quasi-static test. There are two types of energy absorption capability, which are specific energy absorption (SEA) and volumetric energy absorption. SEA is described as cross-section areas in which the material is in contact with the top platen at any deformation, or in other words, SEA can be defined as the total of absorbed energy per unit mass. Meanwhile, an essential parameter for energy absorber system design where space is restrained is known as volumetric energy absorption capability.

The crashworthiness of the specimens is measured by the load-deformation curves of the energy absorption device [7]. Crashworthiness is defined as the ability of materials to absorb impact energy by means of controlled failure mechanisms and modes. During energy absorption, the materials need to be able to maintain a gradual deterioration in the load profile [4]. Mahdi et al. [5] discussed that the crushing ability can be categorized into several stages as shown in Fig. 3.1. Stage I is the total energy absorbed during the precrush stage. Stage II represents the total energy absorbed during the postcrushed stage. Finally, stage III indicates the total energy absorbed during the densification stage. However, the value in stage III is normally trivial; thus, it is being ignored. Several factors have been identified to affect the energy absorption capability of a composite material such as processing, test conditions, fiber and matrix types, and fiber architecture and specimen geometry.

3.3 Failure mechanisms

3.3.1 Definition

The failure mechanisms of polymer composites have been reported by many researchers [2,4,8]. Most of the researchers agreed that the failure modes of polymer

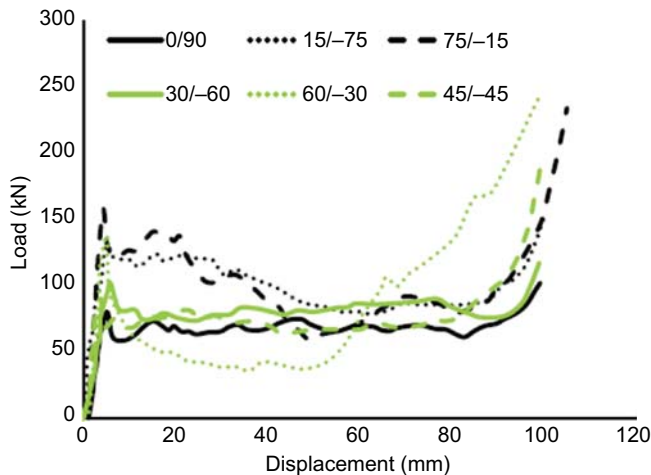


Figure 3.1 Average load-displacement curves for each fiber orientation of the composite tubes tested using quasi-static crushing tests [5].

composites involve catastrophic and progressive failure modes. Jacobs et al. [4] reported that there are two common failure modes known as catastrophic and progressive failure modes. Catastrophic failure is categorized by two conditions: it starts with a rapid increase in load to a peak value and followed by a failure load. In this situation, it is difficult to inhibit injury to the passengers because the actual amount of energy absorbed is much less and the peak load is also very high. In the case of catastrophic failure, the energy absorber structures must be designed heavier than structures designed for progressive failure energy absorbers.

Progressive failure can be obtained given that a trigger at one end of the tube is provided. The trigger will act as a stress concentrator to start a failure at a certain location within the structure and reduce the onset load peak. The stable collapse occurs after failure initiation. The trigger allows the failure to progress in a controlled predictable manner within the structure at the loading speed. In terms of energy absorption, the energy absorbed in catastrophic failure is lower than that in the progressive crushing.

However, a few researchers have specified the failure modes of polymer composite materials with different geometries into more than two categories. These categories have been explained based on experimental observations from a very extensive experimental treatment. Table 3.1 represents the failure mechanism categories reported by several researchers.

3.4 Strain rate

3.4.1 Definition

Strain rate is defined as the change in strain (deformation) of a material with respect to time. High-strain rates are events that occur over a short period of time and tend to

Table 3.1 Failure mechanism categories reported in literature

Geometries	Failure modes	Descriptions	References
Circular tubes	Mode I	• Pure regular progressive mode occurring from one end dominated the crushing process of the axially crushed composite circular tubes. • This mode is associated with crush mechanism that involves extensive matrix deformation and matrix fragmentation. • Fiber microfracture in small zones that moved progressively through the structure was also observed in this mode. The load-deformation behavior of this type of failure is shown to be very stable along the post crush stage. • This type of failure mode exhibited high-energy absorption.	[5]
	Mode II	• This mode of failure is a combination of progressive and local buckling modes. • The crushing process was initiated by matrix cracking. • After the first highest peak, a progressive failure mode was initially developed and followed by both matrix fragmentation and transverse shearing. • At this stage, the main energy absorption mechanism is matrix fragmentation and fiber debonding. • This stage exhibited an unstable load carrying capacity.	
	Mode III	• This stage exhibited a sudden drop-off in load carrying capacity immediately after the first highest peak. • The drop off was observed to be greater than in Modes I and II due to voids that acted as stress raiser. • Matrix cracking caused buckling of transverse fiber. Transverse shear caused the magnitude of the load carrying capacity to drop significantly. The shear was noted to be growing in the circumferential direction.	

Table 3.1 Failure mechanism categories reported in literature—cont'd

Geometries	Failure modes	Descriptions	References
Cone structure	Mode I	- The load carrying capacity increased progressively after the lowest point of load indicating resistance to load. Under progressive crushing, most specimens exhibited a combination of four failure modes identified as fiber debonding, fiber fracture, matrix cracking and delamination. - The progressive failure mode starts at the top end of the structure and followed by a localized loading which resulted in increasing load. - The structure also remains intact without debris splitting after being crushed and demonstrates postcrushing.	[8]
	Mode II	- Mode II type of failure is characterized by little and longer longitudinal cracks and fiber fractures. - The stresses concentration is increasing at the crush zone area as the crushing increase which led to the start of the continuous degradation on the material elastic properties. - Matrix cracks occur when the concentrated stress exceed the matrix strength. Besides, the adjacent region is affected due to the decrease in the mechanical properties at the crushed region.	
	Mode III	This mode of failure associated with brittle fracturing crushing mode. Instantly after the crush proceeds, the top end of the solid starts to fracture and split in small debris.	
Axisymmetric tubes of different geometries	Mode I	Progressive crushing with microfragmentation of the composite materials associated with large amounts of crush energy	[2]
Tubes and circular and square frusta of small semiapical angles.	Mode Ia	Mushrooming failures, mainly characterized by progressive collapse through the formation of continuous fronds which spread outwards and inwards with the simultaneous development of axial splits at the external fronds	

Continued

Table 3.1 Failure mechanism categories reported in literature—cont’d

Geometries	Failure modes	Descriptions	References
Axially loaded circular and square frusta of larger semiapical angles.	Mode Ib	• Characterized by the inversion of the shell wall inwards	
Circular frusta with a 15° semiapical angle	Mode Ic	• Characterized by an outwards inversion of the shell wall	
Circular and square frusta with 20°–30° semiapical angles	Mode II	• Brittle fracture of the component resulting in catastrophic failure with little energy absorption. • Characterized by the development of a spiral or a longitudinal crack propagating along the shell circumference. • Axially collapsed square frusta are associated with the formation of longitudinal corner cracks.	
Tubes and frusta with small semiapical angles and relatively small wall-thicknesses	Mode III	• Progressive folding and hinging similar to the crushing behavior of thin-walled metal and plastic tubes. • Designated as mid-length collapse mode. • Characterized by the formation of circumferential fracturing of the material at a distance from the loaded end of the specimens approximately equal to the mid-height of the shell; catastrophic failure by cracking and separation of the shell into irregular shapes is involved.	
Very thin tubes and frusta not exceeding 15° semiapical angles.	Mode IV	• Progressive folding and hinging similar to the crushing behavior of thin-walled metal and plastic tubes, showing a very low energy absorbing capacity.	

favor the elastic properties of materials. However, low strain rates are events that occur over longer time frame due to the impact resistance or toughness of the materials [9].

3.4.2 Test methods to determine the effect of strain rate in polymer composites

The effect of strain rate on the energy absorption and failure modes of polymer composites have been discussed in the literature [6,10–12]. The quasi-static and impact tests are the test methods commonly conducted on the polymer composites to study the energy-absorbing capacity and modes of collapse. This information is important for the applications where the energy-absorbing capacity is the main technical concern. They are useful in providing an indication of the suitability of polymer composites to be used in many applications such as in automotive and aerospace, which requires good energy absorption capability.

3.4.2.1 Quasi-static compression test

A quasi-static test is described as energy absorption capability of the composite when they are crushed under axial loads. The quasi-static tests are performed using a hydraulic press where the specimen is crushed at a very low crosshead speed between two parallel steel platens as shown in Fig. 3.2. The energy absorption capability of rate-sensitive materials depends on the compression speed. Thus, the determination of energy absorption using a quasi-static test does not indicate the crashworthiness of a structure [4].

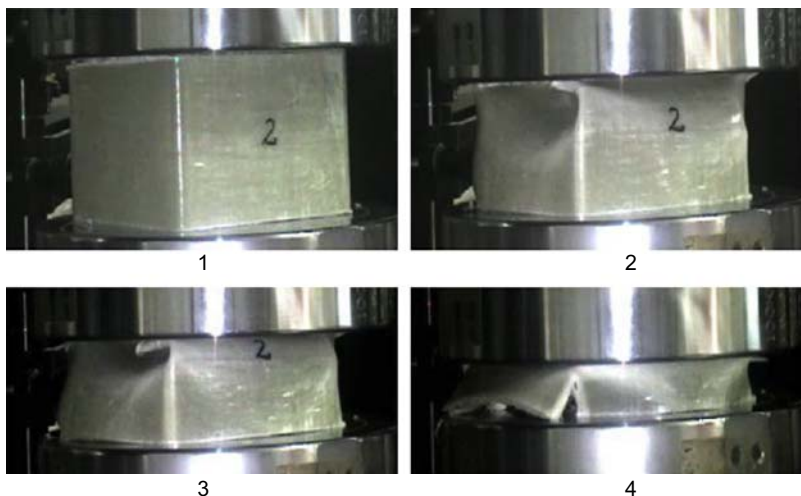


Figure 3.2 Quasi-static compression test [7].

3.4.2.2 Impact test

Impact testing includes the change in crushing speed as a specimen that absorbs the impact energy and can reproduce the behavior of a structure in an actual crash. The impact test crushing process occurred in a split second. The corresponding dynamic ones can be performed by direct impact using a drop-hammer or an impactor (Fig. 3.3) [4].

3.5 The effect of strain rate on the failure mechanisms and energy absorption

The energy absorption and failure mechanisms of polymer composites are affected by the strain rate during the test. There are several types of polymer composites available such as synthetic fiber-reinforced thermoplastic/thermoset composites, and natural fiber-reinforced thermoplastic/thermoset composites as well as thermoplastic/thermoset-based hybrid composites. The effect of strain rate on the failure mechanisms and energy absorption of each type of polymer composites will be discussed as follows:

3.5.1 Synthetic fibers reinforced thermoset composites

Many studies have been done on the effects of strain rate on the synthetic reinforced thermoset composites. Table 3.2 illustrates research works on energy absorption and failure mode of synthetic fiber-reinforced thermoset composites reported in the literature.

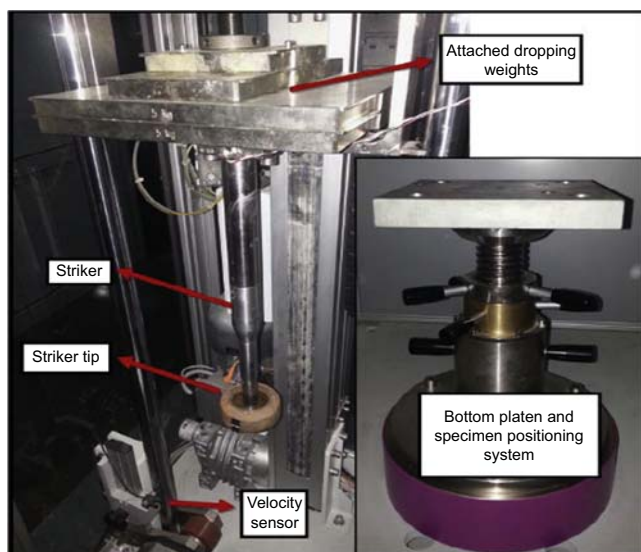


Figure 3.3 Drop-weight test apparatus [13].

Table 3.2 Research works on the energy absorption and failure mode of synthetic fibers reinforced thermoset composites reported in literature

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Carbon fabric	Epoxy	Quasi-static crush test	31.86	1,420	87,110	Meso-scale tubes	Splaying mode Trigger mechanism 45° outside chamfer,	[1]
8 layers of E-glass Woven fabric (15°/−75° and 75°/−15° fiber orientation)	Epoxy	Quasi-static crush test	156.31	7,927	—	Circular tubes	Catastrophic failure mode	[5]
Carbon fiber woven fabric	Epoxy	Quasi-static test	254.60	8,846	38,100	Square cross-section with internal tubes	Mode I, Mode II, and Mode III (Hull classification)	[14]
		Dynamic axial compression test	32.30	2,541	26,900			
Unidirectional carbon fiber	Polyester	Quasi-static test (crush under the inner type jig)	≈ 120	—	80,000 ± 2,200	Unidirectional square tubes with two layer of hoed strand glass mat	Progressive crushing mode	[17]
		Compression test (crush under the inner type jig)	≈ 140	—	95,400 ± 1,400			

Continued

Table 3.2 Research works on the energy absorption and failure mode of synthetic fibers reinforced thermoset composites reported in literature—cont'd

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Carbon fiber	Epoxy	Quasi-static test	≈ 140	—	—	—	—	[15]
E-glass fiber woven fabric	Polyester	Quasi-static test	—	—	—		—	[12]
Unidirectional E-glass fabric	Polyester	Quasi-static test	9.34	—	28.8		2 mm Conical circular type — Y tubes	Progressive mode
Carbon	Epoxy	Quasi-static	35.69	700	—	Half circle segment with two flanges (made up of a rectangular and a quarter-circle segments)	Catastrophic mode and local buckling mode	[10]
		Impact test					Stable progressive crushing	
Glass	Polyester	Quasi-static	≈ 26.02	—		—	Progressive collapse: fragmentation and splaying mode	Hemispherical shells
		Impact test	≈ 29	1,151.7	—			
Carbon	Epoxy	Quasi-static test	87.65	—	75,430	Brittle fracture	Cylindrical specimen	[11]

Several researchers have reported on the influence of strain rate on carbon-epoxy composites [1,10,11,14,15]. Meanwhile, a study on the glass-polyester composites has been discussed by other researchers [6,12,16]. Most of these works have reported on the effect of strain rates through the quasi-static and impact tests.

Johnson and David [1] articulated material models for each loading system under the quasi-static and dynamic loads to obtain input parameters for the material models to determine the strain rate effects on crush response. The dynamic material properties of carbon-epoxy composite energy absorber systems have been implemented in the Finite Element Analysis (FEA). The findings showed that stacking sequence configurations are critical, especially when the strain rate dependent part of the material system is predominantly loaded.

Mamalis et al. [14] discussed the difference between the dynamic and static axial compressed of square carbon fiber-reinforced plastics (CFRP) tubes. They observed three mechanisms of brittle collapse from the series of static and dynamic compression tests conducted, which are (1) the progressive end-crushing mode I was stable, while the other two failure modes were unstable; (2) local tube-wall buckling mode II; and (3) mid-length collapse mode III. They also concluded that the influence of the strain rate was relatively obvious in most cases. The first conclusion was the peak load in the case of dynamic tests was higher than the peak load of the corresponding static test. Secondly, the same occurrences also happened in the case of the absorbed amount of crash energy for almost all test cases with two exceptions [14].

Kara et al. [12] studied the high-strain rate and quasi-static deformations of a symmetric E-glass/polyester composite in two directions: through-thickness and in-plane directions. They have reported that both directions were found to be strain-rate sensitive as shown in compressive stress-strain behaviors of the composite. For the high-strain rate deformation test in the in-plane direction, an increase in strain rate led to increasing the modulus and maximum stress. In terms of a failure mode for in-plane direction, the failure occurred by axial splitting followed by 45 degrees inclined crack surface formation. In the case of through-thickness direction, similar failure modes were observed at the quasi-static and high-strain rates. The failure occurred with a nearly 45 degrees crack surface dividing the specimen into two pieces with axial splitting damage mode. They concluded that the failure mechanisms, as well as damage initiation and progression, were successfully predicted via developed numerical models.

An experimental investigation under both quasi-static and drop-hammer loading was carried out by Gupta et al. [6] to investigate the energy absorption capacities and failure modes of the glass/polyester composite hemispherical shells. Both quasi-static and impact tests showed that the failure of the composite hemispherical shells was primarily due to the fractured zones found along the meridian and circumferential directions. They also revealed that both load-deformation curves and modes of collapse from quasi-static and impact test results were similar. The findings from both tests also indicated that the mean collapse loads of quasi-static tests are slightly lower than that of impact tests. Conversely, they found that the increase in the thickness of the shell leads to the decrease in the percentage increment.

Tasdemirci et al. [13] experimentally and numerically investigated the dynamic crushing response and quasi-static as well as the energy absorption capability of joint geometry shells consists of a cylindrical segment and hemispherical cap. They concluded that the energy absorption of the hemispherical segment at quasi-static strain rate was lower than the high-strain rate due to the inertial effects. They also confirmed that at higher impact velocities, an increase due to inertia effect was higher than that of the strain rate. The effect of inertia became higher than the effect of strain rate sensitivity at a critical velocity for both hemispherical and cylindrical constituents.

Brighton et al. [11] have performed quasi-static and intermediate rate axial crush tests on tubular specimens of carbon/epoxy (Toray T700/G83C) and glass/polypropylene (Twintex). Both types of the matrix were reported to have a dependence on a strain rate. However, the polypropylene matrix showed a greater dependency on impacting speed than that of the epoxy matrix but with a high degree of uncertainty. The failure mode of the Twintex tubes changed with speed, while the carbon/epoxy tubes exhibited similar failure mechanisms throughout all speeds. It is noteworthy to mention that obvious changes can be observed between the quasi-static and intermediate tests. In the quasi-static test, a ductile folding failure can be seen while more brittle fractures were observed in the intermediate test. The greatest SEA and less dependency on speed can be seen in the 45 degrees chamfer carbon/epoxy tests [11].

3.5.2 Synthetic fibers reinforced thermoplastic composites

Most of the works on the energy absorption and failure mechanisms of the synthetic fiber-reinforced thermoplastic composites are using glass [18–21] and carbon [21–23] fibers with various types of thermoplastic. Table 3.3 shows some of the research works on the energy absorption and failure mode of synthetic fiber-reinforced thermoplastic composites reported in the literature.

The study on the effect of strain rate on the energy absorption and failure modes of the reinforced thermoplastic composite materials has received less attention. Okereke et al. [19] reported on the mechanism of rate-dependent off-axis compression of unidirectional composites made from E-glass fiber and polypropylene. The E-glass/polypropylene composites were subjected to the quasi-static, medium, and high-strain rates from 10^{-3} to 10^3 s^{-1} . The study also reported on the effects of strain rates on the evolution of different failure modes of the composites. In the quasi-static test results, the evolution of damage for quasi-static and medium strain rate tests was consistent along with a clearly defined fracture plane. However, the study showed a rate-dependent evolution of damage manifested by the presence of many crack fronts or fracture planes at higher rates.

The other work discussed on the influence of strain rate has been published by Brown et al. [24]. In their study, the effects of strain rate on the tensile, shear, and compression behavior of a commingled E-glass/polypropylene woven fabric composite over a strain rate range of 10^{-3} – 10^2 s^{-1} were reported. They used a special compression fixture for the dynamic compression and quasi-static tests. The results

Table 3.3 Research works on the energy absorption and failure mode of synthetic fibers reinforced thermoplastic composites reported in literature

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Plain weave Carbon	Poly(ethylene 2,6-naphthalate) (PEN)	Impact test	1.8	8.7	—	Square plate		[23]
Plain weave Basalt			3.3	24	—			
Plain weave Aramid			6.5	38.2	—			
Twill weave Vectran			2.8	29.4	—			
Plain weave type glass woven fabric	Polypropylene (PP)	Impact test	3.732	3.4	—	Square plate		[18]
Isotropic E-glass fibers (Plytron)	Polypropylene	Quasi-static test	—	—	—	—	At low and medium strain rates, interlaminar matrix failure exhibiting two distinct evolution patterns: Type I — initiates from the center of the specimen and evolves leading to an	[19]

Continued

Table 3.3 Research works on the energy absorption and failure mode of synthetic fibers reinforced thermoplastic composites reported in literature—cont'd

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Carbon fiber	PEEK	Axial compression test	153.1	—	226,800	Circular tubes	inverted V-shaped wedge Type II — initiates from the corners of the specimen and the crack travels diagonally through the specimen leading to an inverted V-shaped wedge. Mode I Splaying mode crush zone (Hull classification)	[22]
	Polyetherimide (PEI)		112.3	—	187,900			
	Polyimide (PI)		106.1	—	167,900			
	Polyarysulfone (PAS)		105.5	—	148,400			
Fiber glass	Polyamide	Axial impact tests	92.3	5,024	55,957	Square composite tube	Ductile crash mode	[20]
			72	4,683	44,875	Hexagonal composite tube		

±75 fiber orientation of glass fiber	Polypropylene	Axial crushing test	—	—	36,000	Circular tubes	Splaying and progressive folding mode	[21]
±45 fiber orientation of Carbon fiber	Polyamide		—	—	61,000		Fragmentation mode	
E-glass	Polypropylene	Quasi-static compression and impact test	—	—	—	Rectangular	At high-strain rates, delamination (splitting) was observed in the outer plies. At quasi-static strain rates, fiber microbuckling was observed. Microbuckling leads to fiber kinking followed by failure of the fibers and matrix through the thickness of the specimen along a distinct shear band fracture plane.	[24]

indicated that failure mechanism of the composites was comparable under both quasi-static and high-strain rate tests. However, larger damage happened at high-strain rates.

The other literature on the synthetic fiber-reinforced thermoplastic composites has discussed that the impact behavior of thermoplastic composite laminates depends on many factors, such as fiber orientation [21,22], types of thermoplastic [22], and fibers [23], compatibilizer [18] as well as testing temperature [23].

3.5.3 Natural fiber-reinforced thermoset composites

Currently, the awareness of environmentally friendly materials has been emerging. Natural fibers are suitable materials to be used as reinforcement in polymer composites due to their abundance, low density, and cost, renewability, biodegradable, and high specific strength compared to glass fibers [7]. The most important criteria in designing structures are high specific strength and stiffness as well as good energy absorption capability [7,25]. However, despite the advantages of natural fibers as reinforcement in polymer composites, natural fibers are rarely used in investigating the energy absorption capability and failure modes of composite structures. Table 3.4 presents research works on the energy absorption and failure mode of natural fiber-reinforced thermoset composites reported in the literature. Several natural fibers that have been used to study the energy absorption capability and failure modes are natural silk [7,26], oil palm [8], coir [8], banana [27], kenaf [28], hemp [29–31], flax [31], and jute [31]. All of these natural fibers were reinforced in thermoset composites.

Among all of the works reported in the literature regarding natural fiber-reinforced thermoset composites, a limited study has discussed the effects of strain rate on the energy absorption and failure modes of these composites [30,31]. The behavior of hemp fiber epoxy composites subjected to a low-velocity impact loading, using instrumented falling weight impact equipment was presented by Caprino et al. [30]. The main focus of the work reported by Meredith et al. [31] showed the potential for natural fibers to replace synthetic fibers for more sustainable energy absorption structures. They investigated the energy absorption of three different composites; flax, jute, and hemp-reinforced epoxy composites. The energy absorption of the composites with different impact velocity was also presented [31].

Works on the effects of geometry [28], impact energy [29], fiber sizes, and content [27] on the thermoplastic composites reinforced with natural fiber also have been reported in the literature. Eshkoor et al. [7] investigated the SEA of both triggered and nontriggered of plain-woven silk-reinforced epoxy composite tubes. The energy absorption capability and failure mechanism of the oil palm fiber and coir fiber-reinforced polyester composite solid cones with different vertex angles varied from 0 to 60 degrees were discussed by Mahdi et al. [8].

3.5.4 Natural fibers reinforced thermoplastic composites

Thermoplastic composites reinforced with natural fiber have a potential in sustainable energy impact absorption structures. However, not many studies have been reported on the impact properties of natural fiber-reinforced thermoplastic composites. Table 3.5

Table 3.4 Research works on the energy absorption and failure mode of natural fibers reinforced thermoset composites reported in literature

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Plain-woven natural silk fabric with 40 wraps and 30 weft yarns	Epoxy	Quasi-static test	≈ 14	≈ 125	≈ 5,200	Rectangular tubes	Mode I and Mode II (Mamalis classification) Hinge formation (Hull classification)	[7]
Oil Palm fiber	Polyester	Quasi-static test	307.41	—	619,250	Solid cone	Mode I (progressive mode)	[8]
Coir fiber			403.03	—	576,610		Mode II	
1% Banana fiber with 30 mm fiber length	Polyester	Compression test	9.20	23,090	4,050	Rectangular bar tubes	—	[27]
Non-woven kenaf fibers	Epoxy	Quasi-static axial crushing test	28.06	9,200	—	Hexagonal tubes with angle of $b = 45^\circ$	Local buckling mode	[28]
12 layers of woven natural silk with external triggers	Epoxy	Quasi-static test	14.36	113.15	5,433.33	Square section tubes	Catastrophic mode	[26]

Continued

Table 3.4 Research works on the energy absorption and failure mode of natural fibers reinforced thermoset composites reported in literature—cont’d

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Woven hemp fiber mat	Unsaturated polyester	Falling impact test	≈ 3.8	39.39	—	Square plates	—	[29]
Unidirectional hemp fabric	Epoxy	Falling weight impact test	≈ 1–9	≈ 30	—	Square specimens	—	[30]
Bidirectional hemp fabric	Epoxy	Drop-weight impact test and quasi-static	≈ 2–12	≈ 58	—	Conical test specimens	Progressive collapse	[31]
Unwoven hemp	Epoxy		—	—	54,300		Brittle fracture	
Woven flax			—	—	48,500		Brittle fracture	
Woven jute			—	—	32,600		Brittle fracture	

Table 3.5 Research works on the energy absorption and failure mode of natural fibers reinforced thermoplastic composites reported in literature

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Unidirectional and bidirectional hemp	Poly (lactic acid)	Falling impact test	—	≈ 60	—	Square plate	Indentation	[30]
Bamboo fabric	Polypropylene	Drop-weight impact	1.23	16.57	—	Rectangular plate	—	[32]
Bamboo fabric	Poly (lactic acid)	Drop-weight impact	0.54	8.74	—	Rectangular plate	—	
Jute fabric	Poly (lactic acid)	Drop-weight impact	1.84	8	—	Square plate	Fiber failures and delamination	[33]

indicates research works on the energy absorption and failure mode of natural fiber-reinforced thermoplastic composites reported in the literature. The behavior of hemp fiber polylactic acid (PLA) composites subjected to a low-velocity impact loading using instrumented falling weight-impact equipment was presented by Caprino et al. [30]. They performed experiments on composites made from hemp fibers, unidirectional and bidirectional, with PLA resin. It is noteworthy that threshold values found for hemp reinforced PLA composites are higher than those of carbon and glass composites. These results proved that greater energies are required for the damage initiation in natural fiber composite laminates.

Rawi et al. [32] studied the energy absorption and crack propagation of polypropylene (PP) and PLA composites reinforced with bamboo fabric. They reported that bamboo fabric-PP and bamboo fabric-PLA composites absorbed slight energy in the initiation process and utmost energy was absorbed in the damage propagation process. They also concluded that differences in the behavior of PP and PLA contributed to different crack behavior between bamboo fabric-PP and bamboo fabric-PLA composites. PLA is known to be more brittle than PP.

Low-velocity impact test was carried out on the jute fabric-PLA composites up to penetration by Papa et al. [33]. Ultrasonic Non-Destructive Inspection (NDI) technique and visual inspection have been used to identify the internal damages of the composites. The results from the tests performed showed that the phenomena such as fiber breakage and delamination also occurred at low energy levels. However, only impact tests at 15 J caused severe impact damages on the biocomposite samples.

3.5.5 Thermoset-based hybrid composites

Hybridization is defined as the ability to tailor several types of materials either reinforcement or matrix to optimize the material properties for certain structural applications requirements. Table 3.6 lists research work on the energy absorption and failure mode of thermoset-based hybrid composites reported in the literature.

Mahdi et al. [8] examined the quasi-static crushing behavior of hybrid and nonhybrid natural fiber between oil palm fiber and coir fiber composite with different solid cone vertex angles. They observed that oil palm fiber-reinforced polyester composite (OPFRP) had higher energy absorption compared to coir fiber-reinforced polyester composite (COFRP). In addition, the initial failure indicator factors (IFIF) decreased with the increase in cone vertex angle from 0 to 24 degrees.

Yahaya et al. [34] investigated the ballistic properties and quasi-static penetration of hybrid laminates made of nonwoven kenaf/Kevlar fibers and epoxy resin with a thickness ranging from 3.1 to 10.8 mm. One of the main findings obtained from their study was that more penetration energy were absorbed by kenaf/Kevlar hybrid composites as compared to Kevlar/epoxy and kenaf/epoxy composites. Among the entire hybrid composites studied, maximum penetration force and energy absorption were exhibited by those with kenaf at the innermost layers. However, Kevlar/epoxy showed higher SEA compared to hybrid and kenaf/epoxy composites.

Table 3.6 Research works on the energy absorption and failure mode of thermoset-based hybrid composites reported in literature

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Concept-I Coir fiber-oil palm fiber (COF-OPFRP β 60°)	Polyester	Quasi-static test	198.78	—	—	Solid cone	Matrix crazing and cracking mechanism	[8]
Concept-II Oil palm fiber-coir fiber (OPF-CORP β 60°)			121.21	—	—		Catastrophic failure	
High performance polyethylene (HP-PE)/carbon hybrid composites (77 vol% HP-PE)	Epoxy	Quasi-static test	6.5	—	37,600	Circular tubes	Both types of hybrid failure modes are interlaminar delaminations at the carbon/HP-PE interface and fracture of carbon fibers	[36]
High performance polyethylene (HP-PE)/carbon hybrid composites (55 vol% HP-PE)			8.0		44,600			

Continued

Table 3.6 Research works on the energy absorption and failure mode of thermoset-based hybrid composites reported in literature—cont’d

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Non-woven mat kenaf fibers at the innermost layers/Kevlar	Epoxy	Quasi-static test	17.44	131	—	Square laminate	—	[34]
Non-woven mat kenaf fibers at the outermost layers/Kevlar			13.38	90	—			
Kenaf and Kevlar are at the alternate layers			16.44	88.1	—			
T configuration (4Glass/1Jute/2Glass/1Jute/2Glass/1Jute/2Glass/1Jute/4Glass)	Polyester	Instrumented impact test	—	—	—	Rectangular specimen	Preferential orientation of impact damage across the length of the sample, especially at higher impact energy.	[35]
Q configuration (7 Glass/4 Jute/7 Glass)			—	—	—		The impact-damaged area appears in all cases to be quasi-circular	

De Rosa et al. [35] studied the effects of different impact energy (from 5 to 15J) and configurations between jute and glass fibers on the residual postimpact properties of jute/glass-polyester laminates. The results suggested that at low impact energies (up to 10 J), T hybrids perform better and insignificantly damage the laminate core. However, Q hybrids can endure higher impact energies (12.5 and 15 J).

Peijs and Van Klinken [36] studied the compression performance of HP-PE/carbon hybrid composite tubes with a ± 45 degrees fiber orientation. In this study, the energy absorption and failure modes for two types of HP-PE/carbon hybrid (77 and 55 vol% of HP-PE) were investigated. The failure modes of these hybrids are summarized in Table 3.6. The findings also indicated that the increasing amount of HP-PE fiber results in the decrease in the SEA of the composites.

3.5.6 Thermoplastic-based hybrid composites

Table 3.7 displays some research conducted on the energy absorption and failure modes of thermoset-based hybrid composites reported in the literature. Not much work has discussed the effects of strain rate on the energy absorption and failure modes of thermoset-based hybrid composites. Several works on thermoplastic-based hybrid composites have discussed the effects of impact speed [37], temperature [38], and types of fillers [39] on the energy absorption and failure modes of the different types of hybrid composites.

Tjong et al. [37] aimed to investigate the Charpy drop-weight impact behavior of short glass fibers (SGF)/Styrene ethylene butylene styrene (SEBS)/polypropylene (PP) and SGF/Maleated styrene ethylene butylene styrene (SEBS-g-MA)/PP hybrids. The findings from the test indicated that at low-impact speed, SGF/SEBS-g-MA/PP hybrids exhibit low-impact strength as compared to SGF/SEBS/PP hybrids. In contrast, the impact energy of SGF/SEBS-g-MA/PP hybrids was marginally greater than that of SGF/SEBS/PP hybrids.

Dubary et al. [38] reported on the influence of temperature on the damage tolerance and impact behavior of hybrid carbon and glass fiber woven-ply reinforced Polyether Ether Ketone (PEEK) thermoplastic (TP) laminates for aeronautical applications. The findings showed that temperature has trivial effects on the composite impact behavior such as maximum force or maximum deflection.

On the other hand, Haro et al. [39] investigated the effects of the addition of micro- and nanofillers on the dynamic impact response and energy absorption under the ballistic impact of Kevlar short fiber-reinforced high-density polyethylene. The natural microfillers (chonta palm wood and potato flour) and synthetic nanofillers (colloidal silica and gamma alumina) were used to produce the hybrid composites. The findings from the research suggested that the results of the dynamic impact test of the hybrid composites discovered enhanced impact resistance with the addition of synthetic nanofillers (silica and alumina) compared to composites with natural microfillers.

Table 3.7 Research works on the energy absorption and failure mode of thermoplastic-based hybrid composites reported in literature

Reinforcements	Matrix	Type of test	Peak load (kN)	Energy absorption (J)	Specific energy absorption (SEA) (J/kg)	Geometry	Failure modes	References
Glass fibers	SEBS/PP	Instrumented drop-weight impact	≈ 0.75	—	—	Rectangular flat	Fiber pull out and fiber debonding	[37]
Glass fibers	SEBS-g-MA/PP		≈ 0.9	—	—	Rectangular specimen	Fiber rupture, delamination, meta-delamination, matrix-cracking	
Glass fibers/carbon fibers	Polyether Ether Ketone (PEEK)	Drop-weight impact test	≈ 10	—	—	Rectangular specimen	Fiber rupture, delamination, meta-delamination, matrix-cracking	[38]
(10 wt%) Kevlar pulp and (20 wt%) Chonta palm microparticles.	High-Density Polyethylene (HDPE)	Impact test	—	59.1 ± 9.0	—	Rectangular specimen		[39]

(10 wt%) Kevlar pulp and (20 wt%) Potato flour microparticles.			—	56.9 ± 9.5	—			
(10 wt%) Kevlar pulp and (20 wt%) colloidal silica (SiO ²) nanoparticles.			—	58.0 ± 8.7	—			
(10 wt%) Kevlar pulp and (20 wt%) gamma alumina (Al ² O ³) nanoparticles			—	62.7 ± 6.5	—			

3.6 Conclusions

The energy absorption and failure modes of thermoset and thermoplastic composites have been extensively investigated by many researchers. Strain rate is identified as one of the factors that affect the energy absorption and failure modes of the polymer composites. To investigate the influence of the strain rate on the energy absorption and failure modes of polymer composites, quasi-static and drop-weight impact tests have been conducted by many researchers. These tests have successfully provided the information on the influence of the strain rate on the energy absorption capability of various types of polymer composites as well as their failure mechanisms. To further understand about the materials that are rate sensitive, researchers had also performed quasi-static and drop-weight impact tests on different types of polymer composites, including thermoplastic and thermoset composites reinforced with synthetic fibers, natural fibers, and synthetic/natural fibers. From the literature study, the utilization of natural fibers as reinforcement in polymer composites has found to be increasing due to awareness of environmentally friendly materials. However, despite the advantages of natural fibers as reinforcement in polymer composites, natural fibers are rarely used in investigating the effect of strain rate on the energy absorption capability and failure modes of composite structures. This indicates that there is still a need for future research to investigate the effects of strain rate of the natural fiber-reinforced thermoset/thermoplastic composites and natural fiber hybrid-based composites. This study is very important because different materials are found to behave differently at different strain rates. The future research in natural fiber composites and natural fiber hybrid-based composites are essential to give better insight on the ability of these composites to be used in many applications especially in automotive and aerospace applications.

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Bolted joint behavior of hybrid composites

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4.1 Introduction

The contemporary reinforcement utilized in composite materials is mainly dominated by glass fibers due to their relatively low cost and high mechanical strength. However, the utilization of glass fibers could result in environmental problems together with causing health hazards to human beings. The non-biodegradable characteristic of glass fibers is particularly the main concern among research communities. The increasing of environmental concern and awareness has aroused the development of sustainable materials such as natural fiber-based composites to substitute the glass fiber-reinforced composites [1,2]. Indeed, natural fibers possess a number of advantages such as being less abrasive and biodegradable, with high fatigue resistance, high specific properties, and low cost over synthetic fibers [3–6]. Despite natural fibers offer several attractive features, the mechanical properties are relatively lower than synthetic fibers. This impedes the use of natural fiber-based composites in a non-load critical application such as the interior part of the vehicle. In fact, the mechanical properties of natural fibers are dependent on their chemical structures such as microfibril angle, cellulose content, and degree of polymerization. Hence, different natural fibers have varying mechanical properties which depend on their chemical structures. The fiber structures consist of one primary cell wall together with three secondary cell walls. The cell wall comprises lignin, pectin, hemicellulose, and microfibril with a certain winding angle. The angle between the microfibril to the fiber axis is considered as microfibrillar angle and this angle is different from one fiber to another. The high microfibrillar angle results in high ductility whereas low microfibrillar angle leads to the high mechanical strength and modulus [7]. The lignin and pectin are required to be removed from the fiber structure as they hinder the stress transfer from the matrix to the cellulose, consequently resulting in lower mechanical properties in the composite materials. Natural fibers can be classified into stem, leaf, and seed fibers. The classification of natural fibers is illustrated in Fig. 4.1. Generally, natural fibers with higher cellulose content and lower microfibrillar angle possess higher mechanical performance, which is commonly obtained in stem fibers [8]. However, several factors have greatly influenced the performance of natural fibers. Extraction method, environment, and harvesting time are among the aspects that determine the mechanical properties of natural fibers.

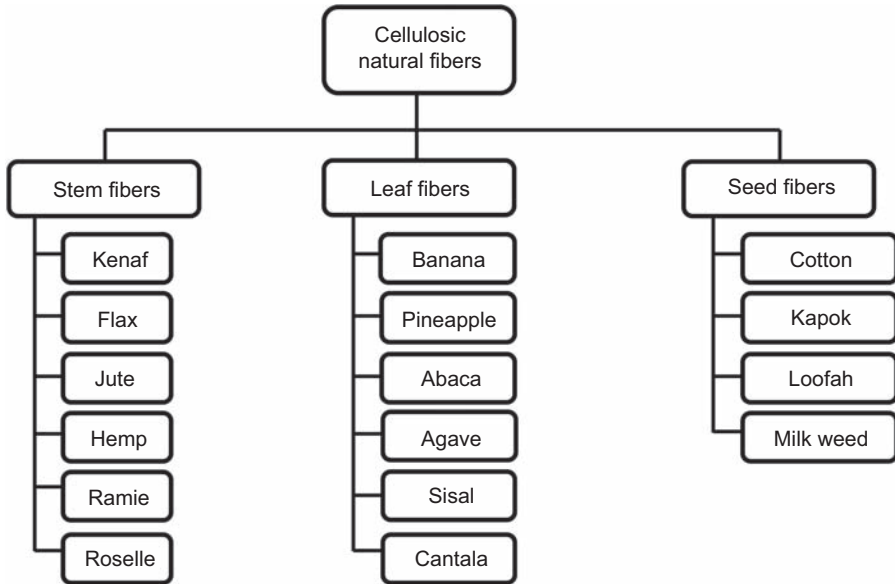


Figure 4.1 Classification of cellulosic plant fibers.

Despite natural fibers offer numerous attractive advantages, the relatively low mechanical properties together with their hydrophilic nature are being considered as the main limiting factors that impede the use of natural fibers in composite materials. The high moisture uptake of natural fiber-based composites results in the reduction in mechanical strength [9]. One of the methods to remedy the drawbacks of natural fiber-based composites is the hybridization with synthetic fibers. Ng et al. [10] attested the improvement in the fatigue resistance and tensile properties of fiber metal laminates with the incorporation of hybrid composites. Although the hybrid composites can be grouped into three main categories, which are synthetic/synthetic, synthetic/natural, and natural/natural hybridization, the most commonly explored hybrid composites are synthetic/natural fiber-reinforced hybrid composites due to the consideration regarding the balance of environmental friendliness and mechanical strength. Apart from the improved mechanical properties, the moisture sensitivity of the composite materials can be reduced through natural/synthetic hybridization. Numerous literature studies have shown the potential of natural/synthetic hybridization in composite materials as well as fiber metal laminates in terms of improved mechanical properties, reduced moisture, and notch sensitivity for structural applications [11–15].

4.2 Overview of hybrid composite materials

Hybrid composite materials have currently received great attention from researchers due to their excellent potential when compared to the non-hybrid single fiber-reinforced

composites. Hybrid composites can be defined as the materials that consist of two or more types of fibers embedded in a single polymer matrix [16]. Due to the different diameters of the fibers in the hybrid composites, more effective stress transfer could take place because of the increase in the fiber-matrix interfacial area [17]. This could result from the improvement in the aspect ratio (length to diameter ratio) of the fibers. Furthermore, the positive hybrid effect could be noticed in such materials as the load could still be bridged to the surrounding high elongation fibers upon the fracture of the fibers having low elongation, thus resulting in enhanced mechanical properties of the composites. In fact, hybrid composites can be considered as the weighted sum of the individual constituents in which a balance of advantage and disadvantages of the constituents could be achieved [18]. It is identified that the advantages of one reinforcement could complement the disadvantages of another reinforcement through the hybridization. Consequently, cost-effective hybrid composites with the required properties could be obtained through the appropriate material selection. Apart from the aforementioned advantages, hybrid composites also provide a lightweight characteristic in comparison to non-hybrid synthetic fiber-reinforced composites, which is due to the significant lower density of natural fibers. The lightweight characteristic is one of the criteria that is particularly important in transportation sectors to reduce fuel dependence and energy consumption. With the improvement in mechanical properties and lightweight characteristic in hybrid composites, the structural performance in transportation sectors could be enhanced without significantly deteriorating the safety measures.

However, the mechanical properties of hybrid composite materials are also strongly dependent on the fiber orientations, fiber length, fiber content, fiber-matrix interfacial adhesion, and the failure strain of each individual fiber. It is demonstrated that the maximum hybrid effect can be obtained if the different types of fibers are strain compatible [19]. In the hybrid composites, a positive or negative hybrid effect could be noticed as the mechanical properties of the materials are generally in between those of non-hybrid fiber-reinforced composites. A positive or negative hybrid effect is used to describe the positive or negative deviation from the mechanical properties of non-hybrid fiber-reinforced composites. The design of the hybrid composites to achieve certain properties is dependent on the requirement for certain applications. The selection of the compatible fibers when designing a hybrid structure is important to obtain the desired properties [20]. The properties of the hybrid system can be determined by the rule of mixture shown in Eq. (4.1) [18].

$$P_H = P_1 V_1 + P_2 V_2 \quad (4.1)$$

Where P_H is the mechanical properties of the hybrid system, P_1 and P_2 are the mechanical properties of the first system and second system, respectively, and V_1 and V_2 are the volume fraction of the corresponding first and second system.

4.3 Structural applications of bolted joint

Composite materials have been widely employed in automotive industries. It is quite impossible to produce structural construction without joining of materials. The assembly of composite materials in structural applications often involve different types of joining methods. However, joining through the adhesive agents, bolted joint and welding are the most commonly used techniques for structural applications. Bolted joint technique has been utilized in a wide variety of structural applications either for metallic alloys or composite materials. Bolted joint is one of the techniques that are used to connect materials together to form mechanical structures. In comparison to adhesive bonding and welding methods, bolted joint has been widely used due to its high load-carrying capacity, ease of assembly, and relatively low cost. Bolted joint comprises bolt, nut and washer to perform its clamping purpose. Their joint integrity is governed by the quantitative representation of the contact pressure distribution at the joint interface. Single and double lap joints are those of the most common form of mechanical joints. Fig. 4.2 shows the typical forms of single and double mechanical lap joints.

Although the joining process is the important step to connect two different parts to produce a construction, the joints are generally the weakest points of the structures that determine their stability. For example, the joints have been the weakest point in the airframe that determines the structural efficiency [21]. Apart from the aerospace application, bolted joints are also required for railway tracks to assemble the rail segment during installation. Nevertheless, the rail joints are also the weak points on the rail track, consequently leading to the short service life. The failure of the joints is generally associated with bolt loosening, cracked bolts, and cracked joint bars, which result in disrupting railroad operation, and sometimes the joints fail catastrophically. Bolt loosening is always the most common problem to occur for the bolted joint. Theoretically, it is possible to provide a proper design to produce a bolted joint that can overcome the unintentional bolt loosening [22]. However, the cyclic load during their service life causes the loosening of bolted joints. The bolts that are exposed to the cyclic loading are usually subjected to the fatigue failure at the threaded locations.

Besides the railway track, bolted joints are also used in many other structural applications such as building, bridges, and automotive components for their structural integrities. The bolted joints induce stress concentration at the vicinity of the holes,

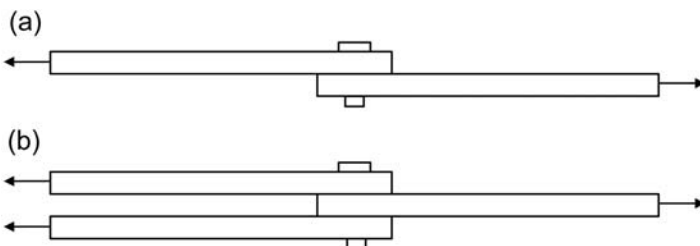


Figure 4.2 Types of mechanical joints (a) Single (b) Double.

eventually leading to the total failure of the structures [23,24]. Therefore, the investigation on the failure strength of the joint is especially important to provide the knowledge regarding the appropriate design of the joint geometry for each application. Several literature studies have shown joint geometric factors have significant effects on the failure strength and failure modes of the bolted joints of composite materials. The influences of the geometric factors of the joint on the failure strength of hybrid composites are briefly discussed in the following section.

4.4 Factors influencing the bearing strength of hybrid composites

There are various factors that can affect the bearing strength of composite materials. Geometrical parameters, joint configuration, loading condition, fastening parameters, and material selection and design are those of the key factors which determine the failure strength and failure mode of bolted joint in composite materials [25]. However, in the majority of the research work, the interaction between the hole diameter and the composite dimension is the most focused region in which the width-to-diameter ratio (W/D), edge-to-diameter ratio (E/D), and the distance between center of two-hole diameter ratio (K/D) are those of the main topics concerned. Overall, the investigation on the failure strength of composite materials has not been fully understood, especially for hybrid composite materials. Geometric parameters of the joint design on hybrid composites are illustrated in Fig. 4.3. Fig. 4.4 illustrates the fixture for a bolted joint test where Fig. 4.4a shows the jig design and Fig. 4.4b demonstrates the specimen setup on the jig. Thermal effect on the bolted joint hybrid composites was identified through the bolted joint test in a heat chamber as shown in Fig. 4.5. The bearing strength of single pin-loaded specimen is defined in Eq. (4.2). The shearing failure strength can be determined using Eq. (4.3).

$$\sigma_b = \frac{P}{tD} \quad (4.2)$$

Where σ_b is the bearing strength, P is the failure load, t is the thickness of the specimen, and D is the diameter of the circular hole.

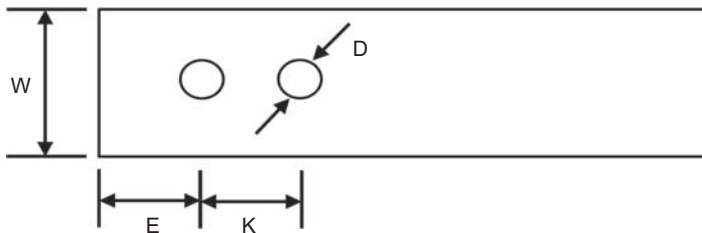


Figure 4.3 Geometric parameters of the joint design.

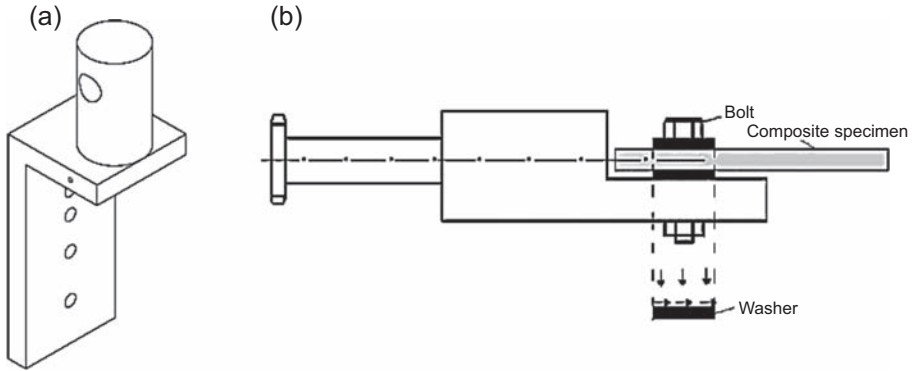


Figure 4.4 Experiment setup (a) jig design and (b) specimen setup.



Figure 4.5 Setup of specimen inside the heat chamber.

$$\tau_{ult} = \frac{P}{2Et} \quad (4.3)$$

Where τ_{ult} the maximum shearing strength and E is the edge distance.

Several experimental investigations have been conducted to study the effect of geometrical parameters on the bolted joint of polymeric composite materials.

Sivakumar et al. [26] studied the failure behavior of kenaf/glass fiber-reinforced hybrid composites with two serial bolted joint holes. The K/D ratio was fixed at 2, 3, 4, and 5, whereas the E/D ratio was in the range of 2–4. The two-hole diameter of the hybrid composites were fixed at 6 mm. From the findings, they noticed the failure strength of the hybrid composites increased with the increase of K/D and E/D ratio. Sivakumar et al. [27] conducted a further experimental investigation on the failure strength of bolted joint kenaf/glass fiber-reinforced hybrid composites with different geometrical parameters, W/D, and E/D ratio. The W/D and E/D ratio of the hybrid composites were both fixed at 3, 4, 5, and 6 while the hole diameter was fixed at 6 mm. They revealed the failure strength of the hybrid composites increased when the W/D and E/D ratio were increased. Furthermore, Sivakumar et al. [28] also investigated the effect of temperature and preload moment on the failure strength of bolted joint kenaf/glass-reinforced hybrid composites. The hybrid composites were subjected to various temperatures of 25°C (room temperature), 40, 50, and 60°C during the bolted joint test. The preload moment for the hybrid composites was fixed at 0, 5.2, and 7.5 Nm. The results showed that the increase in temperature reduced the joint strength of the hybrid composites. This could be due to the softening or damage of the polymer matrix at high temperature. However, it was observed that the increase of preload moment improved the failure strength of hybrid composites. The clamping force resulted from the preload moment avoids the delamination of fiber layers, resulting in improved load carrying capacity of the fibers in the hybrid composites.

Although there are some recent studies which focused on the failure strength of bolted joint of hybrid composites, many studies still focused on non-hybrid fiber-reinforced composites. Pekbey [29] carried out an experimental study on the bolted joint strength of glass/epoxy composites with varied geometrical parameters and preload moments. Similar results were noticed where an increase in the preload moment increased the bearing strength of the composites. Moreover, the increase of W/D and E/D ratio also contributed to the improvement in bearing strength of the materials. Soykok et al. [30] studied the thermal and tightening torque effect on the failure strength of mechanically fastened glass fiber-reinforced epoxy composites. The composites were subjected to the temperatures, ranged from 40 to 80°C with an increment of 10°C. In comparison with room temperature, a noticeable rapid fall in the failure strength of composites was observed when the temperature was increased to 80°C. One of the possible reasons is that the heat applied damages the polymer matrix, causing the reduction in the failure strength. Furthermore, the tightening torque was observed to improve the failure strength of the composites regardless of the temperatures. Ozen and Sayman [23] identified the bearing strength behavior of bolted glass fiber-reinforced epoxy composites with different geometrical parameters, E/D, K/D, and W/D ratio. The effects of preload moment and water immersion were also studied. It was observed that the increase in the geometrical parameter ratio and preload moment increased the failure strength of the composites. The failure strength of the composites with no preload moment reduced after the specimens were immersed in the water for 24 h. However, it is interesting to note that the failure strength of the immersed composites was comparable to those of the unimmersed composites under preload moment. Khashaba et al. [25] investigated the effect of fiber-stacking

sequences on the failure strength of glass fiber-reinforced epoxy composites. Four fiber-stacking sequences in the composites were studied, which include $[0/90]_{2s}$, $[15/-75]_{2s}$, $[30/-60]_{2s}$, and $[45/-45]_{2s}$. The results established the composites with $[0/90]_{2s}$ fiber-stacking sequence exhibited highest failure strength compared to other stacking sequences. However, the composites with fiber-stacking sequence of $[45/-45]_{2s}$ had the maximum failure displacement according to the first peak criterion. Liang et al. [31] studied the effect of stacking sequences and preload moment on the failure strength of bolted joint in glass fiber-reinforced epoxy composites. All the composite laminates with different fiber-stacking sequences and orientations were fabricated through vacuum-assisted resin injection method. The improvement of the failure strength of the bolted joint composite laminates was observed when the tightening torque was applied to the laminates. The laminates stabilized with the increase of tightening torque that increased the failure strength of the composite laminates. Nevertheless, the maximum failure displacement decreased with the increase of tightening torque due to the local stress in the resin around the metal bolts. The combination of fiber orientation of 0, 45 and 90 degrees in the composite laminates showed higher failure strength than the composite laminates with a fiber orientation of 0 and 90 degrees respectively. The aforementioned literature studies are summarized in Table 4.1.

From Table 4.1, it is noticeable that there are very limited studies that highlighted the bolted joint behavior of hybrid composites. It has been identified that the natural

Table 4.1 Reported works on the bolted joint composites

Fiber	Matrix	Parameters	References
Kenaf/ Glass	Polypropylene	K/D and E/D ratio	Sivakumar et al. [26]
Kenaf/ Glass	Polypropylene	W/D and E/D ratio	Sivakumar et al. [27]
Kenaf/ Glass	Polypropylene	Temperature and Preload moment	Sivakumar et al. [28]
Glass	Epoxy	W/D ratio, E/D ratio and Preload Moment	Pekbey [29]
Glass	Epoxy	Temperature and Tightening Torque	Soykok et al. [30]
Glass	Epoxy	E/D ratio, K/D ratio, W/D ratio, Preload Moment, and Water Immersion	Ozen and Sayman [23]
Glass	Epoxy	Fiber-Stacking Sequences	Khashaba et al. [25]
Glass	Epoxy	Fiber-Stacking Sequences and Preload Moment	Liang et al. [31]

fibers are less sensitive toward the notch effects due to their fiber structure. Therefore, natural fibers attest a great potential to be used in bolted joint applications to allow the structural integrity and safety performance. Besides that, most of the literature studies applied glass fibers in the bolted joint composites due to their low cost and relatively high mechanical strength compared to other synthetic fibers. Overall, the using of natural fibers in the bolted joint applications still require a great effort to explore their potential in the future.

4.5 Failure analysis of hybrid composite due to bolted joint

The typical joint failure modes of composite materials are different from those of metals, where the yielding occurs in the metals prior to the total fracture and this is not significantly found in composite materials. Instead of yielding, composite materials are commonly related to the delamination and fiber breakage before final fracture. The failure modes of composite materials highly depend on the parameters, as mentioned earlier. It is undeniable that the factors such as material parameters, fastener parameters, and design parameters also contribute to the different failure modes in bolted joint of composite materials.

In general, net-tension, shear-out, and bearing are those of typical failure modes related to composite materials due to bolted joint failure. The combination of those failure modes is possible in certain situations that are generally dependent on the geometrical parameters of the composite materials. Net-tension and shear-out normally happen in a catastrophic manner, which causes the sudden fracture or failure of the entire structure. In comparison to net-tension and shear-out mode, bearing failure is considered as the local failure in which that occurs in a progressive manner related to the compressive load [32,33]. Due to the progressive bearing failure mode, this failure is usually preferable to provide the highest failure strength and avoid catastrophic failure. Karakuzu et al. [34] have reported that bearing failure mode is the most commonly found in practical applications.

4.5.1 Self-loosening of bolted joint

The service problem associated with the self-loosening of a bolted joint is always the common problem in structural applications. Self-loosening of a bolted joint is different from the net-tension, bearing, and shear-out failure modes in which these failure modes often involve the damage to the structure. Accidents have been reported in aerospace industries due to the self-loosening of a bolted joint. The major cause of the self-loosening that has been identified is due to the lack of preload moment or clamping force that can sustain the load applied during their service life. Because of the importance and complexity of the failure, self-loosening in the bolted joint has been widely explored by researchers. The experimental findings evidenced that the factors of causing self-loosening are related to plastic deformation of the fasteners, slippage,

and fretting wear of the contact surface [35–39]. The vibration during the service life increases the loosening process through the wear and hammering that lead to the lost in the preload and clamping force. Once the preload and friction force dropped to a critical level, this initiates the back off of the nut and the clamping force is totally lost [22]. Nassar et al. [40] and Yang et al. [41] have conducted an experimental and numerical investigation on the self-loosening characteristic of the bolted joint due to the axial cyclic load. The results indicated the plastic deformation of the fastener was the main factor that leads to the self-loosening in the bolted joint. Liu et al. [42] also found the similar observation where the self-loosening of the bolted joint was due to the plastic deformation of the fastener that progressively results in the wear between the contact threads. Apart from the plastic deformation of the fastener, lack of friction force at the contact surface also results in self-loosening. It was reported by Jiang et al. [43] that the self-loosening of the bolted joint due to the rotation of nut happens when the friction force at the contact surface was low.

In practice, various methods have been implemented to avoid the self-loosening and maintain the clamping force of the bolted joint. The methods which are utilized during the bolted joint designing stage include [44]:

- Material selection for those surfaces that possess a high coefficient of friction to improve the friction force between the contact surfaces.
- Minimize the clearance of mating materials.
- Design of joint that can sustain high preload moment.
- Design the joint with high bolt length to diameter ratio.
- Reorientation of the joint to allow the joint can be loaded in axial direction instead of shear loading.

It is theoretically demonstrated that the design of bolted joint which can retain the preload moment to overcome the self-loosening without the using of any locking device is possible. Nevertheless, the self-loosening does happen in the real situation. Therefore, it generally requires the assistance of a fastener-locking device on the joint. The fastener-locking device is installed during the assembly of the structure to avoid the self-loosening of the bolted joint during service life. There are numerous locking methods available, and the performance of these methods depends on the preload moment that can be retained for a given cyclic loading. Many of the locking devices do not absolutely lock the fastener to prevent the rotating of the bolts but they allow a minimal degree of self-loosening under dynamic shear loading [45,46].

4.5.2 Failure criteria

The damage initiation of composite materials can be predicted using failure criteria. The subject of failure criteria for fiber-reinforced plastics either in microscopic or macroscopic scales has attracted large attention [47]. A number of failure models have been recommended to predict the failure in composite materials. However, these models can be classified as interaction or phenomenological. Maximum stress criterion, Hashin failure criteria, and point stress failure criteria are among the major failure models that have been widely utilized.

4.5.2.1 Maximum stress criterion

Maximum stress criterion is one of the most extensively used failure criteria to predict the failure of composite materials as this criterion is less complicated. This criterion is a linear, stress based, and failure mode dependent criterion without stress interaction [48]. Failure occurs once the stress components are higher than the corresponding yield strength either in tension or compression. The maximum stress criterion is according to the following approach in which the failure occurs if one of the conditions is reached.

$$X_T \leq \sigma_{11} \text{ or } \sigma_{11} \leq X_C$$

$$Y_T \leq \sigma_{22} \text{ or } \sigma_{22} \leq Y_C$$

$$\text{or } |\tau_{12}| \geq S_{12}$$

Where X_T is the tensile strength, X_C is the compressive strength, Y_T and Y_C are the tensile and compressive strength in the transverse direction, and S_{12} is the shear strength.

4.5.2.2 Hashin failure criteria

There are some differences between Hashin failure criterion and maximum stress criterion. Hashin failure criterion is a quadratic and physically based failure criterion that takes into account the stress interaction. This failure criterion was developed due to the presence of different failure modes in composite materials. Based on the criterion, failure of the composite materials could occur if one of the following conditions is reached:

$$\frac{\sigma_{11}^2}{X_t^2} + \frac{\tau_{12}^2}{S_{12}^2} \geq 1 \quad \text{if } \sigma_{11} > 0$$

$$\frac{\sigma_{11}}{X_c} \geq 1 \quad \text{if } \sigma_{11} < 0$$

$$\frac{\sigma_{22}^2}{Y_t^2} + \frac{\tau_{12}^2}{S_{12}^2} \geq 1 \quad \text{if } \sigma_{22} > 0$$

$$\left(\frac{\sigma_{22}}{2S_{23}} \right)^2 + \left[\left(\frac{Y_C}{2S_{23}} \right)^2 - 1 \right] \frac{\sigma_{22}}{Y_C} + \left(\frac{\tau_{12}}{S_{12}} \right)^2 \geq 1 \quad \text{if } \sigma_{22} < 0$$

4.5.2.3 Point stress failure criterion

To evaluate the joint strength of composite materials, the stress distribution around the hole must be first evaluated. Theoretical analysis on the bearing strength of pin-loaded joint composite materials was performed by Whitworth et al. [49] based on the two-dimensional point stress failure criteria. The criterion is determined according to the following relationship:

$$\left(\frac{\sigma_1}{X}\right)^2 + \left(\frac{\tau_{12}}{S}\right)^2 = e^2$$

Where σ_1 is the longitudinal stress, τ_{12} is the shear stress, X is the longitudinal strength, and S is the ply shear strength.

In this failure criterion, the failure occurs when the e value is higher than or equals to unity. Furthermore, the characteristic curve for the evaluation pin-loaded composites can be represented using the following equation.

$$r_c(\theta) = R + R_t + (R_c - R_t)\cos \theta$$

Where R is the radius of the hole, R_t and R_c are the characteristic dimension in tension, and compression and the angle θ is ranged from -90 to 90 degrees.

The characteristic dimension in tension and compression are evaluated through applying of the failure criterion to the open hole plate under tension and compression. Point stress failure criterion assumed the failure could occur when the transverse tension and compressive stress at the distance of R_t and R_c reach the tensile and compressive strength of the unnotched materials, respectively. This approach can be used to predict the failure strength of the bolted joint with a two-dimensional finite element model.

4.5.3 Effect of geometrical parameters on the failure modes

The geometrical parameters of the composite materials indeed have a prominent effect on the failure modes under bolted joint test. Any changes in geometric ratio result in different failure modes. The common failure modes observed in the bolted joint test include net-tension, bearing, and shear-out. However, the combination of failure modes is also possible to occur during the bolted joint test. From the literature studies, findings demonstrated the failure modes of the composite materials have a great influence on the bearing strength. Based on the results obtained by Sivakumar et al. [27], the net-tension was commonly observed in the composites with two serial holes when the K/D ratio was high whereas the shear-out failure mode was observed when the E/D ratio was small. The failure modes of the kenaf/glass hybrid composites were summarized in Fig. 4.6. It can be noticed that the combination of bearing and net-tension failure modes was possible when the K/D ratio was fixed at 3 and 5 excluding the case of K/D and E/D ratio equal to 4. The shear-out failure mode was noticed in the outer holes of the hybrid composites when the E/D ratio was fixed at 2. This could be due to the

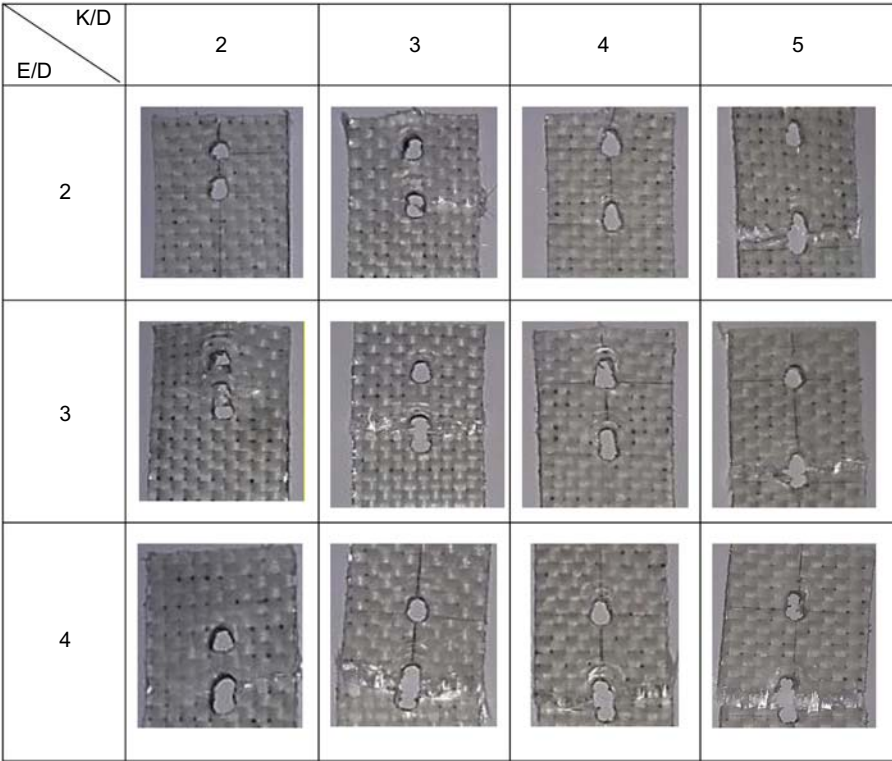


Figure 4.6 Various failure modes from experimental studies for $E/D = 2, 3, 4$, and $K/D = 2, 3, 4$, and 5 .

small distance between the edges to the center of the outer hole, consequently, lead to the development of shear stress around the hole.

Apart from K/D and E/D ratio, the change in W/D ratio also results in different failure modes in composite materials [26]. As the W/D ratio was smaller than 5, net-tension could occur due to the small width of the hybrid composites. However, the combination of net-tension and bearing failure mode was observed when the W/D ratio was equal to 5. There is a transition phase where the failure modes changed from the net-tension to bearing when the W/D ratio was increased from 3 to 6. In comparison to the geometrical parameters, the preload moment and clamping force were identified not to have any significant effect on the failure modes. Sivakumar et al. [28] have demonstrated any changes in the preload moment resulted only in bearing failure mode. However, they demonstrated that the increase in ambient temperature induced more damage to the hybrid composite around the hole, which eventually leads to the notch deformation. Figs. 4.7–4.9 depict the failure mode of the hybrid composites under various preload moments and ambient temperatures. The figures clearly evidence the change in the magnitude of preload moment results in the similar failure mode.

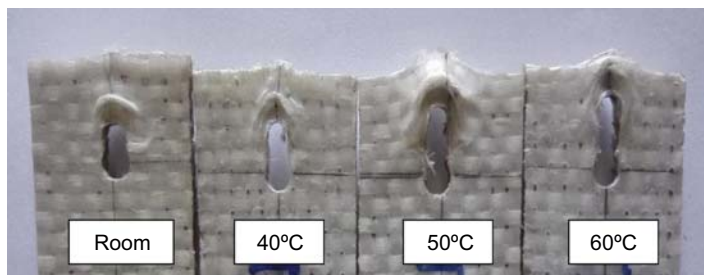


Figure 4.7 Failure mode of hybrid composite at various temperatures and preload moment of 0 Nm.

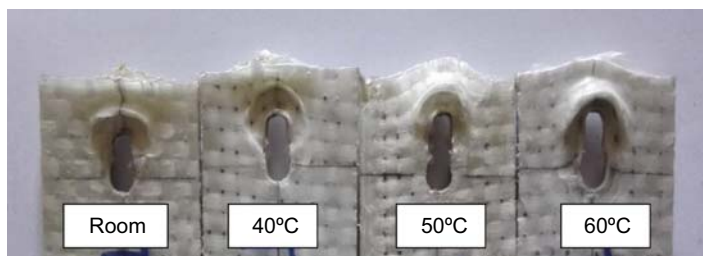


Figure 4.8 Failure mode of hybrid composite at various temperatures and preload moment of 5.2 Nm.

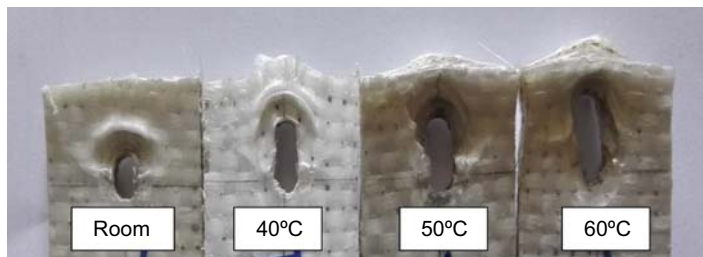


Figure 4.9 Failure mode of hybrid composite at various temperatures and preload moment of 7.5 Nm.

4.6 Conclusions

The structural applications undoubtedly involve bolted joints to assemble the materials together. Based on the studies that have been conducted, it can be concluded that the failure strength and the failure modes are mainly dependent on the ambient temperature and geometrical parameters of the composite materials. The increase of

geometrical ratio improved the failure strength of composite materials regardless of the types of fibers. In spite of the higher failure strength resulted from the higher magnitude of preload moment, the failure mode of composite materials is independent on the preload moment. This could be due to the efficient load transfer between the joint members through the sliding friction. Numerous failure criteria, which can be classified into interaction or phenomenological criteria, have been used to predict the damage initiation of composite materials either in microscopic or macroscopic scales.

The increasing consciousness and awareness toward the utilization of greener materials have led to the development of advanced materials such as natural/synthetic hybrid composites. Due to the presence of natural fibers, the notch sensitivity is reduced as the stress can be further distributed to the adjacent layers of the natural fibers through the sliding friction. This characteristic is particularly vital in the applications where bolted joints are required. To allow more understanding regarding the failure behavior of hybrid composites under bolted joint test, a thorough investigation of the fundamental mechanism and failure strength is necessary. Thus, this chapter gives an overall information in terms of the application of bolted joint, failure analysis on the bolted joint, failure criteria for composite materials, and the factors influencing the properties of the bolted joint. Current research studies are inclined toward the bolted joint behavior of glass fiber-reinforced epoxy composites as mentioned in the previous section. The exploration of greener materials such as hybrid composites in terms of bolted joint properties is still very limited. Further investigation on the hybrid composites is necessary to overcome the knowledge gap and reduce the dependency on synthetic fiber.

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Failure analysis and the optimal toughness design of sheep—wool reinforced epoxy composites

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5.1 Introduction

Fiber-reinforced polymer composite materials are well known for their high mechanical performance characteristics, and they have a high specific modulus, strength, energy absorption, and poor delamination toughness [1]. Due to these properties, laminated composites have been widely used in critical structures, such as aircraft, spacecraft, etc. [2]. Failure in the polymer composites is dependent on several factors. While the matrix, fiber, and fiber-matrix interface constituents have a noticeable impact on the delamination, initiation, and progression of crack, there are several design factors that also play a significant role. These include the composition, composite, and crack size [3]. Delamination of layered fabric-reinforced polymer composite materials characterizes one of the most dominant structural failure modes. The growth of this delamination results in the loss of stiffness and eventually in catastrophic failure. Hence, improved resistance to delamination should assure an extended service life and better mechanical performance of these advanced materials [1]. For most engineering applications, intralaminar fracture toughness is the main parameter used to qualify material systems and is an input parameter for failure analysis [4]. The flaw-free material is extremely difficult to produce, and thus in failure analysis, cracks may be introduced during service. Understanding the crack resistance ability is thus essential [5]. Failure analysis on the microlevel depends on the stress state of the material sample. For example, stress conditions dominate when under tensile load, whereas the failure of ductile material is mainly due to the nucleation, growth, and coalescence of voids [6]. In the design of composite structures open-hole (notched) laminate strength prediction is very necessary. The open-hole tension (OHT) strength of a laminate is an important aspect of the ultimate allowable strength and fracture toughness for composite structure design. Geometrical factors such as the diameter width and thickness of the laminate can play significant roles in the failure modes of OHT laminates [7]. A structural is not homogeneous, and anisotropy of fiber-reinforced composites makes it difficult to use fracture mechanics approach for modeling of the composite structure. An understanding of these design considerations presents significant chances for optimizing composite design to maximize their failure properties to meet the application

requirements [4]. Subsequently, the statistical approach has received more attention than the fracture mechanics approach [8]. Optimization is an important step to find the optimal process parameters based on the results. There are many optimization tools like the gradient search method, the finite element method (FEM) neural network method, and the Taguchi method [9]. Taguchi design is a systematic tool for optimization and for analyzing the influence of control factors on its response [10]. This method was developed for experimental design to investigate how different process parameters affect the mean and variance of a process. Analysis of variance (ANOVA) is used on the collected data to optimize the performance characteristics [11].

In this context, the aim of this study is to produce wool fiber–reinforced polymer composites (WFRPC) with different control factors such as width, thickness, and hole diameter to characterize their failure analysis. OHT composite specimens were prepared as per the ASTM D5766 standard. Very limited results were found during a literature review of failure analysis with natural fiber-reinforced composite using the OHT test. Taguchi experimental design is used to investigate the optimized failure load and fracture toughness WFRPC. Finally, validation of the experimental results with predicted value is done by linear regression.

5.2 Experimental details

5.2.1 *Materials and method*

The natural fiber sheep wool procured from local sources was used as reinforcement with an alkali (NaOH) treatment. The size of wool fiber is around 2–4 mm and matrix material (Lapox L-12) is the liquid epoxy resin based on bisphenol-A and International Union of Pure and Applied Chemistry (IUPAC) oligomeric reaction products named 4, 40-isopropylidene-diphenol with 1-colors-2, 3-epoxypropane [12]. Composite materials 60% sheep wool fabric and 40% epoxy matrix were fabricated by the conventional hand lay-up method [13]. A hydraulic hot press machine was used to prepare the laminated board and fabrication process shown in Fig. 5.1. The roles of the matrix in a fiber-reinforced composite are to retain the fibers in place, transfer stresses between the fibers, and provide a barrier against an adverse environment, such as chemicals and moisture, and to protect the surface of the fibers from mechanical degradation [14]. Length of sheep wool fiber usually ranges from 2 to 35 cm depending on the breed of sheep. In wool processing, wool straight off a sheep contains a high level of grease, which contains valuable lanolin, dirt, dead skins, and vegetable matter. This semigreased wool can be worked into yarn and knitted into water-resistant sweaters. The process of wool from the sheep to the fabric are shearing, cleaning and scouring, grading, sorting, carding, spinning, weaving, and finishing [15].

5.2.2 *Experimental design*

The most important stage in the experimental design is a selection of the control factors. Three parameters, viz., width, thickness, and diameter each at three levels, are

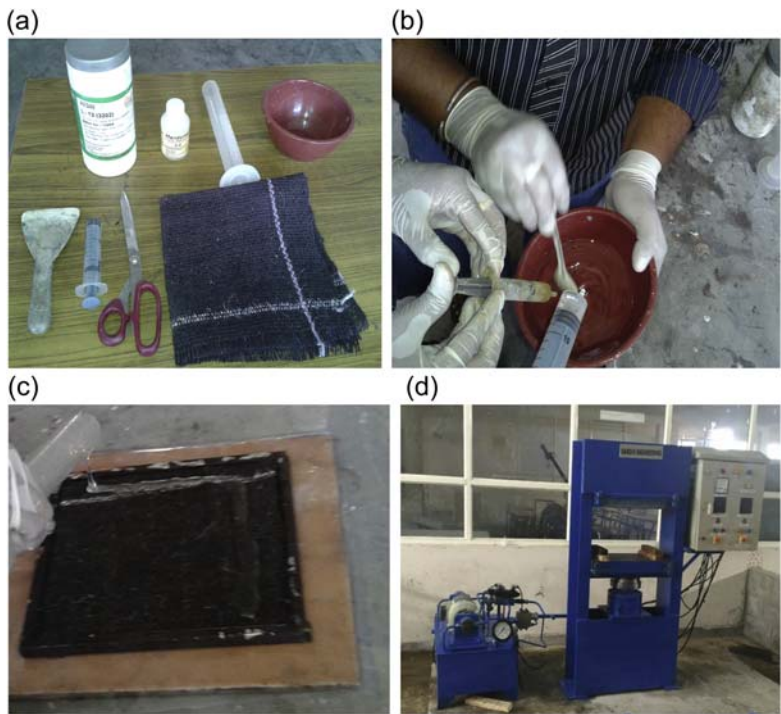


Figure 5.1 Fabrication of laminated composite. (a) Materials and tools. (b) Mixture of epoxy resin with hardener. (c) Placing the layers in the mould. (d) Prepare laminated board to the required thickness (hydraulic hot press machine).

considered in this study, as shown in [Table 5.1](#) in accordance with L9 orthogonal array design ([Table 5.2](#)). A table shows the combination of parameter levels in which each column represents test parameters and each row gives the test condition. The parameters at three levels would require $3^3 = 9$ for the Taguchi factorial design.

5.2.3 Open-hole tension test (OHT)

The open-hole tension test provides values for the tensile strength and fracture toughness. According to ASTM D5766, the composite specimens were prepared for the OHT test as shown in [Fig. 5.2](#). This standard suggests the use of test specimens

Table 5.1 Factors and level combination for open-hole tension (OHT) test specimen

Code	Control factors	Level 1	Level 2	Level 3
A	Width in mm	24	36	48
B	Thickness in mm	7	10	12
C	Diameter in mm	4	6	8

Table 5.2 Experimental design using L9 orthogonal array

Trial	A	B	C
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

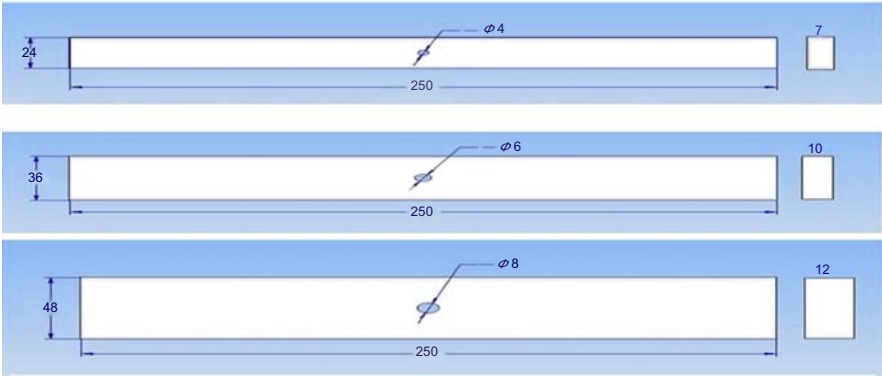


Figure 5.2 Open-hole tension test (OHT) standard sample configurations.

with a length of 250 mm. We can vary width, thickness, and diameter for optimization [16]. The gauge length for each specimen is 200 mm. The specimen was loaded in the universal testing machine until the failure of the specimen. Ultimate load of the composites was measured by determining tensile strength and fracture toughness.

5.3 Results and discussion

Experiments were conducted to describe the tensile strength and fracture toughness in different configurations of the specimen. The OHT test was conducted for different specimen configuration such as varying width, thickness, and hole diameter.

Table 5.3 Experimental results for open-hole tension (OHT) test based on Taguchi orthogonal array (L9)

SL number	Width (mm)	Thickness (mm)	Diameter (mm)	Tensile strength (N/mm ²)	Fracture toughness (Mpa√m)
1	24	7	4	3.57	18.46
2	24	10	6	3.33	19.628
3	24	12	8	4.68	20.23
4	36	7	6	3.80	18.91
5	36	10	8	3.57	23.52
6	36	12	4	4.94	16.114
7	48	7	8	3.93	28.8
8	48	10	4	5.00	20.60
9	48	12	6	5.15	22.76

Experimental results of failure load for each specimen were recorded; from this, tensile strength and fracture toughness was determined by using formula in Table 5.3. The specimen of $W = 48$, $T = 12$, $D = 6$ shows the maximum tensile strength of about 5.15 N/mm^2 , and the specimen of $W = 48$, $T = 7$, $D = 8$ give maximum fracture toughness about $28.8 \text{ Mpa}\sqrt{m}$.

5.3.1 Main effect plots

The main effect plots are the mean response of each level factors connected by the line. When the horizontal line presents, there is no main effect present. When the line is a small deflection from horizontal it may significantly affect the response. Steeper slope in the line illustrates the greater magnitude of the main effect [17]. An effect is defined as the variation in tensile strength and fracture toughness when a factor changes from one level to another. The effect is established by calculating the results at the minimum and maximum values of each factor.

The main effect plots can provide interesting information about the factor influence in the tensile strength and fracture toughness of the WFRPC. The specimens failing by fiber fracture indicated a decreasing strength with increasing hole size, as shown in Fig 5.3. Because initial delamination related with the transverse cracks is observed, for a small hole, they can easily interact, facilitating damage through the thickness and propagating. As the thickness and width increase, it becomes more difficult for delamination at the hole edge [18]. Hence the tensile strength increases slightly on the width and remarkably with the thickness. It is obvious that the fracture toughness of the laminate increases with the increase in the size of the hole, as shown in Fig 5.4.

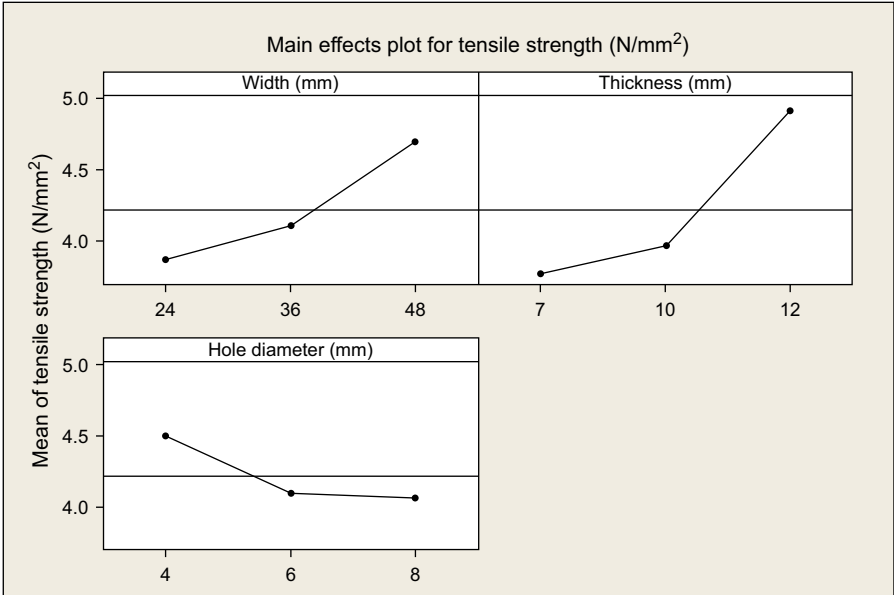


Figure 5.3 Main effect plot for tensile strength (N/mm²).

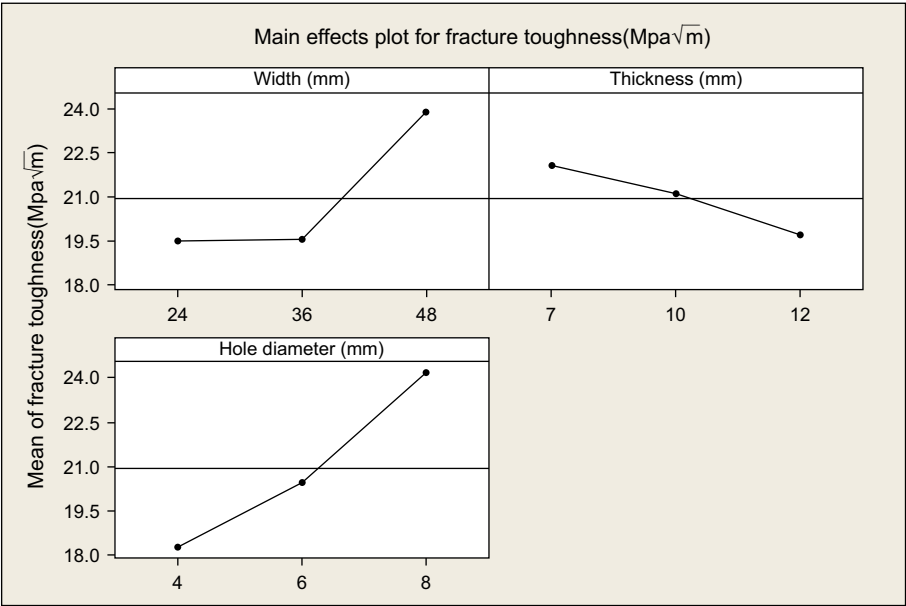


Figure 5.4 Main effect plot for fracture toughness (Mpa√m).

However, for a size of the hole, all wool laminates exhibit highest fracture toughness as indicated by the highest reduction in the failure load. By increasing thickness, the fracture toughness is reduced because of stress concentration will lead to failure of the composite [14].

5.3.2 Analysis of variance (ANOVA) for open-hole tension (OHT) test

Table 5.4 shows the ANOVA for tensile strength. The percentage of the confidence level of width is 26.86%, with a width of 55.91% and a hole diameter of 9.00% on tensile strength. The values clearly show the thickness may affect more and the hole will affect less on load-carrying capacity. The results indicate the tensile strength depends on the thickness of the composite material. The 8.2% of error is due to some fabrication defect.

ANOVA of the experimental results for fracture toughness is shown in Table 5.5. The main factor influencing the fracture toughness is the thickness of 7.77%. The width is 35.72% with a hole diameter of 50.46% confidential level of fracture toughness. It shows that hole diameter has a major effect on fracture toughness. There is a small error about 6.08% due to the fabrication defect.

Table 5.4 Analysis of variance (ANOVA) for tensile strength (N/mm²)

Source	DF	SS	MS	F	P	% of confidence level
Width (mm)	2	1.1018	0.5509	3.24	0.236	26.86
Thickness (mm)	2	2.2931	1.1465	6.74	0.129	55.91
Diameter (mm)	2	0.3658	0.1829	1.07	0.482	9.0
Error	2	0.3403	0.1701			8.2
Total	8	4.1009				100

DF, degrees of freedom; F, variance; MS, Mean square; P, test static; SS, sum of squares.

Table 5.5 Analysis of variance (ANOVA) for fracture toughness (Mpa√m)

Source	DF	SS	MS	F	P	% of confidence level
Width (mm)	2	38.699	19.350	5.87	0.146	35.72
Thickness (mm)	2	8.379	4.189	1.27	0.440	7.77
Diameter (mm)	2	54.660	27.330	8.29	0.108	50.46
Error	2	6.593	3.296			6.08
Total	8	108.332				100

DF, degrees of freedom; F, variance; MS, mean square; P, test statics; SS, sum of squares.

5.3.3 Response surface methodology

Response surface methodology (RSM) is a convenient tool for optimizing processes in the experimental design. The control factors are the input, and the output results are the response variable. A graphical representation of the response with the control factors is called the response surfaces [19].

The response plot of thickness, hole diameter versus tensile strength, and fracture toughness is shown in Figs. 5.5 and 5.6. Increasing the hole diameter decreases the load carrying capacity. This is due to the critical stresses decreasing while the crack length increases [20], and the highest value 5.15 N/mm^2 is found to be at 12 mm thickness and 5.00 N/mm^2 at 4 mm hole diameter. This is because the plastic zone size is considerably larger in thin plates as compared to the thick plate. As the thickness of material increases, the material can withstand more loads [21]. It's clearly observed that fracture toughness decreases with thickness and increases in hole size. It shows

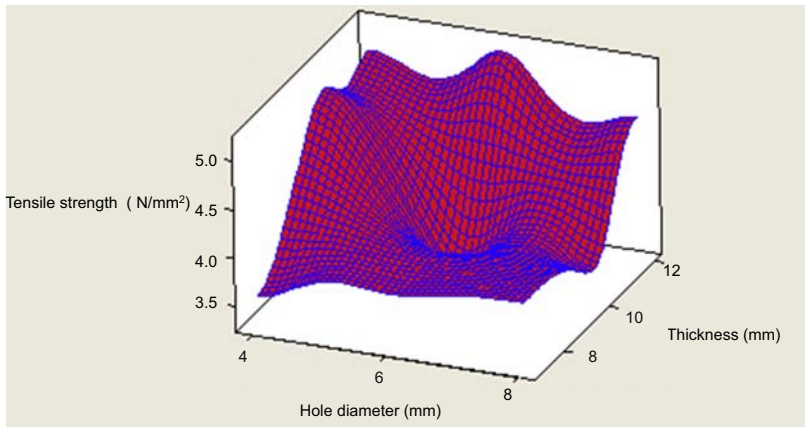


Figure 5.5 Surface plot of tensile strength versus thickness (mm), hole diameter (mm).

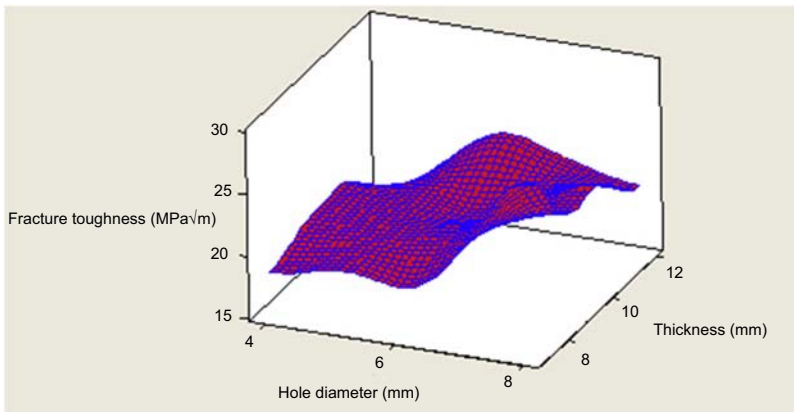


Figure 5.6 Surface plot of fracture toughness versus thickness (mm), hole diameter (mm).

the fracture toughness value $23.52 \text{ MPa}\sqrt{m}$ as the maximum at hole diameter 8 and $28.8 \text{ MPa}\sqrt{m}$ as a minimum value at thickness 7 mm. From the surface analysis, we conclude that fracture toughness of the composite is directly influenced by hole diameter as well as the thickness of the wool fiber–reinforced laminated composite.

5.4 Linear regressions

The confirmation test is the final step in the design of experiment process. The purpose of the confirmation test is to validate the experimental results with predicted values by linear regression. The predicted values can be calculated using mathematical equations, which were developed by the MINITAB software program [22]. The linear regressions equations for tensile strength and fracture toughness as shown in Eqs. (5.1) and (5.2).

$$\text{Tensile strength} = 1.52 + 0.0347(A) + 0.218(B) - 0.111(C) \quad (5.1)$$

$$\text{Fracture toughness} = 9.777 + 0.185(A) - 0.459(B) + 1.49(C) \quad (5.2)$$

A = width in mm, B = thickness in mm, C = diameter of hole in mm.

5.4.1 Experimental validation

Tables 5.6 and 5.7 show the percentage of error from the experimental value corresponding to the predicted values for tensile strength/fracture toughness, respectively.

Table 5.6 Experimental validation of tensile strength (N/mm^2)

Factor combination (width, thickness, diameter)	Experimental tensile strength (N/mm^2)	Predicted tensile strength (N/mm^2)	% Error
24, 7, 4	3.43	3.86	10.9
36, 10, 6	4.283	4.053	5.3
48, 12, 8	4.914	4.55	7.4

Table 5.7 Experimental validation of fracture toughness ($\text{Mpa}\sqrt{m}$)

Factor combination (width, thickness, diameter)	Experimental fracture toughness ($\text{Mpa}\sqrt{m}$)	Predicted fracture toughness ($\text{Mpa}\sqrt{m}$)	% Error
24, 7, 4	18.211	19.89	8.44
36, 10, 6	20.78	20.33	2.16
48, 12, 8	25.53	22.58	9.84

For tensile strength, the difference in error varies from 5.3% to 10.9%, and for fracture toughness, it varies from 2.16% to 9.81%. The optimal combination of 36, 10, and 6 gives the best result for both tensile strength and fracture toughness with a minimum error of 5.3% and 2.16%, respectively.

5.5 Conclusion

In the present study, the optimized failure analysis of wool fiber—reinforced polymer composites have been analyzed experimentally and compared with the predicted values. It can be noted that the composite tensile strength and fracture toughness values are affected by a number of control factors. On the basis of the experimental results and the ANOVA analysis, we discussed the failure of the composite due to the presence of a hole. The obtained results are summarized as follows:

1. The tensile strength increases with thickness (1.5 N/mm^2 to 4.9 N/mm^2) and decreases with the hole diameter (4.5 N/mm^2 to 4 N/mm^2).
2. The fracture toughness increases with the hole diameter ($18 \text{ MPa}\sqrt{m}$ to $24 \text{ MPa}\sqrt{m}$) and decreases with thickness ($22 \text{ MPa}\sqrt{m}$ to $19.5 \text{ MPa}\sqrt{m}$).
3. The thickness of the composite material is approximately 55.91% and the hole diameter is contributing approximately 9.0% of tensile strength.
4. The hole diameter of the composite material is approximately 50.46% and thickness is contributing less than 7.77% on fracture toughness.
5. The percentage of contribution width is 26.86% on tensile strength and 35.72% on fracture toughness.
6. The validation of the experimental results with the predicted values shows that an error less than 10% is acceptable.
7. The RSM graph shows the variation of tensile strength and fracture toughness with control factors.
8. This kind of testing will resolve the problems of rejection in components with holes. Hence, this will be useful in the applications of industrial components.

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Edge crack effect on tensile behavior of diversified wood particulate composites

6

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6.1 Introduction

Reinforcement in a composite material plays a vital role in achieving the desired properties of the composites which suits intended applications. Various forms of fiber reinforcements commonly used in practice are long, short, chopped, micro, and nano level. Out of all the reinforcements, particulate forms of naturally discarded materials are available. Such particulate materials of reinforced composite experimental performance are studied in detail and described in this chapter.

Sawdust particles of 100–300 μm were obtained from carpentry and wood-working processes, used as reinforcement along with the impact modifier, chemical coupling agents in polypropylene matrix. The obtained composites were characterized for mechanical, morphological properties [1]. Block copolymer (Tipplon K948 grade) based wood flour (EFC 1000 grade, 70–150 μm) composites were prepared to investigate micromechanical deformations along with acoustic emission and volume strain measurement [2]. Polypropylene block copolymer with ethylene (8%–11%), wood flour of 210 μm size were combined with interfacial modifications to determine processability, homogeneity, water absorption and mechanical properties of the composites [3].

Through benzylation treatment the surface of the wood flour was modified and reinforced into polypropylene for composite preparation. The work focused on the determination of reaction time influence on degree of modification, effect of reaction on the structure, and properties of wood flour [4]. Commercial grade polypropylene copolymer was combined with 20 μm diameter, 0.5–5 mm length to make untreated composites. Further the effect of styrene-butadiene rubber: impact modifier, malated polypropylene: compatibilizer on composites was studied in terms of tensile, impact properties, and morphological observations [5]. PROPILVEN (PP J600) matrix, hardwood flour, i.e., mahogany, cedar, pine, oak and saki-saki with an average particle size of 20; 40 mesh size was mixed in the preparation of composites for testing mechanically and thermally [6].

Dynamic mechanical analysis, compression, and three-point bend tests were performed on alkali treated eucalyptus saligna wood flour of 250 mesh, 57 μm

reinforced unsaturated polyester composites [7]. Above reinforcement, particles passed through 100 mesh sieve, i.e., average particle diameter of 147 μm was esterified with malic anhydride polypropylene powder along with the compatibilizer, which were used in preparation of composites along with the reinforcement of wood flour to conduct tensile, flexural, and impact tests, and show creep behavior in order to assess long-term loading conditions [8]. Polypropylene composites had two different wood fibers with an aspect ratio about 30 were examined mechanically and compared with the fibers of low aspect ratio [9]. By using rule of mixtures equation elastic modulus of kenaf fiber, wood flour, and polypropylene hybrid composites were determined [10].

Rice husk flour based polypropylene homopolymer composites were made to understand tensile and impact properties [11]. The above composite system was studied by the introduction of compatibilizing agent to investigate its effect on interfacial bonding between hydrophilic wood flour and hydrophobic polypropylene [12]. Through alkaline pulping process banana microfibrils were produced and were reinforced to polyethylene to investigate physicomechanical properties [13]. SRM 100 N grade polypropylene, modified chopped jute fibers were used to study tensile, thermal, and abrasive wear behavior of composites [14]. Sugarcane fiber/glass fiber (chopped form) reinforced polyester composites were studied for the tribological properties [15]. Nutshells of organ particles were reinforced into polypropylene by compounding and casting to characterize tensile properties [16]. Untreated and pre-treated residues from lingo cellulosic raw materials i.e., eucalyptus wood, ground corn cob, and brewery grain were reinforced into low-density polyethylene by melt mixing for the determination of tensile, hardness properties [17]. Particulate fillers like clay and carbon black were used along with the jute fiber reinforced natural rubber composites. The role of silica, carbon black in adhesion/failure criteria of the composites was studied with scanning electron microscopic images [18].

One-centimeter kenaf fibers were reinforced into polypropylene to get composites and tested for tensile, izod impact (unnotched, notched) properties [19]. Short coir with an average diameter of 100–400 μm was chemically modified and reinforced into natural rubber to make composites. Then the addition of silica in the composite system components bonding was investigated along with the fiber orientation [20]. Alkali, dicumyl peroxide, benzoyl peroxide, and permanganate-treated short sisal fiber reinforced LDPE composite sheets were prepared by extrusion and their tensile properties were determined [21].

Miscanthus giganteus/china reed is a woody grass species. Alkali-methanol-anthraquinone pulped fibers reinforced cellulose diacetate composites were characterized for tensile properties [22]. Ground coconut shells with a particle diameter of 50–200 μm were reinforced into epoxy to make composites and were tested for tensile and flexural properties [23]. Theories to predict strength and modulus of particulate polymer composites were discussed. Size, shape, and distribution of inclusion plays key role in macroscopic behavior of composites [24]. Many authors have reported silicate/CNT-reinforced polymer composites, where fillers have a high aspect ratio. Further effects of particle size interfacial adhesion between particles of matrix, stiffness strength, and toughness of the composites having low aspect ratio

fillers were critically discussed [25]. Homo polymer polypropylene—microcrystalline cellulose fiber composites were characterized for thermal and thermomechanical properties [26]. Fully green composites (starch/cellulose nanofiber) were prepared by hot pressing. The mechanical properties of the composite were evaluated along with SEM study [27].

6.2 Discarded particulate natural fiber materials

The present work has focused on collecting a fine wood powder from different trees in a saw mill specially. Heavily grown trees like Teak, Babul, Subabul, Balharshah Teak and Neem are used for various domestic/industrial applications. During preparation of a product/item/article pertaining to specific application wood is cut into number of pieces which result in wood powder formation. Hence the powder obtained from each tree is carefully collected and preserved in polyethylene bags at room temperature. Basic information of common names, botanical names, composition, etc. with regard to these trees is given in the following sections.

6.2.1 *Teak tree*

Teak belongs to verbenaceae family and its vernacular names are tekku-telugu, saguan-hindi, saka-sanskrit, and iti-tamil. Botanically, teak is called *tectonagrandis* and is one of the most invaluable timber yielding plants grown in Central India, Jhansi, Assam, Bengal, Orissa, Sikkim, Saharanpur, Warangal, Adilabad, Medak, Khammam, Cuddapah/Kadapa, Kurnool, Karimnagar, Anantapur, and Nizambad.

It is one of the deciduous plants grown from medium to large height. Teak plants grow/thrive in reasonably good moist, warm tropical conditions [28].

6.2.2 *Babul tree*

Acacia Arabica is the scientific name of babul tree. In 1773 Linnacus first described *Acacia* as belonging to Leguminosae family and subfamily of Mimosoideae. According to the estimates, 1,380 species of *Acacia* exists in world. The common names of *Acacia* are Black Piquant, Casha, Cassie, and Indian Gum Arabic Tree. Regional names of the tree are Black Babul, Indian Gum Arabic Tree—English, Kikar—Hindi, Sak—Punjab, NallaTumma, Tumma—Telugu. The tree is of 2.5–20 m long with dense branches connected to the black colored stems Saurabh Rajvaidhya [29].

6.2.3 *Subabul tree*

Subabul is the Indian Hindi name, and the tree flowers from June to August. This tree belongs to Fabaceae family and botanically called *Leucaena Leucocephala* (Lamk.). In English the tree is commonly called horse tamarind white popinae [30]. These trees grow speedily and are used to revegetate deforested tropical land. It fixes one of the micronutrients in the soil, i.e., nitrogen [31].

6.2.4 Balharshah Teak

This is one of the class of teak trees (refer [Section 6.2.1](#)) grown in India. Wood from this tree is extensively used in household and furniture applications. When the pieces of Balharshah Teak are cut using any wood turning/cutting machines fine particulate powder is obtained.

6.2.5 Neem tree

The Neem tree is the native plant of India and Sri Lanka grown at temperature normally range from 21 to 32°C. The leaves of the tree have various medicinal uses. Neem plant is botanically called *Azadirachta Indica*. Leaves of the plant resemble pinnate 200–400 mm long and 20–30 leaflets of about 30–80 mm, short petioles [32]. It is one of the sacred plants used for devotional activities in India. One of the major uses of this plant is the generation of fresh air by purifying the polluted one, utilization of wood in house for preparation of doors, windows, etc.

6.3 Preparation of particulate composites and testing

Particulate material has the tendency to agglomerate when it is mixed with the liquid resin. So, a low-viscous polyester resin, i.e., Ecmalon 4421, is purchased from Ecmal Resins Hyderabad, Telangana, India and is used as the matrix, and Methyl Ethyl Ketone Peroxide cobalt as a catalyst and accelerator, respectively along with the resin. By using two stages of the hand lay-up method [33], the particulate reinforcements such as Teak, Babul, Subabul, Balharshah Teak, and Neem are successfully mixed with the polyester resin for preparation of the composites.

Tensile, Impact test composite specimens are prepared according to the dimensions of ASTM D 5083-02 and ASTM D 6110-08 respectively. The specimens are tested on Electronic Tensometer (PC 2000 model) further an edge crack of about 5 mm is cut on tensile test specimens to assess the impact of edge crack on tensile properties of composites. Moreover, the thickness of the specimen at five different sections along the length is taken. In addition to the measurement of thickness along length, at each section, three different positions in transverse/width direction is also considered for thickness measurement. The average value of thickness of each tensile test specimen is calculated from the measured data.

6.4 Results and discussion

This section elaborates tensile properties and impact strength of different particulate fiber-reinforced composites. Further, the effect of specimen thickness and edge crack on the tensile performance of the composites is describes. Lastly practical issues associated in fabrication of particulate reinforced composites and the possible reasons of poor performance of composites are also explained.

6.4.1 Tensile and impact properties of particulate composites

Large interest and focus was made to incorporate natural fibers like flax and hemp in polypropylene for various automotive applications of Europe. In the areas of window frames/decking (United States and Europe) wood flour—placed thermoplastics were used in place of wood itself [34]. Another class of materials i.e., thermoset filled composites plays a key role in automobile and structural applications. Two phenomenological steps, i.e., gelation and vitrification, occurs during curing/polymerization of a thermoset polymer. Filler can change the mechanical properties—hardness, stiffness, strength, toughness, thermal properties—such as expansion, conductivity, and thermal decomposition of thermoset resin. The size of a particle affects the mechanical properties of composites to a larger extent [35].

Particulate fiber composites of different trees powders are tensile tested, and the average value of its properties is indicated in Figs. 6.1 and 6.2. The tensile strength of the composites is in the increasing order of Teak, Balharshah, Subabul, Babul,

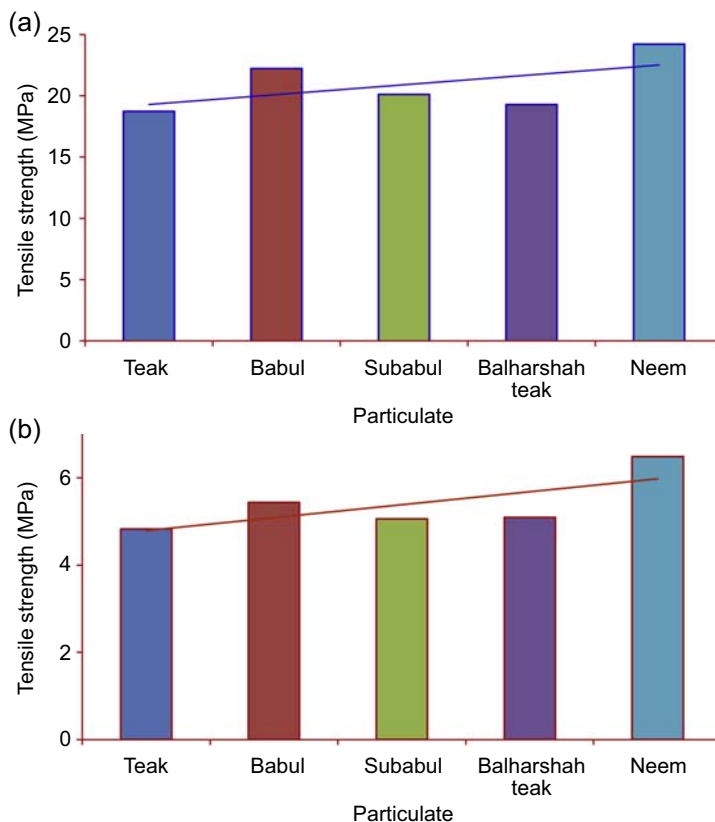


Figure 6.1 Tensile strength of diversified particulate-reinforced polyester composites without and with edge crack.

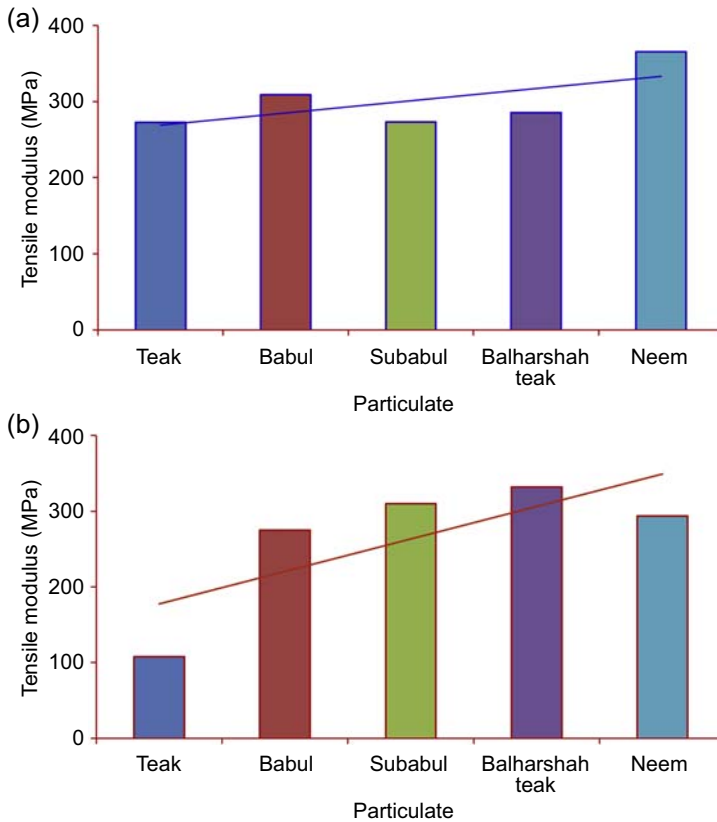


Figure 6.2 Tensile modulus of diversified particulate-reinforced polyester composites without and with edge crack.

and Neem. The highest tensile strength of 24.22 MPa (Fig. 6.1) is achieved in case of Neem particulate fiber-reinforced polyester composites.

Variation in elongation is exhibited by the particulate composites when compared with the load result and a minor change in trend of tensile modulus with respect to tensile strength. Here also Neem particulate fiber-reinforced polyester composites have shown the highest tensile modulus of 365.24 MPa, Fig. 6.2.

Polyester composites reinforced with minimum microfibrillated cellulose (MFC, average diameter: 52.92 μm) content of 1.84% had exhibited highest tensile strength of 22.61 MPa [36]. The value achieved in this work is on par with almost all composites and is 7.12% higher in case of Neem fiber-reinforced polyester composites.

Particulate composite specimens are tested under impact load of the Charpy type. The average impact strength of 10 tested specimens is calculated for obtaining a more accurate result. Impact strength of composites varied from 1.65 to 1.91 KJ/m^2 ; see Fig. 6.3. Out of all the composites experimentally studied in this work, Babul particulate polyester composite showed the highest impact strength of 1.91 KJ/m^2 .

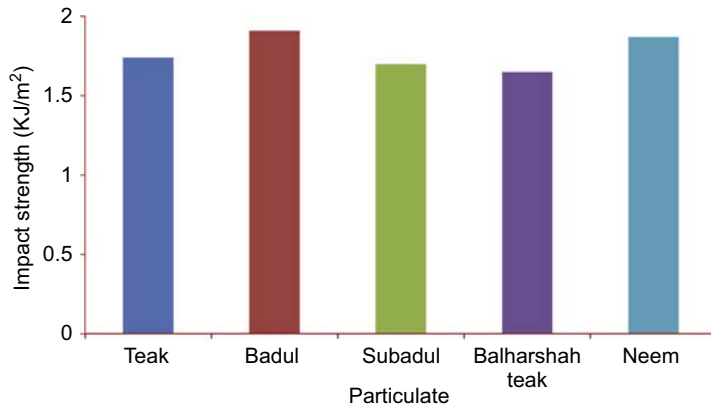


Figure 6.3 Impact strength of diversified particulate-reinforced polyester composites.

Charpy impact strength of 19.37 KJ/m² was achieved in MFC epoxy composites made by Just Mold Fill Immediate Loading (JMFIL) [37]. But the impact strength of composites investigated in this work have shown poor performance and the possible reasons are detailed later in this chapter.

The morphological examination of the tensile and impact-tested specimens is discussed here, and overall, all the types of particulate wood reinforcements are distributed in the matrix more or less evenly and are seen from the SEM images (Figs. 6.4–6.13). From the tested specimens the highest load-bearing specimen is selected for the SEM examination. Teak, Babul, Subabul, Balharshah, and Neem particulates have the diameters 1.43–10.7, 3.3–24.4, 1.79–17.1, 1.71–7.85, 2.15–15 μ m, respectively, and are able to be observed from Figs. 6.4d, 6.5e, 6.6, 6.7, and 6.8d. Similarly, impacted-tested composite specimens with different particulates, viz., Teak, Babul, subabul, Balharshah, and Neem particulate, have the diameters 1.60–23.82, 1.83–11.7, 2.29–8.26, 1.71–7.85, 2–6.13 μ m, respectively, and are shown in Figs. 6.9d, 6.10c, 6.11, 6.12, and 6.13d.

6.4.2 Effect of specimen thickness, edge crack on particulate composites

The thickness of Teak, Babul, Subabul, Balharshah, and Neem particulate fiber-reinforced polyester tensile test composites is given in Tables 6.1–6.5. Although much care is taken in the preparation of composite lamina, a slight unevenness of the table where the mold is placed resulted in uneven lamina thickness during curing. The variation in thickness of the specimens machined from the lamina is responsible for failure of the edge crack specimen not at the position of the crack.

Generally, any specimen has to fail at the crack only when it is loaded under tension. Figs. 6.1b and 6.2b show tensile strength and modulus of edge crack at different particulate polyester composites. Under these conditions Neem and Subabul particulate composites show the highest tensile strength (6.49 MPa) modulus (309.99 MPa), respectively. But the lesser of or variation in thickness has made the

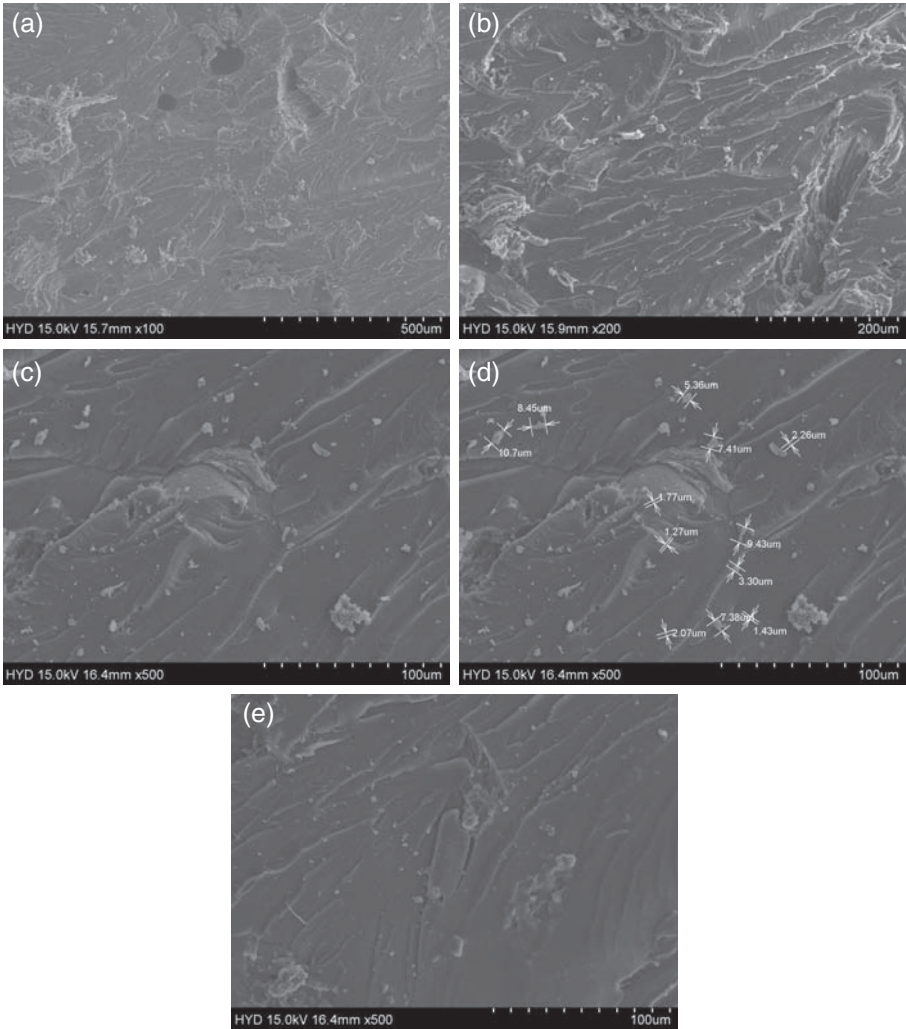


Figure 6.4 Scanning electron microscopy (SEM) images of tensile-tested Teak particulate-reinforced polyester composites.

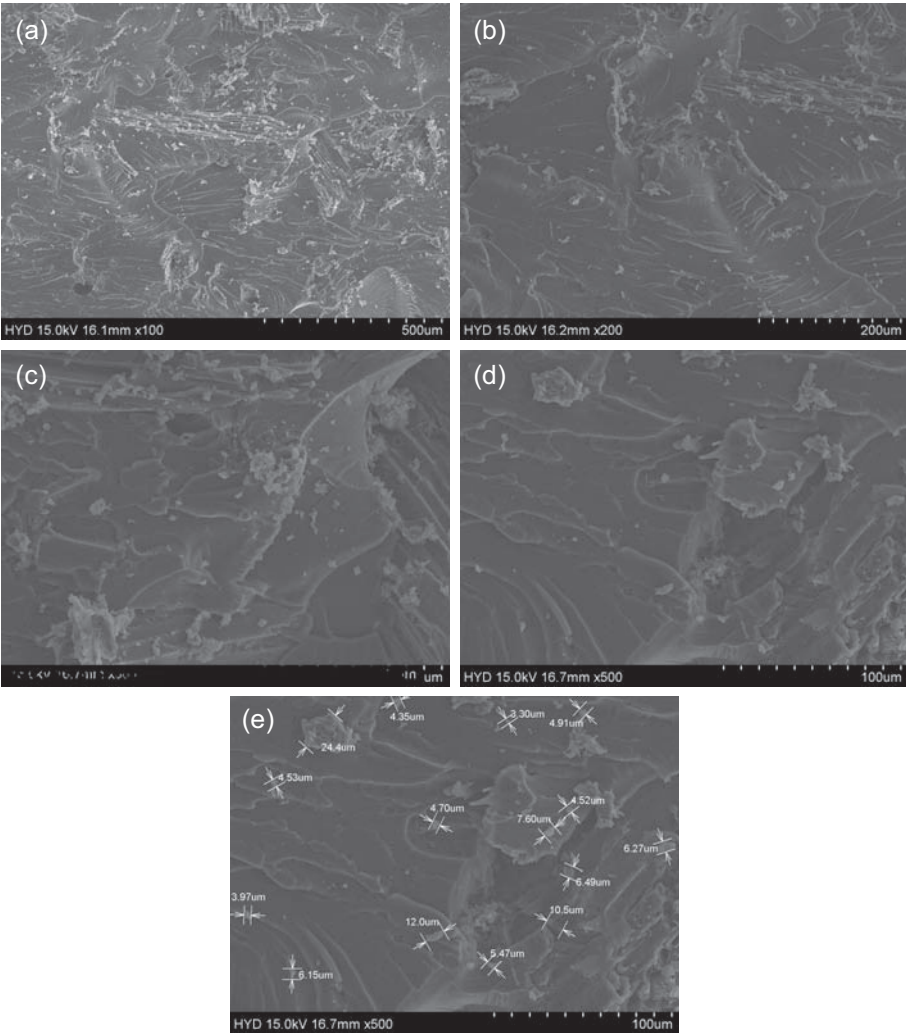


Figure 6.5 Scanning electron microscopy (SEM) images of tensile-tested Babul particulate-reinforced polyester composites.

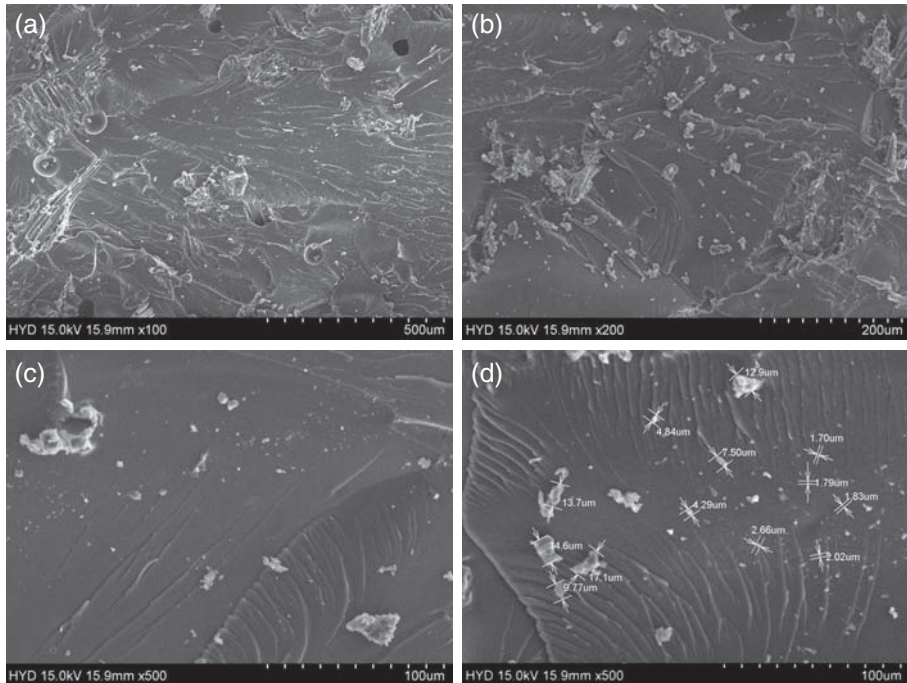


Figure 6.6 Scanning electron microscopy (SEM) images of tensile-tested Subabul particulate-reinforced polyester composites.

composite to fail in that cross-section. Hence, in a composite structure, crack formation and a slight variation in thickness may be quiet dangerous under normal working conditions. The specimens failed in the above said manner is shown in [Fig. 6.14](#).

6.4.3 *Practical problems of particulate composites manufacturing*

To minimize the problem of mixing the particulate properly and to avoid the agglomeration of them, a preliminary step is taken in selecting low viscous transparent polyester resin, i.e., Ecmalon 4421. The resin has the viscosity of 220 centipoise, specific gravity 1.11, gel time 25 min at a temperature of 25°C, as per the data provided by the manufacturer.

Although mechanical stirring is employed at 1500 rpm, wood particulates tend to entangle among them during gelation of polyester resin. Further, all the reinforcements more or less reach the top surface of the mold after about 15 min and are clearly visible from the manufactured/tested composite specimen, [Fig. 6.15](#). The top layer of the specimen bears tensile/impact load rather than the whole specimen. It is presumed that the particulate in the composite lamina should present evenly. But in practicality, the above nature is lacked by the reinforcement, which may result in variation in performance of the composites under mechanical loading.

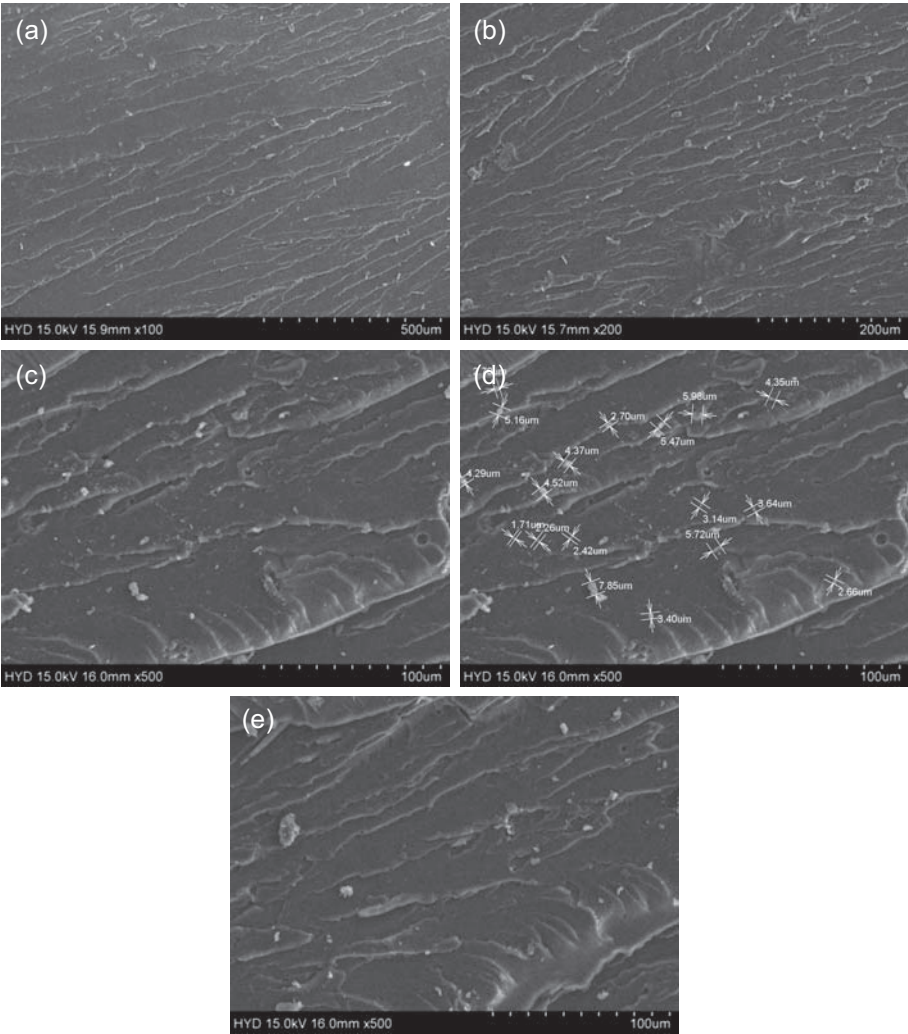


Figure 6.7 Scanning electron microscopy (SEM) images of tensile-tested Balharshah particulate-reinforced polyester composites.

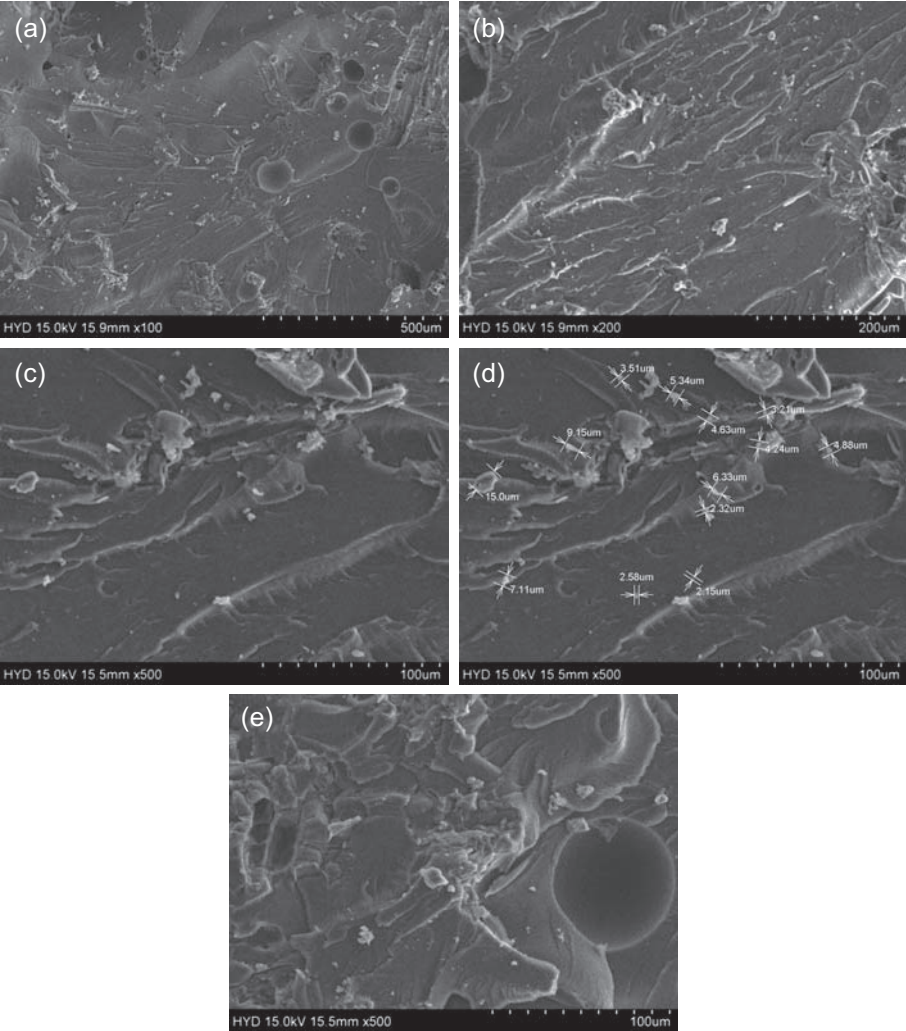


Figure 6.8 Scanning electron microscopy (SEM) images of tensile-tested Neem particulate-reinforced polyester composites.

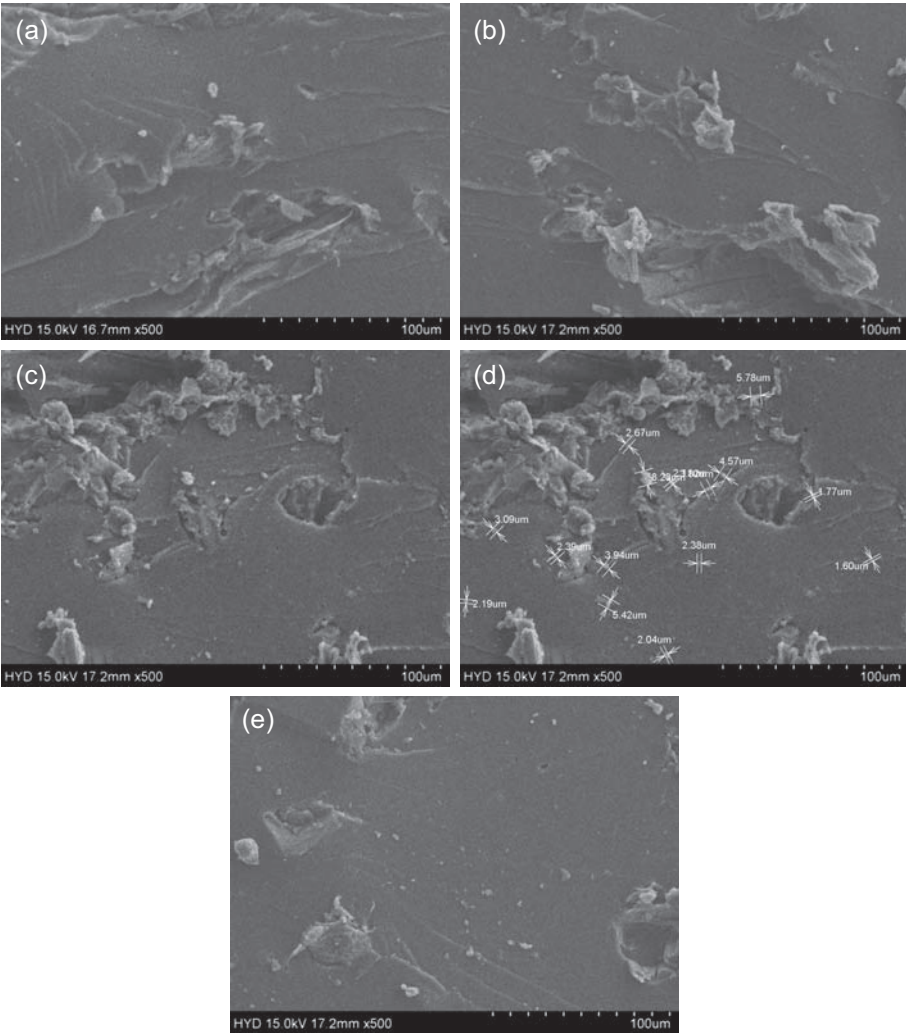


Figure 6.9 Scanning electron microscopy (SEM) images of impact-tested Teak particulate-reinforced polyester composites.

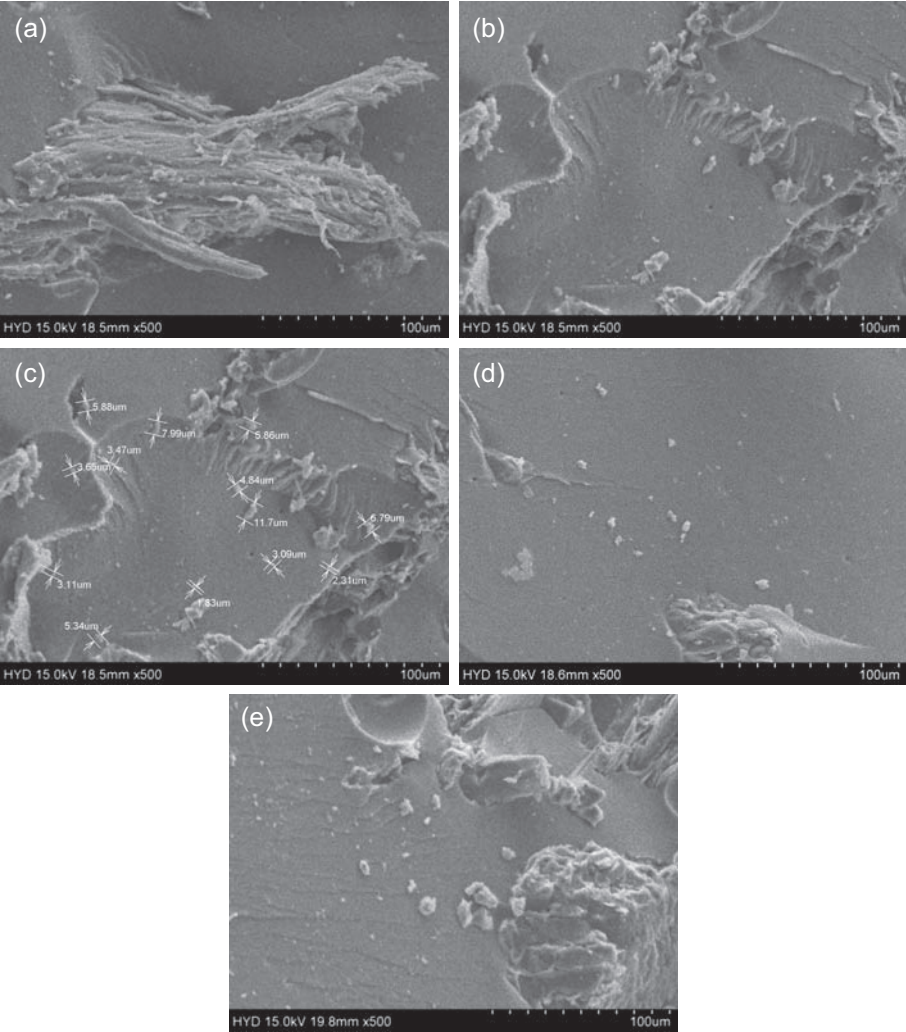


Figure 6.10 Scanning electron microscopy (SEM) images of impact-tested Babul particulate-reinforced polyester composites.

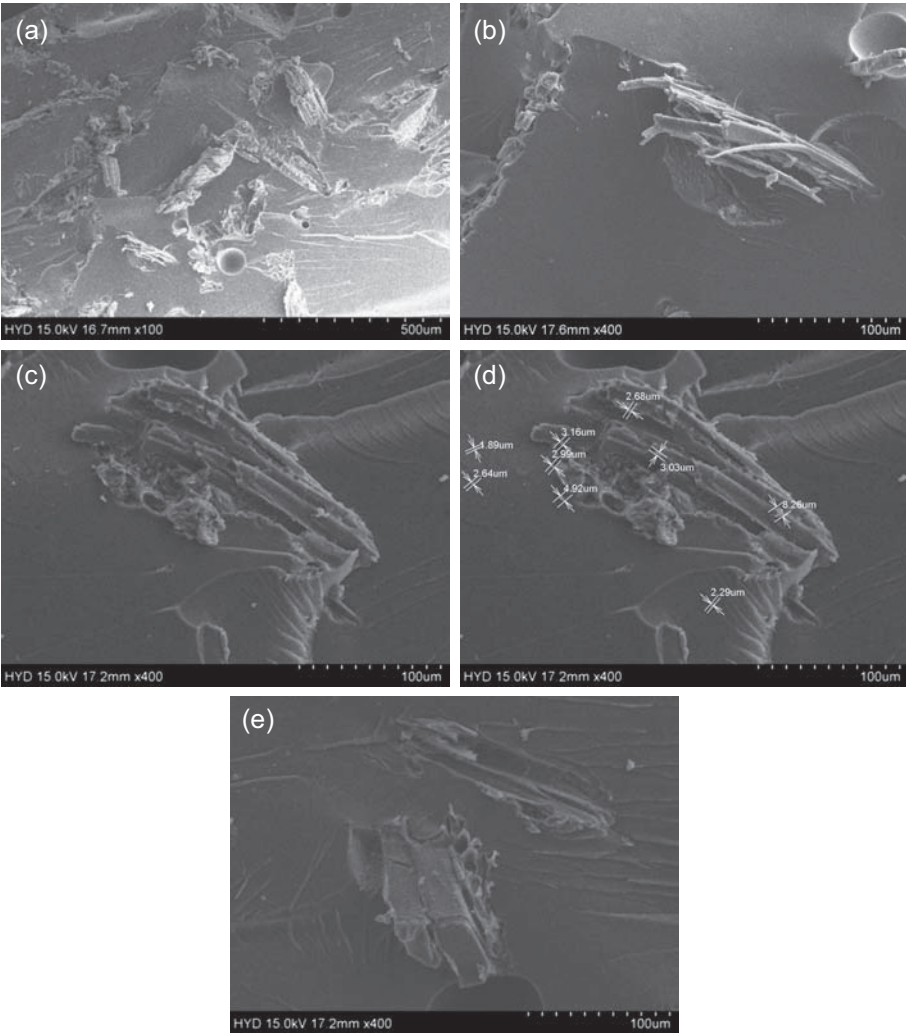


Figure 6.11 Scanning electron microscopy (SEM) images of impact-tested Subabul particulate-reinforced polyester composites.

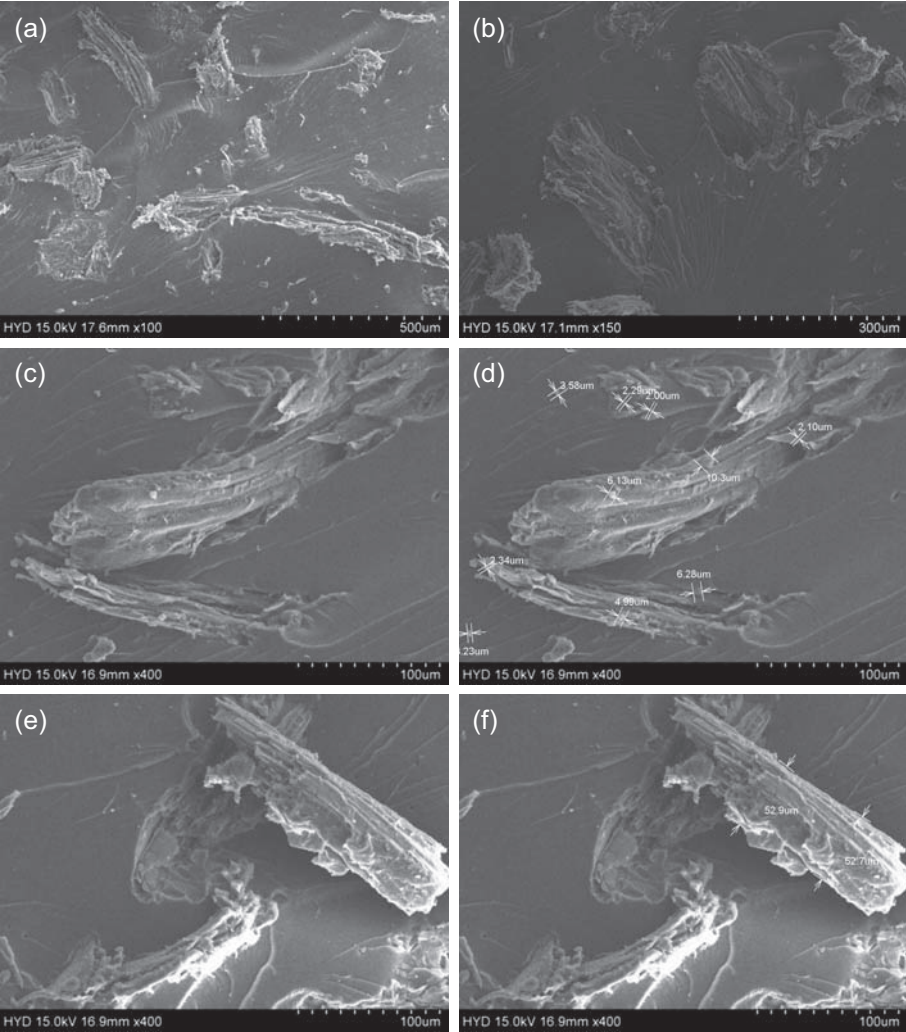


Figure 6.12 Scanning electron microscopy (SEM) images of impact-tested Balharshah particulate-reinforced polyester composites.

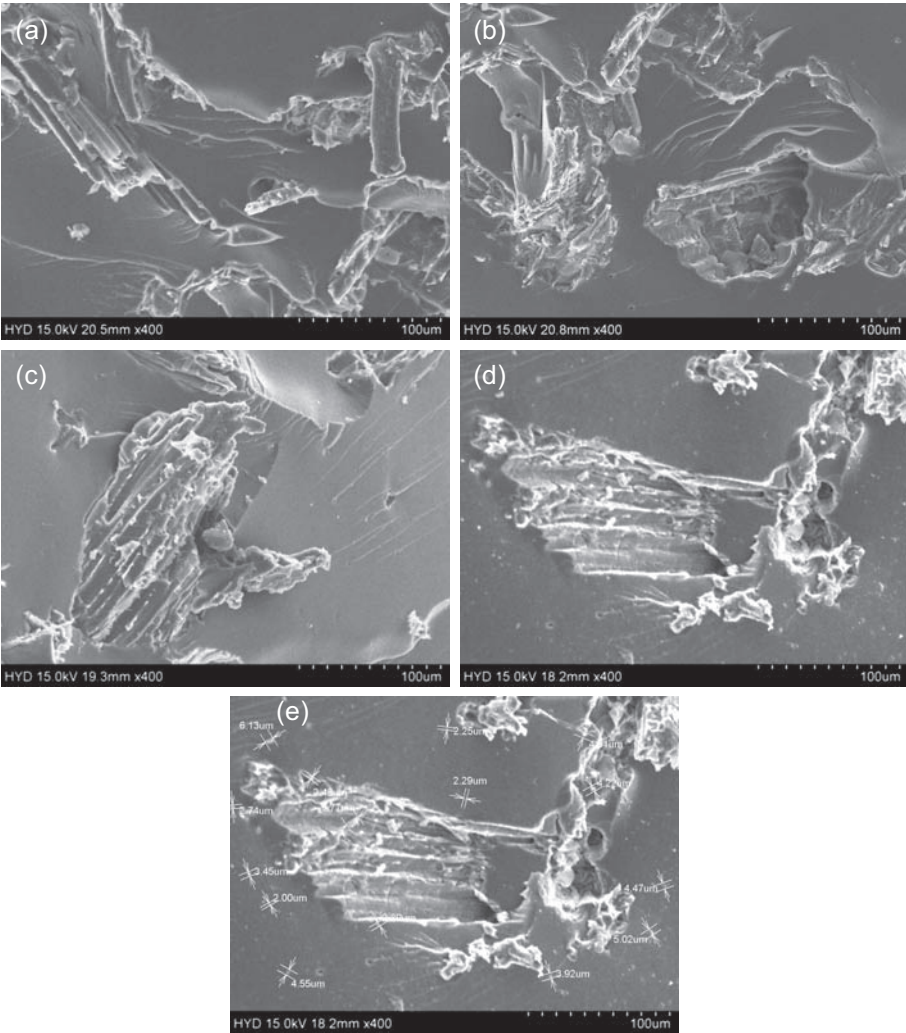


Figure 6.13 Scanning electron microscopy (SEM) images of impact-tested Neem particulate-reinforced polyester composites.

Table 6.1 Thickness of the Teak particulate fiber-reinforced epoxy tensile test composite specimens

Specimen no	Thickness at section 1-1 (mm)			Thickness at section 2-2 (mm)			Thickness at section 3-3 (mm)			Thickness at section 4-4 (mm)			Thickness at section 5-5 (mm)			Average thickness (mm)
	Width position 1			Width position 2			Width position 3			Width position 4			Width position 5			
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	
1.	4.240	4.930	5.115	4.857	5.160	4.861	2.097	2.882	3.935	1.590	1.640	2.197	4.920	4.571	4.335	3.822
2.	3.505	3.428	3.406	5.002	5.216	5.246	4.204	4.023	3.877	4.518	4.830	5.062	4.058	5.248	5.145	4.451
3.	4.219	4.546	4.635	1.473	1.411	1.608	2.822	2.538	2.268	4.746	4.755	4.769	4.533	4.319	4.452	3.540
4.	4.975	4.992	5.005	4.979	5.139	5.144	4.450	4.330	4.042	1.773	2.148	2.733	4.786	4.980	4.929	4.294
5.	4.313	4.302	4.147	4.616	4.754	5.030	4.774	4.511	4.166	4.407	4.369	4.158	5.186	5.193	5.192	4.608

Table 6.2 Thickness of the Babul particulate fiber-reinforced epoxy tensile test composite specimens

Specimen no	Thickness at section 1-1 (mm)			Thickness at section 2-2 (mm)			Thickness at section 3-3 (mm)			Thickness at section 4-4 (mm)			Thickness at section 5-5 (mm)			Average thickness (mm)
	Width position 1			Width position 2			Width position 3			Width position 4			Width position 5			
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	
1.	4.041	4.581	4.977	3.647	4.185	4.791	3.390	3.860	4.655	3.469	3.954	4.485	4.237	4.103	4.326	4.180
2.	4.717	4.745	4.775	3.900	4.504	4.907	3.854	4.040	4.375	4.448	4.612	4.910	4.648	4.764	5.215	4.561
3.	5.324	5.294	5.249	5.361	5.358	5.324	5.358	5.344	5.325	5.175	5.292	5.279	5.104	5.136	5.151	5.272
4.	5.065	5.024	4.578	4.850	4.888	4.906	4.998	5.024	4.889	4.900	5.153	5.203	4.778	5.030	5.305	4.973
5.	4.411	4.389	4.298	4.368	4.475	4.635	4.333	4.323	4.709	4.957	4.985	4.980	5.054	5.137	5.116	4.678

Table 6.3 Thickness of the Subabul particulate fiber-reinforced epoxy tensile test composite specimens

Specimen no	Thickness at section 1-1 (mm)			Thickness at section 2-2 (mm)			Thickness at section 3-3 (mm)			Thickness at section 4-4 (mm)			Thickness at section 5-5 (mm)			Average thickness (mm)
	Width position 1			Width position 2			Width position 3			Width position 4			Width position 5			
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	
1.	2.423	2.894	3.527	2.277	3.443	4.562	2.446	3.839	4.852	3.052	3.992	4.607	4.865	4.900	4.903	3.772
2.	4.411	4.358	4.169	4.805	4.615	4.472	4.383	4.073	3.787	4.467	4.176	4.053	4.993	4.890	4.916	4.438
3.	3.940	3.772	3.626	3.645	3.953	4.157	3.429	3.199	3.450	4.161	4.269	4.275	4.741	4.455	4.296	3.958
4.	4.036	4.009	4.019	3.607	3.779	4.014	4.322	4.488	4.609	4.393	4.479	4.517	4.117	4.509	4.772	4.245
5.	4.255	4.201	4.105	4.376	4.344	4.199	4.639	4.562	4.380	4.542	4.411	4.198	5.007	5.057	4.733	4.467

Table 6.4 Thickness of the Balharsha particulate fiber-reinforced epoxy tensile test composite specimens

Specimen no	Thickness at section 1-1 (mm)			Thickness at section 2-2 (mm)			Thickness at section 3-3 (mm)			Thickness at section 4-4 (mm)			Thickness at section 5-5 (mm)			Average thickness (mm)
	Width position 1			Width position 2			Width position 3			Width position 4			Width position 5			
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	
1.	3.447	3.372	3.368	4.462	4.611	4.579	4.199	4.128	4.118	3.766	3.744	3.903	4.691	4.606	4.268	4.084
2.	4.034	3.939	3.850	3.808	3.595	3.488	3.247	3.291	3.365	4.140	4.135	3.969	4.044	4.972	4.937	3.921
3.	3.344	3.315	3.275	3.606	3.554	3.460	4.301	4.092	3.861	3.573	3.588	3.733	3.975	4.409	4.493	3.772
4.	3.033	3.014	3.157	4.462	3.981	3.588	4.236	4.159	3.998	3.697	3.734	3.739	4.573	4.711	4.676	3.917
5.	4.642	4.258	3.871	4.798	4.109	3.550	4.884	4.292	3.807	4.910	4.360	3.982	5.050	4.884	4.591	4.399

Table 6.5 Thickness of the Neem particulate fiber-reinforced epoxy tensile test composite specimens

Specimen no	Thickness at section 1-1 (mm)			Thickness at section 2-2 (mm)			Thickness at section 3-3 (mm)			Thickness at section 4-4 (mm)			Thickness at section 5-5 (mm)			Average thickness (mm)
	Width position 1			Width position 2			Width position 3			Width position 4			Width position 5			
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	
1.	4.845	4.880	4.912	4.693	4.695	4.857	4.857	4.788	4.747	4.972	4.937	4.922	4.859	4.905	4.833	4.847
2.	4.737	4.652	4.236	4.694	4.615	4.294	4.683	4.598	4.301	4.423	4.536	4.455	4.582	4.491	4.289	4.506
3.	4.763	4.815	4.885	4.715	4.803	4.930	4.773	4.749	4.797	4.895	5.094	5.047	4.720	4.822	4.883	4.846
4.	4.981	5.001	4.977	4.868	4.673	4.240	4.795	4.712	4.178	4.865	4.786	4.768	4.992	4.792	4.266	4.726
5.	4.380	4.699	4.756	4.392	4.041	3.638	4.414	4.323	3.928	4.070	3.710	3.387	4.319	4.344	4.254	4.177

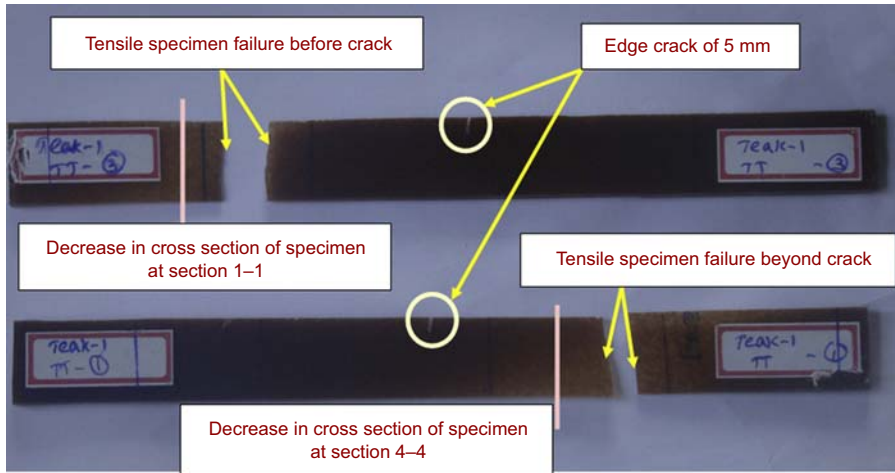


Figure 6.14 Shift of failure in edge crack Teak specimens under tensile load.

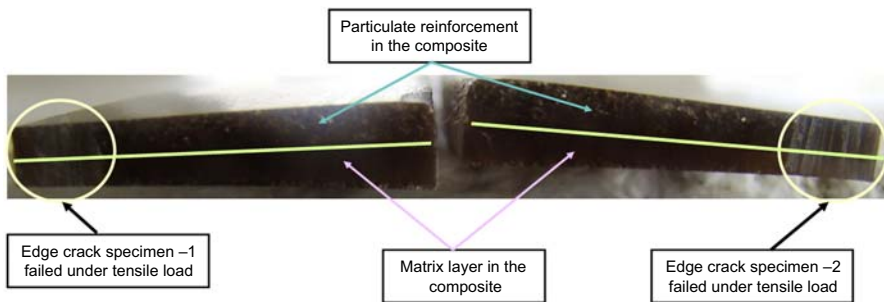


Figure 6.15 Settlement of low-dense wooden particulate reinforcement in polyester composite at the top and bottom layered with matrix.

6.5 Conclusions

Different wooden particulate (Teak, Babul, Subabul, Balharshah, and Neem) polyester composites are successfully manufactured by a two-stage wet lay-up technique. To the practical possible extent, agglomeration of particulate in liquid polyester resin is avoided due to mechanical stirring and is evidenced from SEM. A change in the failure position of the edge crack particulate composition specimen is observed due to variable composite thickness. This class of lightweight particulate composites is suitable in diversified automotive applications.

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Investigation of mechanical testing on hybrid composite materials

7

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7.1 Introduction

Hybrid composites consist of more than two materials combined together to form a new material with better characteristics than the original materials. The composite material consists of a reinforcing fiber which is woven ply, and a matrix material that bonds the plies together and gives stiffness which shapes the fibers into structures. These special properties differ from those of metallic alloys since each of the materials in a composite still maintains its original chemical, physical, and mechanical properties. As a recent trend, composites are widely employed in the aerospace industry. Basically there are two types of fiber composites that have been used in the aerospace industry, which are carbon/epoxy and glass/phenolic. The carbon/epoxy is mostly applied to major load-bearing structures, while glass/phenolic is for cabin furnishings.

One of the important properties that can define a good aeronautical vehicle is the “crashworthiness” of the structures. Crashworthiness design requirements can be described as maintaining a survivable volume, maintaining deceleration loads with respect to the occupants, the retention of items of mass, maintaining an egress path, and how the material reacts when it undergoes an impact [1]. The higher the “crashworthiness” of the material, the less damage the material will sustain after an impact. In the history of aviation, the Wright brothers successfully flew an aircraft that used a cast aluminum engine block on the flyer. At that time, the main issues for the main airframe were weight and static, thus they only used wood with a fabric covering [2]. After technologies improved during the mid-1960s to early 1970s, the military started using high performance composites in their aircraft. One of the earliest uses of high performance composites is on the empennages of F-14 and F-15 fighter aircraft. Boron/epoxy was used for the horizontal stabilators on both aircraft, and additionally, on the rudders and vertical fins for the F-15 aircraft [3].

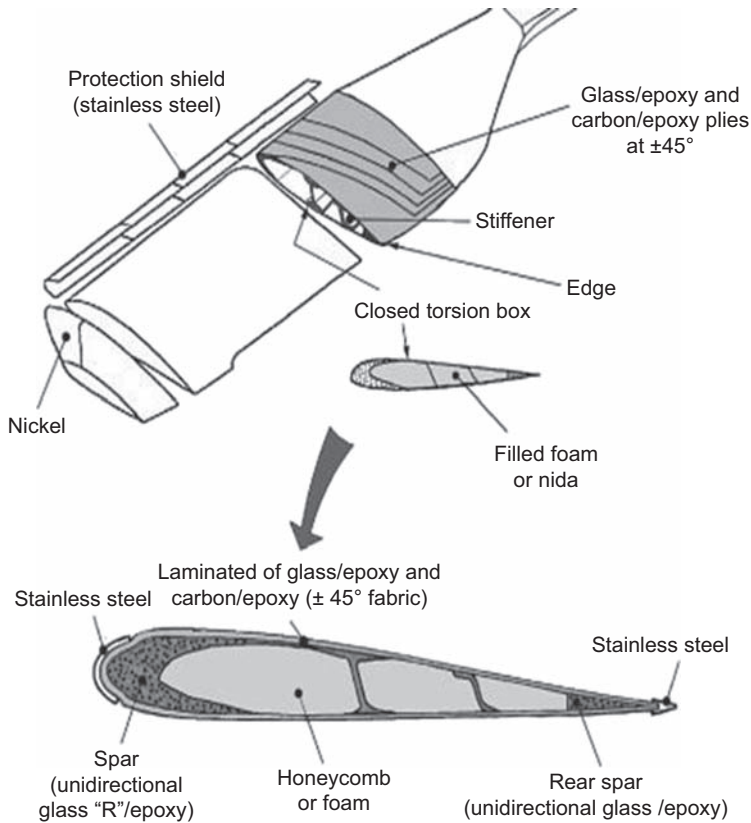


Figure 7.1 Examples of composite materials used in a helicopter blade [4].

Fig. 7.1 shows examples of the application of composite materials in a helicopter blade. The blade consists of different materials to make a complete part. Each layer has special characteristics to fit its own function when a load is applied. Composites are not only used in blades and hubs but implemented in over 50% of the other parts of the rotorcraft structure [4].

When comparing the composites with other materials such as steel and aluminum, there is a significant advantage in terms of strength and stiffness, combined with low density, weight reduction, and reduced maintenance cost. Fig. 7.2 shows the major components of the EC 145 T2 that use composite materials. The new generation of aircraft have a higher percentage of composite materials in the airframe compared to older models. This change in the composition of the materials has greatly improved the performance of each aircraft. There are also positive competitions between companies to continue developing new materials that have better characteristics at a lower cost or make it easier to mass produce the components. Based on the types of reinforcement used, the composites are classified as shown in Fig. 7.3.

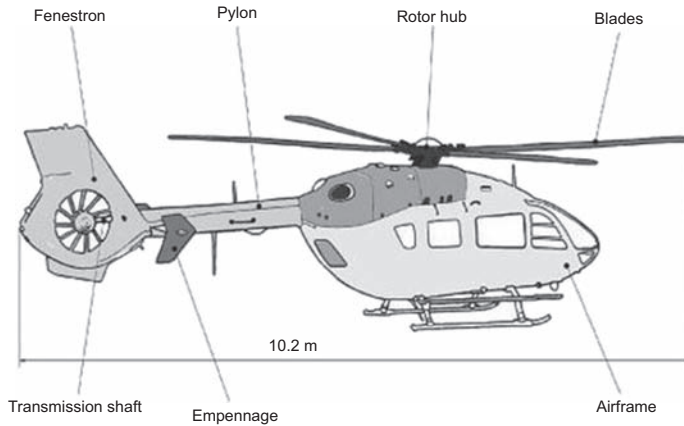


Figure 7.2 Composite components in the helicopter EC 145 T2 [4].

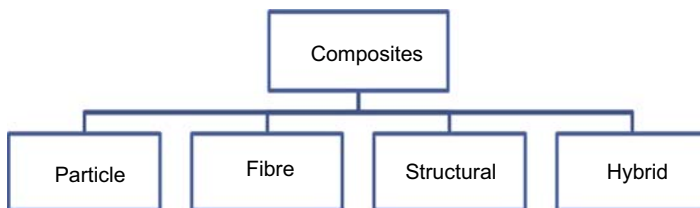


Figure 7.3 Classifications of composites.

The fiber reinforcement provides stiffness and strength to the composite. Fiber composite constituents consist of one or more discontinuous phases called reinforcements embedded into a continuous phase that is called the matrix [5]. The fiber can exist in the form of glass roving, woven fabric, continuous carbon tow, aramid yarn etc. Fibers produce high strength composites because of their small diameter, thus they usually have fewer defects when compared to material produced in bulk.

The matrix is the material that will surround the fiber and protect it from chemicals, mechanical damage (wear), and environmental attack such as humidity. The matrix basically needs to have a lower modulus and greater elongation than the reinforcement, so that it will give the composites a good surface finishing and shape. The factor of failure for a composite is largely affected by the type of matrix used and the compatibility between the matrix and the reinforcement. The matrix used in the composites also contributes to its elongation even though the contribution is not too significant. Matrix selection factors include, for example, chemical, thermal, and electrical properties, as well as flammability, performance, cost, environmental aspects, and manufacturing requirements. The properties of the matrix that affect the properties of the composites are shear strength, tensile strength, and longitudinal compression strength.

An overall view of the advantages and disadvantages of composite materials is as shown in Table 7.1 [6–8].

Table 7.1 Advantages and disadvantages of composite materials

Advantages	Disadvantages
1. Weight reduction (approximately 20%–50%) 2. Fatigue and corrosion resistance 3. Reduced assembly costs 4. High specific stiffness 5. Provide capabilities for part integration. 6. High fatigue strength	1. High material costs 2. Temperature resistance depends on the temperature resistance of the matrix materials 3. Nonvisible impact damage 4. High cost of repairs 5. Absorb moisture that affects the properties and dimensional stability of the composites

The mechanical properties of composites have a range of values even for the same types of material. The reason for this is that there are many different techniques for material production. The method of fabrication will greatly affect the mechanical properties of the material, not to mention the temperature and the curing condition used [9]. To obtain consistent results, the steps involved in the fabrication must be the same for all the specimens. Errors and defects during the fabrication process need to be prevented or minimized to produce the optimum mechanical properties of the material. Table 7.2 shows a comparison of the mechanical properties for a few of the aerospace composites that have been used in many parts of an aircraft.

Table 7.2 Mechanical properties of materials [10]

Material type	Nomenclature	Tensile strength (Ksi)	Modulus (Msi)	Strain (%)	Tensile strength (MPa)
Carbon/Epoxy	T300/934	245	20	1.0–1.2	1689.275
	IM7/8551-7	400	24	1.62	2758
	P75/934	135	44	0.2–0.5	930.825
	AS4/3501-6	100	10	1	689.5
	IM6/3501-6	330	23	1.5	2275.35
Glass/Epoxy	E-glass/934	150–170	6–8	2.75	1034.21–1172.11
Kevlar/Epoxy	K-49/7934	80–85	4	1.85	551.58–586.05
Carbon/PEEK	IM7/APC-2	419	24	1.6	2889.005
Carbon/Phenolic	FM5055	15–20	2.6–2.8	1.0–1.2	103.42–137.90
Cast iron	—	—	—	—	172
Aluminum	—	—	—	—	89.63

7.1.1 Glass fibers

Nearly all continuous glass fibers are made by a direct draw process and are formed by extruding molten glass through a platinum alloy bushing that may contain up to several thousand individual orifices, each being 0.793–3.175 mm in diameter [11]. Glass fibers and a resin matrix are needed by each other. Though the glass fibers themselves are quite strong, they are still vulnerable to damage. Meanwhile, certain plastics are relatively weak but still versatile and tough. Thus combining these two materials will produce a material that is strong, lightweight, and corrosion resistant. Glass fiber is known as a good reinforcement in high performance composite applications due to its combination of good properties and low cost.

Glass fiber has an amorphous structure, which means its properties are the same along the fiber and across the fiber [12]. By varying the amount of raw materials and the processing parameters, different types of glass can be produced. There are differences in composition within the glass type because of the available glass batch raw materials, or from the different environmental constraints at the manufacturing site, or in the melting and forming processes as shown in Table 7.3. However, these fluctuations do not have a significant effect on the physical and chemical properties of the glass type Table 7.3.

7.1.2 Natural fiber composites

Researchers are looking for environmentally friendly materials so that the wastes of the product are not harmful to the planet [13–16]. Natural fiber is categorized based on its origin, i.e., plant, animal, or mineral. All plant fibers contain cellulose as their major structural component, while animal fibers mainly consist of protein. Mineral-based natural fibers exist within the asbestos group of minerals and were once used

Table 7.3 Classification of Glass fibers [15]

Letter designation	Characteristic
A, alkali	Used when the strength, durability, and good electrical resistivity of E-Glass are not required
C, chemical	Chemical stability in corrosive acid environments
D, dielectric	Low dielectric constant for electrical applications
E, electrical	High strength and high electrical resistivity
ECR, Calcium aluminosilicate	High strength, electrical resistivity, and acid corrosion resistance
AR, Alkali resistant	Used in cement substrates and concrete
R, reinforcement	Reinforcement where added strength and acid corrosion resistance are required
S, strength	High strength, modulus, and stability under extreme temperature and corrosive environments

extensively in composites; these are now avoided due to the associated health issues (carcinogenic through inhalation/ingestion) and are banned in many countries.

One example of a natural fiber is jute, which has been found to have a high tensile strength, low cost and a high specific modulus and is also reusable and recyclable [17–20]. Zampaloni et al. [21] have proven that the kenaf–PP composites compression moulded in their study have superior tensile and flexural strength compared to other compression moulded natural fiber composites such as kenaf, sisal [22], and coir-reinforced thermoplastics. Thermoplastic polymer composites with natural fibers added as reinforcing agents have received increasing attention because of the growing environmental awareness in recent years [23]. In addition, a few researchers have previously produced studies on kenaf fiber as well [9,24–26].

Pickering et al. [6] have reported that much higher strengths and stiffness are obtainable with the higher performance plant fibers than with the readily available animal fibers. An exception to this is silk, which can have very high strength, but is relatively expensive, has lower stiffness and is less readily available. Table 7.4 shows the mechanical properties of natural fiber and synthetic fiber.

7.1.3 Research motivation

There are substantial demands for low cost materials with good mechanical properties, which are environmentally friendly. Glass fiber has been applied for quite a long time, but there is now a demand for material that has equal or better properties to replace it. The current EC 130 helicopter door is completely made up from glass fiber composites. Glass fiber has many advantages such as good mechanical properties and light weight but the material is brittle, has weak abrasive-resistance, and the material is not environmentally friendly [27]. Currently, the world is taking great steps in reducing the use of nonrecyclable materials or materials that take a long time to decompose. Many composites use thermosetting resin matrices in their composition, which are hard to recycle because of their cross-linkage and they cannot be remoulded. Glass fiber produces dangerous substances when incinerated, meaning that the only option for the disposal of the material is landfill. Other materials are being used for frames for either air transport or land transport such as aramid or carbon fiber, but the total percentage of glass fiber composites is much higher and much vaster in application. Much research has discovered the benefits of natural fiber, which has been seen as a very good material to replace glass fiber. When natural fiber is used as replacement for glass fiber, this will have a large effect in reducing the percentage and amount of glass fiber used in industry thus reducing the amount of waste. One option that is being suggested is implementing natural fiber with glass fiber, which produces hybrid composites. For the aerospace industry (specifically the EC 130 helicopter door) there is an absence of research using hybrid composites as the main material.

For the purposes of the present research, full glass fiber will be set as the benchmark and the other two test specimens will be utilized with either jute or kenaf. Glass fiber is also known as glass fiber-reinforced plastic (GFRP) [28]. It is a fiber-reinforced polymer that is made of a plastic matrix reinforced by extremely fine fibers of glass. Even though glass fiber has lower strength properties and is less stiff compared to carbon

Table 7.4 Mechanical properties of natural and synthetic fiber [6]

Fiber	Density (g/cm³)	Length (mm)	Failure strain (%)	Tensile strength (MPa)	Stiffness/ Young's modulus (GPa)	Specific tensile strength (MPa/gcm⁻³)	Specific Young's modulus (GPa/gcm⁻³)
Ramie	1.5	900–1200	2.0–3.8	400–936	44–128	270–620	29–85
Flax	1.5	5–900	1.2–3.2	345–1830	27–80	230–1220	18–53
Hemp	1.5	5–55	1.6	550–1110	58–70	370–740	19–47
Jute	1.3–1.5	1.5–120	1.5–1.8	393–800	10–55	300–610	7.1–39
Harakeke	1.3	4.5	4.2–5.8	440–990	14–33	338–761	11–25
Sisal	1.3–1.5	900	2.0–2.5	507–855	9.4–28	362–610	6.7–20
Alfa	1.4	350	1.5–2.4	188–308	18–25	134–220	13–18
Cotton	1.5–1.6	10–60	3.0–10	287–800	5.5–13	190–530	3.7–8.4
Coir	1.2	20–150	15–30	131–220	4–6	110–180	3.3–5
Silk	1.3	Continuous	15–60	100–1500	5–25	100–1500	4–20
Feather	0.9	10–30	6.9	100–203	3–10	112–226	3.3–11
Wool	1.3	38–152	13.2–35	50–315	2.3–5	38–242	1.8–3.8
E-glass	2.5	Continuous	2.5	2000–3000	70	800–1400	29

fiber, it is still most commonly used as it is typically far less brittle and also cheaper than carbon fiber.

The objective of this study is to investigate the effect of the hybridization of kenaf and jute with glass fiber on the tensile and compressive properties of the composites. This study serves as an introduction of hybrid composites into the aerospace industry, especially helicopter manufacturing.

7.2 Methodology

The samples were fabricated into three different configurations which are glass fiber, hybrid kenaf/glass fiber, and hybrid jute/glass fiber. The mechanical properties of the samples were determined via tensile and compression testing.

7.2.1 Material fabrication

Material selection is a very important process as it will narrow down the available options. E-type glass fiber of 200 g/m^2 is selected as a benchmark material as it is the closest to C-type glass fiber of 200 g/m^2 which is the original material for the helicopter door frame. E-type glass fiber has higher strength compared to the C-type [13]. The other reason for the decision is because of the availability of the material. Thus, this selection process involves determining the natural fibers that will be hybridized with the glass fibers.

The samples were fabricated using the hand lay-up process. The matrix used was epoxy type Zeepoxy HL002TA cured with Zeepoxy HL002TB hardener. The resin and hardener were prepared with a ratio of 2:1 [29,30], and were stirred until the solution became nonviscous. A rectangular shaped panel with dimensions of $350 \text{ mm} \times 350 \text{ mm}$ was fabricated in a specific layer sequence. Figs. 7.4–7.6 show the glass fibers, kenaf fibers, and jute fibers, respectively.

The layer configuration is presented in Table 7.5 and shown in Figs. 7.7 and 7.8. Finally, in the curing process, the specimen was covered with another metal plate and then compressed with 6 kN weights on top of the metal plate to spread the epoxy and push the air bubbles to the side of the metal plate. The specimen was allowed to dry for a duration of 48 h (2 days). The cured specimens were cut into specific dimensions following the test requirement. For the tensile test, the specimen was cut into $25 \text{ mm} \times 250 \text{ mm}$ pieces (ASTM D 3039/D 3039M–00/95a) [31]. For the compression test, the specimen was cut into $25 \text{ mm} \times 150 \text{ mm}$ pieces (ASTM D 3410/D 3410M–03) [32]. The cutting process involved using a Computer Numerical Control (CNC) machine and a grinding machine to obtain accurate dimensions and avoid cracking. The thickness for all materials was 4 mm (specimen thickness variation $\pm 2\%$). Fig. 7.9 shows the samples of the composites.

7.2.2 Testing method for tensile and compression test

Tensile and compression tests were performed using a floor model universal testing machine. The testing was carried out using an Instron 3382 (Fig. 7.10) with a capacity

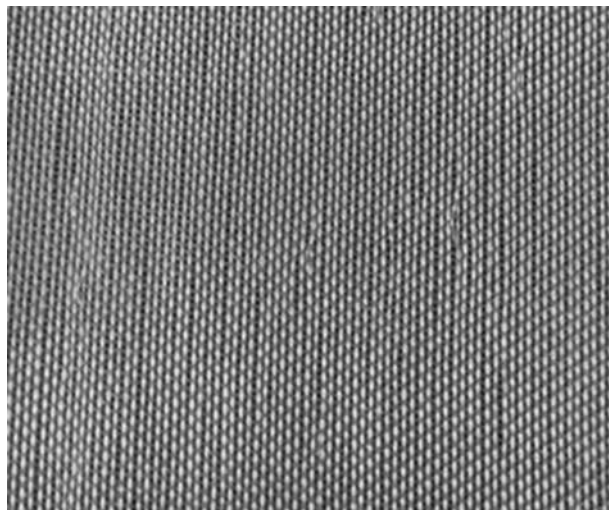


Figure 7.4 Woven glass fibers.



Figure 7.5 Woven kenaf.

of 100 kN, maximum speed of 500 mm/min and a vertical test speed of 1323 mm. The entirety of the tests were recorded by Instron Bluehill software and also included with PC data acquisition, which automatically calculates the obtained data directly. The speed used for all configurations in the tensile and compression tests was 2.0 and 1.5 mm/min, respectively. The specimens were marked and clamped at 50 mm on the top and bottom part of the specimen. The number of specimens used for both tests for each type of composite was five.



Figure 7.6 Woven jute.

Table 7.5 Number of layers for each type

Type	Number of layers
Pure glass fibers composites,	15
Kenaf hybrid composites,	8–1
Jute hybrid composites,	8–2

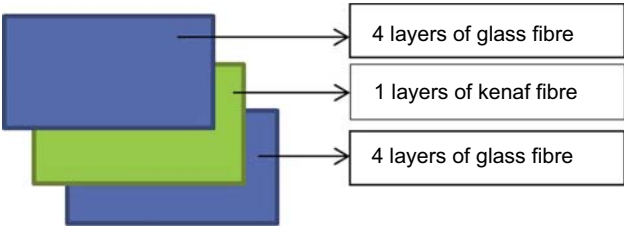
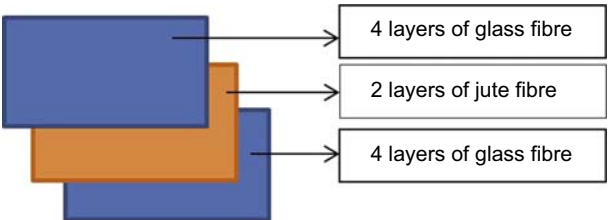


Figure 7.7 Schematic of kenaf hybrid composite configuration.

Figure 7.8 Schematic of jute hybrid composite configuration.



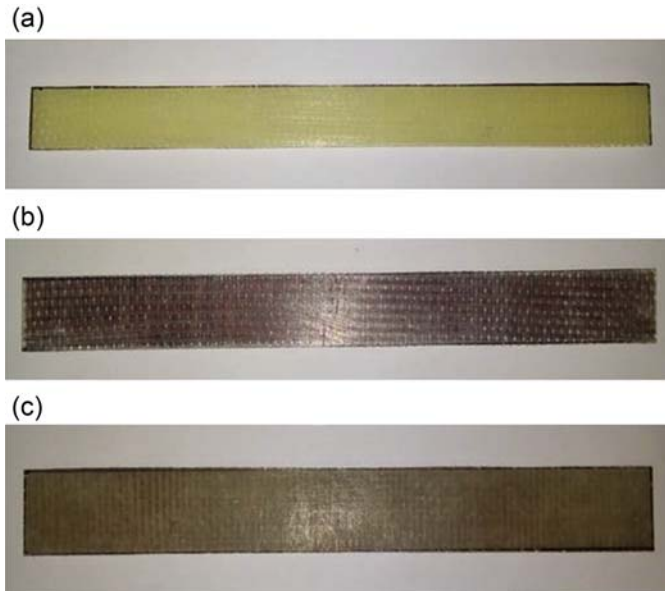


Figure 7.9 Specimen of (a) glass fiber composite, (b) kenaf/glass hybrid composite, and (c) jute/glass hybrid composite.

7.2.3 Tensile test analysis

The specimens were maintained under the same experimental conditions to prevent data variation. When each specimen reached its maximum elongation and break, the data was recorded, e.g., extension, load, tensile stress, and tensile strain. [Table 7.6](#) shows the average of the maximum load, the tensile strength, the tensile strain, and the tensile extension. Mechanical properties of the glass fiber composites, the kenaf/glass hybrid composites, and the jute/glass hybrid composites are tabulated in [Table 7.6](#).

Based on [Table 7.6](#), the kenaf hybrid composites are 4.89% closer in tensile strength to glass fiber composites than jute hybrid composites. Kenaf hybrid composites also have 4.89% and 23.62% longer extension than glass fiber composites and jute hybrid composites, respectively.

[Fig. 7.11](#) shows the average of five specimens for glass fiber composites, kenaf hybrid composites, and jute hybrid composites. The differences in the tensile strength and the tensile strain can be clearly observed and compared based on the graph. Both the kenaf hybrid composites and the jute hybrid composites have a lower tensile strength, but only the kenaf hybrid composites have a higher tensile strain than glass fiber composites.

[Fig. 7.12](#) shows a comparison of the tensile stress of glass fiber composites, kenaf hybrid composites, and jute hybrid composites, together with another material from a previous researcher [\[10\]](#). This comparison is presented to give a clear view concerning how the materials in this project differ from those used in other research studies. Only



Figure 7.10 Specimen is clamped by the machine.

jute hybrid composites are lower than aluminum, while glass fiber composites and kenaf hybrids are still better in terms of tensile stress.

Fig. 7.13 shows a comparison of the average tensile modulus for each specimen of the three types of composite. The tensile modulus describes the stiffness of the

Table 7.6 Average value of mechanical properties

Type	Maximum load (N)	Tensile strength (MPa)	Tensile strain (%)	Tensile extension (mm)
Glass fiber composites	15008.71	124.24	3.061	4.47
Kenaf/glass hybrid composites	11034.49	94.92	3.329	4.70
Jute/glass hybrid composites	9923.75	88.84	2.548	3.59

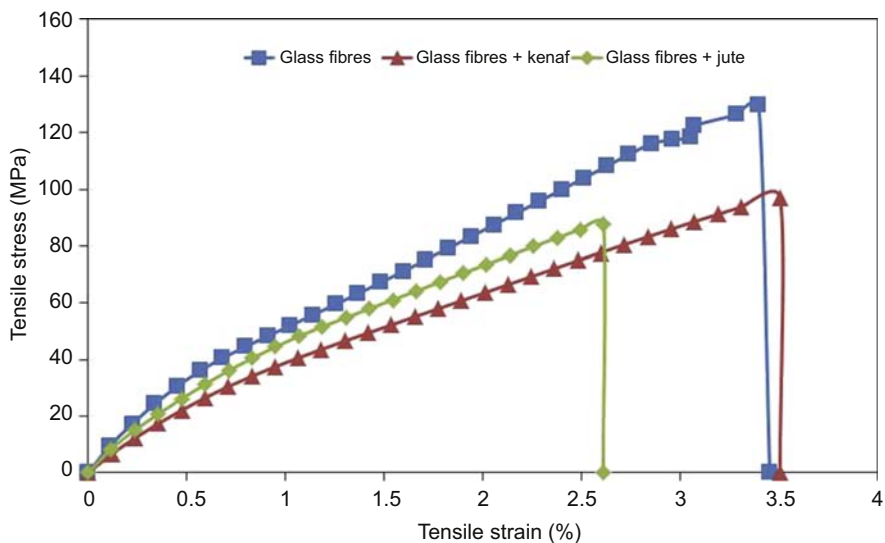


Figure 7.11 Tensile stress (MPa) versus tensile strain (%) for glass fibers/kenaf hybrid/jute hybrid composites.

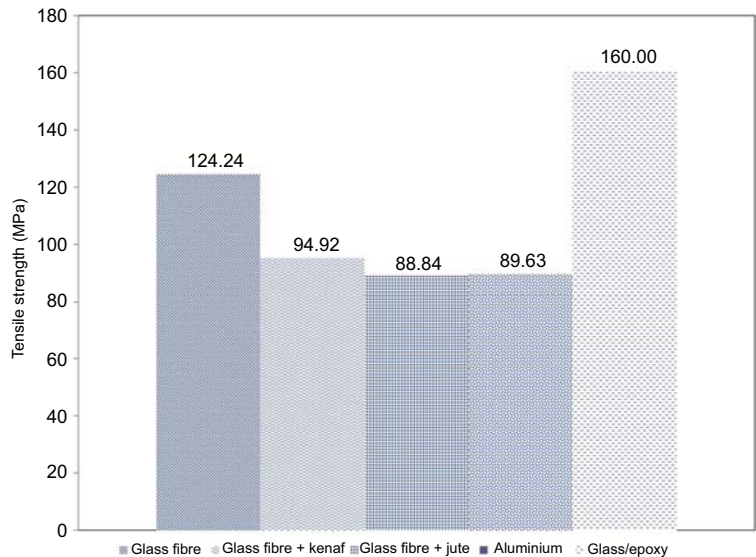


Figure 7.12 Tensile strength (MPa) for various materials.

material, i.e., the higher the value of the tensile modulus the stiffer the material. Materials with a high tensile modulus typically can withstand high stress and have a higher ultimate tensile strength but usually have low extension before break (and vice versa for materials with low tensile modulus). A higher tensile modulus does

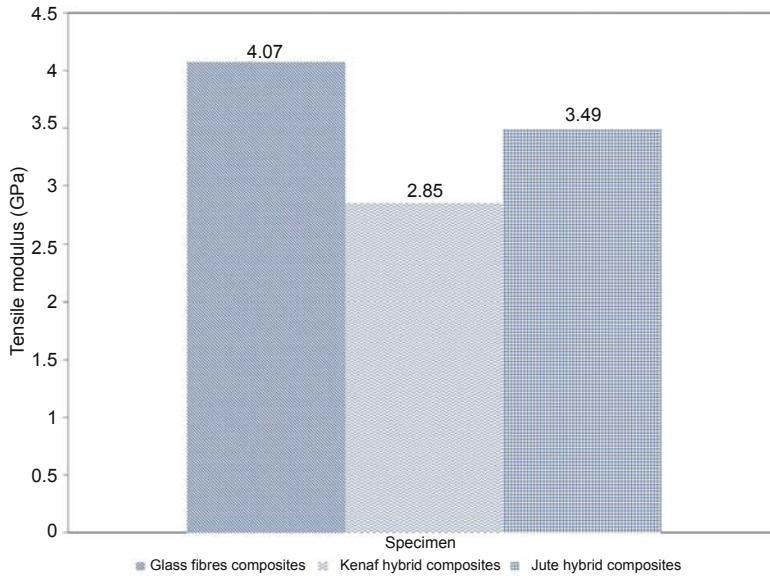


Figure 7.13 Average tensile modulus (GPa).

not mean that the material is better than a material with a lower tensile modulus because it depends on where the material will be applied [33].

Based on the results, we can see that the glass fiber composites have the highest average tensile modulus of 4.07 GPa, followed by jute hybrid composites with 3.49 GPa and lastly kenaf hybrid composites with 2.85 GPa. Jute hybrid composites are stiffer than kenaf hybrid composites even though kenaf hybrid composites have the higher tensile strength.

Fig. 7.14 shows the differences in percentage for glass fiber composites, kenaf hybrid composites and jute hybrid composites. Kenaf hybrid composites have

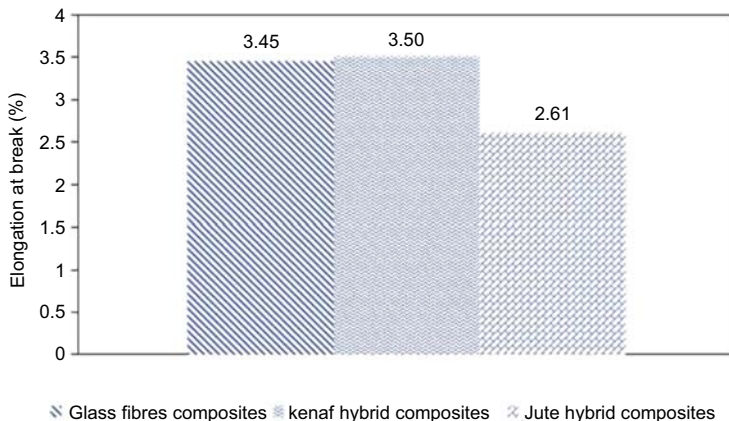


Figure 7.14 Elongation at break (%) for glass fiber/kenaf hybrid/jute hybrid composites.

0.05% and 0.81% higher elongation at break compared to glass fiber composites and jute hybrid composites, respectively. For the same thickness, the kenaf hybrid composites' tensile strength is 23.60% lower than that of the glass fiber composites, while the jute hybrid composites' tensile strength is 28.49% lower than that of the glass fiber composites. Based on the overall results of the tensile test, kenaf hybrid composites can withstand higher stress while having longer elongation and taking more time before break than jute hybrid composites.

There is huge difference in the values for the overall mechanical properties when compared to other research studies [6,10]. The reason for this difference is probably because other researchers have used different techniques for fabrication, or the fabrication was performed at different temperatures. Compression moulding or resin transfer moulding have been used, which distribute the resin equally to each ply of the fiber by using pressure and controlling the temperature [6]. This explains why the materials have far better mechanical properties than those obtained by the traditional hand lay-up method used in this research. Differences in the temperature for curing also have a large impact on the results. The process error during hand lay-up may be a factor that influences the results. The traditional hand lay-up method is relatively easy to handle, gives design flexibility in shaping, and has a lower cost for fabrication. However, with respect to the consistency of fabrication, the resin may not be equally distributed along every part of the fiber, which causes some parts to have more resin than others. The loads that are applied on top during the curing process and the moisture in the surrounding air will also have an effect on the cured specimen.

7.2.4 Compression test analysis

Compression testing is conducted to determine the specimen's properties under compressive loading (e.g., compressing, squashing, crushing or planar). These embody the elastic limit, which for a "Hookean" material is approximately the same as the proportional limit, and is additionally referred to as the yield point or yield strength, the compressive modulus and the compressive strength. Buckling is the main failure effect arising during the compression test. Buckling causes the fibers in a material to be opened up or become misaligned, thus breaking the bond between the fibers. Compression testing that uses a long column straight beam with a rectangular cross-section and applied to a compressive axial load can be analyzed using the theories of tension or compression loads, as long as the beam remains straight; however, if the deflection suddenly becomes large and leads to catastrophic failure, buckling theory has to be applied for the analysis.

Table 7.7 shows the average values of the maximum load, the maximum yield strength, and the compressive strength of the composites. Based on Table 7.7, the compressive strength of kenaf/glass hybrid composites is 8.67% higher than jute/glass hybrid composites.

Fig. 7.15 shows the differences in the stress-strain curve of the compression test for glass fiber composites, kenaf/glass hybrid composites and kenaf/glass hybrid composites. The difference in the strain of the hybrid composites compared to the glass fiber composites can clearly be seen.

Table 7.7 Average value of mechanical properties of the composites

Type	Maximum load (N)	Yield strength (Offset 0.2%) (MPa)	Compressive strength (Mpa)
Glass fibers composites	5937.04	39.75	56.83
Kenaf/glass hybrid composites	4548.95	35.96	45.07
Jute/glass hybrid composites	4048.49	27.54	40.14

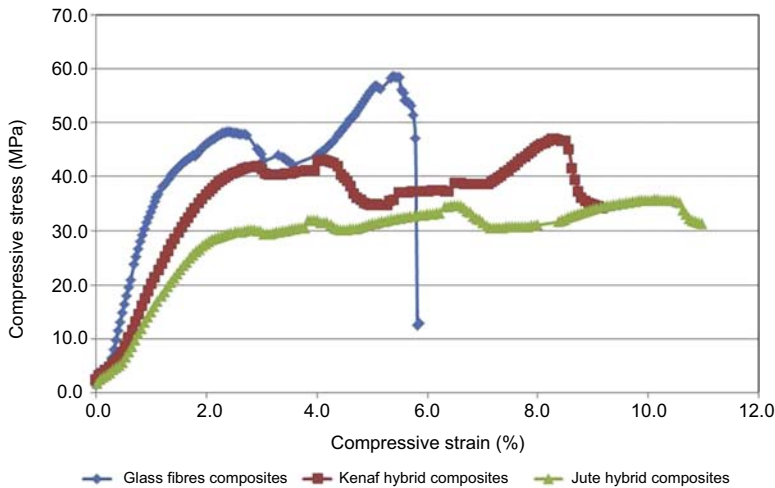


Figure 7.15 Compressive stress (MPa) versus Compressive strain (%) for glass fiber/kenaf hybrid/jute hybrid composites.

Fig. 7.16 shows a comparison of the compressive strength values of glass fiber composites, kenaf hybrid composites and jute hybrid composites together with another material used by a previous researcher.

Table 7.8 shows the average values of the compressive modulus for each type of composite. Fig. 7.17 shows the average compressive modulus based on the compression test. The results show that the glass fiber composites have the highest average compressive modulus of 1.53 GPa, followed by kenaf/glass hybrid composites with 0.94 GPa, and lastly, jute/glass hybrid composites with 0.54 GPa. Jute hybrid composites are the easiest to compress with the longest compressive extension and the lowest compressive modulus, which shows that jute/glass hybrid composites are most elastic when in compression.

Fig. 7.18 shows the percentage of elongation at break for glass fiber composites, kenaf hybrid composites, and jute hybrid composites. Jute hybrid composites have

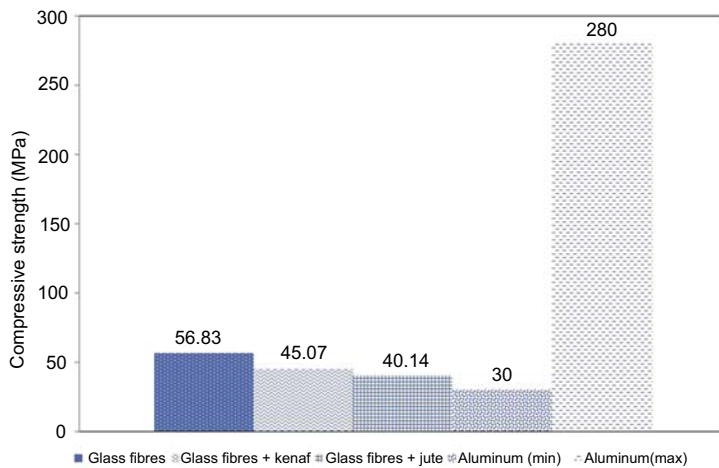


Figure 7.16 Comparisons of compressive strength (MPa).

the highest percentage with 7.4% elongation, followed by kenaf hybrid composites with 4.8%, and glass fiber composites with 3.7%.

There is a huge difference in the values for the overall mechanical properties when compared to other research [6,10]. The difference in the results obtained is probably due to different fabrication techniques or different temperatures, such as those being used in compressed moulding and resin transfer moulding [6]. This explains why the materials have far better mechanical properties than the materials fabricated using the traditional hand lay-up method being used in this research. Differences in the temperature for curing have a large impact on the results.

7.3 Failure analysis on tensile and compression

The strength of composite is highly dependent on the resin/matrix and fiber-reinforced properties. Besides that, the interfacial bonding strength between matrix and fiber surface can give better performance on mechanical properties. A comparison study from previous output can prevail the failure mechanism occur in tensile or compression test. The Scanning Electron Microscopy (SEM) is employed to study the effect of the morphological features of the fiber-matrix interface on composite structure.

Figs. 7.19 and 7.20 depict the fiber morphology of woven kenaf and random chopped kenaf respectively. Generally, both figures show that the interaction between the fiber and the matrix is a bonded interaction without resin penetration occurring in the fiber. There are obvious gaps in both figures indicating poor bonding interaction between the fiber and the matrix resin [9].

The resin matrix is induced into the kenaf fiber gaps as shown in Fig. 7.19. Hence, this gives additional strength and a greater bonding surface area between the resin and the fiber. Fig. 7.20 shows that the fiber's sizes are mixed and randomly arranged. The

Table 7.8 Average values of Compressive Modulus for the compression test

Type	Maximum load (N)	Yield strength (Offset 0.2%) (MPa)	Compressive strength (Mpa)	Extension (mm)	Average compressive strain (%)	Average compressive modulus (GPa)
Glass fibers composites	5937.04	39.75	56.83	1.38	3.70	1.53
Kenaf/glass hybrid composites	4548.95	35.96	45.07	1.10	4.80	0.94
Jute/glass hybrid composites	4048.49	27.54	40.14	1.75	7.4	0.54

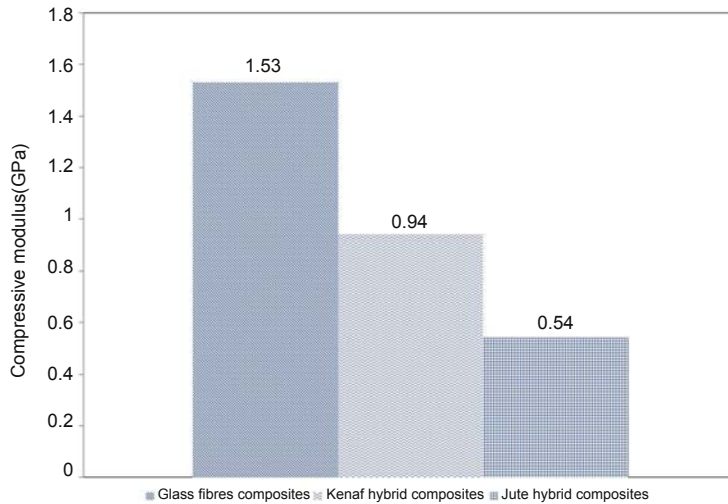


Figure 7.17 Average compressive modulus (GPa) for the compression test.

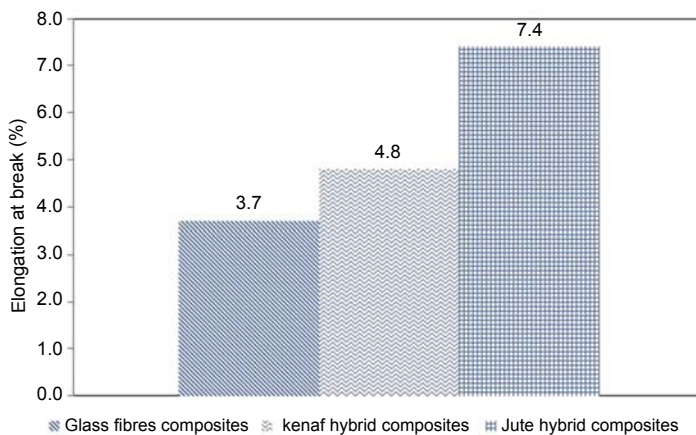


Figure 7.18 Elongation at break (%) for glass fibers/kenaf hybrid/jute hybrid composites.

bigger fibers may decrease the bonding surface area, hence decreasing the bonding strength. The fracture mechanisms occurring for woven and random chopped samples are: pulled out, detached and debonded from the epoxy matrix as depicted in Figs. 7.20 and 7.21. Fig. 7.19 shows matrix crack in the composite which can reduce the composite strength.

Senthilkumar et al. [22] reported finding on sisal fiber. The failure mechanism and fiber morphology was properly discussed and reported. Fig. 7.21a shows the SEM fractograph of the $90^\circ/0^\circ/0^\circ/90^\circ$ oriented sisal fiber composites after the impact test. This figure clearly shows the 90 degrees oriented fibers placed at the extreme side and 0 degree oriented fibers placed next to the 90 degrees fibers. The failure

Figure 7.19 Scanning electron microscope (SEM) analysis on woven kenaf [9].

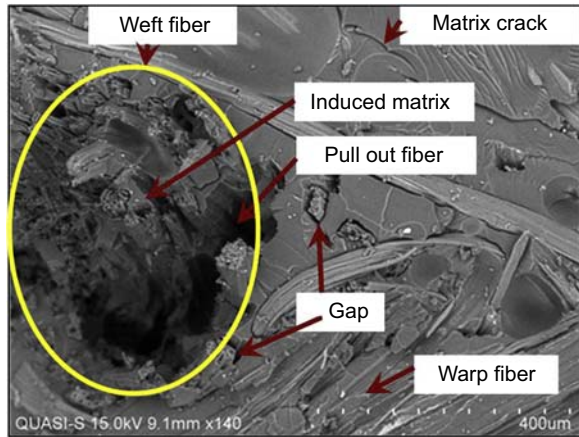
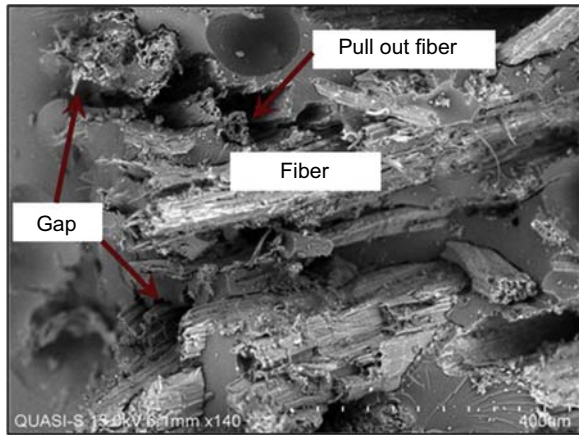
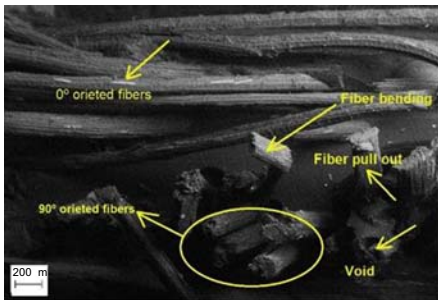


Figure 7.20 Scanning electron microscope (SEM) analysis on random chopped kenaf [9].



(a)



(b)

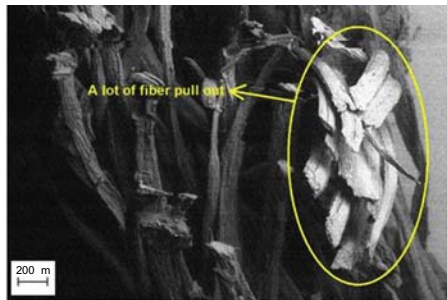


Figure 7.21 Scanning electron microscope (SEM) studies on the effect of 90°/0°/0°/90° (a), and 0°/90°/45°/45°/90°/0° (b) fiber orientation on the impact strength of sisal fiber composites [24].

mechanisms for the impact loading were fiber fracture, fiber bending, fiber pull out, and voids. A poor interfacial bond between the fiber to matrix caused void formation and earlier failure in composites.

Moreover, fiber bending could be the failure mechanism arising as a result of the lesser impact strengths of fiber oriented composites. Fig. 7.21b exhibits the fracture details of the $0^\circ/90^\circ/45^\circ/45^\circ/90^\circ/0^\circ$ sisal oriented composite. The failure mechanism is fiber pull out. This required energy dissipation of a larger magnitude, which was exhibited in the higher impact strength.

7.4 Conclusions

The application of composite materials in helicopters has been relatively late compared to other types of aircraft. This is probably because the helicopter contains complicated structures and is less used as a transport aircraft. The world is searching for a solution to reducing the usage of nonbiodegradable material. Applying natural fiber in composite materials is one way of reducing the use of synthetic fiber, thus causing the material to become more environmentally friendly.

The helicopter needs strong materials to fulfill its purpose. Many types of natural fiber have been investigated for their properties, such as kenaf, jute, sisal, etc. The results show that the materials are promising for partial or full implementation as the main material in the structure. Combining different types of material (what been called hybrid material) is a good solution as the combination helps to overcome the weaknesses of each of the component materials.

The materials and the method of fabrication are really important in the production of a good material. The material will not have the maximum performance if it is fabricated without the proper handling of each of the steps needed in the completion of the structure. The advantages of the traditional hand lay-up method are that it is relatively easy to handle, gives design flexibility in shaping, and has a lower cost for fabrication. The disadvantages of the hand lay-up method are in the consistency of fabrication, and the condition of the surroundings during fabrication can easily change the properties of the end product.

Based on the tensile testing conducted, the results show that the average maximum tensile loads of glass fiber composites, kenaf hybrid composites, and jute hybrid composites are 15008.71, 11034.49, and 9923.75 N, respectively. While the average tensile strengths of glass fiber composites, kenaf hybrid composites and jute hybrid composites are 124.24, 94.92, and 88.84 MPa respectively. The tensile modulus values for glass fiber composites, kenaf hybrid composites, and jute hybrid composites are 4.07, 2.85, and 3.49 GPa, respectively. Overall, the tensile tests show that kenaf hybrid composites can withstand more loads than jute hybrid composites but still have a lower maximum load than glass fiber composites. Kenaf hybrid composites have longer extension before failure when compared to the other two types, which means that kenaf hybrid composites have a longer time before failure occurs.

Based on the compression testing conducted, the results show that the average maximum compressive loads of glass fiber composites, kenaf hybrid composites,

and jute hybrid composites are 5937.04, 4548.95, and 4048.49 N, respectively. While the average compressive strengths of glass fiber composites, kenaf hybrid composites, and jute hybrid composites are 56.83, 45.07, and 40.14 MPa, respectively. The compressive modulus values for glass fiber composites, kenaf hybrid composites, and jute hybrid composites are 1.53, 0.94, and 0.54 GPa, respectively. The highest percentage of compressive strength to tensile strength is for kenaf hybrid composites with 47.48%.

Overall, this research study is just the beginning for proposing a new material which incorporates natural fiber with synthetic fiber to produce hybrid composites. The quality of fabrication involved in the traditional hand lay-up method will greatly affect the quality of the material. Therefore, the steps in the process of the hand lay-up method must be consistently and carefully followed to reduce error.

The failure mechanism that may occur in composite is influenced by several factors such as fiber orientation, interfacial bonding, and microcracks. A few mechanisms which cause failure to the structure such as fiber fracture, fiber bending, fiber pull out, voids, pulled out, detached, and debonded.

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Cracks, microcracks, and fracture toughness of polymer composites: formation, testing method, nondestructive detection, and modifications

8

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8.1 Introduction

A composite can be defined as a material consisting of two or more different constituents. The different constituents can encompass different elements or different phases. However, the material can be considered as a composite only when the elements and phases have their own different physical properties compared to the matrix material or phase. These differences in properties should also be great enough to influence the original properties of the material [1]. An example of a composite material is the polymer matrix composite. The polymer is the matrix material, and the fiber is incorporated into the matrix usually as reinforcement. Commercial polymer on its own usually contains additives such as binders, fillers, pigments, and other elements that further enhance its properties for use. However, these additions of additive materials do not turn the polymer into a composite since its physical properties are not greatly changed by the additives [1,2]. The application of polymers in day-to-day use is limited by the low values of their mechanical properties, for example, low stiffness and strength. To widen their application possibility, fibers are frequently added as reinforcement to improve these properties for higher-strength demand applications [3].

The addition of these fibers, be it in continuous or particle form, into the polymer turns it into a composite material. This is due to the fact that the physical properties of the fibers are completely different compared to the matrix. The addition of the fibers turns the composite into a stronger, less ductile material. In theory, the matrix polymer is expected to support the fibers and keep them in their proper position, transfer the load to other fibers, protect the fiber from damage during manufacturing and use, and prevent cracks in the fiber from propagating through the entire composite. The polymer matrix

also has major control over the electrical properties and chemical behavior of the composite. Fibers on the other hand are usually stronger, stiff, and have high melting temperature if they are expected to be used in an elevated temperature environment [2]. However, how the fibers incorporate themselves into the polymer, whether the bonds between the fibers and the polymers are strong enough, whether the fibers in particulate form are homogeneously distributed into the matrix, and whether they are in continuous form, the direction of these fibers in the matrix is the major parameter that determines the final values of the composite's physical, mechanical, and electrical properties.

Looking at the interaction between the fibers and the polymer matrix is the key to understanding the formation of cracks and microcracks in polymer composite material. The existence of these failure initiation sites, in turn, determines the fracture behavior of the composite material. After understanding the formation and how it influences the fracture behavior of the composite, detection using nondestructive testing and advances in polymer and polymer composites to improve the material's resistance to cracks will be discussed in detail in this chapter.

8.2 Formation of cracks and microcracks in polymeric composites

In polymer composite, defects (e.g., cracks and microcracks) are complicated since defect formation can be dominated by several factors: the materials (matrix and fiber, chemical compatibility, loading, fiber orientation and size), the manufacturing process (hand lay-up, vacuum, curing, etc.), and use (corrosion, mechanical stress, thermal mismatch, etc.). Once the cracks or microcracks are formed, the next evolution will be the growth of cracks until the material fails. For a polymer composite with fiber loading in particulate form, the cracks formed and their growth will be very much different compared to composites with a laminated structure. A comparison between laminated composites, i.e., between unidirectional fibers and multidirectional fibers, will give different crack formation and growth behavior as well. Different chemical reactions between the matrix and the fibers will influence the strength of the bond between them, which also influences the formation and growth of cracks. Even if there is no defect formation at the starting point of the matrix and fiber interaction, and the manufacturing process is perfect, during use, defects can still form originating from the residual stress of the thermal mismatch between the matrix and the fiber or from the disintegration of the materials from corrosion or from mechanical loading over time.

For example, in the fiber-dominated defect [1], microcracks can form when the fiber ends become separated from the matrix at a very small load. This usually happens in discontinuous-fiber-reinforced polymer composites where the fiber ends become a point for stress concentrations. A similar situation can also arise for continuous fiber if the fiber breaks at its weakest cross-section. The first microcrack at the fiber end will have several alternative effects. One alternative outcome is that the cracks will propagate along the fiber's interface to the matrix and the composite will fail. Another alternative

situation is that the crack will propagate out in a direction normal to the fibers across to other fibers leading to immediate composite fracture. If both of these situation effects are suppressed, an increased load will result in further separation between the fiber ends and the matrix, forming more microcracks at other sites. Each of the cracks formed becomes a stress concentration site and influences the mode of crack propagation until the composite fails completely.

A study by Ponomavera et al. [4] found that in the case of a particle-reinforced polymer composite, the shape of the particle influences the defect that is formed in the composite. In the study, the matrices used were polypropylene with talc (lamellar shape), wollastonite (acicular shape), and glass spheres (round) as reinforcement. The results of the analysis showed that the samples with talc and wollastonite exhibited a brittle failure mode, while the sample with glass spheres is ductile. It can be summarized from this study that the sharp edges or points of the particulate become the stress concentrator, which encourages the formation of microcracks in the polymer composite.

Sometimes the atmosphere surrounding the composite in use can be the source of cracks, e.g., the phenomenon called environmental stress cracking (ESC). In this case, the polymer composite in use might be immersed in a liquid environment and subjected to stress. Over time, with material degradation, microcracks will start to form in the material. Liquid will then seep into these cracks and further reduce the strength at the area. The microcracks will then propagate faster with this deterioration and this finally results in material failure, which takes place at lower stress forces compared to the normal environment [5].

In cases where the fiber is in a woven form, and hence the composite is actually made up of layers of woven mat encapsulated by the matrix, the interfaces between these woven mats and the matrix is the weakest point. The performance in these composites greatly depends on the interlaminar characteristics. Delamination, which is also a type of crack propagation in composites, will occur when the peel stresses exceed the tensile strength of the matrix [6]. However, in a study by Krishnan and Roy Xu [7], delamination for woven mat in polymer composites is categorized under notch propagation and not crack propagation due to the blunt point at the tip of failure that propagates, which results in the different energy value needed for failure to happen. Fig. 8.1 show the impact-induced delamination in composite laminates.

8.3 Fracture mechanics in polymeric composites

Fracture mechanics is defined as a topic related to the behavior of materials containing cracks or other small flaws. The word flaw in this context refers to small pores, inclusions and microcracks. The fracture toughness measures the maximum stress that a material can withstand if it contains a crack with known size and geometry [9]. This analysis is crucial and complicated in composite materials since the interaction of the different types of materials and fibers can be a source of flaws. For example, in the particulate-reinforced polymer composite, in order for the fracture toughness to be

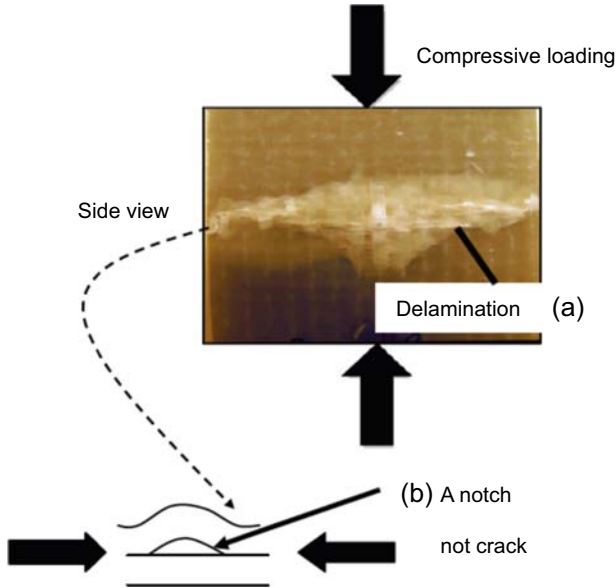


Figure 8.1 (a) Impact-induced delamination inside a fibrous composite laminate propagates under compressive load during a compression-after-impact experiment [8]. (b) the propagating delamination front along the composite interface is a notch, not a mathematically sharp crack [7].

acceptable for the composite mix, certain criteria for the particulate's properties have to be taken into consideration. Some of the requirements are as stated below [3]:

1. The particle should be of small size (less than $5\ \mu\text{m}$)
2. The aspect ratio should be close to 1 (Fig. 8.2 shows the meaning of aspect ratio)
3. The particles must deboned prior to the yield strain of the matrix
4. The particles should be dispersed homogeneously in the matrix (agglomeration must be avoided or minimized)

Formation of microcracks and voids makes the situation worse. However, in the prediction of fracture toughness, a controlled crack is introduced into the composite material and its crack propagation during the fracture test is analyzed to predict the fracture

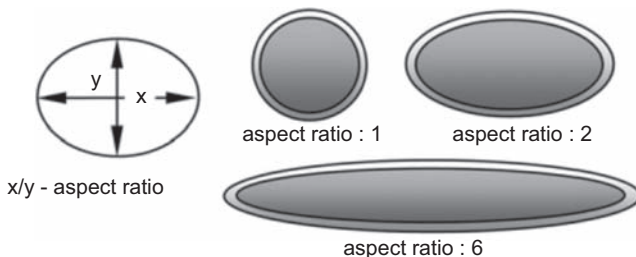


Figure 8.2 Aspect ratio calculation [3].

toughness of the composite material system. The following topic will discuss the method for fracture toughness experiments and the analysis method normally used for polymer composite material.

8.3.1 Fracture toughness in polymeric composites

Fracture toughness is represented by the symbol K_{IC} and is the critical value of the stress intensity factor at a crack-tip necessary to produce catastrophic failure under simple uniaxial loading. The subscript “I” stands for mode I (crack opening under normal tensile stress perpendicular to the crack) loading, and “C” stands for critical. A simple example is a balloon with a small pinhole defect. When it is blown until the internal pressure of the balloon reaches a critical value, catastrophic failure originates at the pinhole and the balloon will pop [10]. The fracture toughness can also be measured in Mode II (shear). The fracture toughness unit is $\text{MPa (m)}^{1/2}$. Fig. 8.3 shows a typical measurement of K_{IC} , and Table 8.1 lists the values of K_{IC} for various materials. It is worth noting that the value K_{IC} is normally associated with a plane strain condition, where the sample thickness is relatively large compared to the notch dimension. For thin samples (plane stress conditions), fracture toughness is denoted by K_C , and it is a sensitive function with respect to specimen thickness. Plane strain conditions are applicable when the thickness of the sample $\geq 2.5 (K_{IC}/\text{Yield strength})^2$ [10]. Some sources also use the symbol G instead of K to symbolize fracture toughness.

Highly brittle materials with little or no ability to deform plastically at the crack-tip will present low K_{IC} values and are susceptible to catastrophic failures. Meanwhile, the

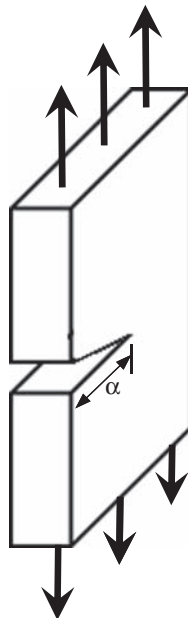


Figure 8.3 Schematic of fracture toughness test.

Table 8.1 Typical values of fracture toughness for various polymeric materials [10]

Material	$K_{IC} \text{ (MPa(m))}^{1/2}$
Polyethylene (High density)	2
Polyethylene (Low density)	1
Polypropylene	3
Polystyrene	2
Polyesters	0.5
Polyamides (nylon 66)	3
Polycarbonates	1.0–2.6
Epoxy	0.3–0.5

highly ductile materials will deform substantially at the crack area prior to fracture. The main importance of fracture mechanics is basically to identify the materials of intermediate ductility that are susceptible to catastrophic failure below their yield strength due to the stress concentrating effect of structural flaws. For example, in designing a pressure vessel, it is convenient to plot operating stress which is related to the operating pressure as a function of flaw size. In manufacturing, components can be inspected using nondestructive testing to ensure that there is no flaw existing in the component above an acceptable size [10]. The different types of the available nondestructive testing will be discussed later in the following topic.

8.3.2 Mode I fracture toughness analysis method

Generally, in any material engineering, the specification of the sample and the method used for fracture toughness analysis is guided by the ASTM standard. In polymer composite laminates, for Mode I analysis, ASTM D5528 is the standard normally used. The data obtained will basically be used to study crack propagation with a double cantilever beam test. The equations and specification used is as stated below:

$$G_I = -\frac{1}{B} \frac{dU}{da} \quad (8.1)$$

where G_I is the energy release rate or loss of energy in the test specimen per unit of specimen width for an infinitesimal increase in delamination length, U is the total elastic energy in the test specimen, B is the specimen width and a is the delamination length. For ASTM D5528, the standard requires the fabrication of a beam-type sample with a precrack. Fig. 8.4 shows the schematic of the sample. Fig. 8.5 shows a snapshot of a sample with delamination growth. The thickness of the laminates is critical to avoid large deflection effects. The sample normally comprises an even

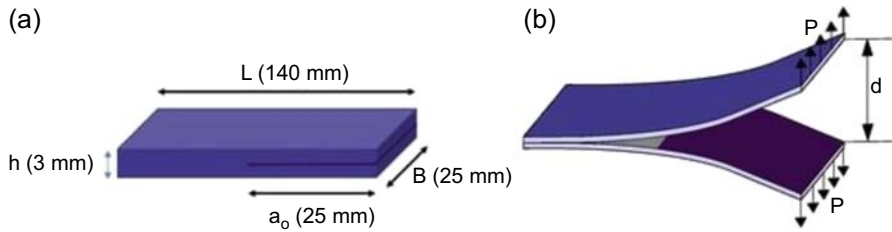


Figure 8.4 Schematic of the sample (a) without loading, (b) during loading [6].

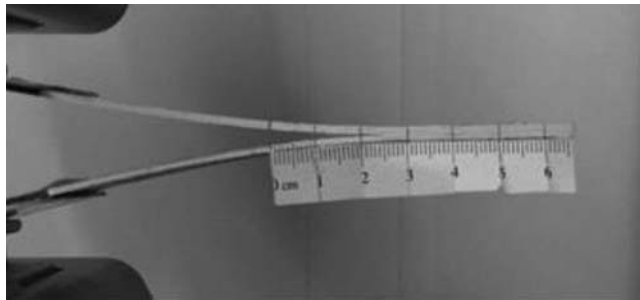


Figure 8.5 Snapshot of delamination growth in sample [6].

number of layers so that the midplane is inserted with nonstick film with a thickness less than $13\ \mu\text{m}$ to form the crack. The standard proposed for the sample length is 125 mm and 20–25 mm in width. The specimen is loaded via end-blocks or piano hinges [6].

For an example of the analysis, in a work by Wu et al. [11] on an epoxy carbon fiber laminated composite, the sample dimension is 140 mm length $\times$ 20 mm width. The side faces are polished, painted white and marked from the edge of the insert film with vertical lines every 1 mm up to 50 mm for ease and accuracy of measurement using direct visual observation with an image magnifier. The tests were carried out using an Instron 5969 universal testing machine equipped with a 10 kN load cell. The calculation used the modified beam theory equation as below:

$$G_I = \frac{3P\delta}{2b(a + I\Delta I)} \quad (8.2)$$

where P is the applied load, δ is the displacement, b is the specimen width, a is the crack length, and $I\Delta I$ is calculated using the compliance calibration method as defined in the standard of ASTM D5528. Three methods were used in this study to determine the G_{IC} value at the initiation of delamination: visual observation, onset of nonlinearity and 5% offset/maximum load (the symbols used are G_{IC-Vis} , G_{IC-NL} and G_{IC-MAX}). Following initiation of delamination propagation values, $G_{IC-PROP}$ was determined as a function of crack length a .



Figure 8.6 Photograph of Mode I test [12].

In comparison, a research study conducted by Vijay et al. [12] on the mode I interlaminar fracture toughness of a woven carbon fabric reinforced polymer composite used samples with dimensions 180 mm length, 25 mm width and 20 mm crack length as in Fig. 8.6; this is also according to ASTM D5528. Piano hinges and the specimen were grit blasted with sandpaper before bonding and cleaned with carbon tetra chloride impregnated soft paper. Steel hinge end tabs were glued to the surfaces of the beam at the notch for application of the load to the specimen during loading. The sides were painted white to permit visual crack-tip location. The tests were performed using Instron EN 6033 with a cross-head speed of 10 mm/min. Load versus displacement values were recorded to measure G_{IC} . The equation used is as below:

$$G_{IC} = \frac{3P\delta}{2b(a + \Delta)} \quad (8.3)$$

The equation is similar to the one used by Wu et al. [11] and the symbol denotes the same meaning except for Δ in this case, which was determined experimentally by generating a least squares plot of the cube root of compliance, $C^{1/3}$ as a function of delamination length. However, the mode I interlaminar fracture toughness values, G_{IC} , were obtained directly from the computerized Instron machine (DIN EN ISO 7500-1).

8.3.3 Mode II fracture toughness analysis method

In another work by Wu et al. [11], the Mode II strain-energy release rates were measured using a four-point bend end-notched flexure (4-ENF). The configuration and dimensions of the specimen are as in Fig. 8.7. The spans for the loading rollers and the supporting rollers were 60 and 100 mm respectively. Therefore, the effective initial delamination length (a_0) was 30 mm. The sides of the specimen were polished

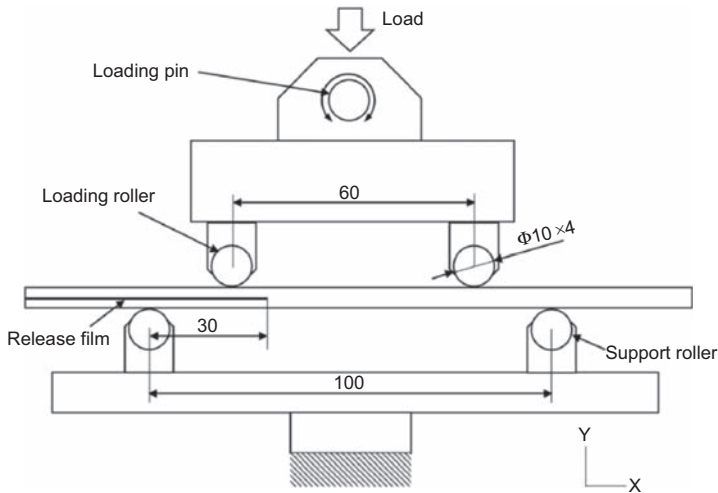


Figure 8.7 Fixture for 4-ENF Mode II testing. All dimensions in mm [11].

and painted white with markings every 1 mm for 50 mm. The tests were conducted using an Instron 5969 universal testing machine fitted with a 10 kN load cell. The cross-head displacement speed was 5 mm/min for precracking (to extend the started crack beyond the resin pocket at the end of the insert), 0.5 mm/min for the remaining length and 10 mm/min for the return of the cross-head after the delamination length reached around 40 mm. The G_{II} data was calculated using the equation below:

$$G_{II} = \frac{P^2}{2b} \frac{\partial C}{\partial a} \quad (8.4)$$

where P is the load, b is the specimen width, a is the delamination length and $\partial C/\partial a$ is the gradient from a compliance curve plotted according to modified beam theory. Similar to Mode I, the critical values of $G_{IIC-VIS}$, G_{IIC-NL} , $G_{IIC-MAX}$ and $G_{IIC-PROP}$ were determined.

As comparison, in a study by Vijay et al. [12], Mode II analysis was carried out using the three-point bending test as described in ASTM D3878. The span length is 60 mm, the width is 11.3 mm and the thickness is 3.1 mm, with an average initial crack length of 18.5 mm. Fig. 8.8 shows a schematic view of the apparatus. The load level, the displacement and the crack length were recorded from the test. The cross-head velocity is 10 mm/min using the Instron machine EN 6033. G_{IIC} represents the critical strain-energy release rate for the crack growth of the insertion of precrack length. G_{IIC} values were calculated from the initial crack length and the load displacement curve using the highest load and displacement level in the equation below:

$$G_{IIC} = \frac{9P_c \delta_c a^2}{2b(2L^3 + 3a^3)} \quad (8.5)$$



Figure 8.8 Three-point bending test for Mode II [12].

where P_c is the maximum load, L is the span length, a is the delamination crack length, b is the specimen width and δ_c is the maximum displacement.

8.4 Detection of cracks and microcracks using the nondestructive testing method in a polymeric composite

The main issue with cracks and microcracks in composite materials is the detection and measurement of the flaw. In experiments, to understand the fracture behavior of the material in depth, it is imperative that a material with a known flaw amount and size before and after testing is used. This information is beneficial and important for researchers to be able to engineer materials for certain applications. The usual method used in experiments is direct visual observation, such as the comparison of photographs after a certain duration or loading. Another situation where the information about the cracks and microcracks is beneficial will be when the material is in use. Knowledge about this can help the engineers to predict when the structure will fail and this knowledge will prove useful in avoiding catastrophic damage and even save lives. For direct visual observation, the position of the crack must be visible to the naked eye, hence this is impossible in application since exposing the cracks will damage the component as well. In order to estimate correctly the flaw's quantitative features, several nondestructive testing (NDT) methods can be applied. NDT can be used to measure the subsurface and internal cracks that are formed. However, some methods prove to be more accurate, cost-saving and technologically possible for use at the moment compared to others. The following topic will discuss the available methods that are being used and are also currently under research for polymer composites.

8.4.1 Nuclear magnetic resonance imaging method in polymeric composite

Nuclear magnetic resonance imaging (NMRI) can be used as a quality evaluation technique in assuring the quality and integrity of the manufactured component. It can be considered as a type of chemical microscope in generating images. This method is famously used in the medical field to study anomalies in human bodies. However, recent studies have also applied this method in materials engineering. The main constraint in the application for materials study is due to the high incurred cost [13]. This issue can be solved as the technology in nuclear magnetism evolves.

How NMRI works is based on the following theories as explained by Tarpani et al. [14]. Basically, the nuclear particles of both protons and neutrons have their intrinsic angular momentum (p) and intrinsic magnetic moment (μ). Because of these traits, they will behave as infinitely small magnets with a north and a south pole. Both p and μ are vectors with magnitudes and directions in space. The magnitude of these vectors is characterized by the nuclear spin (I). The I value for both protons and neutrons is $I = 1/2$. The net magnetic moment of an atomic nucleus is the sum of the magnetic moments of all the protons and the neutrons in the nucleus. This means that an atomic nucleus with a net magnetic moment behaves like a little magnet as well, with the angular momentum vector having rotary movement around its axis. This is also known as the motion of precession or Larmor precession. This is fundamental to the development of the NMR phenomenon. The sensitivity of the atomic species to NMR depends on the following equation:

$$\mu = \gamma \times p \quad (8.6)$$

The proportionality constant γ is known as the magnetogyric ratio. Each atomic nucleus has a unique value of γ , in units of $\text{s}^{-1} \cdot \text{T}$, where T = Tesla, the unit of magnetic field strength. Hydrogen has $\gamma = 26.8 \text{ s}^{-1} \cdot \text{T}$, which is the highest one known, making the element most sensitive to the NMR effect. In an NMR experiment, the object is placed under the external action of a strong, static magnetic field. A superconducting magnet, which remains continuously active, normally provides the static magnetic field (B_0) and, accordingly, the magnetic moments associated with the nuclear spin of the target object tend to align parallel or antiparallel to B_0 , following the state energy of its protons and/or neutrons. Submitted to a second oscillating, external magnetic field, called radio-frequency (RF), the spins absorb energy when hit and excite a resonant frequency. A coil standing nearby the analyzed object can detect the magnetization derived from the precession motion of the nuclear spin, which will decay over time. The signal captured by the coil perpendicular to the magnetization plane is called Free Induction Decay (FID). In Tarpani et al.'s [14] work, the potential of NMRI use for in vitro conditions to study translaminar and delamination for a continuous carbon fiber reinforced laminated polymer was investigated. Fig. 8.9 shows the schematic position of the sample inside the NMR supermagnet and Fig. 8.10 shows the images that were captured.

From the study, it can be deduced that, generally, the failure in a composite can be detected using the NMRI technique. However, several views need to be studied to

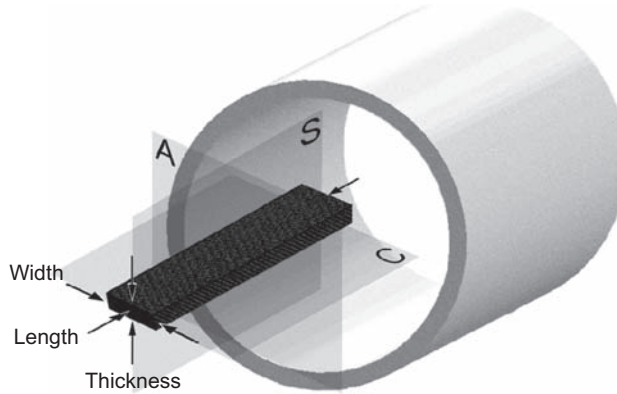


Figure 8.9 Schematic of the position for a tablet-like test piece inside the nuclear magnetic resonance (NMR) super-magnet where the main tomographic planes evaluated are as follows: A(Axial), S (Sagittal) and C (Coronal) [14].

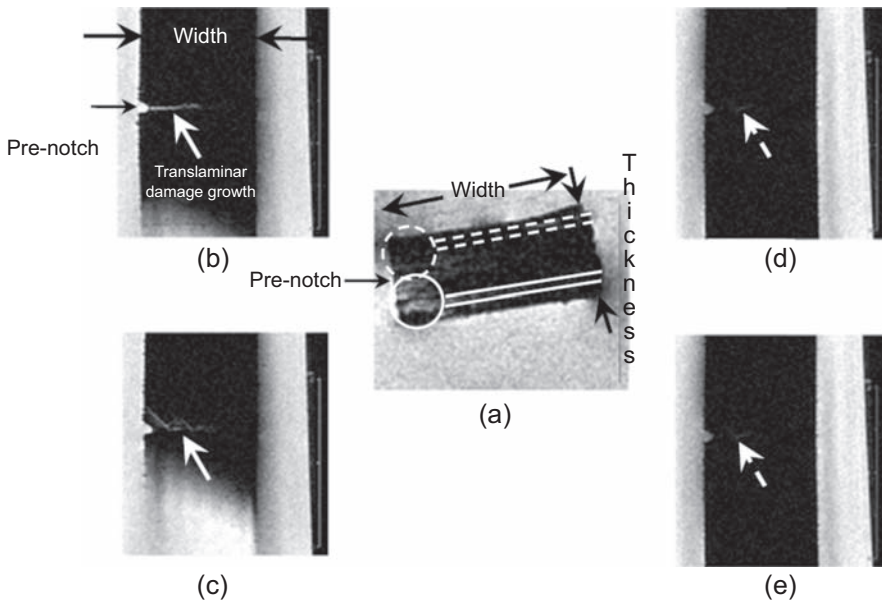


Figure 8.10 Nuclear magnetic resonance (NMR) images of the EPX-FC composite laminate with a translaminar fracture, as obtained via the FLASH-3D protocol: (a) AXIAL view, (b) and (c) Coronal views near to one of the lateral sides of the specimen showing extensive cracking starting from the pre-notch tip (d) and (e) Coronal views adjacent to the opposite lateral side of the specimen displaying almost no damage growth from the pre-notch tip [14].

comprehensively and accurately analyze the dimension and nature of the failure in the composite, especially if it involves unbalanced translaminar cracks with respect to the sample thickness. Hence, if this is the type of failure involved, the analysis will be more costly and time consuming and might not be worth the cost incurred unless the technology can be improved to be cheaper.

8.4.2 The X-radiography method in a polymeric composite

X-radiography produces a shadowgraph of the internal structure of a part. It works like a medical chest X-ray. It is commonly used in industry to inspect castings and welding joints. The method works when a certain intensity of the X-ray beam, I , is transmitted through a thickness of material, x , given by Beer's law in Eq. (8.7):

$$I = I_0 e^{-\mu x} \quad (8.7)$$

In this equation I_0 is the incident beam intensity and μ is the linear absorption coefficient of the material. The intensity is proportionate to the number of photons in the beam and is distinct from the energy of the photons in the beam. The absorption coefficient is a function of the beam energy and the element composition. The dependence of the linear absorption coefficient on the elemental composition is given in Table 8.2. The table shows that μ increases with the atomic number of the element. This is the main reason why low-atomic-number materials will appear relatively transparent and high-atomic-number materials appear more opaque [10].

8.4.3 The ultrasonic method in a polymeric composite

Ultrasonic testing is based on a portion of the acoustic spectrum (typically 1–25 MHz) with frequencies well above those of the audible range (20–20,000 Hz). The ultrasonic wave is mechanical in nature and requires a transmitting medium. Most typical engineering materials are relatively transparent to ultrasonic waves. The key item in this testing is the reflection of the ultrasonic waves at the interfaces of dissimilar materials. A high degree of reflection by a typical flaw, such as an internal crack, is the basis for defect inspection. This method is not suitable for use on complex shape components and there is a tendency for the ultrasonic waves to scatter due to microstructural features such as porosity and precipitates [10].

The application of ultrasonic defect detection as a nondestructive testing method for polymer composites is being explored by researchers. However, the structure of polymer composites makes it difficult for the method to be applied. The anisotropic properties of the materials, the different configuration of the fibers in the matrix, and the different processes make it difficult for the measurement to be precise. In several experiments,

Table 8.2 Linear absorption coefficient of various elements for an X-ray beam with energy = 100 keV [15]

Element	Atomic number	μ (mm ⁻¹)
Aluminum	13	0.0459
Iron	26	0.293
Nickel	28	0.396
Tungsten	74	8.15

the polymer composite is assumed to be homogeneous, and the process uses low frequency and high power to overcome the high specific acoustic impedance to disclose internal defects such as large voids and delamination. Another challenging aspect for polymer composites in this regard is the location and nature of the defect. The weakest part in this type of composite is the interaction between the fibers and the matrix, which means that delamination is a high probability. The issue arises when the existence of the crack in this delamination process is being muted down by the compression forces applied on the sample/structure, which results in crack closure [16]. This reduces the probability of measuring the true size of the crack and the consequence can be catastrophic.

Achenbach [17] mentioned that many applications of ultrasonic have used water coupling in an immersion tank. The effectiveness of this technique depends on the capability to focus the ultrasound. However, the conventional ultrasonic focused transducer is rather inflexible in its direction and the focal length is fixed. Direct focusing on a flaw is not possible without a great deal of scanning. A time delay steered array, which generates a focal spot in the acoustic beam at a specified beam angle, can be added to improve the capability of the equipment. It consists of transducers placed geometrically, which are capable of exciting individual transducers in a predetermined time sequence. The manipulation of these time delays is possible through electronic means, which result in faster imaging. However, the time delay steered array has its weaknesses when it comes to detecting and characterizing discrete defects. First of all, the location of the crack is unknown, secondly, any material variations can affect the focal point depth to make it differ from theoretical calculation, and thirdly, the correction of the arrival time of the received signals may contain errors that result in phase aberrations and hence reduce the effectiveness of the array. This is something that researchers are working on in order to improve the effectiveness of this testing method.

8.4.4 The acoustic emission method in a polymeric composite

An acoustic microscope consists of four main components: the acoustic probe, the pulse-mode measurement system for transmitting and receiving signals, the mechanical system for the alignment and movement of the specimen, and a computer for controlling the system and processing the recorded wave forms [17]. Abo-El-Ezz [18] studied the possibility of using an acoustic emission wave to monitor crack propagation in a polymeric material. Acoustic emission is a measurement of the stress waves generated as the cracks propagate in the material. It is more commonly used for studying metal due to the fact that polymeric material gives out lower energy levels when the cracks propagate. This makes it difficult for the monitoring process to differentiate whether the signal given out is from crack propagation or interference from the surrounding environment, e.g., mechanical noise from the equipment. Other than this, the frequency range for polymers is also large (between 10 and 500 kHz). Based on the study, the setup is most important to help differentiate whether the wave detected is actually the true data, and the result is most prominent when the crack propagation is nearing failure. Fig. 8.11 illustrates the setup of the equipment on the material and Fig. 8.12 shows the signal detected for each loading sequence. This

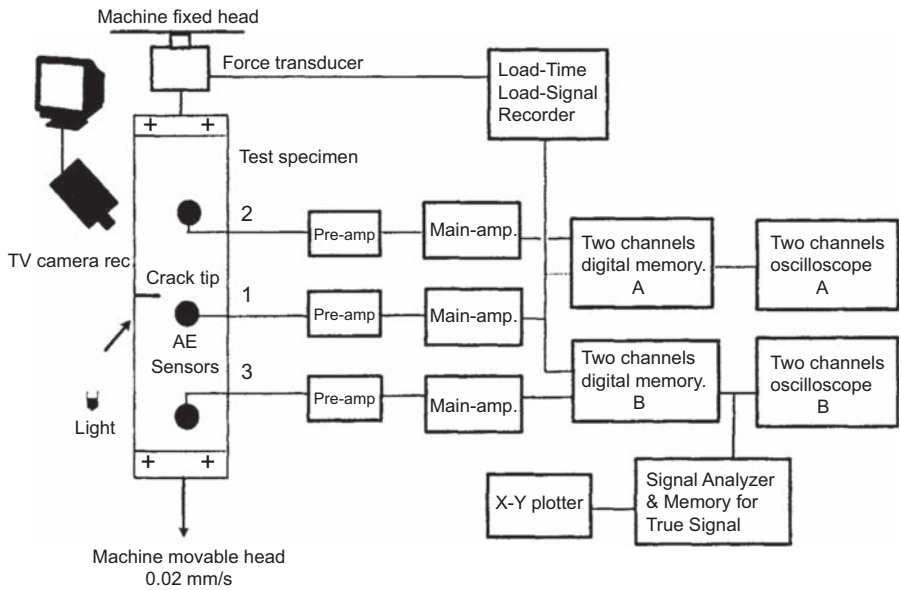


Figure 8.11 Schematic diagram for the measuring system [18].

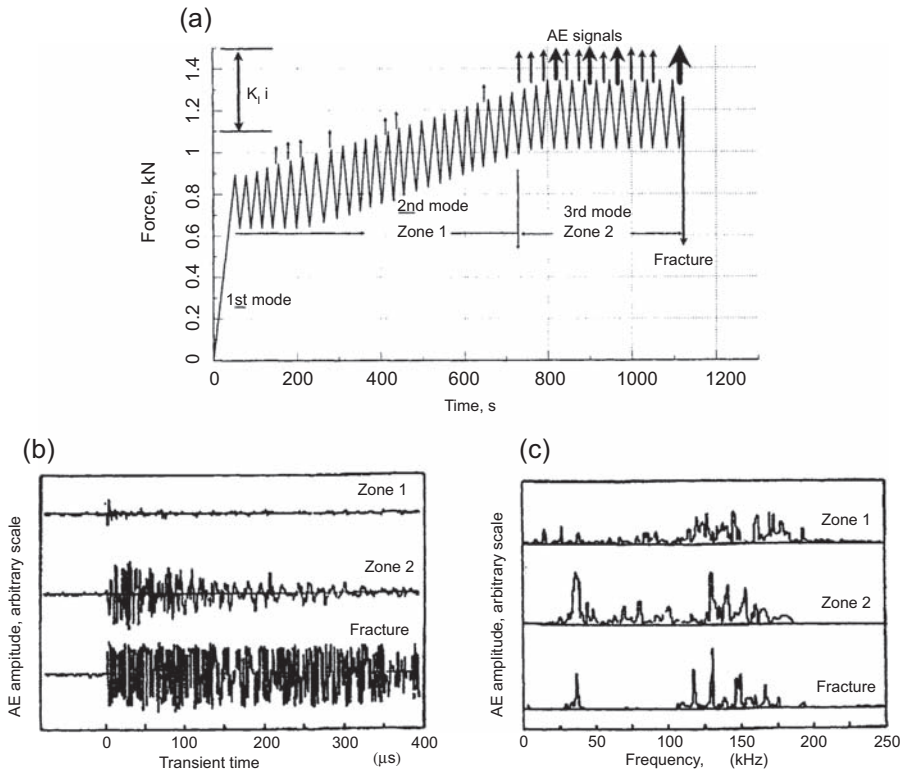


Figure 8.12 (a) The applied load spectrum versus time and the position at which the acoustic emission (AE) signals were detected, (b) Typical AE signals detected in zone 1, zone 2, and at fracture, and (c) Frequency range of the AE waves shown in (b) [18].

method will need to be optimized for polymer composites. The other weakness of this method is that the sensor has to be located near to the crack-tip, which limits the application of the setup for use only in a controlled experimental environment. However, if all the weaknesses can be resolved, this method can be used to monitor the structural health of polymeric composites in working conditions.

8.5 Current modifications to improve fracture toughness for polymeric composites

Progress has been made in the field of composite materials in general to increase their fracture toughness property so that their application can be widened. Based on the crack propagation study, several microstructural techniques have been identified, which significantly affect the fracture toughness for general composites. Two methods have been identified which are transformation toughening and crack arrest.

For the first theory, transformation toughening, taking an example of ceramic material, [Fig. 8.13](#) illustrates the transformation toughening mechanism in partially stabilized zirconia (PSZ). Using second-phase particles of tetragonal zirconia in a matrix of cubic zirconia is one method to improve the toughness. Any propagating crack will create a local stress field that induces transformation of the tetragonal

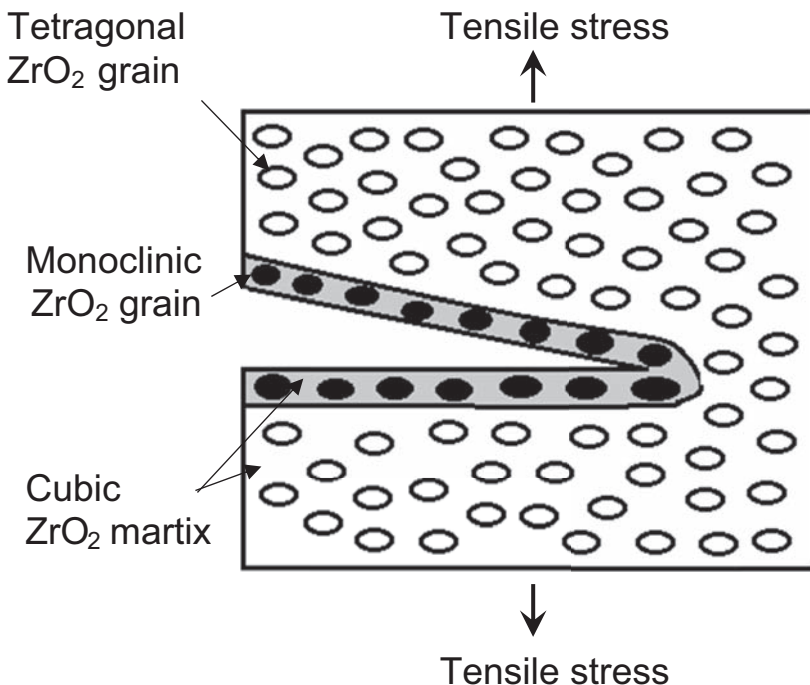


Figure 8.13 Schematic of transformation toughening in partially stabilized zirconia.

zirconia particles to the monoclinic structure in that area. The slightly larger specific volume of the monoclinic phase causes an effective compressive load locally and, in turn, will squeeze the crack closed [10].

An example of crack arrest is shown in Fig. 8.14. The microcracks that formed due to the internal stresses during the processing of a ceramic were able to blunt the tip of the advancing crack. The expression associated with Griffith cracks (Eq. 8.8) indicates that the larger tip radius can dramatically reduce the local stresses at the crack-tip, thus slowing down the propagation of the crack.

Applicable to purely brittle materials, the Griffith crack model can be used. Alan Arnold Griffith lived from 1893 to 1963. He was a British engineer. Griffith's career was spent primarily in aeronautical engineering. He was one of the pioneers to suggest that the gas turbine would be a feasible propulsion system for aircraft. In 1920, he published his research on the strength of glass fibers, which makes his name immortal in the field of materials engineering. Based on his Griffith's crack model, he assumed that in real materials there would be many elliptical cracks at the surface and/or in the interior [10]. From his equation, shown in Eq. (8.8), the highest stress (σ_m) at the tip of such a crack is:

$$\sigma_m \approx 2\sigma(c/\rho)^{1/2} \quad (8.8)$$

In this equation, σ is the applied stress, c is the crack length and ρ is the radius of the crack-tip. Since the crack-tip radius can be as small as an interatomic spacing, the stress

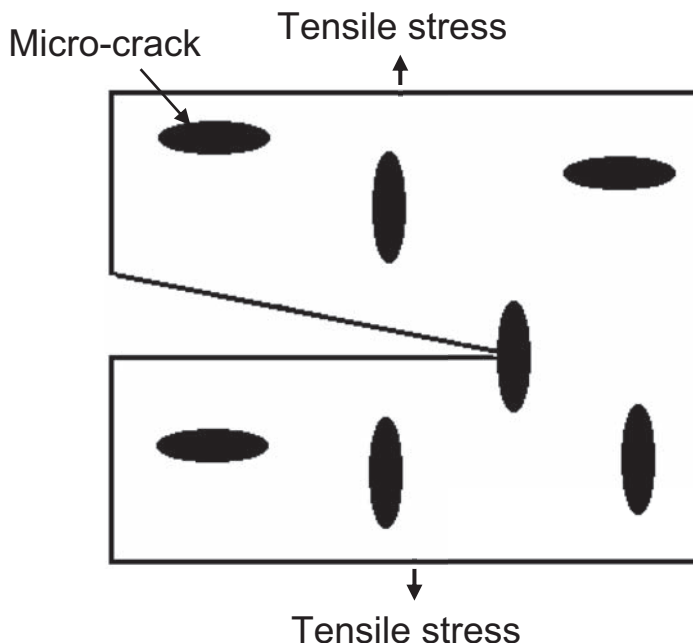


Figure 8.14 Crack arrest by microcracks.

intensification can be large. Routine production and handling of brittle materials makes this model indispensable. This is the reason why brittle materials are weak in tension but stronger in compression, since the compressive loads tend to close, not open the flaws [10]. However, for polymer composites, the available options for toughness improvement would differ compared to metal and ceramic composites due to the different interactions, chemistry and configurations between the matrix and the fibers. These methods can be applicable to some extent in particle-reinforced polymer composites with modifications. For continuous long fibers in the matrix, the above methods will not be applicable. For example, in a laminated composite, it was mentioned by Chaudhry et al. [6] that, based on their literature review, improvement in fracture toughness for a laminated polymer composite can be achieved through (1) a strong resin matrix, (2) an interlayer of micro- and nanofillers, and (3) stitching, z-pinning, or weaving techniques. Other than fracture toughness, crack propagation, mode of failure, etc. are also important in order to engineer a polymer composite for a wide range of applications. Two examples of the current research on this topic are discussed in the following sections.

8.5.1 *Interphase/mesophase between the fibers and the matrix*

In polymer composites, one of the issues faced is unstable crack propagation in the structure and also the weak strength between the fiber and the matrix. The interphase, also known as the mesophase, is introduced around the fiber in this modification. The interphase formed can be a result of the naturally different chemical and mechanical properties of the matrix and the fiber. However, an interphase layer can also be introduced artificially by special chemical treatment or coating of the fiber prior to mixing with the matrix. This layer is expected to increase the bond between the fiber and the matrix, acting as a crucial medium in transferring the applied load from the matrix to the fibers and playing an important role in damage evolution in the composite. One of the damage control mechanisms of the interphase is the dampening/blunting of the crack-tip, which will slow down the crack propagation. For example, in a continuous fiber polymer composite, in a study conducted by Pengfei Wang et al. [19], carbon fiber-reinforced plastic (CFRP) laminates without any coating, coated with graphene oxide (GO-CFRP), and coated with graphene oxide and polydopamine (PDA&GO-CFRP) were compared in terms of how the cracks propagate and their fracture toughness in Mode I and Mode II failure. The study found that the coating acts as an interphase layer to increase the stability of the crack propagation of the composites in a Mode I experimental setup. Fig. 8.15 illustrates the stability of the crack propagation with the modification in Mode I.

Based on the analysis, it was found that the crack propagates in a continuous manner without a significant jump. Crack blunting may be the dominant factor in controlling the crack propagation. Accumulation of plastic deformation around the crack-tip changes the behavior to the stick-slip crack propagation type. This is also effective in improving the delamination resistance, similar to the stitching effect in a laminated composite. Fig. 8.16 shows a schematic of the crack propagation and morphologies of the failed

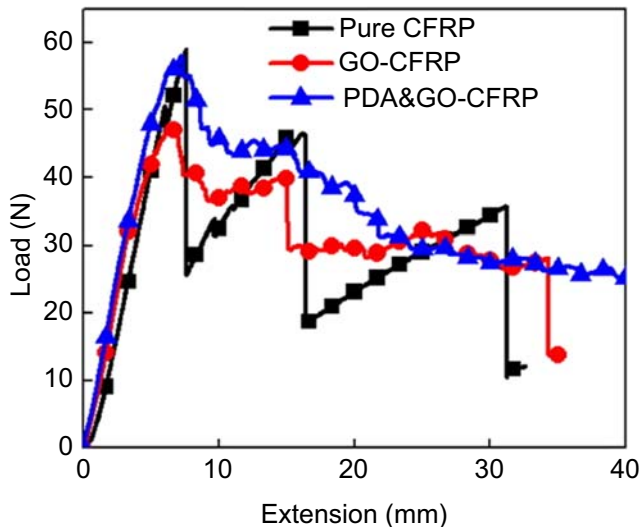


Figure 8.15 Load extension curves for untreated and coated CFRP [19].

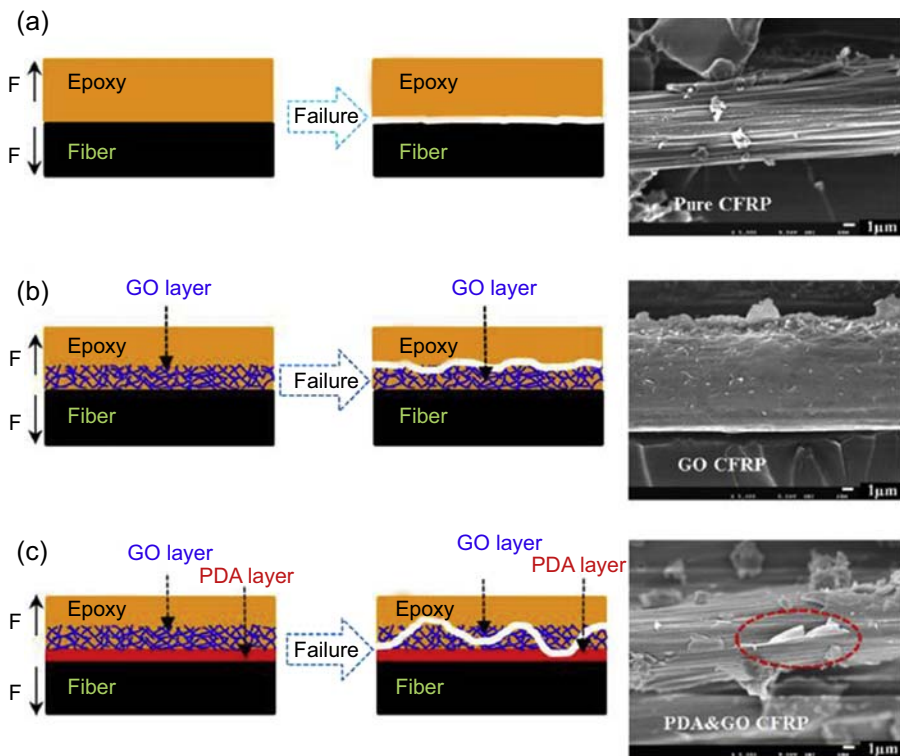


Figure 8.16 Schematic diagrams and micromorphologies of the failure at the fiber/epoxy interface of (a) pure CFRP; (b) GO sized CFRP and, (c) PDA&GO sized CFRP [19].

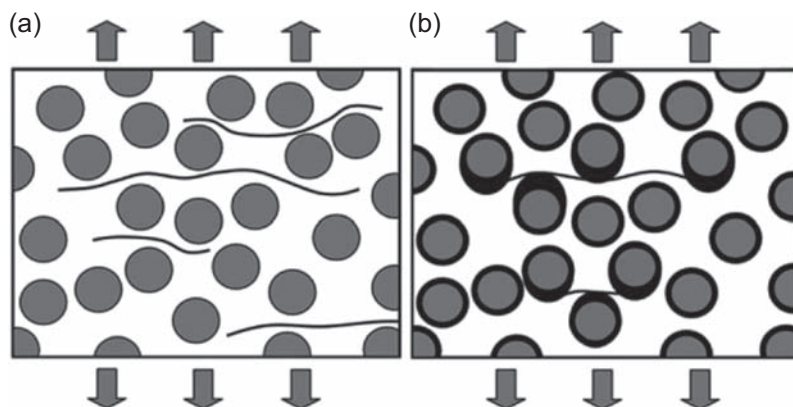


Figure 8.17 (a) microcrack propagation in the case of rigid particles without an interphase and (b) micro-crack propagation in the case of coated particles [3].

fiber/epoxy interface. For the pure CFRP samples, the epoxy separated cleanly from the fiber. However, for the GO coated and PDA&GO coated CFRP, the crack goes through a disordered GO peeling/tearing layer, and the fiber at the cracked surface was found to be encapsulated by GOs and the epoxy medium. This GO peeling/tearing process in crack propagation helps to increase the crack propagation resistance.

However, the results for this experiment do not really work for crack behavior in the sliding mode (Mode II). The data shows that the coated CFRP crack propagation starts at a lower force compared to the untreated samples. Even though the crack propagation is more continuous prior to failure for the coated CFRP, similar to the Mode I result, the fact that the crack started early means that further investigation is needed to assess the interaction of this coating for Mode II shear failure.

The application of an interphase layer is also applicable for a particulate polymer composite. Fig. 8.17 below shows the interaction of the interphase layer in a particulate-reinforced polymer matrix with crack propagation.

In a discussion by Hutař et al. [3], it was mentioned that if we assume perfect adhesion between the particulate and the matrix, with the particulate shape assumed to be a perfect sphere, microcrack propagation will tend to avoid the rigid particle and grows preferentially in the soft matrix. However, in the event that we have a soft interphase layer, microcrack propagation will be attracted to this interphase layer. Once the crack-tip touches the interphase, the interphase is damaged due to the high stress concentration at the crack-tip and the matrix and the particle can be fully debonded. As a consequence, the crack is blunted with the stress field reduced and the crack remains arrested near the particle. The blunting of microcracks with debonding will contribute to an increase in the fracture toughness of the composite.

8.5.2 Interlayer of micro- and nanofiller

Another method that has been explored by researchers for fracture toughness improvement in a laminated polymer composite is an interlayer of micro- and nanofiller. In this

method, the micro- and nanofiller is distributed evenly between the woven fiber layer and the matrix, especially in-between the layers that have been identified to be most prone to delamination. Or in some cases, the filler was mixed together with the matrix prior to the lay-up process. The major issue with this method is the uneven distribution of the fillers. Agglomeration or lumps of filler will reduce the fracture toughness instead of increasing the value. Since some types of polymer composite deal with woven fiber, the weave pattern can also influence the distribution of the filler.

In a study conducted by Chaudhry et al. [6], carbon nanotubes (CNT) were sprayed onto prepreg layers in the delamination plane using an airbrush. Prior to spraying, a mixture of the 0.2 wt% CNT and an ethanol solution was sonicated in an ultrasonic bath for an hour to ensure uniform dispersion of the CNT. The data was collected using the Mode I failure method. Table 8.3 shows the calculation for the represented CNT percentage for samples A, B, and C, based on the research. Fig. 8.18 shows the Mode I analysis of load versus deflection for the three sample types.

Chaudhry et al. [6] found that the CNT inclusion is able to improve the fracture toughness value with a maximum CNT loading of 1 g/m². Above this value of CNT

Table 8.3 Sample name and corresponding carbon nanotube (CNT) areal density [6]

Sample name	CNT areal density (g/m ²)	Calculated CNT mass fraction (%) for entire composite
A (baseline)	0	—
B	0.5	0.12
C	1	0.24

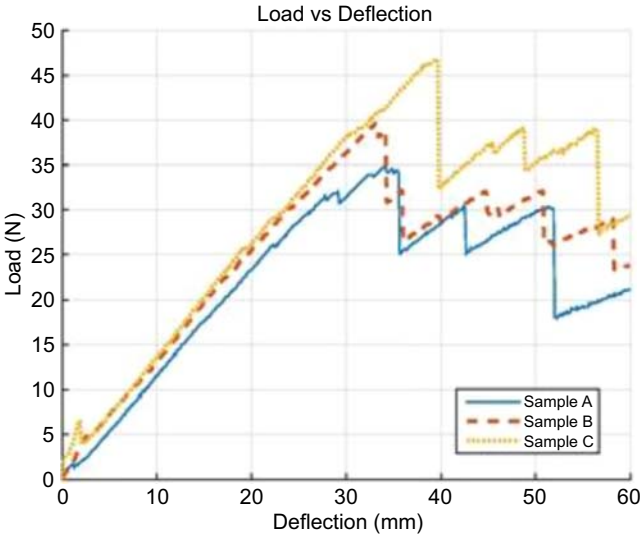


Figure 8.18 Load versus displacement curves [6].

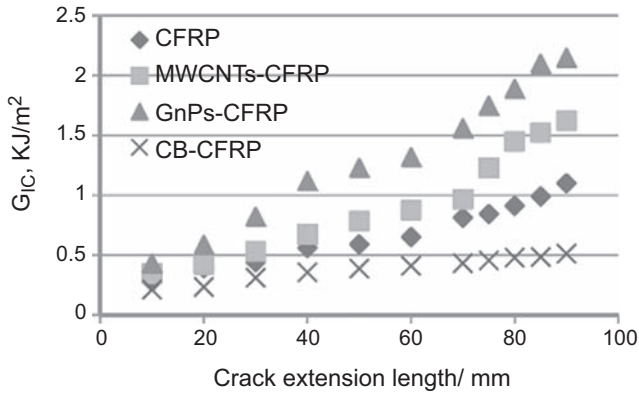


Figure 8.19 Variation of interlaminar fracture toughness (GIC) with crack extension length under the DCB test of composites [12].

loading, the viscosity of the epoxy matrix decreased and resulted in epoxy matrix degradation due to the high amount of ethanol. Other than that, the agglomeration of the CNT worsens the performance. The CNT rich regions fail to provide resistance to the crack growth, hence reducing the fracture toughness. Basically, CNT can increase the fracture toughness in a lamellar composite. However, the performance is based on the method used to disperse the CNT at the layer and the maximum amount of CNT that can be contained in one space at a time.

For some experiments, the nanofillers were mixed together with the matrix to improve the toughness of the composite. The idea is that a tougher matrix is equal to a higher fracture toughness in the polymer composite. In a study by Vijay et al. [12], up to 3% nanofillers, such as multiwalled carbon nanotubes (MWCNT), graphene nanoplatelets (GnPs) and carbon black (CB) were mixed into the epoxy resin for the preparation of CFRP. The Mode I and Mode II data of the composites were collected to compare the fracture toughness values. Fig. 8.19 shows the fracture toughness for Mode I. From the results, the inclusion of the nanofillers (such as MWCNT and GnPs) was able to increase the fracture toughness value for both Mode I and Mode II. However, there is an adverse effect for CB due to CB's lower surface area, lower modulus of elasticity and agglomeration.

8.6 Conclusion

Microcrack and crack formation and propagation are important aspects to be studied in materials. This is due to the material's capability to withstand stress without failure during application, which is highly related to it. For polymer composite material, due to the different properties of the reinforcement and the matrix, the topic of study becomes more complicated. The discussion in this chapter mentioned that the formation and propagation of the cracks were highly dependent on the selection of matrix and fiber,

how it is being layered, the manufacturing process that the polymer composite goes through, and the environment that it is exposed to afterwards. Microcracks and cracks were categorized as defects in materials. To quantify the defect, fracture toughness is one of the properties that can give it value. Measurement of fracture toughness is guided by the ASTM standard. Mode I analysis usually refers to the tensile stress effect on crack propagation while Mode II is more related to the shear stress applied for crack propagation. To fully understand the formation and propagation of microcracks and cracks, nondestructive testing was one of the options that can be used to measure the location, shape, and size of the defects in real time. Nuclear magnetic resonance imaging method, the x-radiography method, the ultrasonic method, and the acoustic emission method were some of the available nondestructive testing used on polymer composite material. Each and every method have its own strength and limitations. In order to improve the fracture toughness of the polymer composite, modification on the material is one of the option available. Two methods of material modification have been established to improve the fracture toughness property. One method is the formation of interphase layer between the fiber and the matrix, and another is the addition of micro- or nanofiller into the polymer composite.

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Shape memory polymer and its composites as morphing materials

9

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9.1 Introduction

Shape memory polymer (SMP) is a type of smart material that can remember its permanent shape from a deformed shape. SMPs are widely used as actuator due to their ability to transform shape following an external stimulus. The unique ability of SMP has advanced the scope of many scientific fields, especially in medicine and engineering. Polyurethane (PU) polymer is the most researched SMP material since it was discovered to have the same shape memory behavior as the shape memory alloy (SMA). Other SMP materials include epoxy and polystyrene. SMPs are actively responsive to external stimuli such as heat, light, electricity, pH, and the magnetic field [1]. Some SMPs can have a combination of two or more responses at the same time. This promising property places SMP as a suitable candidate for smart structures and systems that operate at different conditions and require multiple stimuli responses. The broad possibilities of SMP application in industry include actuators, self-deployable structures, and morphing structures. SMP has the potential to be the skin material for morphing aircraft as described in reference [2]. However, there are a few limitations that may challenge the utilization of SMPs, such as low thermal conductivity, inertness to electromagnetic stimuli and lower strength compared to the already established, and well researched, SMA. A variety of approaches have been developed to improve the properties of SMP and have led to the development of shape memory polymer composites (SMPC). Reinforcement of SMPC can incorporate either fillers or fiber types to enhance the properties of pure SMP. By integrating an electrically conductive filler such as carbon nanotubes (CNTs), carbon nanofibre (CNF), graphene, conductive fiber and ferromagnetic particles into the SMP resin, recovery of SMP can be induced through Joule heating. When electrical current is applied across the SMP, the resistance in conductive filler heats up the SMP according to Joule's law and ultimately triggers the recovery process. Many significant developments have been obtained from research on electrically conductive SMPs; Lu et al. provided a

systematic review of carbon-based reinforcement in electrically conductive SMP [3]. The purpose of this review is to highlight recent advances and the development of carbon-based reinforced SMPC emphasizing the works done in the development of morphing wing materials. The review also focuses on the development of graphene-reinforced SMPC which shows good prospects for producing SMP with improved mechanical and electrical performance. Selected works in SMP research are summarized, and a short look into the future aspect is discussed at the end of this article.

9.2 Concept of shape memory polymer

Shape memory materials (SMM) are materials that can deform into a temporary shape after a thermal process above a certain temperature i.e., the glass transition temperature or melting temperature. Past this transition temperature, the SMM will morph into a soft state and can be deformed easily by applying a load. As it is cooled down under an applied load, the material will maintain a temporary shape in a stable manner even when the load is removed [2]. The SMM will assume its original shape when a certain stimulus is applied to the deformed structure due to elastic energy stored during programming [4–9]. The shaping and returning to initial shape processes are called programming and the recovery process respectively. SMA, SMP, and shape memory ceramic (SMC) are the classifications of SMM [10,11]. Shape memory effect in SMAs is stimulated by thermal induction. The alloy structure is cooled down from the austenitic phase to the martensitic phase where the stiffness is reduced and the structure can be easily deformed [2]. When a load is applied in this martensitic phase, SMA can retain the temporary shape and only return to the original shape when heated above the austenite temperature in the austenite phase. On the other hand, the transition temperature for SMP is usually the glass temperature. This is when the polymer structure becomes rubbery with low stiffness, enabling large deformations. When it is cooled below the glass temperature, the SMP's stiffness increases drastically [12]. SMC on the other hand can be either a reversible phase transformation resembling the SMA, or a multiphase system which resembles the SMP. Typical stimuli for SMPC are temperature, light, electric field, magnetic field, pH, or enzymes [1]. The most common stimuli on a shape memory material are temperature and light. Thermal-responsive SMPs were among the first to be discovered [13]. To this date, there have been investigations and research on thermal-responsive SMP and its properties [14–16]. The focus of these researchers was on the thermomechanical behavior and response of the thermally induced SMP [1]. Electroresponsive SMPs are materials that have electrically conductive fillers integrated within the SMP matrices to produce structures that are sensitive to electricity [1,17,18]. On the other hand, light responsive materials can be categorized into two types. The first are SMP that only respond to a certain light wavelength and are not influenced by the thermal effect of light, and the second category is SMP that absorb heat from the light and induce thermal heating to implement shape memory response [1,12,13,19,20]. The shape memory process described earlier is classified as a one-way shape memory effect in which the shape-changing effect is not reversible. In theory, any one-way shape memory cycles can be run indefinitely, however, the programming process always requires external

mechanical stress to form the temporary shape. In contrast, a two-way shape memory effect is a material where the structure can reversibly change shape without any external mechanical stress [4]. An example of a two-way shape memory effect can be seen in SMA and liquid crystalline elastomers [21–23]. An extension of the dual-shape memory effect, triple-SMM can fix and recover a total of three shapes, including the permanent shape, all in one cycle. Bellin et al. discovered two distinct thermal transition temperatures that can be used to obtain the triple-shape memory effect [24]. A cross-linked polymer with two distinctive glass transition temperatures can be independently shaped into two temporary shapes with two programming cycles and two recovery cycles [25].

9.3 History of shape memory polymer

Shape memory research has increased significantly over the last few decades prompting interesting developments of novel SMPs. Historically, SMP research can be traced back to 1940. Liu et al. suggested that the emergence of shape memory effects was first recorded in a United States patent in 1941 by L. B. Vernon in which it claimed that a dental material (methacrylic acid ester resin) possessed “elastic memory” [26]. This was backed by Xie and Hager et al. as it was mentioned in their review paper [4,27]. Interestingly, the patent was recorded even before appearance of the first SMA [28]. From there, the research and development of SMP does not receive much further attention until the 1960s. The development of cross-linked polyethylene tubes and films that shrink in response to heat recognized the importance of SMP [29–31]. The use of the term SMP was first reported in 1984 by CDF Chimie Company with the invention of polynorbornene-based SMP [6]. Regardless of the early history in SMP discovery, the effect of shape memory in polymers remained alien to the scientific community. Limited research projects on the materials were associated with a lack of possible applications of the material [4]. The advancement of research on SMP remained inactive until 2000, when Lendlein et al. proposed a self-tightening plastic surgery-stitch using SMP for minimum surgery after-effect [13]. Liu shows that the amount of research works on SMP worldwide has been growing since the 1990s by the number of total publications [26]. Back then, research works in SMP were mainly to prepare the material for various industrial applications without emphasizing the fundamental theory involved. Bellin et al.’s work in 2006 found that two thermal transition properties of SMP could be tailored independently to recover two temporary shapes within one shape memory cycle [24]. That particular functionality has driven industrial research to harness more tailored applications. In compliment to the academic research, focus mainly lies in biomedical applications, which started to investigate the theory and principle mechanisms of these materials. This is also motivated by the large degree of research funding apportioned to biomedical research in general. The discovery of PU SMP by Mitsubishi Heavy Industries Ltd. sparked a new interest in SMP material due to the flexibility of urethane, which can be easily tuned for various purposes and industrial applications [4]. Research of PU has

remained quite active until today for the same reasons, although many new SMP materials have been discovered since.

9.4 Reinforcement of shape memory polymer (SMP)

Although SMP has advantages in various aspects of its structure, there are a few obvious flaws that could be improved, which are low mechanical strength and shape recovery stress [32,33]. The topics have been major areas of research over the last decade. The addition of reinforcement such as fiber or filler is an alternative method to obtain SMP with increased stiffness and better recovery stress [6,9,34]. For micro-sized fillers such as chopped carbon and glass fiber, an increase in stiffness can be obtained by the addition of 40–50 wt% [35]. In the case of nanofiller, only 20 wt% of SiC is required to achieve a 50% increase of epoxy SMP recovery force [36]. There is also a certain case where the addition of filler improved the recovery force in the polymer but was accompanied by a decrease in recovery strain [9]. Adding 30 wt% of carbon black into the shape memory polyurethane (SMPU) led to a decrease in shape recovery rate from 98% to 65%. However, addition of carbon nanotube to the same polymer showed an increase in both recovery of strain and stress, while the addition of nanoclay showed an increase of 25% of shape recovery stress and only a small decrease in recovery strain ratio [37,38]. Mechanical behavior of SMP can be tailored by either modification of the molecular structure by addition of reinforcement into the matrix to form composites. SMP composites can be classified by the type of reinforcement that they employ. The most common types of reinforcement are particle type reinforcement and fiber type reinforcement.

SMP filled with particles such as carbon black, CNT, carbon nanofibre (CNF), SiC, Na, clay, and short continuous fibers, can be classified as particle reinforced SMPC. Other than the reinforcement material, particle-filled SMP also develop other functions such as high electrical conductivity, magnetic responsiveness and light responsiveness, which classifies them as multifunctional materials. Carbon nanomaterials have been proven to have exceptional mechanical strength and elastic modulus and are thus attractive as reinforcing materials to improve mechanical strength and shape recovery in SMPs. CNT and CNF are widely used as fillers to produce conductive SMP composites. There are several methods used to fabricate SMP incorporating carbon nanoparticles. The first one is to mix the nanofillers directly with the polymer matrix. The second method is to blend surface modified CNT or oxidized CNF with the matrix polymer. The third method is to convert either CNT or CNF into thin paper or film and incorporate that into the matrix. An additional method to fabricate CNT-reinforced composites is by aligning the CNT inside polymer matrix in the presence of an electric/magnetic field, or by crosslinking CNT with the polymer matrix. On the other hand, a hybrid filler of CNF can also be used to fabricate composite polymers. There are a few challenges that always arise in incorporating CNT into a polymer matrix, among which dispersion and bonding of CNT with the polymer matrix are among the most difficult ones.

Yoo et al. prepared a conductive PU and multiwalled CNT (MWCNT) composite by an in situ polymerization process for an actuator. Three methods were used to fabricate the composite films. The composite exhibited a maximum conductivity of 0.28 Scm^{-1} at room temperature. This was sufficient to heat the composite beyond the melting temperature of the soft segment domain. They showed that the sample could recover its original shape in almost 40 s with a bending mode when a 50 V electric field was applied [39]. Shape recovery via electric current rather than via heating can be beneficial to the application of conductive SMP composites as actuators. Viry et al. showed that assembling CNT in the form of pure or composite fiber was an effective way to orient the CNT in a preferred direction. The CNT-PVC composite fiber was prepared and tested for mechanical and shape memory behavior. The sample produced showed large stress generation and enhanced temperature memory phenomena [40]. Assembly of CNT on the macroscopic scale to optimize material properties can be achieved through the development of fibers. This allows macroscopic orientation of nanotubes into preferred directions. Wu et al. prepared CNT/epoxy with various CNT contents to study their thermomechanical property. The tensile test result showed that SMP with 2 wt% CNT had higher tensile strength than 1 and 3 wt% CNT composites. They also showed that CNT content had no effect on shape recovery speed at 60°C [41]. In order to fully exploit the potential of CNT or MWCNT in polymer composites, it is critical to ensure even dispersion of the nanoparticles in the matrix solution. Lu and Gao showed that better CNT distribution can be achieved by in situ polymerization compared to direct mixing [42]. Yu et al. used CNT filler to bridge gaps and produce conductive channels for electric current. Their study showed that the electrical resistivity of a composite with CNT chains was 100 times lower than that containing random CNTs [43]. Yu et al. compared the reinforcement effect of cup-stacked CNT (CSCNT) and multi-walled CNT on a styrene-based shape memory composite. The composites containing different fiber contents (0.5, 1, 1.5, 2 wt%) were fabricated and tested for mechanical and shape memory behavior. From their research, they found that CSCNT exhibited superior interface bonding and load transfer efficiency between the SMP matrix and fillers [18]. Abishera et al. investigated reversible plasticity shape memory effects on epoxy composites reinforced with nanotubes. They showed that the addition of MWCNT increased the mechanical and reversibility plasticity shape memory properties like shape fixity, response temperature, and response speed. Varying different parameters such as MWCNT content, transition region, and programming conditions, the composite can be effectively designed to meet specific requirements [44]. Table 9.1 summarizes the properties of the mentioned SMP composites incorporated with CNT. For research that fabricated variable content fillers, only the optimum sample was mentioned.

Similar to CNT, CNF have received considerable interest due to their excellent thermal and electrical properties. Additionally, the mechanical properties of CNF are better than carbon fiber. Lu et al. studied the effect of CNF on the shape recovery of SMP composites. CNF was blended with Veriflex E2 to improve the thermal conductivity of the SMP and accelerate the electroactive response. The electrical resistivity of the SMP composite was lowered from 1.954 to $0.96 \text{ }\Omega\text{cm}$ by the addition of 2.5 g CNF into the SMP resin. A faster response from electrical resistive heating was observed,

Table 9.1 Comparison of properties of shape memory polymer (SMP) composite reinforced with carbon nanotubes (CNT)

Author	Polymer matrix	Filler type	Fabrication method	Filler quantity (wt%)	Tensile strength	Electrical resistance/ conductivity	Recovery speed
Yoo et al. [39]	PU	MW-CNT	In situ	3	—	0.28 Scm ⁻¹	40 s at 50 V
Viry, L. et al. [40]	PVC	CNT	Aligned	15	120 MPa	0.1 Ωcm	9 s at 150°C
Wu, X. et al. [41]	Epoxy	CNT	Blend	2	42 MPa	—	180 s at 60°C
Yu, K. et al. [43]	Styrene	CSCNT	Blend	2	38.12 MPa	—	77.3 s at 7.5 V
Abishera, R. et al. [44]	Epoxy	MW-CNT	Direct Mix	2	32.06 MPa	—	—

however, the shape recovery ratio was reduced due to interfacial friction between the SMP macromolecular segments, carbon nanopaper, and CNF [45]. Incorporating CNF paper into SMP resulted in better shape memory effects than blending the SMP matrix with a conductive filler. Lu et al. demonstrated a shape memory recovery experiment using SMP with 1.2 g CNF conductive paper, and obtained almost 100% recoverability when 12 V was applied for 140 s. Flexural tests conducted showed that SMP composites coated with CNF paper had higher flexural strength than pure SMP, and the mechanical strength depended on the weight fraction of CNF [46]. However, due to the heterogeneous dispersion of CNF paper in the SMP, the flexural strength showed no regular pattern. It is expected that a more controlled dispersion of CNF paper could be used to program the recovery process of the SMP.

Graphene was discovered almost a decade ago and has presented an interesting feature as a reinforcing material [47] since its discovery in 2007. Popularized as the strongest material, researchers showed high interest in using graphene for applications in various industries, including the aerospace industry. Graphene is chemically analogous to CNT in terms of its properties and structure. Currently, there are three strategies for incorporating graphene into an SMP. This is either done by direct mixing of graphene with the polymer matrix, blending oxidized graphene with the polymer matrix, or by converting graphene into film form and incorporating that into the polymer matrix [3]. It was found that oxidized graphene generated up to 30% higher modulus increase in SMP composites compared to raw graphene [48]. Graphene has high electrical conductivity, hence can effectively enhance the conductivity of SMPs. Choi showed that 1.0 wt% graphene doped into a polyurethane SMP had an electrical conductivity of $10\text{--}2\text{ Scm}^{-1}$ and required 100 V electrical voltage to induce actuation via resistive heating [49]. Tan et al. fabricated a series of graphene oxide (GO)-filled SMPU nanofibers and studied the composites. Mechanical tests obtained indicated an increase in Young's Modulus as GO content increased, with a 4.0 wt% (12.9 MPa) of GO having quintuple the modulus of 0.0 wt% (2.8 MPa) [50]. Wang et al. investigated the effect of graphene content on thermoelectric responsive shape memory hydro-epoxy (H-EP) for actuators. The results showed that graphene content of 2.0 wt% produced the highest increase of bending strength at about 47% larger than the composite with 0.0 wt% graphene. Further analysis with SEM showed that at 3.0 wt%, the graphene appeared to agglomerate and fold, compared to a uniformly dispersed graphene at 2.0 wt% [11]. Jiu et al. prepared shape memory PU containing graphene nanosheet (GNS) by in situ polymerization ranging from 1.0 to 8.0 wt% to be compared in terms of thermal, electrical, mechanical, and shape memory properties. They showed that 2.0 wt% graphene in shape memory PU resulted in fine dispersion and high mechanical strength [51]. Williams et al. investigated graphene-reinforced SMP epoxy applications in aircraft structures. Their result showed improved shape memory recovery of thermally actuated SMP epoxies at a T_g near 168°C when graphene nanofiller was added. Calculations based on thermomechanical data showed a reduction of crosslinking density and an increase of elasticity as a result of graphene addition [52]. Table 9.2 summarizes the properties of graphene composites obtained from the research outlined in this chapter.

Table 9.2 Comparison of properties of shape memory polymer (SMP) composite reinforced with graphene

Author	Polymer matrix	Filler type	Filler quantity (wt%)	Tensile strength	Electrical resistance/ conductivity	Recovery speed
Jiu, H. et al. [51]	PU	GNS	4.0	47.5 ± 11.5 MPa	18.2^{-4} Scm ⁻¹	95% for 8 s at 80 V
Tan, L. et al. [50]	PU	GO	4.0	5.9 ± 1.8 MPa	—	92.1% for 300 s at 40°C
Wang, Y. et al. [11]	Epoxy	HP- Graphene	2.0	86.4 MPa	21 Ωcm	120 s at 55°C 90 s at 80 V

9.5 Applications of shape memory polymer (SMP)/shape memory polymer composites (SMPC)

Aircraft are designed to be able to perform more than one mission during flight. A conventional fixed-wing aircraft can only have perfect flight performance on one mission of its flight envelope, while having only acceptable performance in other flight missions. The idea of in-flight shape change is a solution to realize a multimission aircraft design. Aircraft performance measures such as speed, range, maneuverability and fuel consumption can be improved by using different shapes. Gomez et al. gives a good review of the unmanned aerial vehicle (UAV) morphing design concept and development. The methods described are explained in detail including their advantages and disadvantages [53]. Developing morphing materials is one of the crucial challenges in designing multi-mission aircraft. Incorporating SMPs into aircraft design is an interesting strategy to create a morphing aircraft. SMP can change its shape upon reaction to stimuli in a low stiffness state and also withstand the aerodynamic loading in a high stiffness state. This property of SMP meets the requirement for morphing skin material. SMP morphing skins are able to withstand aerodynamic pressure in the glassy state and undergo large deformations (up to 100% in strain in some cases) in their elastic mode and can thus act as morphing structures [54,55]. Thill et al. presented a detailed review of morphing skin development, concepts, and technology [2]. The review focused on structures that transform in a span-wise direction, and morphing via chord flexibility.

McKnight and Henry have conducted a study to demonstrate a new variable concept for morphing structures using SMP. The structure novel concept uses a laminate of two contradicting elements consist of a high stiffness element and variable stiffness elements. The function of the constant stiffness element is to provide structural rigidity, while the function of the variable stiffness element is to provide a variable connectivity between the high stiffness elements. In the structural mode, the variable stiffness elements act as a traditional composite structure, while in the morphing mode, the variable stiffness elements become soft and generate a disconnection between the high stiffness elements. In this mode, the composite can generate large axial transformations and bending deformations through body translation of the stiff elements [54]. A representation of this concept is illustrated in Fig. 9.1, which shows how a large deformation is achieved.

This study uses polyurethane SMP as the variable stiffness material and high-yield sprung steel as the constant stiffness materials. The samples were fabricated as multi-layer composites where individual shape memory thermoplastic sheets were sandwiched between adhesion-enhanced high-carbon steel shims. The study assumed that the rigid body elements performed a body translation while the SMP deformed in shear strain. The initial study showed that the geometry of the composite significantly influenced the deformation of the composite structure, and the key parameters were the length and thickness of the stiff elements, and the vertical spacing and horizontal gap between these elements. A number of samples were fabricated based on these parameters and tested to determine the ratio of peak to minimum stiffness via dynamic mechanical analysis (DMA).

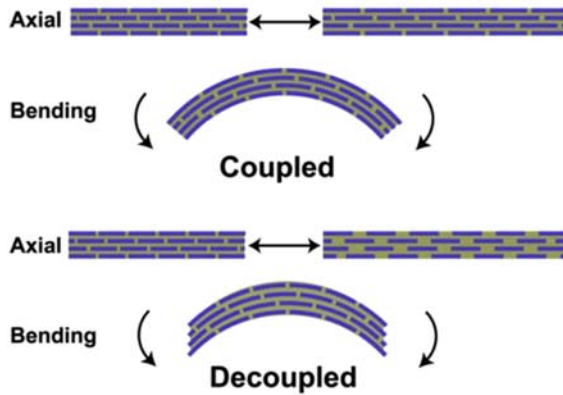


Figure 9.1 Generic concept of the deformation mechanism obtained using variable stiffness elements as connecting materials between high stiffness elements. The blue hexagon represent a high stiffness materials [54].

It was found that the ratio varied from 15 up to 64 for different geometry parameters. These values can be used to determine the sample that is most appropriate for the morphing application. In an application where structural rigidity is required amid transformation, a sample with a large stiffness variable may not be desired, but in other applications, for example, a space mission where gravity is not an important factor, a material with a large stiffness variable may be useful due to smaller actuation and lower power system requirements.

Leng et al. designed a morphing wing that performs two deformations during deployment; shape recovery of the wing from a bent state, and shape recovery of the SMP filler from a compressed state. A styrene-based SMP reinforced with short carbon fiber filler was used as the material for morphing. The designed morphing wing consisted of three parts that were the SMP filler, a metal skeleton, and an SMP plate. An experiment conducted on the metal skeleton showed that the structure recovered to its original shape after 45 s following a 100 V DC voltage [56]. The transformation was steady and gentle and did not generate undesired mechanical impact compared to conventional morphing technology. This SMP filler was also tested for shape recovery via resistance heating from a compressed state. Fig. 9.2 shows that the filler recovered its shape precisely in the air with up to 100% recovery rate to form a standard airfoil within 98 s [57].

Using this morphing technology, aircraft wings could be deformed into a smaller shape and be conveniently stowed in a limited space. The design is also lightweight and cost effective, which has the potential to overcome the drawback of conventional morphing technology. In addition, Yin et al. developed a variable-camber wing concept and performed tests to measure the deflection of the wing using the fiber Bragg grating (FBG) method. The FBG sensor was bonded to the upper surface to measure strain and shape bending of the wing design. Results obtained showed that the reconstructed shape from the FBG sensor compared very well with the theoretical results [58].

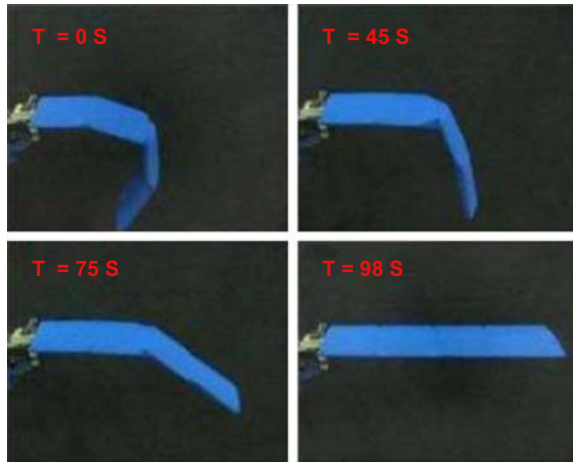


Figure 9.2 Shape recovery of morphing wing with shape memory polymer filler [57].

Reed and colleagues from the Cornerstone Research Group (CRG) designed and patented a shape memory composite named Veritex, designed after the shape memory thermoset, Veriflex, to develop an adaptive wing structure aimed at enhancing the capability of a UAV at loitering condition. The materials had an activation temperature that could be tailored ranging from 47 to 106°C [59]. Fig. 9.3 shows the demonstration of Veritex transforming from temporary rolled out shape to its initial cast aerofoil shape.

This ability shows an extreme flexibility and adaptive capability of the materials for morphing applications. The skin activation system was one of the challenges in developing a morphing wing using SMP due to the need for external stimuli in activating the

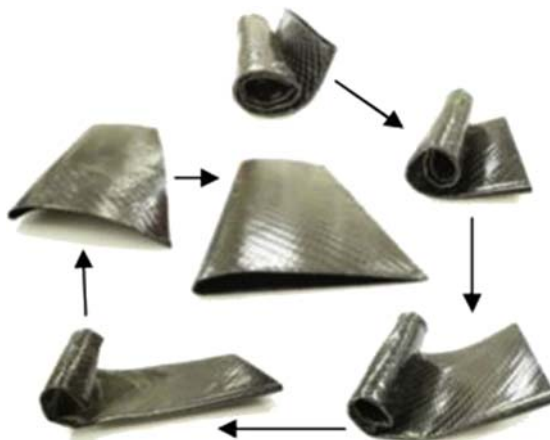


Figure 9.3 Demonstration of wing-shaped Vetirex returning to original state [59].

shape recovery process. In their study, Reed et al. initially integrated a heating wire into the skin and analyzed its effectiveness for the morphing structure. The approach seemed to be an effective one, but a few issues needed to be addressed before incorporating it into the polymer matrix. This included an inherent separation between the heating wire and the polymer upon elongation. An investigation was carried out using different wire thicknesses as well as combinations of wire spacing, size, and power settings to achieve optimum elongation while minimizing activation time. Following this approach, CNF conductive fillers were incorporated and evaluated in terms of conductivity and maximum elongation. CNF showed some desirable characteristics, especially conductivity in the range necessary for the resistive heating of SMP. However, the composite showed a logarithmic increase in resistivity as elongation increased. This leads to a complete loss of conductivity before the minimum elongation of 100%. Use of nichrome-nickel wire fortunately, enabled the materials to deform up to 100% elongation and enabled greater conductivity compared to a CNF-filled SMP. Fig. 9.4 shows a nichrome embedded into the polymer which changes color from black to white when activated.

Yu et al. developed a morphing wing concept based on styrene-based SMP reinforced with carbon fiber. The wing consisted of a carbon fiber-reinforced SMP skin and SMP filler with through holes. The carbon fiber skin provided most recovery stress for the wing, however, when the skin is in a soft state, the SMP filler was necessary to sustain aerodynamic loads. Two-dimensional aerodynamic analysis was obtained from computational modeling and the results showed that the carbon fiber-SMP provided sufficient stiffness to undergo aerodynamic load at 100 ms^{-1} cruise speed. Three-dimensional analysis found that the rectangular wing experienced a bending deformation with a maximum displacement of 27.562 mm at the tip which was acceptable given the dimensions of the wing [60]. Elastic steel slices (ESS) and SMA wire were used to improve shape recovery properties and reinforce the pure SMP. Figs. 9.5–9.7 show a snapshot series of the composite skin deployment. The result showed that both samples had significant increases in deployment speed compared to carbon fiber-SMP.

Yu et al. investigated a variable sweep morphing wing using carbon fiber fabric-reinforced SMP as a hinge actuator to control the wing deformation. A series of experiments were conducted to investigate the mechanical properties of the SMP composite. DMA tests revealed that the storage modulus, E' and loss factor, $\tan \delta$ were dependent on temperature, and the transition temperature, T_g of the SMP was 64°C [61]. Since

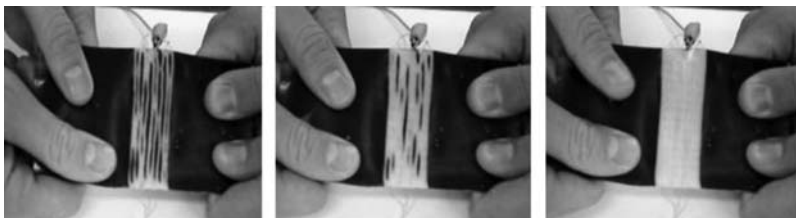


Figure 9.4 Nichrome embedded into shape memory polymer (SMP) [59].

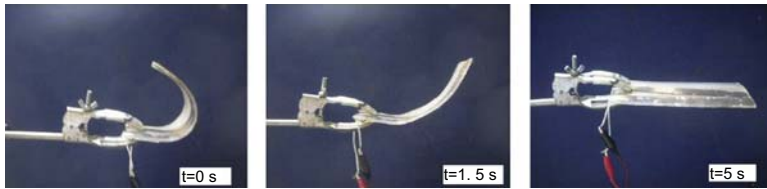


Figure 9.5 Recovery of carbon fiber–shape memory polymer (CF–SMP) composite skin [60].



Figure 9.6 Recovery of ESS-SMP Composite skin [60]. ESS, elastic steel slice.



Figure 9.7 Recovery of shape memory alloy (SMA) wire–shape memory polymer (SMP) composite skin [60].

the SMP was used as an actuator in the wing morphing, a twist recoverability test was conducted to study the time dependency of the twist recovery property of the SMP. The result showed that the SMP composite had a good shape recovery, with increased recovery ratio and increased recovery time. A computational model was developed to study the relationship of the SMP hinge initial angle and wing sweep angle. Results obtained by analysis were validated with a prototype of the wing structure and showed good agreement, indicating that the design enabled wing morphing and altered its configuration by heating of the SMPC hinge.

Sun et al. manufactured, simulated, and tested spandex-reinforced SMPC based morphing skins for variable-camber wing structures. The wing structure consisted of a leading edge, a trailing edge, metal sheet pneumatic artificial muscle (PAM) actuators and an SMP skin. The structure was tested for its shape memory properties and reaction under aerodynamic pressure. An airbag was used to apply aerodynamic load to the SMP skin. The experiment showed that a 10 mm SMPC skin could withstand aerodynamic load at Mach 0.6 flight in high modulus state and alter its shape at Mach 0.1 in low modulus state [62]. The designed wing structure was driven by PAM actuators and required 200 s of preheating in order to morph. Although experimental results tallied with simulation, there was a need to experiment with the morphing design under a real aerodynamic load which could be realized by a wind tunnel

experiment or flight test. Further analysis of the SMPC material is required to understand in detail the SMPC mechanism as a wing skin.

Sun et al. later investigated the effect of incorporating elastic fiber into a styrene-based SMP to improve the toughness and tear strength of the SMPC. The enhanced SMPC was used in the variable-camber morphing wing mentioned before. After initial investigation with different elastic fiber contents via DMA, tensile tests and high temperature and tear strength tests were conducted. DMA revealed that incorporating higher fiber content in the SMP would decrease the T_g and reduced the storage modulus of the composite in the glassy state. This result was supported by the tensile test, which showed a decrease in strength at room temperature. However, the incorporation of elastic fiber increased the strength of composite in the rubbery state above T_g [63]. This enabled a safer use of SMP at higher temperatures. The 2 mm thick SMPCs with 20 vol% fibers was selected to be used as the upper and lower surface of the wing. This skin was able to withstand aerodynamic loading in the glassy state and change shape in the rubbery state. However, the lower skin must be prestretched before installing to avoid wrinkles. Smooth skin morph was achieved in the soft state when the SMP was heated above T_g by a 9.39 W resistance heating film for 200 s. A maximum trailing edge deflection angle of 1.69 degree was obtained when 0.6 MPa PAM input pressure was applied [63]. The resultant variable-camber wing design using SMPC skin could be used for aircraft taking off and landing, employing a different camber for each flight regime. However, further tests should be conducted to experiment with the morphing wing design in various flight conditions such as low temperature, sand, dust, rain, and hail.

9.6 Conclusion

One apparent disadvantage of SMPs is low stiffness compared to SMA and shape memory ceramics. SMPs also have lower shape recovery properties for implementation in morphing structures. It is, therefore, mandatory to conduct a study on reinforcing the material to make it suitable for this particular application. This article reviews the progress and recent developments in SMP composites, incorporating carbon-based fillers for reinforcement and enabling thermal actuation through Joule heating. A selection of experimental processes and results are presented and discussed in order to accommodate recent efforts in the development of incorporating CNT, CNF, and graphene in SMPC to obtain shape recovery. As can be seen, many SMPs described in this review were developed as actuators for morphing structures. However, SMP also has the potential to be developed as a morphing skin, as studied by Yu et al. [60] and Sun et al. [62,63]. Incorporating SMP as a skin material could also reduce the overall mass of the wing design, compared to current skin materials. More research needs to be directed at enhancing the strength of SMPs to withstand load at higher aerodynamic loading. This would enable SMPs to be applied to full-scale aircraft that require more strength for skin materials in the hard state, and also be able to perform shape changes in the soft state. Graphene fillers could produce SMPCs with better mechanical properties compared to CNT and CNF fillers. A synergistic effect of graphene and

another type of filler could be investigated to enhance the performance of SMPCs, especially in terms of strength and shape recovery. Hybrid composites with natural fibers could potentially produce a composite with similar properties at a lower cost, and with the additional benefit of being more environmentally friendly. Research on graphene and other carbon-based reinforced SMPCs is expected to drastically increase the development of SMPs and expand the application scope of smart materials.

Acknowledgments

The authors would like to acknowledge the Aerospace Manufacturing Research Centre (AMRC) and the Laboratory of Biocomposite Technology, Institute of Tropical Forestry and Forest Products (INTROP-HiCOE) for the facilities provided to complete this research. The authors also would like to thank Universiti Putra Malaysia for the close collaboration provided through the Grant Putra GP-IPS/2018/9490602. Special thanks to Institute of Tropical Forestry and Forest Product (INTROP) – HiCOE.

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Fabric-Reinforced Cementitious Matrix (FRCM) composites: mechanical behavior and application to masonry walls

10

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10.1 Introduction

In last decades, composite materials for construction and retrofitting of engineering structures have received a growing interest, which has motivated large research programs and widespread application in engineering practice. The worldwide consensus on the use of fiber-reinforced composite systems in construction industry is mainly inspired by high tensile strength-to-weight ratio, relative ease of installation, and high resistance to corrosion. Among several classes of composite materials, Fiber-Reinforced Polymers (FRPs) have captured most of attention by civil engineers and are used in both construction (e.g., in the form of pultruded profiles, sandwich panels, or reinforcing bars) and structural retrofitting (e.g., in the form of laminates or near-surface mounted bars) [1]. Nevertheless, FRPs have a number of drawbacks due to their resin-based (and hence organic) matrix, including sensitivity to low/high temperatures, vapor impermeability, flammability, inapplicability on wet surfaces or at low temperatures, and poor bond to historical masonry substrates. Such limitations can be overcome by a novel class of fiber-reinforced composites having an inorganic matrix, the characteristics of which significantly promoted their recent application in external retrofitting of concrete and masonry constructions. This class of composites is herein termed Fabric-Reinforced Cementitious Matrix (FRCM) and their mechanical behavior mainly depends on the composite action of single/multiple fiber grids embedded within the matrix, as well as their interaction with the substrate. The matrix consists of a cement- or lime-based mortar, or also a geopolymer, which may include chopped fibers and/or low-content polymeric addition. The composite grid is made of artificial fibers (e.g., aramid, basalt, carbon, glass, polyparaphenylene benzobisoxazole (PBO), steel) or natural fibers (e.g., jute, flax, hemp). According to a wide variety of grid-matrix configurations, FRCM composites have been also identified in the literature as Textile Reinforced Mortar (TRM), Textile Reinforced Concrete (TRC), Cementitious Matrix-Grid (CMG), Inorganic Matrix-Grid (IMG), or Steel Reinforced Grout (SRG). The choice of the matrix and grid along with the determination of total

thickness of the composite system generally depends on the type of substrate and target mechanical performance after retrofitting. For instance, cement mortar is more frequently used for concrete constructions, whereas lime mortar is mainly employed for historical masonry buildings. Indeed, the interaction between similar materials ensures better chemical, thermal, and mechanical compatibility between the strengthening system and the substrate.

Kolsch [2] performed a pioneering investigation on the first generation of FRCM composites in which the organic matrix was simply replaced by a cement-based mortar and the reinforcing material was made of continuous fiber sheets. As a consequence of high viscosity and granular structure of mortar, fiber sheets were not adequately impregnated by the matrix. Therefore, such a technology was improved by replacing continuous fiber sheets by fiber meshes made of continuous fiber yarns with bidirectional or multidirectional geometric configuration. Nonmetallic and natural fibers can be dry, coated, or impregnated, usually by means of a bituminous or polymeric agent. In the case of dry fiber yarns, the mechanical behavior of the FRCM system is further improved in terms of composite matrix-grid action as a result of the intrusion of the mortar within the yarn.

A number of theoretical and experimental investigations have shown the high effectiveness of FRCM systems for confinement, shear, and flexural strengthening of both reinforced concrete (RC) and masonry structural elements. Several studies on RC columns, beams, and slabs were carried out by, among others, Triantafillou and Papanicolaou [3,4], Brückner et al. [5], D'Ambrisi and Focacci [6], Ombres [7], De Caso y Basalo et al. [8], and Loreto et al. [9].

A lot of investigations have been carried out on masonry structural components, most of which consisting of loadbearing walls. In masonry applications, FRCM strengthening systems allow additional requirements to be met, such as chemical, physical, and mechanical compatibility to the substrate, durability, low invasiveness, and reversibility. The last two requirements are mandatory for historical constructions according to current conservation and risk mitigation rules. Prota et al. [10] and Papanicolaou et al. [11] performed the first experimental programs on the effectiveness of FRCM in the case of unreinforced masonry (URM) walls subjected to in-plane loading. The experimental tests in [10] were numerically simulated by Lignola et al. [12]. Conversely, Papanicolaou et al. [13], Harajli et al. [14], and Babaeidarabad et al. [15] assessed the out-of-plane behavior of URM walls with FRCM strengthening. Parisi et al. [16] showed that lime-based mortar can be more effective than its cement-based counterpart when applied to tuff stone masonry (TSM) walls, as a consequence of higher composite-substrate compatibility. That type of inorganic matrix was used by Augenti et al. [17] in the first worldwide experimental tests on full scale URM walls with openings, which were numerically and analytically investigated by, for instance, Parisi et al. [18,19]. Razavizadeh et al. [20] characterized the bond performance of masonry units with SRG strengthening by means of experimental tests and numerical simulations. The same strengthening system was also investigated by De Santis and de Felice [21]. Numerical simulations and experimental tests on FRCM-strengthened URM walls subjected to in-plane loading were also performed by Garofano et al. [22] and Marcari et al. [23]. In addition, the performance of FRCM systems in the

case of URM walls subjected to concentric compressive loading was analyzed by Fossetti and Minafò [24], whereas Bernat et al. [25] and Cevallos et al. [26] studied externally strengthened walls under eccentric compressive loading. Other challenging fields of investigation deal with the effectiveness of FRCM systems with natural fibers [27,28] and the performance of FRCM strengthening techniques at the scale of structural systems [29,30].

All theoretical and analytical studies have shown the major role played by the tensile properties of the FRCM [31,32] and the bond between the FRCM and masonry substrate [33–35]. As in the case of FRP composites, fibers mostly provide tensile strength and stiffness, whereas the matrix allows stress distribution among fibers and provides the fibers protection against environmental actions. Nevertheless, the main difference between FRCM and FRP systems is that the former are influenced by fiber-matrix debonding, rather than potential local buckling phenomena. Indeed, internal slippage at the fiber-matrix interface can strongly affect the tensile behavior of the FRCM, depending on the textile-mortar combination. This is reflected by the response of the composite to uniaxial tensile loading after mortar cracking, which activates a complex nonlinear behavior in which fibers do not typically reach tensile rupture. Recent reviews on different FRCM variants and their application to masonry structural elements have been made by Caggegi et al. [36] for both PBO and aramid FRCMs, Carozzi et al. [37] for carbon FRCM, Leone et al. [38] for glass FRCM, and Lignola et al. [39] for basalt FRCM.

This chapter aims to provide a comprehensive discussion on mechanical behavior of FRCM systems and FRCM-strengthened masonry walls, including important issues related to experimental characterization, capacity modeling, and nonlinear simulation. Such a discussion is deemed strongly important, also because of recent codification activities on FRCM qualification and design for retrofit applications. In the United States of America, the American Concrete Institute (ACI) issued the first guidelines on design and construction of FRCM systems for retrofit of concrete and masonry structures [40]. In response to a widespread application of this strengthening technology after the 2009 L'Aquila, Italy, earthquake, the Italian Superior Council of Public Works launched a similar codification activity on identification, qualification, and quality control of FRCM composites for structural retrofitting of existing constructions.

10.2 Mechanical behavior of FRCM composites

Artificial and natural fibers used in FRCM systems are typically employed in the form of fabrics, grids, or meshes made of a composite material [23,29,32,41,42]. This particular geometry has a double purpose: firstly, the bidirectional shape allows for a better strain distribution throughout the target surface, and secondly, the larger contact area with the matrix and the substrate increases the bond efficiency between the FRCM constituents. In the FRCM application process, the dry reinforcing grid or mesh is applied onto the masonry or RC substrate following the placement of a first layer of inorganic matrix. Afterwards, the grid is pressed against the matrix which acts as a bonding agent between the substrate and the grid itself. Mechanical joints can be

also considered for an effective anchorage of the reinforcing system to the substrate. The final stage of the FRCM application process consists in the placement of a further layer of inorganic matrix which covers the reinforcing grid, providing environmental protection and finishing features. Given this specific configuration of the FRCM application, it appears that the mechanical properties of the single constituents, i.e., fabric/grid and matrix, are potentially involved in the load transfer process between the external reinforcement and the substrate. In particular, the low tensile strength of the inorganic matrix within such systems generally leads to cracking phenomena which commonly affect the interfacial stress transfer between the inner reinforcement, the matrix and the substrate. In addition, possible tension stiffening effects along with effects of adding short fibers in the matrix composition should be properly correlated to the macroscopic mechanical behavior of FRCM.

At the scale of constituent materials, several test procedures have been developed so far to characterize the stress–strain behavior of dry fabric. Specimens can include single or multiple yarns of the fabric material immersed into a mortar or FRP parallelepiped [31,32,34]. The typical experimental behavior is described by load–displacement curves with an elastic phase followed by yarn failure or, in some cases, by a sudden decrease of strength with further slippage of the grid at constant stress. Tensile tests carried out on inorganic matrices still represent a challenge in the field. Several authors have reported on tensile properties' determination by means of flexural tests and indirect tensile tests, whereas only in very few cases direct tensile tests have been performed. It should be pointed out that also the available information on test methods and mechanical performance of FRCM composites is still limited. Indeed, the mechanical performance of FRCM systems still deserves suitable investigations with the final aim of providing reliable parameters such as design values of limit strains, strength, and stiffness as well as bond strength and design limitations.

Composite materials used in the form of reinforcing fabrics/grids/meshes are characterized by very high tensile strength and stiffness along the fiber direction (e.g., carbon, glass, or PBO fabrics), whereas inorganic matrices generally exhibit a poor tensile behavior with severe cracking and low tensile strength. When an FRCM specimen is subjected to tensile loading, the fabric absorbs most of tensile stresses, whereas the matrix has the role of protecting the fibers and distributing the stresses among them. Nonetheless, as the matrix cracking process takes place quickly within the specimen, the tensile behavior of the FRCM is strongly influenced by the subsequent mechanical interaction between the inorganic matrix and the fabric itself. This particular feature is rather new if compared to the tensile behavior of conventional FRPs in which the polymeric matrix is able to accommodate large strains up to fiber breakage.

The constitutive behavior of the FRCM subjected to tensile loads can be typically derived from uniaxial tensile tests on prismatic specimens. The outcomes of those tests are generally used to estimate the mechanical properties of the FRCM for retrofit purposes, such as strength, stiffness, and strain capacity at various performance levels. In contrast to conventional testing techniques adopted for FRPs, there is not yet a commonly accepted testing procedure for characterizing the tensile behavior of FRCM composites. Major technical issues are related to the gripping system used for anchoring the specimen to the testing machine. Among different gripping systems

available [31], the scientific community has conventionally focused the attention on the following configurations: (1) clamping grips (Fig. 10.1a) and (2) clevis system (Fig. 10.1b). In case (1), the two ends of the FRCM specimen are fixed into the grips of the testing machine by means of metallic or fiber-reinforced tabs that are applied using epoxy resin; the tabs facilitate a more homogeneous stress distribution and avoid local damage to the inorganic matrix. By using a low grip pressure (sometimes referred to as “soft clamping”), the clamps produce compressive stresses normal to the plane at the end of the specimens and allow for a very limited torsional rotation. In case (2), metal tabs with a proper bonding length are fixed to the FRCM specimen ends with epoxy resin. The grip system is then set up to provide a pinned-end support [43], allowing multiple degrees of freedom for the specimen. That particular testing configuration reduces bending moments at the specimen ends and allows for possible slippage of the fabric from the matrix. In case (1), such a slippage is prevented by the applied clamping pressure.

The stress states generated by the two grip systems are certainly different. In case (2), only shear stresses are transferred to the specimen, whereas in case (1) the clamping grips generate compression and shear in the specimen to avoid slippage between fabric and matrix at the specimen ends. The influence of the two different testing procedures is mainly related to the failure modes of the FRCM specimens, which will be described in detail in the following. Axial deformations can be typically measured by strain gauges, linear variable differential transformers (LVDTs) or extensometers with

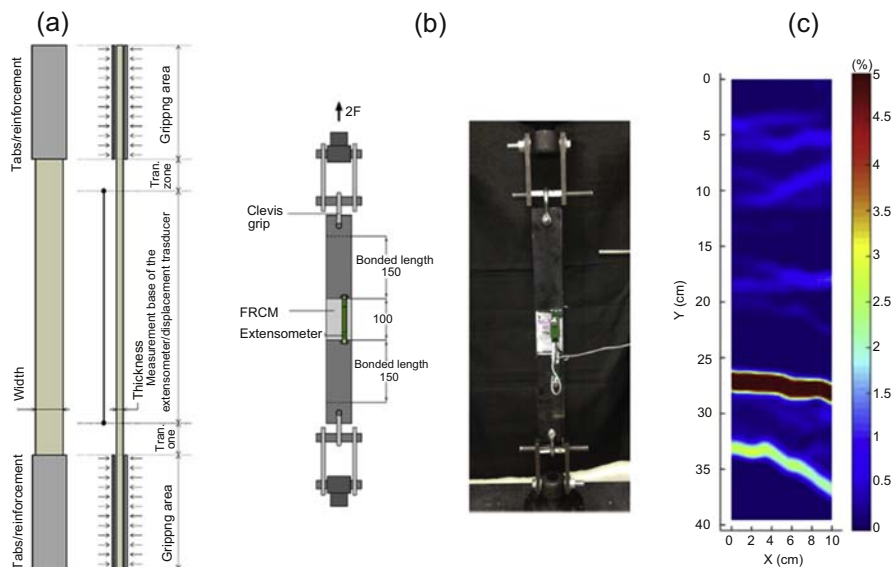


Figure 10.1 Experimental features of tensile tests on Fabric-Reinforced Cementitious Matrix (FRCM) composites: (a) clamping grips [38]; (b) clevis system [46]; (c) digital image correlation technique for strain measurement [44].

minimum gauge length of 50 mm [43]. Nonetheless, those methods provide only locally accurate information about strain measurements and are not suitable when severe or multiple cracking takes place in the FRCM specimen. Recently, photogrammetry and digital image analysis or correlation have been successfully implemented as refined methods to provide a complete overview of the crack formation in the specimen [44] (Fig. 10.1c).

Stress–strain curves of FRCM specimens are conventionally obtained from uniaxial tensile forces divided by the cross-sectional area of the embedded fabric (A_f) rather than by the area of the full specimen (i.e., width by thickness). This procedure is typically implemented to avoid the dependency of the tensile stress on the actual dimensions of the cracked cross-section. The resulting behavior of FRCM under tensile loading is commonly idealized as a trilinear curve [45] representing three physical states of the FRCM specimen (Fig. 10.2). The initial stress–strain phase (stage I) is linear and represents the uncracked state of the FRCM specimen; in that state, the FRCM behaves as a composite material in which stiffness is dominated by the matrix properties, given the greater amount of matrix compared to the fabric. Stage II of the

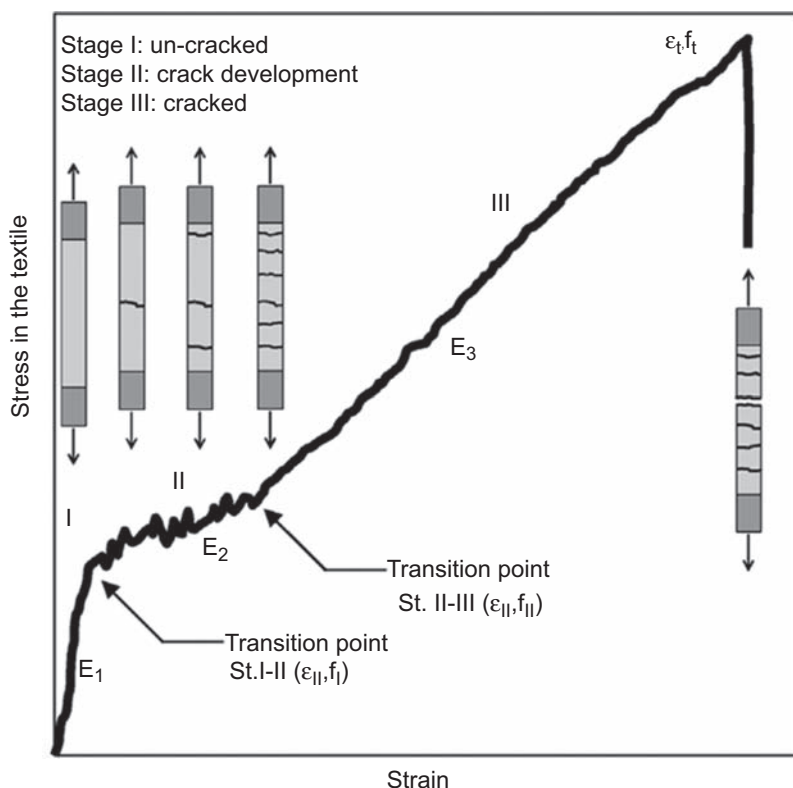


Figure 10.2 Typical stress–strain curves of Fabric-Reinforced Cementitious Matrix (FRCM) subjected to tensile loading [45].

stress–strain curve is associated with the formation of transverse cracks (i.e., normal to the loading direction) within the inorganic matrix. Those cracks can propagate along the specimen or concentrate in a given location, mainly depending on the tensile properties of the matrix and relative amount with respect to the inner fabric. Stage II is characterized by a significant decrease of stiffness due to the loss of a large portion of resisting area. The extent and slope of that part of stress–strain curve vary as a function of the fabric–matrix bond and volume percentage of the fibers still involved in the load transfer process from the matrix. Stage III of the stress–strain curve is characterized by widening of existing matrix cracks and resulting further loading of the fabric. Indeed, in that stage, the fabric withstands most of the overall tensile load and, consequently, the slope of the curve generally reflects the Young’s modulus of the dry fabric. However, when the uncracked matrix between consecutive cracks still contributes to carry the tensile load (as a tension stiffening effect), the slope of the stress–strain curve of the FRCM is the same of the dry fabric, but the curve runs at a slightly higher stress compared to the dry fabric itself. On the other hand, if the tensile strength of the inorganic matrix is very low, the second segment of the curve is very limited and, consequently, a bilinear behavior is often observed.

In the final part of the stress–strain curve, existing cracks become wider up to the ultimate FRCM failure that is potentially associated with three main failure modes (Fig. 10.3):

- attainment of tensile strength of the fabric (brittle failure);
- slippage of the fabric from the matrix (pseudoductility or possible softening);
- combination of previous modes.

In this respect, it is worth noting that FRCM ultimate failure is influenced by several factors. Among them, it has been observed that the testing configuration, particularly the grip system, plays a major role in the determination of failure mode induced by tensile loading. Fabric slippage is maximized in the case of pure shear load transfer (clevis grip system), whereas fabric breakage is achieved only with high clamping pressures (clamping grip system).

Following these considerations, Arboleda et al. [31] concluded that the clevis grip system is more suitable for reproducing the in-service behavior of the FRCM material. This is represented by an initial elastic stage followed by a second branch of the stress–strain curve characterized by a lower slope attributable to matrix cracking and fabric–matrix slippage. On the other side, those researchers reported that the

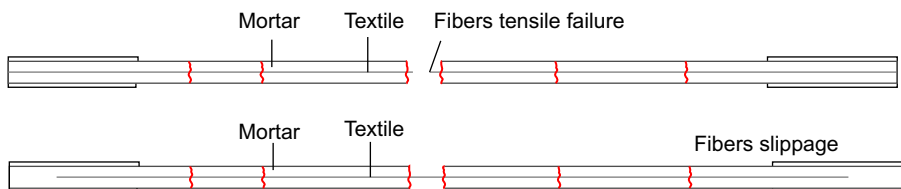


Figure 10.3 Schematization of typical failure modes occurring when Fabric-Reinforced Cementitious Matrix (FRCM) are subjected to tensile loads [37].

clamping grip is able to achieve a complete characterization of the FRCM (i.e., the trilinear approximation of the stress–strain diagram in Fig. 10.2), which allows for the tensile failure of each constituent material of the composite (matrix and fabric).

Besides failure modes, Carozzi et al. [37] pointed out that the testing procedure (clamped or clevis grip system), instrumentation for strain measurement, specimen preparation, and curing conditions are responsible for a large variability in the measurement of both the ultimate tensile stress and slippage initiation limit of the FRCM. Further factors involved in the characterization of the tensile constitutive behavior of the FRCM include: volume fraction of fabric, fabric geometry and spacing, and fabric impregnation or coatings affecting frictional forces between reinforcement and matrix. This latter aspect was studied by Donnini et al. [46] who investigated the effect of different organic coating treatments and an additional quartz sand layer on the bond behavior between a carbon fabric and cement-based matrix. The experimental outcomes showed that the tensile failure mode of the systems investigated could be changed from slippage of the yarns inside the matrix to fibers rupture by improving the interfacial adhesion, specifically by using a coating made of flexible epoxy and sand (Fig. 10.4).

Given the large number of factors affecting the stress–strain behavior of FRCM materials, several authors have tried to characterize the mechanical variability in relation to available types of fabric reinforcement and matrix. For instance, Ascione et al. [45] reported on direct tensile tests that were carried out on five FRCM composites with a clamping grip system, comprising four different textiles (basalt, carbon, glass and steel) and two matrices (cement and lime mortars). Among the FRCM composites, they observed remarkable differences in the uncracked and cracked stages of the corresponding stress–strain curves depending on reinforcement stiffness. The highest stiffness was observed in the case of steel reinforcement and was responsible for the

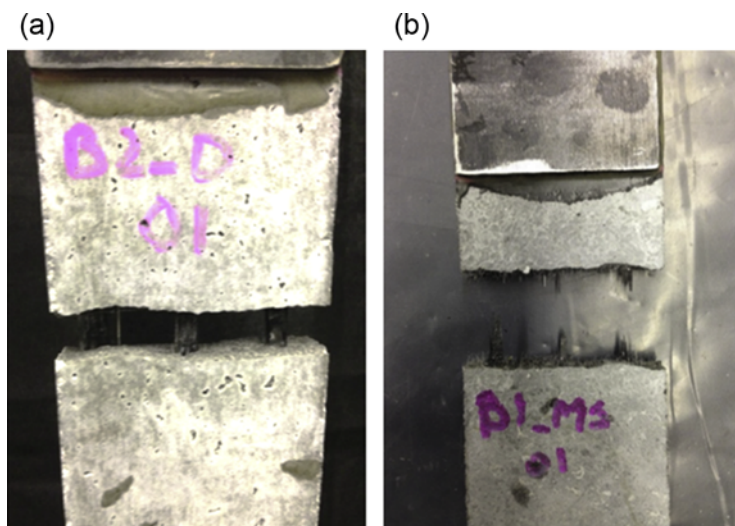


Figure 10.4 (a) Slippage of carbon fabric from cementitious matrix; (b) fabric fracture due to improved interface bond [46].

earliest and widest matrix cracking with negligible transition stage from the cracked to the uncracked stage. This feature was also attributed to the better interlocking with the matrix provided by some reinforcement types (e.g., steel cords). Those reinforcements allow a better crack distribution with respect to that observed on systems with poor fabric-to-mortar bond (e.g., glass, basalt, aramid composite grids) for which crack development is the dominant damage mechanism. Similar conclusions were drawn by De Santis et al. [32] who carried out direct tensile tests on eight types of FRCM composites obtained from two textiles and five matrices. It was shown that the mortar contribution in the first two stages of the stress–strain curves is effective only in composites with relatively weak and flexible fabrics (e.g., those made of natural, glass, or aramid fibers). Conversely, mortar plays an almost negligible role when using very stiff fabrics compared to the mortar properties (e.g., high modulus steel cords in combination with hydraulic lime mortar).

In Fig. 10.5, representative average stress–strain curves retrieved from different FRCM systems subjected to direct tensile tests are shown. The difference between FRCM systems relies in the fabric or mortar type, number of fabric layers, grip system and nature of fibers. It can be observed that FRCM systems based on natural fibers (Sisal-FRCM) have very low stiffness and strength if compared to other FRCM systems made of synthetic fibers. PBO-FRCM systems have the highest tensile stress up to a failure strain of approximately 1.5%. However, the behavior of PBO-FRCM systems is different depending on whether the test is carried out by means of clevis grip system (continuous blue line) or clamping grip system (dotted blue line), providing a stiffer response in the latter case because fabric slippage within the matrix is avoided. This difference is less evident in the case of carbon FRCM (C-FRCM), as shown by red lines in Fig. 10.5. The presence of multiple layers in basalt FRCM

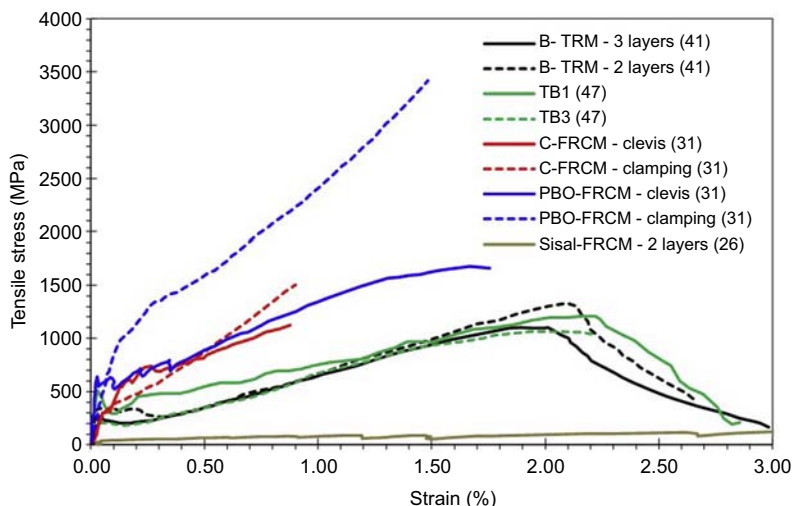


Figure 10.5 Representative average stress–strain curves of different Fabric-Reinforced Cementitious Matrix (FRCM) systems subjected to direct tensile tests.

(denoted as B-TRM, TB1, and TB3 by the authors of tests) does not significantly affect the overall tensile response of the specimens (see the black and green lines in Fig. 10.5). However, in the same cases, there is a softening branch taking place at an axial strain of approximately 2% that can be attributed to the relative slippage between fabric layers and matrix. Table 10.1 provides an overview on strength, limit strains and cracked elastic moduli of different FRCM systems.

Generally, experimental outcomes of tensile tests on FRCM composites are analyzed either with analytical or numerical models [37,47]. The Aveston-Cooper-Kelly (ACK) theory [48] was implemented to define a theoretical stress–strain behavior of an FRCM composite made of a brittle matrix, in which the fabric–matrix bond remains intact in stage I and then is reduced in stage II after that the matrix cracks. According to that model, stage III is representative of dry fabric tensile behavior. The resulting stress–strain behavior is a trilinear curve. Such a simplified model does not take into account possible slippage phenomena between fabric and matrix. Nonlinear numerical simulations with 3D finite element (FE) codes demonstrated a better capability of reproducing experimental results. This is because FE numerical models allows the choice of a bond-slip curve in order to reproduce bond stresses and slippage along matrix–fabric interfaces.

10.3 Mechanical behavior of masonry walls with FRCM strengthening

One of the most important issues in seismic risk mitigation is the high vulnerability of masonry structures. As a matter of fact, a large fraction of seismic damage to existing masonry buildings with lacking or bad seismic detailing (e.g., floor-to-wall and wall-to-wall connections, transverse connections through the thickness of loadbearing walls, lintel above openings) is associated with local collapse mechanisms of some structural subassemblages such as single walls or building corners. If such failure modes are prevented by seismic design (case of modern masonry buildings) or retrofitting, a sort of “box-type” global response of the structure can be activated and damage to loadbearing walls occurs only in their own longitudinal plane. Therefore, it is strongly important to characterize the effectiveness of retrofit solutions against both in-plane and out-of-plane seismic actions, which act in the lateral direction with respect to the vertical axis of walls. This motivates the following discussion on mechanical behavior of URM walls with FRCM strengthening in subsections 10.3.1 and 10.3.2 that critically review experimental and analytical studies on nonlinear behavior of FRCM-strengthened walls subjected to in-plane and out-of-plane loads, respectively.

10.3.1 Nonlinear behavior under in-plane lateral loading

10.3.1.1 Experimental outcomes

Experimental and analytical investigations on URM walls with externally bonded FRCM systems were mainly focused on nonlinear response to in-plane loading. The

Table 10.1 Experimental values of tensile strength, limit strains, and cracked elastic modulus of FRCM systems

Source	FRCM configuration	Grip system	Matrix	Fabric	f_I	ϵ_I	ϵ_{II}	f_u	E_3	ϵ_u
					[MPa]	[%]	[%]	[MPa]	[GPa]	[%]
[26]	Sisal-FRCM	Clamped	Hydraulic lime mortar	Sisal	49	N.A.	N.A.	249	N.A.	7.30
[31]	C-FRCM	Clamped	Cement-based mortar	Carbon	482	0.060	0.24	1492	186	0.74
	PBO-FRCM	Clamped		PBO	890	0.082	0.50	3316	216	1.69
	C-FRCM	Clevis		Carbon	458	0.102	N.A.	1031	N.A.	0.99
[41]	PBO-FRCM	Clevis	Cement-based mortar	PBO	375	0.017	N.A.	1664	N.A.	1.75
	B-TRM (3 layers)	Clamped		Basalt	271	0.045	0.25	1195	57	2.10
	B-TRM (2 layers)	Clamped			308	0.018	0.31	1256	59	1.96
[47]	TB1	Clamped	Cement-based mortar	Basalt	373	0.034	0.40	1088	43	2.15
	TB3	Clamped		Carbon	206	0.028	0.21	1195	57	2.10

Note: N.A. indicates that either the mechanical property was not experimentally characterized (case of artificial fibers) or it cannot be determined on a monotonic curve (case of natural fibers).

majority of experimental programs consisted of diagonal compression tests on masonry panels strengthened on a single side or both sides, with or without transverse connections (Fig. 10.6a).

Prota et al. [10] tested 12 TSM panels strengthened through an FRCM composite composed of a cement-based matrix and a coated alkali-resistant glass grid. All specimens were subjected to diagonal compression loading with monotonically increasing axial displacement. The performance of strengthened masonry specimens with several FRCM configurations (eight in total) were compared to that of four as-built masonry specimens. Experimental data, especially the shear strength at zero confining stress of the as-built or strengthened masonry, were processed according to ASTM E519-81 standard [49]. The peak shear strength at zero confining stress is here denoted as τ_0 . First of all, it was found that the FRCM system can reduce the high anisotropy of as-built TSM panels, increasing shear strength and pseudoductility particularly when a double layer of FRCM strengthening is applied to both sides. In the same strengthening configuration, a moderate increase in initial stiffness was measured through an estimate of shear modulus for the whole FRCM-masonry system. Nonetheless, the importance of initial stiffness is limited to the elastic performance of masonry structures under low-intensity earthquakes. When TSM panels were strengthened on a single side, a parasite out-of-plane bending deformation was observed as a consequence of eccentric axial forces in composite grids. That was a source of asymmetric behavior, which apparently produced a shear modulus lower than the mean value of as-built masonry panels. In terms of failure modes, FRCM-strengthened panels suffered the crushing of tuff stones in the proximity of the corner steel toes used to apply diagonal compression loading. The out-of-plane bending deformation of TSM panels with single-side strengthening generated a more brittle failure that was followed by a lower increase in strength. Conversely, double-side strengthening configurations induced a uniform cracking that improved the shear performance in terms of strength and pseudoductility. No debonding failure was observed, thus allowing a complete exploitation of the FRCM composite in the tensile state produced by diagonal compression loading. In all tests, FRCM strengthening met additional retrofit requirements for historical

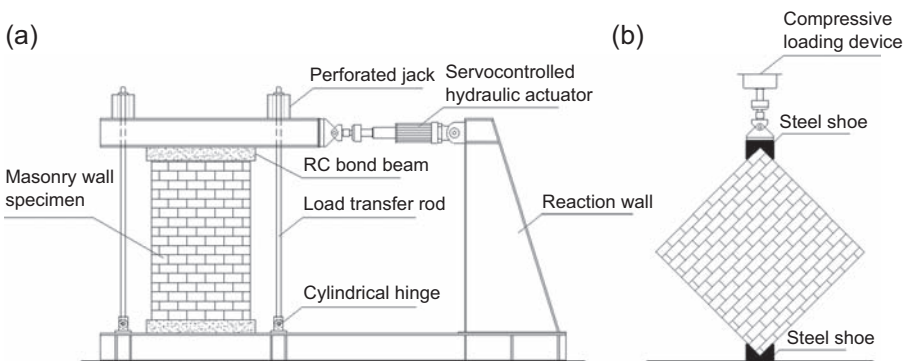


Figure 10.6 Typical setup of (a) diagonal compression tests and (b) shear-compression tests on masonry panels.

masonry structures, such as compatibility with substrate (e.g., no debonding) and reversibility. Based on experimental results, Prota et al. [10] recommended shear strength amplification factors equal to 1.7 and 2 in the case of single-side and double-side FRCM configurations, respectively.

Papanicolaou et al. [11] investigated the shear behavior of FRCM-strengthened clay brick masonry (CBM) under cyclic in-plane loading. Those researchers tested 22 specimens grouped in three series. Series A specimens (shear walls) were subjected to shear-compression testing with displacement control, assuming a compressive load equal to 10% of ultimate load (Fig. 10.6b). Series B and C specimens (beam-columns and beams, respectively) were tested in three-point bending. The in-plane cyclic capacity was investigated under varying matrix (mortar vs. resin), number of textile layers, and compressive load on shear walls and beam-columns. The FRCM composite was found to produce a lateral strength increase between 12% and 30%, depending on the number of textile layers and the axial load level. By contrast, the in-plane displacement capacity increased at approximately 14%–31%. From a comparative viewpoint, the FRCM systems generated a 65%–70% lower increase in strength compared to its FRP counterpart with the same textile configuration. The opposite outcome was found in terms of deformation capacity, because the FRCM was more effective than FRP systems of approximately 15%–30%, 135%, and 350% in the case of shear walls, beam-columns, and beams, respectively. As expected, the strength generally increased with the number of layers and the axial load level, the opposite occurring in terms of deformation capacity.

Papanicolaou et al. [50] extended the previous work in [11] and experimentally evaluated the effectiveness of double-side FRCM strengthening on single-wythe walls consisting of either hollow clay bricks or stone blocks. Both in-plane and out-of-plane loading cases were considered, assessing the influence of the number of grid layers (one or two), the type of fibers (carbon, glass, basalt, polypropylene and polyester), the type of matrix (mortar with different compositions or epoxy resin), and the compressive stress level on specimens subjected to in-plane loading. In the case of shear wall specimens subjected to low axial load levels and with moderate slenderness, the in-plane cyclic response was influenced by the following damage sequence: (1) extensive toe cracking; (2) tensile fracture of the FRCM grid in the most tensioned parts of wall toes; (3) crushing of masonry in compression; and (4) local buckling of the FRCM grid in the same parts of wall toes. Higher in-plane stiffness and strength, along with slightly lower deformation capacity, were found in the case of specimens with double-layer overlays that did not experience tensile fracture of fibers before masonry toe crushing or textile buckling.

Parisi et al. [16] carried out diagonal compression tests with displacement control on both as-built and strengthened TSM panels, the latter having the following alternative layouts: single-side strengthening; single-side strengthening with passing-through SFRP ties; and double-side strengthening. As-built specimens experienced the classical stair-stepped cracking induced by diagonal compression. Single-side strengthened specimens with/without passing-through SFRP ties suffered small cracks on the strengthened side and large cracks on the as-built side, which were associated with out-of-plane bending deformation as shown in Prota et al. [10]. In the absence

of SFRP ties, out-of-plane bending induced pseudoductility ratios lower than those corresponding to other FRCM configurations, although ensuring an increase in both shear strength and pseudoductility. In any case, SFRP transverse ties improved both in-plane capacity parameters of TSM panels with single-side strengthening. When the FRCM was applied to both sides of TSM specimens, a further improvement in shear behavior was observed, allowing no out-of-plane deflections in the postpeak softening stage. All tests showed the absence of debonding failure, even if the FRCM system was not mechanically anchored to the masonry. In that study, the authors processed experimental data according to ASTM [51] and RILEM [52] standards, separately, to assess the in-plane capacity under two different assumptions on stress distribution within specimens. Mean values of shear strength and pseudoductility ratio of as-built specimens were in agreement with previous tests by Prota et al. [10] on the same type of URM specimens. Nevertheless, the FRCM composite with lime-based matrix mortar was more effective than its cement-based counterpart, allowing higher amplification of shear strength and pseudoductility (the latter in the cases of single-side strengthening with SFRP ties and double-side strengthening). Dealing with pseudoductility of TSM panels with single-side strengthening and without SFRP ties, the cement-based matrix was found to be better than its lime-based counterpart. The mean shear strength increase was equal to 90% in the case of single-side strengthening, 140% in the case of single-side strengthening with SFRP ties, and 210% in the case of double-side strengthening. From the standpoint of pseudoductility, Parisi et al. [16] used two strongly different methods applied to monotonic (or envelope) shear stress–strain diagrams:

- a “direct approach” in which the pseudoductility ratio is assumed to be $\mu_\gamma = \varepsilon_u/\varepsilon_e$, where ε_u is the ultimate shear strain associated with a 20% strength drop on the postpeak softening branch and ε_e is a nominal limit elastic shear strain associated with τ_0 ;
- a “bilinear idealization approach” in which ε_e was associated with the corner point of a bilinear approximation of the experimental stress–strain diagram.

The first approach led to mean pseudoductility amplifications approximately equal to 10%, 600%, and 130% in the case of single-side strengthening, single-side strengthening with SFRP ties, and double-side strengthening, respectively. The same FRCM configurations were found to induce 50%, 530%, and 120% amplifications, when the bilinear idealization approach was used. In the case of cement-based matrix, Prota et al. [10] estimated a mean pseudoductility ratio equal to 3.5 that provides a mean increase of approximately 40% regardless of the FRCM configuration (i.e., externally bonded reinforcement applied on a single side or both sides).

Garofano et al. [22] performed shear-compression tests on as-built and strengthened masonry walls composed of adobe bricks and mud mortar. External composite reinforcement was applied on both sides of walls and consisted of polyester or polypropylene grids embedded in mud mortar layers, resulting in a total thickness of 200 mm. Compared to X-shaped crack patterns of as-built walls, a more widespread damage involving both masonry and matrix was observed on strengthened walls. Debonding failure occurred only at the end of tests, so the bond performance of the strengthening system was not critical to the in-plane behavior of masonry walls.

The lateral strength increased of approximately 20% and 30% in the cases of polyester and polypropylene grids, respectively. For the same strengthening solutions, the increase in displacement capacity was equal to 23% and 83%.

Marcari et al. [23] carried out axial and diagonal compression tests on TSM panels with and without FRCM strengthening, the latter on single side and both sides. Compared to the specimens tested by Prota et al. [10] and Parisi et al. [16], the FRCM grid was made of basalt fibers rather than glass fibers. The FRCM matrix consisted of a hydraulic lime mortar. Experimental data of diagonal compression tests in terms of shear stresses were again processed according to ASTM E 519-07 standard [52]. Shear stress–strain diagrams were approximated as bilinear to obtain pseudoductility ratios. Diagonal compression tests confirmed the out-of-plane bending deformation of single side–strengthened panels, one of them suffering not only cracks in the masonry and FRCM matrix, but also tensile rupture of basalt rovings that crossed the panel diagonal subjected to compressive loading in the midheight of the specimen. By contrast, masonry panels strengthened on both sides experienced a more widespread distribution of small cracks, indicating a more even distribution of stresses throughout the specimen. On average, the shear strength increase was between 36% (case of single-side strengthening) and 62% (case of double-side strengthening). The increase in shear modulus was approximately equal to 13% and 39% in the case of single- and double-side strengthening configurations. Finally, for the same FRCM configurations, the mean pseudoductility ratio increased of 130% and 50%, showing higher dispersion in the former case.

Dealing with FRCM systems with natural fiber grids, Menna et al. [28] tested 12 masonry panels in both as-built and strengthened configurations. Two variants of masonry units were considered, that is, tuff stones and solid clay bricks. For each type of masonry, two variants of double-side, single-textile layer, FRCM strengthening consisting of a bidirectional, hemp fiber composite grid were used according to the type of matrix, that is, pozzolana-based mortar and natural hydraulic lime mortar. In the former case, the total thickness of the FRCM was approximately equal to 15 mm on each side, whereas in the latter case it increased to about 40 mm. Furthermore, the FRCM was anchored to the masonry through T- and L-shaped ties, respectively. It is also noted that a proper low-viscosity epoxy resin was used to impregnate dry hemp cords of the grid to maximize the loadbearing contribution and load transfer between hemp fibers and the inorganic matrix. That technological process allowed a good flexibility of the composite grid, which can be easily bended and rolled for onsite applications. Diagonal compression test results were processed according to both ASTM E 519-07 [51] and RILEM [52] standards. The average increase in shear strength of TSM specimens was equal to 86% and 182% in the cases of FRCM with pozzolana-based and natural hydraulic lime mortar matrix, respectively. For the same variants of FRCM system, the strength increase shown by CBM specimens was equal to 431% and 441%.

10.3.1.2 Theoretical models

In order to deepen the significant scatter in the experimental data by Prota et al. [10] in view of capacity modeling of FRCM-strengthened masonry walls, Lignola et al. [12]

carried out nonlinear FE analyses with micromodeling approach, assessing the sensitivity of shear performance to mechanical properties of masonry and FRCM constituents. A satisfactory agreement between numerical simulations and experimental data was found. The effects of potential workmanship defects were also investigated in the form of partially filled mortar joints of masonry, which were found to be a source of inherent variability in the overall performance of strengthened panels to diagonal compression loading. In detail, when masonry joints are fully filled with mortar, a more uniform cracking of TSM panels takes place and the shear strength increase due to the FRCM is maximized. By contrast, in the case of partially filled joints, cracking tends to concentrate along the diagonal line of loading. Numerical simulations confirmed the experimental observation that the FRCM strengthening can reduce the anisotropy of masonry and sensitivity to construction defects because both stresses and cracks are more uniformly distributed, providing higher shear strength and inelastic deformation capacity.

Based on the outcomes of full scale lateral loading tests on perforated TSM walls with and without FRCM strengthening on both sides of masonry spandrels [17], Parisi et al. [18] calibrated a nonlinear micromechanical FE model and proposed a closed-form analytical model for the prediction of the flexural capacity of URM walls subjected to in-plane lateral loading. The novelty of that formulation was that existing American guidelines assumed the FRCM to be effective only as shear reinforcement [40], considering the in-plane shear capacity of the strengthened wall equal to:

$$\phi_v V_{ns} = \phi_v (V_m + V_f) \quad (10.1)$$

where: V_m is the shear strength of the URM wall computed according to existing formulations [53]; and V_f is the FRCM contribution to shear strength. According to the American safety format [40], Eq. (10.1) includes a strength reduction factor for shear $\phi_v = 0.75$. The shear strength contribution of the FRCM is computed as follows:

$$V_f = 2n A_f L f_{fv} \quad (10.2)$$

where: n is the number of grid layers; A_f is the area of mesh reinforcement by unit width effective in shear; L is the wall length evaluated in the direction of lateral loading; and $f_{fv} = E_f \epsilon_{fv}$ is the design tensile strength of the FRCM system, assuming that E_f and $\epsilon_{fv} \leq 0.004$ are the Young's modulus and ultimate axial strain in tension. In this respect, it is noted that the ACI 549.4R-13 guidelines [40] assume a bilinear elastic behavior for the FRCM composite in uniaxial tension. The first branch of the stress-strain diagram is associated with an initial stage up to cracking of the matrix, whereas the second branch simulates the cracked behavior of the composite. Therefore, in the ACI capacity model, E_f denotes the tensile Young's modulus of the cracked FRCM.

Nonetheless, if the composite is well anchored, it was shown that the flexural contribution to the in-plane capacity can be significant. Parisi et al. [18] developed a flexural capacity model under the following assumptions: (1) plane cross-sections remain plane after flexural deformation, although in the presence of shearing deformation according

to the Timoshenko beam theory; (2) masonry is regarded as a no-tensile-resistant (NTR) homogeneous material with elastic-perfectly plastic behavior in compression defined by the limit elastic strain $\epsilon_{e,m}$, ultimate strain $\epsilon_{u,m}$, and strength $f_{d,m}$; (3) the FRCM matrix is an NTR material with linear elastic behavior in compression up to ultimate strength $f_{c,M}$ and axial strain $\epsilon_{c,M}$; (4) the FRCM grid does not provide compressive strength and shows linear elastic behavior in tension up to ultimate strength $f_{t,F}$ and axial strain $\epsilon_{t,F}$; and (5) bond strength of matrix is sufficient to delay brittle debonding failure modes after that ultimate limit state (ULS) is reached, allowing strain transfer to the grid (Fig. 10.7).

In that formulation, the thickness of the masonry wall and FRCM system are respectively denoted as t_m and t_r , whereas the cross-sectional resistant area of the i -th yarn under the neutral axis and its distance from the compressed edge were denoted as A_i and d_i , respectively. The FRCM constituents are thus separately modeled and both mechanical and geometrical nonlinearities are taken into account. The flexural capacity of the FRCM-strengthened wall at ULS is governed by the following balance equations:

$$N_d = \int_0^c \sigma_m(\epsilon) t_m dx + N_{sides} \int_0^c \sigma_M(\epsilon) t_r dx - n N_{sides} \sum_i \sigma_{F_i}(\epsilon) A_i \quad (10.3)$$

$$M_u = \frac{f_{d,m} t_m c}{12 \mu_c^2} [3 L \mu_c (2 \mu_c - 1) - 2 c (3 \mu_c^2 - 3 \mu_c + 1)] + f_{c,M} t_r c \left(\frac{L}{2} - \frac{c}{3} \right) + 2 \sum_i E_f \epsilon(d_i) A_i \left(d_i - \frac{L}{2} \right) \quad (10.4)$$

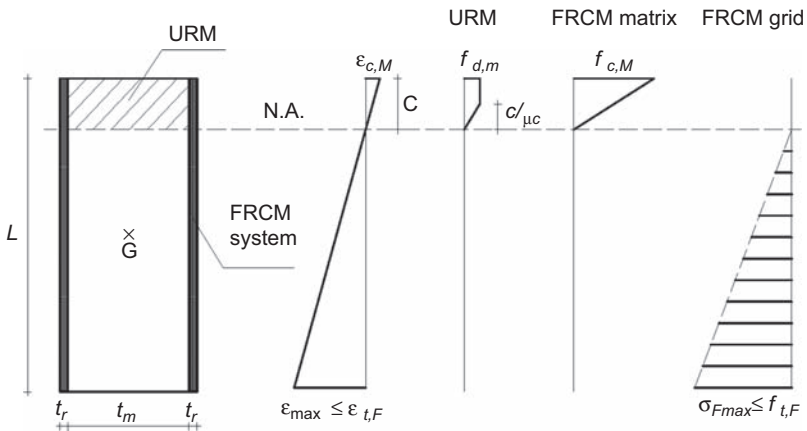


Figure 10.7 Flexural capacity model of Fabric-Reinforced Cementitious Matrix (FRCM)-strengthened cross-section of unreinforced masonry (URM) wall subjected to in-plane lateral loading.

where N_d is the design compressive load on the wall; c is the neutral axis depth; x is the distance of the generic cross-sectional fiber from the compressed edge of the cross-section; $\sigma_m(\epsilon)$, $\sigma_M(\epsilon)$, and $\sigma_{Fi}(\epsilon)$ are the normal stresses in masonry, inorganic matrix, and i -th yarn, respectively, corresponding to the cross-sectional strain $\epsilon(x)$; μ_c is the pseudoductility ratio of the FRCM-strengthened cross-section in compression; and M_u is the ultimate bending moment. In the case of (1) FRCM system with single grid layer (i.e., $n = 1$) placed on both wall sides (i.e., $N_{sides} = 2$) and (2) low axial load levels, Eqs. (10.3) and (10.4) can be simplified as follows:

$$N_d = f_{d,mtmc} \left(1 - \frac{1}{2\mu_c} \right) + f_{c,Mtrc} - 2 \sum_i E_f \epsilon(d_i) A_i \quad (10.5)$$

$$M_u = \frac{f_{d,mtmc}}{12\mu_c^2} [3h\mu_c(2\mu_c - 1) - 2c(3\mu_c^2 - 3\mu_c + 1)] + f_{c,Mtrc} \left(\frac{h}{2} - \frac{c}{3} \right) + \sum_i E_f \epsilon(d_i) A_i \left(d_i - \frac{h}{2} \right) \quad (10.6)$$

considering that the axial strain capacity in compression can be limited by the ultimate strain of the FRCM matrix $\epsilon_{c,M}$ (that means $\mu_c = \epsilon_{c,M}/\epsilon_{e,m}$). Given N_d , one can evaluate the neutral axis depth by numerically solving Eq. (10.3) or (10.5) because the cross-sectional area in tension depends on the number of grid yarns providing flexural strength to the masonry panel (geometrical nonlinearity). Then, the bending moment capacity of the strengthened cross-section can be computed through Eq. (10.4) or (10.6) and the in-plane lateral strength corresponding to the flexural resisting mechanism of the wall can be predicted as $V_{nf} = M_u/h_0$, being h_0 the distance between the wall cross-sections subjected to zero and maximum bending moment. Accordingly, the in-plane lateral strength of the FRCM-strengthened masonry wall can be analytically defined through the following equation:

$$V_n = \min(V_{ns}, V_{nf}) \quad (10.7)$$

10.3.2 Nonlinear behavior under out-of-plane lateral loading

10.3.2.1 Experimental findings

Harajli et al. [14] investigated the out-of-plane behavior of masonry wall specimens by means of four-point bending tests (Fig. 10.8a), which were carried out in both monotonic and cyclic fashions. Specimens were 1.2 m-long and 1 m-wide and were simply supported with clear span equal to 1000 mm. Three types of masonry units were considered, that is, hollow concrete blocks, clay bricks and sandstones. The following constituents were used for the FRCM strengthening system: (1) matrix consisting of lime-cement mortar (both in its normal- and high-strength variants) or cement mortar; and (2) textile made of either bitumen coated E-glass fibers or coated/uncoated basalt fibers. All strengthened specimens experienced a mixed failure mode consisting of (1)

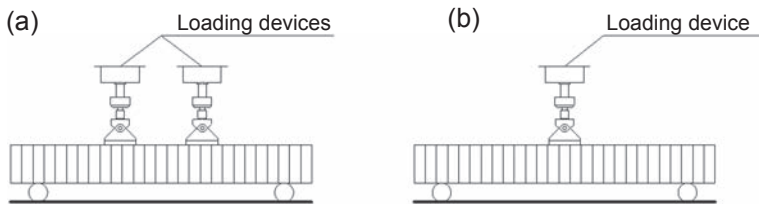


Figure 10.8 Typical setup of (a) four-point bending tests and (b) three-point bending tests on masonry panels.

wide cracks within masonry, (2) transverse debonding from the substrate due to relative slippage among masonry units, and (3) a combination of transverse shear and tensile fracture of textile fibers. Particularly the specimens with cement mortar (that is stiffer than lime-cement mortar) suffered a single crack within masonry, resulting in a premature shear fracturing of the FRCM textile at the location of the crack and lower load capacity. It is worth noting that those researchers found a load capacity increase lower than that theoretically expected for fibers able to develop their peak tensile strength. Based on a simple capacity model, it was estimated that, in failure conditions, fibers were subjected to a maximum tensile strain ranging between 20% and 70% of the ultimate strain, with mean of 48%. That outcome was motivated by a progressive fracture process of fibers in a combined shear-tension mode. Strengthened specimens subjected to monotonic loading showed a significant increase in both load and displacement capacities, which was rather the same over all masonry units under investigation. The FRCM strengthening of specimens subjected to cyclic loading produced a substantial increase in both load and displacement capacities, low degradation in stiffness and strength, and relevant energy absorption and dissipation capacities, highlighting a significant performance improvement compared to that of URM walls. Lime-cement mortar allowed a better cyclic behavior than cement mortar because of partial fracturing of the textile. The FRCM system made of coated basalt textile and lime mortar allowed the least stiffness and strength degradation, the highest energy absorption and dissipation capacities, and the best combination of load capacity and ductility under out-of-plane loading.

Papanicolaou et al. [13] tested two series of CBM specimens representing piers (series A) and walls (series B) by means of cyclic, three-point bending tests (Fig. 10.8b). CBM was made of perforated clay bricks and cement mortar. An external reinforcement system was applied on both sides of specimens using both inorganic and resin-based matrices as well as single- and double-textile layers. As far as series A specimens (i.e., piers) are concerned, the FRCM allowed an even distribution of fine cracks on the coated surface in the direction parallel to masonry bed joints, opposed to the case of resin-based composites. Therefore, the lower tensile capacity of the cementitious matrix compared to its resin-based counterpart produced a widespread cracking that allowed the specimens to experience higher deflections at failure and to develop a higher load capacity. Damage to strengthened specimens consisted of X-shaped cracks in bricks, gradual crushing of brick webs, and gradual FRCM debonding from the masonry substrate. The increase in load and displacement capacities generated by the

FRCM strengthening with single-textile layer was respectively 22% and 141% higher than that observed in the case of resin-based matrix. When two textile layers were applied, those ratios were equal to 17% and 61%, respectively. In the case of series B specimens (i.e., walls), the as-built masonry wall suffered a single crack at the mid-span under monotonic out-of-plane loading. That crack propagated stepwise in adjacent bed joints (that is, along the longitudinal axis of the specimen) without inducing damage to bricks. All specimens suffered diagonal cracking during the early cycles of the response, as observed in series A. That type of cracking produced the failure of specimens with two textile layers. By contrast, the failure of specimens with a single-textile layer was caused by tensile fracture of the FRCM system at the midspan. The increase in load and displacement capacities of the FRCM-strengthened specimens was 19% and 14% lower than that observed in the case of resin-based matrix, indicating a lower effectiveness of FRCM systems when failure is controlled by tensile failure of the externally bonded reinforcement. This was not the case of specimens with two textile layers, which showed an increase in load and displacement capacities equal to 13% and 39% higher than those related to their resin-based counterparts. A double number of textile layers produced an increase in load and displacement capacities that was 52% and 33% higher than that evaluated for a single-textile layer, and this is motivated by a failure controlled by the masonry wall.

Papanicolaou et al. [50] widened their previous investigation, assessing the difference in nonlinear performance of FRCM-strengthened specimens with geogrids and basalt fiber grids. Those tests confirmed that FRCM systems can outperform their FRP counterparts in terms of both load and displacement capacities if tensile fracture of the textile reinforcement does not occur. Regardless of the strengthening configuration, adequately anchored FRCM systems showed an increase in load and displacement capacities equal to 400% and 130%, respectively. This occurred even in the weakest FRCM configuration with geogrid and low-strength mortar. The strengthening effectiveness was maximized by the fact that the textile terminated beneath the end bearings of the specimen, so that it was mechanically anchored because of clamping. In real-world applications, a good anchorage of the FRCM on the masonry wall could be achieved through mechanical fasteners or fiber-bundle spike anchors.

Bernat et al. [25] performed eccentric compression tests on FRCM-strengthened walls in order to investigate their nonlinear performance against out-of-plane, second-order deformation produced by eccentric gravity loads. Thus, such an experimental program did not focus on the out-of-plane lateral capacity considered in seismic applications, but it deals with slender loadbearing walls and/or highly eccentric compressive loads. Three FRCM configurations were considered as follows: glass fiber grid with Portland-based mortar, glass fiber grid with lime-based mortar, and carbon fiber grid with pozzolana-based mortar. In addition, four types of masonry mortar were taken into account. Irrespective of the FRCM type, the loadbearing capacity of masonry walls increased of approximately 100%. No debonding failure was observed, whereas the wall failure was controlled by masonry crushing. The FRCM strengthening also reduced the dispersion of wall response and increased both the in-plane and out-of-plane stiffness, the former chiefly depending on the type of FRCM mortar and the latter on the type and amount of fibers.

Babaeidarabad et al. [15] assessed the out-of-plane capacity of as-built and FRCM-strengthened CBM walls under uniform lateral loading, which was applied through a vertical airbag with maximum pressure of 0.14 MPa (Fig. 10.9). The airbag was protected through a 6 mm-thick plywood sheet on each side and was placed between the wall specimen and a reaction wall. In addition, to three as-built walls assumed as control specimens, those researchers tested three walls strengthened with 1-ply FRCM and three walls strengthened with 4-ply FRCM. The FRCM was made of carbon fibers and cement mortar, and the strengthening system covered the whole tension face of each wall. As-built walls suffered flexural failure after developing cracks located at the mortar joints near midheight. Strengthened walls suffered flexural failure and shear failure in the case of 1-ply and 4-ply reinforcement, respectively. Flexural failure consisted in the formation of a horizontal crack, which was followed by fabric slippage within the matrix at midheight that continued to open as the load increased until fabric ruptured at wall collapse. Shear failure consisted of hairline vertical cracks in the masonry substrate close to the supports, which developed at a slope of approximately 45 degrees. Therefore, 4-ply reinforcement was considered somehow excessive because shear controlled the failure mode of strengthened walls. The mean ultimate moment capacity increased of 175% and 653% in the case of 1-ply and 4-ply FRCM configurations, respectively. Moment–deflection curves highlighted a bilinear out-of-plane behavior composed of a first stage controlled by tensile cracking of the masonry mortar and a second stage influenced by the tensile contribution of the FRCM reinforcement. The slope of the second branch increased with the FRCM amount. The strengthening system increased both the out-of-plane stiffness and pseudoductility, the latter property decreasing as the reinforcement ratio increased. The

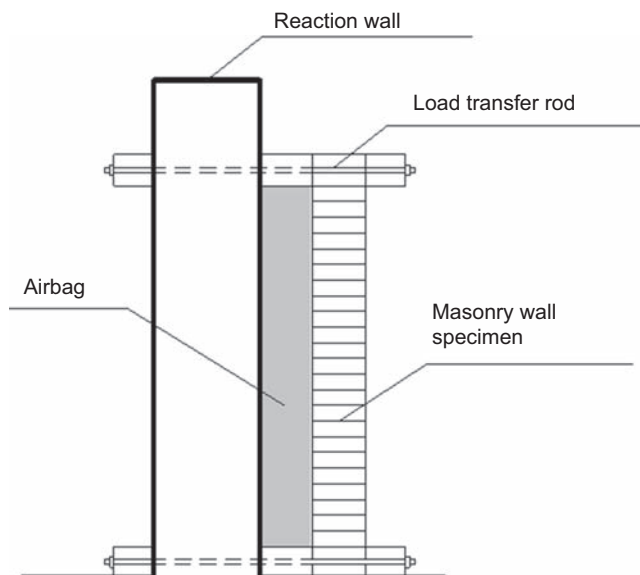


Figure 10.9 Setup of airbag out-of-plane tests on masonry panels.

mean ultimate deflections of the strengthened walls with 1-ply and 4-ply FRCM was approximately 18 and 7 times higher than that reached by as-built walls.

Bernat-Maso et al. [54] investigated the effectiveness of FRCM systems with sprayed mortar in increasing the out-of-plane capacity of masonry walls and in saving application time. The as-built masonry was composed of solid clay bricks. Out-of-plane behavior was evaluated by means of three-point bending tests, considering different variants of mortar (cement, lime, pozzolana-based, projectable plastering), fiber grid (glass, carbon, steel, basalt), strengthening sides (single, double), and textile layers (one, two). It was noticed that the strengthening mortar has a significant influence on the failure mode. The following failure modes were observed: (1) flexural collapse controlled by the tensile rupture of the FRCM grid; (2) mixed failure consisting of flexural cracking of the FRCM and shear sliding failure of masonry; (3) shear sliding failure of masonry; and (4) FRCM delamination after flexural cracking (debonding beginning at the midspan crack and spreading toward the supports). While as-built CBM specimens did not withstand their own weight, strengthened specimens developed significantly higher capacity. The FRCM with cement, lime, or pozzolana-based mortar allowed a higher increase in loadbearing capacity, compared to that based on plastering mortar. No significant drawbacks were observed in the case of sprayed mortar because the latter fully passed through the grid allowing a satisfactory bond performance of the FRCM system. The application of the FRCM on both compression and tension sides of walls did not distinctly improve the out-of-plane behavior compared to single-side configurations. The increase in bending moment capacity was approximately equal to 225% and 80% in the case of basalt and glass fiber grids, respectively. That experimental program confirmed that doubling the amount of reinforcement does not duplicate the capacity of the strengthened masonry wall.

Cevallos et al. [26] further investigated the nonlinear behavior of CBM walls under eccentric gravity loads that can produce second-order out-of-plane deformations. Three types of masonry were prepared based on clay bricks bonded with a natural hydraulic lime mortar combined with a flax- or PBO-FRCM composite. Compared to the PBO fabric, the flax fabric allowed higher levels of deformation capacity to be achieved, mitigating the risk of debonding failure. Indeed, debonding was observed only on walls strengthened with PBO-FRCM. The ultimate load capacity of walls strengthened with flax- and PBO-FRCM systems was 120%–165% and 128%–153% higher than that of as-built walls.

10.3.2.2 Theoretical models

To predict the increase in the out-of-plane loadbearing capacity of masonry walls strengthened with FRCM systems, the ACI 549.4R-13 guidelines [40] provide a simplified analytical model based on the following main assumptions: (1) strain compatibility between masonry and FRCM composite material; (2) plane sections remain plane after loading; and (3) bilinear tensile behavior of the FRCM up to failure in which the FRCM contribution before cracking is neglected. A number of failure modes are considered for an URM wall with FRCM strengthening, as follows: (1) masonry crushing in compression; (2) FRCM debonding from the masonry substrate; and

(3) slippage of the fiber mesh within the matrix. The design tensile strain ε_{fd} of the FRCM is assumed as an upper bound to the effective tensile strain ε_{fe} of the composite material at failure, as follows:

$$\varepsilon_{fe} = \varepsilon_{fd} \leq 0.012 \quad (10.8)$$

Therefore, the effective tensile stress at failure in the FRCM reinforcement is computed as $f_{fe} = E_f \varepsilon_{fe}$. The design bending moment capacity is thus defined on the basis of the nominal bending moment capacity M_n through the following equation:

$$\phi_m M_n = \phi_m (M_m + M_f) \quad (10.9)$$

where $\phi_m = 0.6$ is the strength reduction factor for flexure; M_m is the contribution of the URM if a nonzero axial load is applied; and M_f is the contribution of the FRCM composite. In double-side FRCM configurations, the contribution of the composite material on the compression side is neglected. Furthermore, the contribution of the FRCM on the out-of-plane shear strength is neglected as well. In terms of boundary conditions and resisting mechanism, the ACI 549.4R-13 guidelines [40] assume that the masonry wall behaves as a simply supported element with negligible influence of arching effects. As in the case of shear strengthening applications on in-plane loaded walls, those guidelines state that the flexural strength increase should not exceed 50% of the capacity of the as-built masonry wall. In addition, the maximum force transferred by the FRCM is limited to 87.6 kN/m.

Based on eccentric compression tests, Bernat et al. [25] developed a simplified analytical model to assess the ultimate load of FRCM-strengthened masonry walls under gravity loads. Although such an issue does not fall within the scope of this chapter, it is worth noting that failure of those walls was evaluated at sectional level through maximum moment–axial load interaction curves because only in few cases the wall collapsed after a transformation into a mechanism. The failure was defined as attainment of a limit stress (i.e., tensile strength of the FRCM or compressive strength of masonry) under the assumptions of linear strain distribution through the thickness and perfect FRCM-to-masonry bond. The distribution of stresses was assumed to be linear when sectional failure was governed by the tensile failure of the FRCM. Otherwise, a uniform distribution of stresses over 80% of neutral axis depth and tensile force in the FRCM (stress-block capacity model) were considered when sectional failure was controlled by masonry crushing in compression. Despite this high level of simplification, theoretical results were found to be in good agreement with experimental data. That analytical model was also used by Bernat-Maso et al. [55] to evaluate the out-of-plane load capacity of walls. Nevertheless, the same researchers developed a nonlinear FE model that correctly simulated the experimental results according to their 30% scatter. A sensitivity analysis mainly showed that the tensile strength of the FRCM matrix plays a key role and double-side strengthening of walls reduces the out-of-plane deformation, increasing the gravity load capacity.

As per ACI 549.4R-13 guidelines [40], Babaeidarabad et al. [15] performed a sectional analysis of FRCM-strengthened walls to compare analytical predictions to

experimental results in terms of flexural capacity. In addition, to the assumption of plane section after flexural deformation, those researchers assumed (1) perfect bond of the FRCM to the masonry substrate as well as the grid to the matrix, and (2) parabolic distribution of compressive stresses within masonry. Analytical results underestimated the experimental database. Design values of out-of-plane flexural capacity were even more conservative when the ACI guidelines' limitations and the flexural strength reduction factor were applied.

After a comprehensive collection of experimental data on out-of-plane loaded walls before and after FRCM strengthening, Menna et al. [56] developed a sectional capacity model that extends the nonlinear capacity model proposed by Parisi et al. [57] for URM cross-sections. The capacity model allows tensile cracking of the FRCM matrix and gradual debonding of the fiber grid from the matrix to be considered, thus including both tensile and compressive softening of masonry and tensile softening of the FRCM system. The analytical capacity model allowed a good reproduction of the experimental moment–curvature response of masonry sections strengthened through a PBO-FRCM system, which experienced nonlinear softening after that the peak bending moment was reached.

10.4 Conclusions

In this chapter, the authors have discussed on one hand the macromechanical behavior and modeling of FRCM composites, and on the other the effectiveness of FRCM strengthening systems in increasing the capacity of URM walls subjected to in-plane or out-of-plane lateral loading. Special emphasis has been given to the improvement of nonlinear response to earthquake actions, because they often produce heavy damage to existing masonry constructions and huge life losses.

Most of structural engineering research has focused on FRCM composites subjected to uniaxial tensile loading, because they are basically expected to work as tensile-resistant materials. Based on a detailed review of experimental and theoretical studies, the following conclusions can be drawn:

- The macromechanical behavior of an FRCM system under monotonic tensile loading can be generally described through a trilinear stress–strain diagram in which the first branch considers the uncracked state of the matrix and maximum stiffness, the second branch takes into account the progressive cracking of the matrix with a significant stiffness drop, and the final branch reflects the widening of matrix cracks and a stiffness approximately equal to that of the fabric. Tensile failure can be either brittle when tensile strength of the fabric is attained or quasi brittle when relative fabric-matrix slippage occurs.
- The experimental characterization of the tensile behavior is significantly sensitive to the setup adopted, and particularly to the grip system applied to the specimen ends. On one hand, clevis grip systems should be preferred over their clamping counterparts because the former allow for a more realistic boundary condition in which a gradual fabric-matrix slippage can take place. On the other hand, clamping grip systems allow a complete macromechanical characterization because the tensile failure of each constituent can be reached.

- The relative stiffness of matrix and fabric constituents has an impact on the overall stress–strain behavior, inducing large crack propagation or concentration depending on the low or high Young’s modulus of the fabric reinforcement, respectively.

In the second part of this chapter, research studies on FRCM-strengthened masonry walls subjected to in-plane or out-of-plane lateral loading have been reviewed. Dealing with the in-plane capacity of those structural components, the authors have mainly found that:

- FRCM systems applied to URM walls subjected to diagonal compression can produce a shear strength increase between 36% and 441%, along with an increase in pseudoductility ranging from 10% to 130%. Besides, the load and displacement capacities of FRCM-strengthened walls subjected to shear-compression loading can increase of approximately 12%–30% and 14%–83%, respectively. This large scatter mainly depends on the fact that the lower is the capacity of as-built masonry, the higher is the capacity increase achievable with the FRCM.
- FRCM composites applied onto a single side of masonry walls generate secondary out-of-plane bending deformations, resulting in a more brittle failure and a lower increase of the in-plane capacity compared to double-side (i.e., symmetric) strengthening applications.
- The percentage of FRCM reinforcement has a minor influence on the elastic behavior of masonry walls, particularly before matrix cracking.
- FRCM systems mainly influence the postpeak behavior, increasing both the residual lateral strength and deformation capacity after cracking associated with a stress redistribution in tension.

As far as the out-of-plane capacity of FRCM-strengthened masonry walls is concerned, the available studies allow the following aspects to be outlined:

- Most of FRCM strengthening systems are able to provide a considerable gain in terms of both flexural and displacement capacities, the former between 80% and 653% and the latter between 130% and 1700%. However, significantly increasing the number of fabric layers can produce an undesirable shear failure with a lower increase in pseudoductility.
- Failure is often characterized by a progressive loss of strength after that the peak load capacity is reached, producing a satisfactory deformation capacity. In this respect, FRCM grids with low stiffness (as, for instance, those made of natural fibers) allow a more uniform distribution of tensile cracks on the strengthened side. By contrast, stiff grids induce a brittle failure consisting in the formation of a single crack at the midheight of the masonry wall, resulting in grid sliding within the FRCM system and/or matrix detachment from the grid.

Although extensive experimental research on both the in-plane and out-of-plane behavior of URM walls strengthened with FRCM systems has been carried out, a comprehensive theoretical understanding is still lacking and deserves further attention by the scientific community. This should be aimed at providing practitioners with sound mechanics-based engineering models that allow reliable and sustainable strengthening solutions for seismic risk mitigation of existing masonry structures.

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Failure analysis in hybrid composites prepared using industrial wastes

11

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11.1 Introduction

Composite material can replace conventional material almost everywhere because of its light weight and durability. In 1990's composite materials were developed with synthetic fibers as its reinforcement phase, due to environmental causes and various reasons like biodegradability, synthetic fiber reinforced composite materials lost its market in the pursuit of replacing conventional material. To overcome the composite material's disadvantages, researchers introduced natural fiber as the reinforcement material for fabricating the composite material. Although natural fibers are environmentally friendly, in many heavy loading applications these natural fiber-reinforced composite materials cannot withstand heavy loading. For this purpose, researchers have found a few methods to enhance the mechanical properties of the natural fiber-reinforced composite materials. Among them, hybridization was one of the methods used by researchers to overcome the negatives of one fiber with the introduction of secondary reinforcement. Synthetic fiber used as the reinforcement for the matrix material were aramid, glass, carbon, and Kevlar fiber, though synthetic fiber has many good properties like high strength, stiffness, good wear resistance, and high fatigue resistance. There are also major negatives like high cost and poor recyclability and biodegradability, which limit researchers' use of these kind of synthetic fiber [1]. From 1960 to 1990, coir fiber, banana fiber, sisal fiber, pineapple leaf fiber, palmyrah fiber, talipot fiber, spatha fiber, rachis fiber, rachilla fiber, and peitole bark fiber were the natural fibers used with polyester and epoxy resins to fabricate the composite. Also few combination of hybridization was used to enhance the mechanical properties of the composite fabricated. Cost of the materials can be reduced with the use of natural fiber as reinforcement to the composite fabrication [2]. To incorporate the benefits of the synthetic fibers and to inherent the drawbacks of natural fibers, a hybrid composite was fabricated. Mechanical properties of the hybrid composite were found to increase as the volume fraction of the synthetic fiber increase up to a certain optimum value, and after that a negative hybridization effect occurs. Fatigue sensitivity of the hybrid composite increases when increasing the content of natural fiber. Main drawbacks of natural fiber are its moisture absorbing nature. For avoiding these defects, researchers

used synthetic fiber as the protective layer and kept natural fiber inside synthetic fiber while fabricating the hybrid composites [3]. In this chapter, detailed research on the failure analysis of the hybrid composite with and without industrial waste fillers is discussed. Various matrix material, reinforcement material, fabrication methods, and analysis techniques used by researchers to prepare a highly effective composite material were discussed. Use of industrial waste as filler material to enhance the mechanical properties of the composite is also discussed. Different industrial wastes which are harmful to the environment when disposed of were utilized as the secondary reinforcement for fabrication of hybrid composite material. Influence of filler material on the mechanical performance and wear behavior of the fabricated composite is discussed in detail in this chapter.

11.2 Materials and methods

11.2.1 *Matrixes used in hybrid composites*

Composite material was fabricated by combining two phases of materials, namely, the matrix and reinforcement phases. The matrix phase of the composite material was chosen depending upon the requirement of the composite to be fabricated. Very commonly used and commercially available matrix materials for fabrication of polymer composite are polyester resin, epoxy resin, and vinyl ester resin. Various grades were chosen depending on the availability of specific matrix material used by the researchers for fabricating their desired composite. Epoxy resin was used as the matrix material for fabricating the carbon fiber-reinforced composite pipes. Nano filler like carbon nanotubes and boron nitride nanoplates were used as the filler materials to prevent the failure causes during bursting. It was observed that fatigue performance of the nano-filled epoxy composite pipes was better compared with the normal carbon fiber-reinforced epoxy composite [4]. Hybridization was also done by mixing the matrix material. Epoxy/vinyl ester resin and epoxy/unsaturated polyester, i.e., hybrid resins, were used as matrix material to fabricate composite material [5]. TDE-86 epoxy resin with hardener methyl tetrahydrophthalic anhydride and accelerant dimethyl benzyl amine was used as the matrix material in finding the tensile response of braided architecture of the composite fabricated [6]. EpoxAmite 100 (thermoset matrix material) and homopolymer polypropylene PP granules (thermoplastic matrix material) were used as the matrix material. Both thermoset and thermoplastic hybrid composite was fabricated and compared for the tensile strength. In many cases epoxy resin was used as the matrix material to restrict the stiffening effect in the natural fiber [7]. A DISTITRON VEef 220 STZ vinyl ester resin with low volatile content, low styrene, and low air hazard was used as the matrix material [8]. Epoxy resin (Araldite LY 556) and hardener (Ara-dour HY 951) were used as matrix material [16]. Commercial grade epoxy resin Type: LY556 was used to fabricate glass/sisal hybrid composite [17]. This section described the various matrix materials with their chemical grade used by researchers. The matrix material was chosen depending upon the requirement of the final composite product, and in certain cases it was chosen depending on the availability.

11.2.2 Reinforcement used in hybrid composites

Reinforcement material was added to the matrix material to enhance the physical properties of the final composite material. Two kind of reinforcement material was used mostly by the researcher's, namely synthetic fiber and natural fiber. Secondary reinforcement was added to the composite material to further enhance the properties of the composite. When two or more reinforcement material were added to the matrix material, then this kind of composite was called as hybrid composite. Carbon fiber was used as the reinforcement material for fabricating hybrid epoxy composite [4]. Unidirectional carbon fiber and carbon/glass fiber hybrid composite were fabricated to enhance the toughness of the prepared composite [5]. T700-12K carbon fiber and 1420D Kevlar 49 fiber were braided and used as the reinforcement material for hybrid epoxy composite. The braided architecture of the carbon and aramid fiber was varied to obtain the best tensile property for the prepared hybrid carbon aramid fiber-reinforced epoxy composite [6]. Fiber configuration was used while arranging the reinforcing material for fabrication purposes; the fiber configuration was kenaf/glass/kenaf and glass/kenaf/glass reinforced to the epoxy resin to prepare natural fiber-reinforced hybrid composite [7]. Since the basalt fiber have the density twice that of flax fiber, the thickness of the flax fiber was taken as 0.53 mm with tolerance of 0.02 mm whereas the thickness of the basalt was 0.54 mm with the same tolerance limit [8]. Fiber materials are glass fibers woven into a roving mat of 610 gsm and carbon fibers woven into a roving mat of 450 gsm put together to fabricate hybrid composite [16]. Almost all the researches carried on the fiber-reinforced composite, the fiber length, fiber type, fiber orientation, fiber stacking in case of hybrid composites, and fiber treatment, which were varied to evaluate the fabricated composite materials performance. It was inferred from the research that the reinforcement material influences majorly the mechanical properties of the composite prepared. Therefore, it was understood that choosing the fiber type and fiber properties plays a major role for making the composite more liable to the product produced.

11.2.3 Types of industrial wastes for hybrid composite manufacturing

To utilize the industrial waste and to avail the advantageous properties of the industrial waste, researchers uses it as the secondary reinforcement in the fabrication of composite material. Insoluble waste residue called red mud from the alumina production is an industrial waste which is easily available from the aluminum manufacturer [19,21]. Fig. 11.1 shows the different industrial waste used as the filler in composite material to enhance the mechanical property as well as to improve the wear resistance of the fabricated composite. Local marine litter such as kelp brown algae (*Eklonia* spp.) and bivalve mollusk shells (*Veneridae* spp.) were used as secondary biofillers for fabricating wood fiber-reinforced polypropylene hybrid composite (Fig. 11.2). From the results it was inferred that biofillers enhance the mechanical and moisture resistance of the fabricated composites [20]. This overcomes the disadvantage of the natural fiber composite by improving the moisture resistance by adding bio-fillers to the composite.

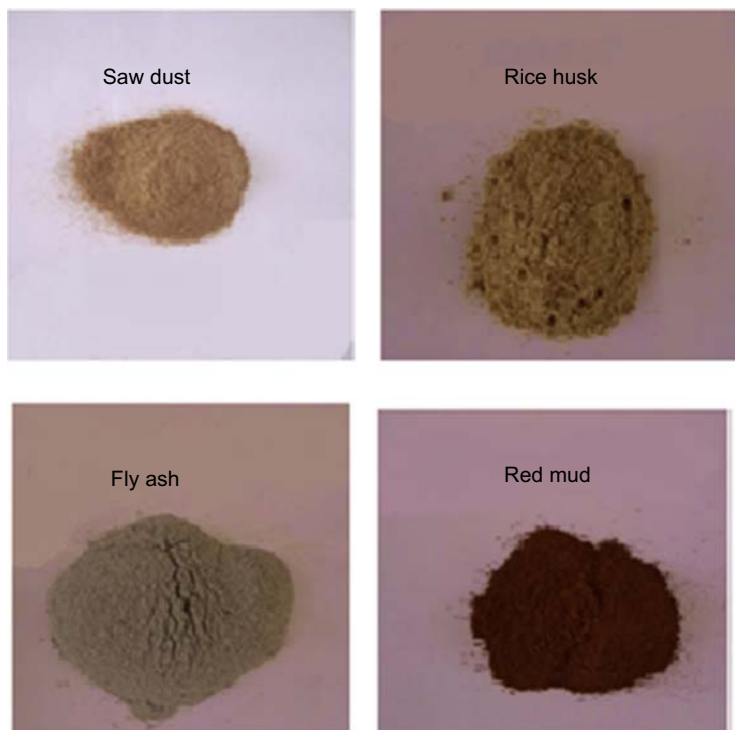


Figure 11.1 Various industrial waste used as reinforcement [22].



Figure 11.2 Marine litter: kelp brown algae and bivalve mollusc shell [20].

Solid waste from the iron ore during separation of iron from it is called blast furnace slag. Blast furnace slag is the industrial waste which was used as the filler material for fabricating short glass fiber-reinforced polypropylene hybrid composites [24]. Fly ash-filled woven jute/glass fabric hybrid composite shows better erosion wear resistance, tensile, and flexural strength compared with the hybrid composite without filler [25].

11.2.4 Hybrid composite preparation methods

There are many methods to fabricate the composite materials, and it varies according to necessity of where the composite is to be utilized, shape of the composite and dimension of the composite, type of reinforcement material, and availability of resources. Very common methods used to fabricate the hybrid composite materials include the hand lay-up technique [5], resin transfer moulding [6], hot press compression moulding [7], resin infusion [8], compression moulding at room temperature [16], and the injection moulding technique [23]. Figs. 11.3 and 11.4 shows the various machines used for fabrication of hybrid composite materials.

11.2.5 Failure analysis methods

Failure in the composite material may be because of various events, namely, the type of the reinforcements, type of the matrix material, fiber orientation, fiber contribution, fiber concentration, fabrication methods, and reinforcement architecture. The acoustic emission (AE) multivariable technique was used to find the failure mechanism and the typical damage that happens during tensile-tested hybrid composites. Depending upon the amplitude of the AE signals from the NOESIS software, the damage mechanism was derived for the composite under study. Fig. 11.5 shows the amplitude to the AE signals for deriving the damage mode of the fabricated composite [10]. The hydrostatic behavior of the composite makes it difficult to use classic models for failure prediction; for that, a modified Arcan's device and the Drucker–Prager model were used to predict the failure in the hybrid composites. Experimental results obtained from the mechanical testing were applied to von Mises and Drucker–Prager models, and the

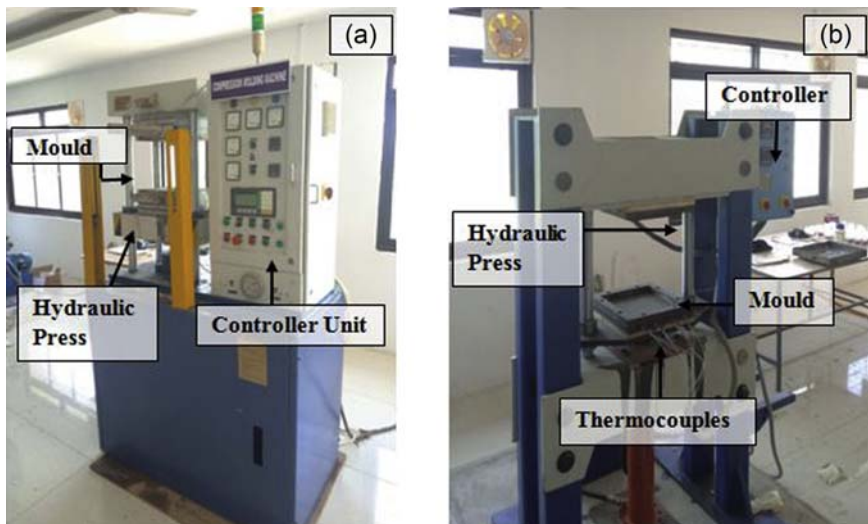


Figure 11.3 (a) Compression moulding machine; (b) hot press mould compression machine available at the Centre for Composite Materials, Kalasalingam Academy of Research and Education.



Figure 11.4 Injection moulding machine available at Centre for Composite Materials, Kalasalingam Academy of Research and Education.

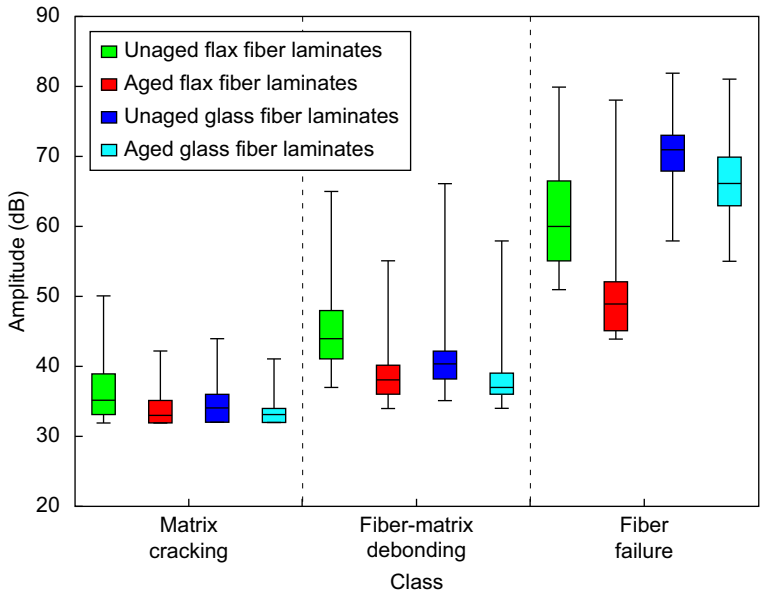


Figure 11.5 Amplitude ranges of acoustic emission (AE) signals versus the three damage mechanisms for unaged and aged nonhybrid composites [10].

failure modes were analyzed for the hybrid composite [11]. Onset failure of the hybrid composite can be predicted from a greater distance with the AE technique [12]. During failure or damage prediction in the AE technique of hybrid composite materials, the parameters like amplitude, rise time, cumulative counts, cumulative energy, rise angle (RA), and felicity ratio (FR) were monitored for damage propagation during

mechanical testing of the hybrid composite specimens [13]. Maximum load the fabricated hybrid helmet composite withstands can be analyzed using ANSYS software. Using ANSYS software, the 3D image of the hybrid composite helmet is analyzed by giving von Mises stress to the design, assigning the load to the point of impact on the helmet in the 3D image gives the maximum permissible load [14].

A Weibull distribution was used to describe the fatigue life and the impact performance of the cementitious composite. The Weibull distribution was adequate enough to give the number of impact causes the initiation of crack to the specimen to the point at which the failure occurred from the impact test data. Fig. 11.6 shows the Weibull distribution of impact energy at the initiation of crack and at failure of the composite

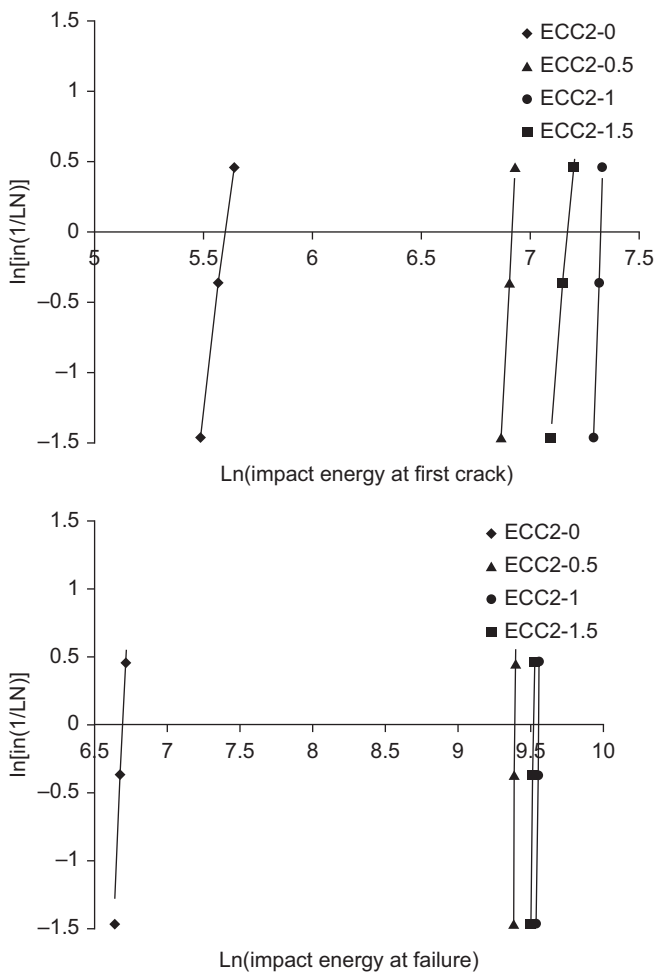


Figure 11.6 Weibull Distribution of impact energy at the initiation of crack and impact energy during failure of Engineered Cementitious Composite (ECC) specimen [15].

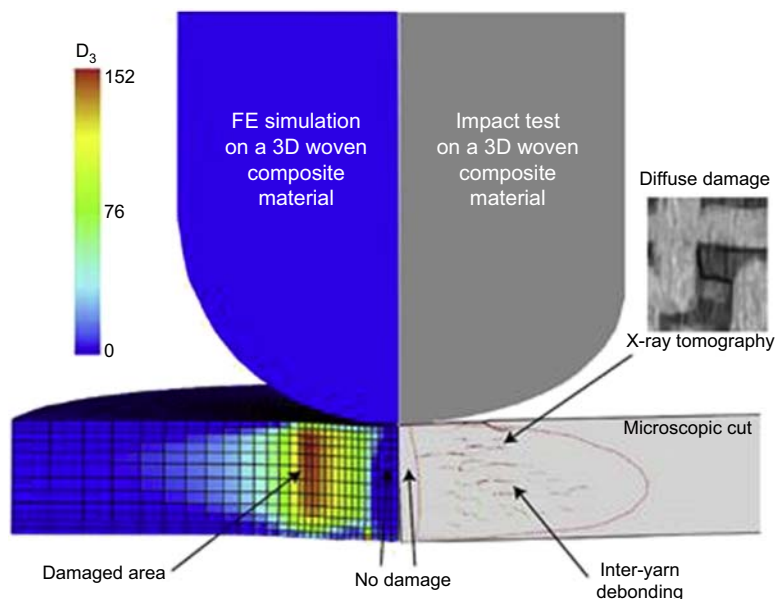


Figure 11.7 Microscopic cut and X-ray tomography of the impacted 3D woven composite plate [26].

specimen [15,16]. Failure mechanism of the mechanical tested hybrid composite was studied using scanning electron microscopic (SEM) images from the JEOL JSM-6480LV SEM [17].

X-ray tomography and microscopic observation was used to determine the damage mechanism of the 3D woven composite plate after impact loading. Matrix cracking and intern-yarn debonding was observed on the impact-tested 3D woven composite specimen. Fig. 11.7 shows the diffuse damage occurred on the impact-tested 3D woven composite plate using X-ray tomography. Finite element simulation on an impact-tested 3D woven composite specimen shows damages, whereas no damage can be seen on the X-ray tomography [26].

11.3 Mechanical performance studies on the hybrid composites

11.3.1 Tensile behavior of the hybrid composites

Tensile strength is the one of the important properties for describing the mechanical performance of the material. Especially for the newly prepared composite material, researchers tend to study the mechanical properties of the material. A universal testing machine was used to determine the tensile strength of the test composite specimen. ASTM D3039 standards were used to measure the axial tensile strength of the

fabricated hybrid carbon aramid epoxy composite with 3D braided architecture. Four different architectures were followed to fabricate the hybrid composite, and the tensile test was conducted to compare which has superior tensile properties. Tensile strength was high when the hybrid architecture was Kevlar braided with a 29.6 degrees axial arrangement of carbon fiber, with 16.7% volume fraction to 58.3% volume fraction of aramid fiber to the epoxy resin. With carbon fiber as axial yarn, the tensile properties improve. It was inferred that the arrangement, i.e., the architecture of the fiber, influence the tensile property of the hybrid epoxy composite [6]. Tensile strength and tensile modulus of the thermoset hybrid composite were 15% and 17% more compared to the thermoplastic hybrid composites. The fiber configuration glass/kenaf/glass shows better tensile property to the kenaf/glass/kenaf configuration. There is improvement of 24% in tensile strength and 14% in tensile modulus for glass/kenaf/glass reinforced thermoset hybrid composite [7]. According to the ASTM D3039-14, the tensile test was performed on the flax basalt hybrid vinyl ester composite. The results showed that the values of the tensile strength and tensile modulus of the hybrid composite was intermediate to the flax and basalt individual reinforcement to the vinyl ester composite [8]. Tensile strength of the carbon fiber-reinforced composite was 150.07 MPa and it gets slightly increased to 158.95 MPa for carbon/E-glass hybrid composite and further it gets enhanced to 167.67 MPa for the carbon/kenaf hybrid composite [9]. When adding shape memory alloy fiber to the cementitious composite, the hybridization enhances the tensile strength of the specimen and this addition can only enhance the tensile strength to certain levels i.e., 1% addition of Shape memory Alloy (SMA) fiber enhances the tensile strength of the specimen beyond this, and when adding 1.5% of SMA fiber, the tensile strength decreases [15]. Tensile strength of the composite is depends on the fiber direction i.e., the direction in which the fiber is placed during the fabrication process influences the tensile strength of the fabricated hybrid composite. The tensile strength of the longitudinally placed fiber was high compared to the transverse direction of the fiber arrangement [17].

11.3.2 Flexural behavior of the hybrid composites

Flexural properties of the composite were measured according to ISO 14125:2011. The hybridization was done with both the matrix and the reinforcement material to observe the flexural properties for various combinations of matrix and reinforcement material. It was observed that the epoxy/unsaturated polyester combination as matrix material had better flexural strength compared with epoxy/vinyl ester resin as matrix material. More than the hybrid carbon/glass fiber with the hybrid epoxy/unsaturated composite the carbon fiber-reinforced epoxy/unsaturated polyester gave better flexural strength, and it proves that the hybridization to the matrix material can contribute to the flexural property of the fabricated composite [4]. Three-point bending tests following ASTM D790-10 were performed on the fabricated hybrid composite. Hybridization of basalt and flax fiber to the vinyl ester composite does not create any drastic enhancement to the flexural properties [8]. Flexural strength of the natural fiber-hybrid composite shows better results than the synthetic fiber-reinforced hybrid composite, the carbon kenaf hybrid composite having 102% improvement whereas the carbon glass

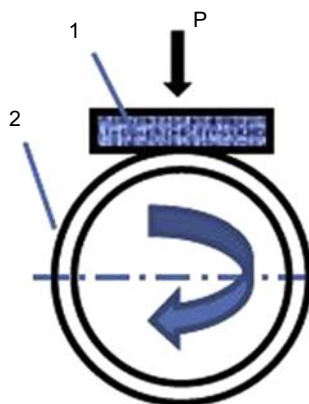
hybrid shows only 53% improvement compared to the neat carbon fiber flexural strength [9]. A flexural test on longitudinally placed fiber shows better results than fiber placed in transverse direction [17]. Replacing the 50% volume of glass fiber with carbon fiber enhances the flexural property of the fabricated epoxy composite, the flexural strength increases by 83%, and the flexural modulus increases by 112%; hence, it was inferred that the hybridization of epoxy composite drastically enhances the flexural property of the composite prepared [18].

11.3.3 Impact behavior of the hybrid composites

ASTM D7136/D7136M-15 was followed to conduct falling weight impact test on the fabricated hybrid composite. The impact strength of the hybrid basalt flax fiber-reinforced vinyl ester composite showed good impact resistance compared to the individual reinforcement of the basalt and flax fiber to the vinyl ester resin. It was observed that the brittleness and stiffness of the basalt fiber is reduced when hybridization is done to the composite material; also, it provides greater flexibility to fabricated composite material [8]. A drop weight impact test was performed on the natural fiber-reinforced epoxy hybrid composite. The hybrid composite was made as a helmet and testing was carried out to check the maximum permissible load the helmet can withstand. It was inferred that the helmet can withstand a 68.57 KN load, but after that the material becomes damaged—this was observed using ANSYS software. Maximum impact energy absorbed by the hybrid composite helmet was found to be 1397.913 KJ [14]. Impact resistance of the engineered cementitious composite increases during addition of PVA fiber to the composite, further when introducing secondary reinforcement with shape memory alloy fiber, again the impact resistance increases. When the specimen is heat treated, the crack initiates at the point where the heat treatment happened. It is inferred that the impact resistance of the cementitious composite increases when hybridization happens and the heat treatment to the specimen makes it easier to crack at first impact itself. In hybridization of glass fiber with sisal fiber to the epoxy matrix, the flexural strength decreases as the content of sisal gets more in the fiber mixture combination due to the high flexural property of the glass fiber [15]. Although the tensile and flexural strength of the hybrid sisal/glass fiber-reinforced epoxy composite decreases upon adding more sisal fiber content to hybridization, impact energy absorption increases when adding sisal fiber content during hybridization of epoxy composite [17].

11.3.4 Wear behavior of the hybrid composites

A reciprocating sliding wear test was conducted on the polyetheretherketone (PEEK) based hybrid composite. Graphite, Short carbon fiber, TiO_2 , ZnS , and SiO_2 were used for hybridization of the PEEK composite. Various combinations of reinforcements were added to the matrix material and the wear studies were conducted. By varying the sliding velocity and contact pressure, wear resistance of the two various combinations and neat PEEK hybrid composite were studied, and it was observed that for the highest contact pressure nanoparticle-added hybrid composite possesses good wear



1-- testing blocks, 2— counter parts

Figure 11.8 Block on ring wear tester [28].

resistance compared to the neat PEEK composite. No significant changes in the wear occur for lower contact pressure and sliding velocity [27]. When adding polytetrafluoroethylene (PTFE) yarn for hybridization of carbon fiber-reinforced epoxy composite the volume wear, the coefficient of friction and the temperature raise increases by 91%, 40%, and 64% compared with the values obtained for the normal carbon fiber-reinforced epoxy composite. For evaluating the wear behavior of the fabricated composite block on ring sliding wear tester was used and it is shown in Fig. 11.8 [28].

11.4 Effect of industrial waste-filled hybrid composites on the mechanical performance

11.4.1 Tensile behavior of the industrial waste filled hybrid composites

When adding red mud filler to the sisal fiber, banana fiber, and glass fiber-reinforced unsaturated polyester composite, the tensile strength of the prepared hybrid composite weakened due to the fibrilization process, i.e., detachment of fiber from the matrix under tensile loading condition [19]. ASTM D638 was followed for tensile testing hybrid silkworm fiber Waste Silk Fiber (WSF)/poplar wood flour (PWF) reinforced recycled polycarbonate composite (RPC). Results from the experiments showed that the tensile strength of the fabricated hybrid composite improves when the Waste Silk Fiber (WSF) content increase even at the absence of the PWF: 62% increase in the tensile strength for WSF filled composite and a 120% increase in tensile strength for PWF-filled RPC [23]. Tensile strength of the pure polypropylene was 39 MPa. When adding the filler to the matrix material to 20 wt% of short glass-fiber reinforcement, the tensile strength decreases gradually from 180.2 to 120.76 MPa. Inclusion of

the blast furnace slag to the polypropylene hybrid composite decreases the tensile strength of the composite [24].

11.4.2 Flexural behavior of the industrial waste-filled hybrid composites

The flexural strength of the banana fiber-reinforced composite and sisal reinforced composite increases upon adding of red mud as secondary reinforced material to the polyester composite. Fig. 11.9 shows the effect of red mud filler to the synthetic and natural fiber-reinforced polyester composite. When adding red mud to the sisal fiber, banana fiber, and glass fiber-reinforced polyester composite, the flexural strength improves by 12%, 6%, and 14% [19]. An experiment was carried out for red mud-filled untreated, NaOH-treated and silane-treated banana fiber-reinforced polyester composite. Flexural studies on the prepared samples showed that the flexural strength of the both NaOH- and silane-treated banana fiber decreases when adding red mud as filler for fabrication of hybrid polyester composite, whereas for untreated banana fiber-reinforced polyester composite, the flexural strength increases slightly when adding red mud as filler in the fabrication process [21]. There is also a 28% and 59% increase in flexural moduli for waste SWF- and PWF-filled RPC [23]. The flexural strength of the blast furnace slag filled short glass fiber-reinforced polypropylene hybrid composite was greatly affected by the filler content due to its high hardness and three times higher density than the pure polypropylene matrix [24].

11.4.3 Impact behavior of the industrial waste-filled hybrid composites

In sisal/banana fiber hybrid polyester composite, results from the impact test shows that red mud-filled hybrid composite enhances the impact energy of the fabricated

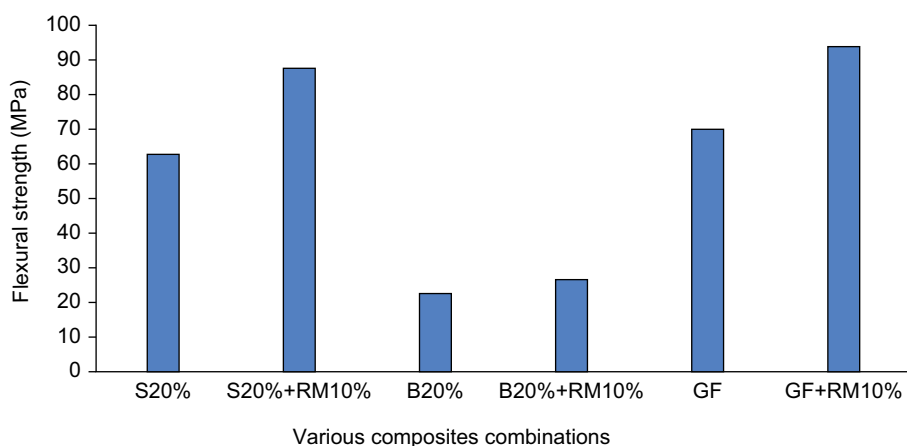


Figure 11.9 Effect of red mud filler to the sisal and banana fiber-reinforced polyester composite [19].

composites. Fiber and matrix interaction gets improved after introducing red mud filler to the hybrid composites [19]. Impact tests were carried according to ASTM D256 with the dimension of $65 \times 13 \times 3 \text{ mm}^3$. When adding red mud, i.e., as the red mud content to the banana fiber-reinforced polyester composite increases, the impact strength of the final hybrid composite increases [21]. As the waste SWF content increases the impact strength of the fabricated hybrid RPC decreases, and the impact strength increases as the PWF content increases by 134%. It was observed that there was significant improvement in the impact strength of the hybrid RPC as the filler content increases [23]. Impact strength of the blast furnace slag filled composite will have good impact resistance due to the high hardness of the material [24].

11.4.4 Wear behavior of the industrial waste-filled hybrid composites

The wear rate of the blast furnace slag filled short glass fiber-reinforced hybrid polypropylene composite was analyzed using Taguchi L_{16} orthogonal array by varying the process parameters of a pin on the disk apparatus, such as sliding velocity, sliding distance, normal load, and filler content. The best combination of process parameters and filler content to achieve the least wear rate was investigated using the Taguchi analysis, and it was found that sliding velocity was the predominant factor affecting the wear of the hybrid composite. Next to sliding velocity, filler content play the major role in controlling the wear to the composite specimen. It was inferred that the blast furnace slag is the best filler particle to enhance the wear resistance of the polypropylene hybrid composite [24]. The standard pin on a disk apparatus was used to perform a dry slide wear behavior test on the hybrid composite, and Taguchi orthogonal array was used to frame the experiment for conducting wear studies on the fabricated hybrid composites [26].

11.5 Failure analysis of the hybrid composites with industrial wastes

Fabricated hybrid composites were taken for mechanical tensile testing, flexural testing, and impact testing, and during such testing conditions, the prepared hybrid composite undergoes certain failure mechanisms. Certain techniques were followed to analyze the damages and the failure caused due to the mechanical testing. The SEM image shows the failure mechanism of the hybrid polypropylene composite. Thus, SEM analysis becomes the very widely used failure analysis technique commonly used by most of the researchers to predict the failure mechanism caused during mechanical testing of the hybrid composite materials. Fig. 11.10a and b shows the SEM images describes the failure mechanism of the mechanical tested hybrid polypropylene composite [7].

Fig. 11.11 shows the SEM image of tensile-tested industrial waste (rice husk) filled polyester and epoxy composite specimen. From the SEM analysis it was inferred that the polyester hybrid composite enhances its tensile strength when rice

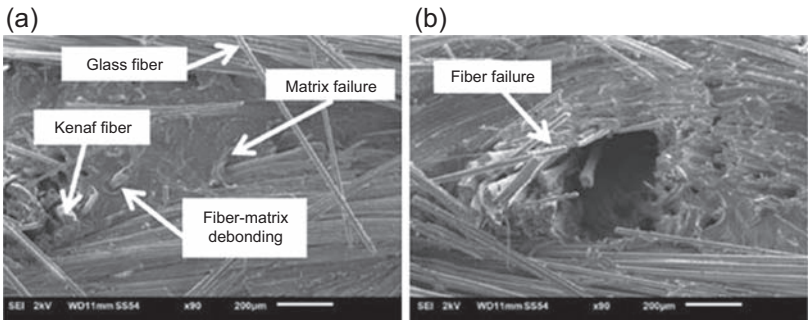


Figure 11.10 Failure mechanism of (KGK) pp hybrid composite under scanning electron microscope (SEM): (a) matrix failure; (b) fiber failure [7].

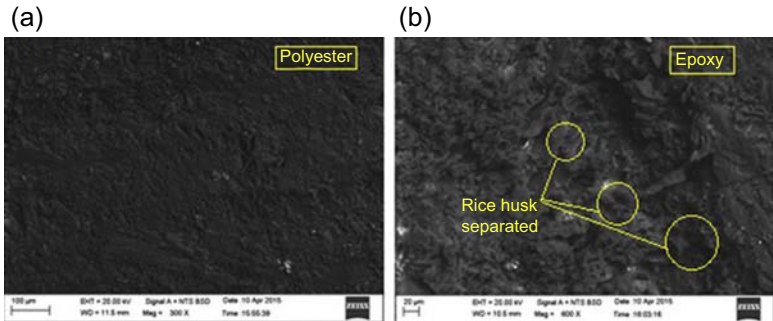


Figure 11.11 Scanning electron microscope (SEM) images (magnification (a) 100 mm (b) 20 mm) on tensile strength of ricehusk reinforced polymer composites [22].

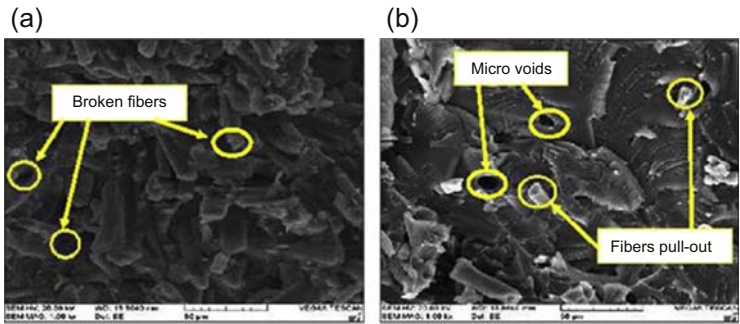


Figure 11.12 Scanning electron microscope (SEM) micrographs of tensile fracture surfaces of the composite types of (a) W0S30 and (b) W30S0 [23].

husk is added as filler material and SEM image of rice husk—filled epoxy hybrid composite shows debris of rice husk, which means the adhesion was poor between the matrix and the filler material, leading to a decrease in tensile strength after an increase in filler content [22].

Waste silk fiber and PWF-reinforced hybrid RPC was fabricated with a counterrotating twin screw extruder. Various damages and failure mechanism during mechanical testing of the composite were analyzed using SEM images shown in Fig. 11.12. It was inferred that the addition of industrial waste filler to the RPC enhances the tensile, flexural, and the impact strength of the specimen compared with the neat matrix material [23].

The composite contact with the counterbody leads to the protrusion of the filler particle from the matrix, and the matrix detachment in the hybrid composite was the wear mechanism found using SEM [24].

11.6 Conclusions

- The potential reuse of industrial waste along with the composite manufacturing drastically reduce the disposal-related issues which cause major environmental hazards.
- Through the incorporation of industrial waste, along with the hybrid composites, good mechanical performance was evident in such a way that the tensile, flexural, and impact strength increases.
- The failure analysis of the composites is needed to suit each composite for a specific application and function effectively without any specific failure. This was also discussed and analyzed in detail in this chapter.

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Index

‘Note: Page numbers followed by “f” indicate figures, “t” indicate tables.’

A

A-FEM. *See* Augmented finite-element-method (A-FEM)
Absorption coefficient, 169
Acetone, 9–10
ACI. *See* American Concrete Institute (ACI)
ACK theory. *See* Aveston-Cooper-Kelly theory (ACK theory)
Acoustic emission (AE), 170–172
 amplitude ranges, 234f
 method in polymer composite, 170–172, 171f
 multivariable technique, 233–235
Acoustic spectrum, 169
Actuation, 187
AE. *See* Acoustic emission (AE)
Aircraft, 189
 wings, 190
Aluminum, mechanical properties of, 138t
American Concrete Institute (ACI), 201
Analysis of variance (ANOVA), 97–98, 103t
 for OHT test, 103
ANOVA. *See* Analysis of variance (ANOVA)
ANSYS software, 233–235
Arcan’s device, 233–235
Aspect ratio, 80–81
 calculation, 160f
ASTM D3039 standards, 236–237
ASTM D5528 standard, 162–163
ASTM D638 standard, 239–240
ASTM D7136/D7136M-15 standard, 238
Augmented finite-element-method (A-FEM), 20
Aveston-Cooper-Kelly theory (ACK theory), 208
Axial compression tests, 213

Axial deformations, 203–204
Azadirachta Indica, 112

B

Babul particulate fiber-reinforced epoxy tensile test composite thickness, 126t
Babul tree, 111
Balharshah particulate fiber-reinforced epoxy tensile test composite thickness, 127t
Balharshah teak, 112
Bamboo fabric–PLA composites, 70
Bamboo fabric–PP composites, 70
Bearing strength of hybrid composites, 83–87
 experiment setup, 84f
 geometric parameters of joint design, 83f
 setup of specimen inside heat chamber, 84f
 works on bolted joint composites, 86t
Bilinear idealization approach, 212
Biodegradable characteristic, 79
Bivalve mollusk shells (*Veneridae* spp.), 231
Blast furnace slag, 232, 241
Bolted joint, structural applications of, 82–83, 82f
Bolted joint behavior of hybrid composites
 bearing strength of hybrid composites, 83–87
 classification of cellulosic plant fibers, 80f
 failure analysis of hybrid composite to bolted joint, 87–91
 hybrid composite materials, 80–81
 structural applications of bolted joint, 82–83
Bond behavior, 206
Boron nitride nanoplates, 230
Buckling, 147

C

- C-FRCM. *See* Carbon FRCM (C-FRCM)
- Carbon black (CB), 178
- Carbon fiber, 231
- Carbon fiber-reinforced plastics (CFRP), 12–13, 61, 174, 175f
- Carbon FRCM (C-FRCM), 207–208
- Carbon nanofibre (CNF), 181–182, 184–187
- Carbon nanomaterials, 184
- Carbon nanotubes (CNTs), 5–6, 177, 177t, 181–182
 - CNT-PVC composite fiber, 185
 - paper-based nanocomposites, 7–9
 - direct synthesis, 8–9
 - domino pushing method, 7–8, 9f
 - spin-coating method, 7, 8f
 - vacuum/pressure filtration, 7
- Catastrophic failure modes, 52–53
- CB. *See* Carbon black (CB)
- CBM. *See* Clay brick masonry (CBM)
- CCP sampling. *See* Central-cut ply's sampling (CCP sampling)
- Cellulosic plant fibers, 80f
- Cement mortar, 199–200
- Cement-or lime-based mortar, 199–200
- Cementitious Matrix-Grid (CMG), 199–200
- Central-cut ply's sampling (CCP sampling), 12–13
- CFRP. *See* Carbon fiber-reinforced plastics (CFRP)
- Chemical composition, 30
- Chemical vapor deposition technique (CVD technique), 8–9
- Clamping grips, 202–203
- Clay brick masonry (CBM), 211
- Clevis systems, 202–203
- CMG. *See* Cementitious Matrix-Grid (CMG)
- CNC machine. *See* Computer Numerical Control machine (CNC machine)
- CNF. *See* Carbon nanofibre (CNF)
- CNTs. *See* Carbon nanotubes (CNTs)
- COFRP. *See* Coir fiber-reinforced polyester composite (COFRP)
- Cohesive zone models (CZMs), 15, 18–19
 - developments, 19
 - early CZM approaches, 18–19
- Coir fiber-reinforced polyester composite (COFRP), 70
- Compatibilizer, 109–110
- Composite(s), 157. *See also* Hybrid composites
 - based on filled and reinforced epoxy, 40–42
 - based on filled epoxy, 37–38
 - deformation of epoxy filled with clay particles, 38f
 - materials, 79, 202, 229–230
 - advantages and disadvantages, 136t
 - rupture and damage mechanisms based on epoxy, 31–37
 - mechanisms of unidirectional composites, 32
 - rupture of laminated composites, 36–37, 37f
 - unidirectional composite submitted to longitudinal tensile, 33–35
 - unidirectional composite submitted to transverse tensile, 35–36
- Compression, 90
 - failure analysis, 149–153
 - moulding machine, 233f
 - test
 - analysis, 147–149
 - testing method, 140–141
- Compressive modulus, average values of, 150t
- Computational model, 192–193
- Computer Numerical Control machine (CNC machine), 140
- Constant stiffness element, 189
- Cornerstone Research Group (CRG), 191
- Crack
 - detection using NDT method, 166–172
 - formation in polymer composites, 158–159
 - propagation, 160–161
- Crack tip opening displacement (CTOD), 2
- Crashworthiness, 52
- CRG. *See* Cornerstone Research Group (CRG)
- Cross-linked polyethylene tubes and films, 183–184
- Cross-linked polymer, 182–183
- CTOD. *See* Crack tip opening displacement (CTOD)
- Cup-stacked CNT (CSCNT), 185

CVD technique. *See* Chemical vapor deposition technique (CVD technique)

CZMs. *See* Cohesive zone models (CZMs)

D

1420D Kevlar 49 fiber, 231

DC. *See* Direct current (DC)

Deformation, 189, 190f

behavior of epoxy-based composite materials

epoxy reinforced composites, 37–42

mechanisms of rupture and damage of composites, 31–37

thermosetting matrix, 30

rupture criteria, 42–47

criteria in principal axes, 43

criteria of maximum deformation, 43–44

criteria of maximum stress, 44–45

deformation criteria outside principal axes, 43–44

interactive criteria, 46–47

Delamination, 1–2, 13–15, 159, 160f, 169–170

Diagonal compression tests, 213

Direct current (DC), 10

Discontinuous-fiber-reinforced polymer composites, 158–159

Displacement curves, 177f

DISTITRON VEef 220 STZ vinyl ester resin, 230

DMA. *See* Dynamic mechanical analysis (DMA)

Domino pushing method, 7–8, 9f

Double-cantilever beam, 12

Double-side strengthening, 211–212

Drafting process, 13

Drop weight impact test, 238

Drucker–Prager model, 233–235

Dual-shape memory effect, 182–183

Dynamic mechanical analysis (DMA), 189

E

E/D. *See* Edge-to-diameter ratio (E/D)

Edge-to-diameter ratio (E/D), 83–84, 91f

EG. *See* Extended graphite (EG)

Elastic memory, 183–184

Elastic steel slices (ESS), 192

Electrophoretic deposition method (EPD method), 10, 11f

Electroresponsive SMPs, 182–183

ELS. *See* End load split (ELS)

ENCB. *See* End notch cantilever beams (ENCB)

End load split (ELS), 12–13

End notch cantilever beams (ENCB), 12–13

End notch flexures (ENF), 12–13

Energy absorption

capability, 52

average load-displacement curves, 53f
strain rate effect, 58–73

ENF. *See* End notch flexures (ENF)

4-ENF. *See* Four-point bending end-notched flexure (4-ENF)

Environmental stress cracking (ESC), 159

EPD method. *See* Electrophoretic deposition method (EPD method)

EpoxAmite 100, 230

Epoxy, 30, 30f

advantages and disadvantages, 30, 31t
characteristics, 30

mechanical characteristics of epoxy matrix, 31t

chemical composition, 30

epoxy/unsaturated polyester, 230

epoxy/vinyl ester resin, 230

reinforced composites, 37–42

composite based on filled epoxy, 37–38

composites based on filled and reinforced epoxy, 40–42

fiber reinforced epoxy composites, 38–40

resin, 230

rupture and damage mechanisms of composites, 31–37

ESC. *See* Environmental stress cracking (ESC)

ESS. *See* Elastic steel slices (ESS)

Ethanol, 7–8

Extended finite element method (XFEM), 15, 19–20

Extended graphite (EG), 4–5

Extraction method, 79

F

- Fabric-Reinforced Cementitious Matrix composites (FRCM composites), 199–200
 - average stress–strain curves, 207f
 - experimental features of tensile tests on, 203f
 - failure modes, 205f
 - mechanical behavior, 201–208
 - of masonry walls with FRCM strengthening, 208–222
- Fabricated hybrid composites, 241
- Failure analysis/mechanisms, 52–53, 54t–56t, 88–90
 - Hashin failure criteria, 89
 - of hybrid composite to bolted joint, 87–91
 - failure criteria, 88–90
 - effect of geometrical parameters on failure modes, 90–91
 - self-loosening of bolted joint, 87–88
 - in hybrid composites
 - hybrid composite preparation methods, 233
 - effect of industrial waste-filled hybrid composites, 239–241
 - with industrial wastes, 241–243
 - industrial wastes for hybrid composite manufacturing, 231–232
 - materials and methods, 230–236
 - matrixes used in hybrid composites, 230
 - mechanical performance studies on hybrid composites, 236–239
 - reinforcement used in hybrid composites, 231
 - maximum stress criterion, 89
 - point stress failure criterion, 90
 - strain rate effect, 58–73
- Failure modes, 205f, 206
 - geometrical parameter effect, 90–91, 92f
- Fatigue delamination of CFRP-matrix composites
 - load control delamination fatigue, 11–13
 - methods for prediction delamination growth, 13–20
 - processing of polymer-matrix composite, 3–10
- Fatigue-life model, 13–15
- FBG method. *See* Fiber Bragg grating method (FBG method)
- FE. *See* Finite element (FE)
- FEA. *See* Finite-element analysis (FEA)
- Felicity ratio (FR), 233–235
- FEM. *See* Finite element methods (FEM)
- Fiber Bragg grating method (FBG method), 190
- Fiber reinforced epoxy composites, 38–40
 - complex modulus vs. frequencies, 40f
 - strain vs. clay content of composites, 40f
- Fiber-reinforced polymers (FRPs), 12, 17, 199–200
- Fiber(s), 174–176, 184
 - fiber-dominated defect, 158–159
 - fiber-stacking sequences, 85–86
 - rupture, 33
- FID. *See* Free Induction Decay (FID)
- Filler, 184
 - content, 241
- Filtration process, 7
- Finite element (FE), 208
- Finite element methods (FEM), 2, 97–98
- Finite-element analysis (FEA), 17, 61
- Flexural behavior of hybrid composites, 237–238
- Flexural capacity of FRCM-strengthened wall at ULS, 215–216, 215f
- Flexural tests, 185–187
- Fly ash–filled woven jute/glass fabric hybrid composite, 232
- Four-point bending end-notched flexure (4-ENF), 12–13, 164–165, 165f
- FR. *See* Felicity ratio (FR)
- Fracture mechanics
 - fracture-mechanics based methods, 15, 17–18
 - in polymer composites, 159–166
- Fracture toughness, 97–98
 - ANOVA for, 103t
 - effect plot for, 102f
 - experimental validation, 105t
 - for polymer composites, 161–162, 172–178
 - interlayer of micro-and nanofiller, 176–178
 - interphase/mesophase between fibers and matrix, 174–176
 - mode I fracture toughness analysis method, 162–164

- mode II fracture toughness analysis
 - method, 164–166
- in polymeric composites, 161–162, 161f, 162t
- surface plot, 104f
- FRCM composites. *See* Fabric-Reinforced Cementitious Matrix composites (FRCM composites)
- Free Induction Decay (FID), 167
- FRPs. *See* Fiber-reinforced polymers (FRPs)

G

- Geometrical parameters effect on failure
 - modes, 90–91
- Geopolymer, 199–200
- Glass fiber-reinforced composites. *See* Glass fibers
- Glass fiber-reinforced plastic (GFRP).
 - See* Glass fibers
- Glass fibers, 12–13, 79, 137–140, 137t
- Glass/epoxy composites, 85–86
- GnPs. *See* Graphene nanoplatelets (GnPs)
- GNS. *See* Graphene nanosheet (GNS)
- GO. *See* Graphene-oxide (GO)
- GO-CFRP. *See* Graphene oxide-CFRP (GO-CFRP)
- GO-filled SMPU nanofibers. *See* Graphene oxide-filled SMPU nanofibers (GO-filled SMPU nanofibers)
- Graphene, 187
- Graphene nanoplatelets (GnPs), 178
- Graphene nanosheet (GNS), 187
- Graphene oxide-CFRP (GO-CFRP), 174
- Graphene oxide-filled SMPU nanofibers (GO-filled SMPU nanofibers), 187
- Graphene paper-based nanocomposites, 9–10
 - EPD method, 10, 11f
 - vacuum filtration, 9–10
- Graphene-oxide (GO), 9–10
- Graphite peeling, 9–10
- Griffith cracks, 173

H

- H-EP. *See* Hydro-epoxy (H-EP)
- Hand lay-up method, 147
- Hashin failure criteria, 88–89
- High density polyethylene nanocomposites (HDPE nanocomposites), 5–6

- Hill criteria, 46
- Hoffman criteria, 47
- Homopolymer polypropylene PP granules, 230
- Horizontal crack formation, 219–220
- Horse tamarind white popinae, 111
- Hot press mould compression machine, 233f
- Hybrid composites. *See also* Composite(s); Wood particulate composites
 - bearing strength of, 83–87
 - failure analysis to bolted joint, 87–91
 - industrial wastes for, 231–232
 - materials, 80–81, 134f
 - composite classifications, 135f
 - composite components in helicopter EC 145 T2, 135f
 - compression test analysis, 147–149
 - failure analysis on tensile and compression, 149–153
 - glass fibers, 137, 137t
 - material fabrication, 140
 - natural fiber composites, 137–138
 - research motivation, 138–140
 - tensile test analysis, 143–147
 - testing method for tensile and compression test, 140–141
- matrixes, 230
- mechanical performance studies, 236–239
 - flexural behavior of hybrid composites, 237–238
 - impact behavior of hybrid composites, 238
 - tensile behavior of hybrid composites, 236–237
 - wear behavior of hybrid composites, 238–239
- preparation methods, 233
- reinforcement, 231
- Hybrid resins, 230
- Hybridization, 70, 229–230
- Hydro-epoxy (H-EP), 187

I

- IFIF. *See* Initial failure indicator factors (IFIF)
- IMG. *See* Inorganic Matrix-Grid (IMG)
- Impact behavior of hybrid composites, 238
- Impact test, 58
 - drop-weight test apparatus, 58f
 - 3D woven composite specimen, 236, 236f

- In situ polymerization process, 5–6, 5f
In-plane lateral strength of FRM-strengthened masonry wall, 215–216
Industrial wastes, 229–230
 failure analysis of hybrid composites with, 241–243
 for hybrid composite manufacturing, 231–232
 industrial waste-filled hybrid composite effect, 239–241
 flexural behavior, 240
 impact behavior, 240–241
 effect of red mud filler, 240f
 tensile behavior, 239–240
 wear behavior, 241
Initial failure indicator factors (IFIF), 70
Injection moulding machine, 234f
Inorganic Matrix-Grid (IMG), 199–200
Inorganic/organic nanocomposite, 5–6
Interactive criteria, 46–47
 Hill criteria, 46
 Hoffman criteria, 47
 Tsai-hill criteria, 46
Interlayer of micro-and nanofiller, 176–178
Intern-yarn debonding, 236
International Union of Pure and Applied Chemistry (IUPAC), 98
Interphase/mesophase between fibers and matrix, 174–176
- J**
Just Mold Fill Immediate Loading (JMFIL), 115
- K**
K/D ratio, 90–91, 91f
Kelp brown algae (*Eklonia* spp.), 231
- L**
LEFM. *See* Linear elastic fracture mechanics (LEFM)
Leucaena Leucocephala (Lamk.), 111
Lignin, 79
Linear absorption coefficient, 169, 169t
Linear elastic fracture mechanics (LEFM), 15
Linear regressions, 105–106
 experimental validation, 105–106, 105t
- Linear variable differential transformers (LVDTs), 203–204
Load control delamination fatigue, 11–13
 mode I, 12
 mode II, 12–13
Load curves, 177f
Local marine litter, 231, 232f
Longitudinal cracking, 32
Longitudinal tensile, unidirectional
 composite submitted to, 33–35
Low grip pressure. *See* Soft clamping LVDTs. *See* Linear variable differential transformers (LVDTs)
- M**
Material fabrication, 140
Material selection, 140
Matrix, 174–176
 cracking, 236
 polymer, 157–158
Maximum stress criterion, 88–90
Melting process, 6–7, 6f
Mesophase, 174
MFC. *See* Microfibrillated cellulose (MFC)
Micro-filler, 176–178
Microcracks, 173
 crack arrest by, 173f
 detection using NDT method, 166–172
 formation in polymer composites, 158–159
Microfibrillar angle, 79
Microfibrillated cellulose (MFC), 114
Miscanthus giganteus/china reed, 110–111
Moment–deflection curves, 219–220
Morphing wing, 190, 192
Mortar cracking, 201
Multiwalled carbon nanotubes (MWCNTs), 4–5, 178, 185
- N**
Nano filler like carbon nanotubes, 230
Nanocomposite, 4–5
Nanofiller, 176–178
Nanoparticles, 3
Natural fibers, 79, 138, 231
 composites, 137–138
 mechanical properties, 139t
 natural fiber-reinforced thermoset composites, 66

- research works on energy absorption and failure mode, 67t–68t
 - reinforced thermoplastic composites, 66–70
 - research works on energy absorption and failure mode, 69t
 - Natural microfillers, 73
 - Natural/natural hybridization, 80
 - NDI technique. *See* Non-Destructive Inspection technique (NDI technique)
 - NDT methods. *See* Nondestructive testing methods (NDT methods)
 - Neem particulate fiber-reinforced epoxy tensile test composite, thickness of, 128t
 - Neem tree, 112
 - NMR. *See* Nuclear magnetic resonance (NMR)
 - NMRI. *See* Nuclear magnetic resonance imaging (NMRI)
 - No-tensile-resistant homogeneous material (NTR homogeneous material), 214–215
 - NOESIS software, 233–235
 - Non-Destructive Inspection technique (NDI technique), 70
 - Non-hybrid
 - single fiber-reinforced composites, 80–81
 - synthetic fiber-reinforced composites, 80–81
 - Nondestructive testing methods (NDT methods), 166–172
 - acoustic emission method in polymer composite, 170–172
 - NMRI method in polymer composite, 167–168
 - ultrasonic method in polymer composite, 169–170
 - X-radiography method in polymer composite, 169
 - Nonlinear behavior
 - under in-plane lateral loading, 208–216
 - experimental outcomes, 208–213
 - theoretical models, 213–216
 - under out-of-plane lateral loading, 216–222
 - airbag out-of-plane tests on masonry panels, 219f
 - experimental findings, 216–220
 - theoretical models, 220–222
 - NTR homogeneous material. *See* No-tensile-resistant homogeneous material (NTR homogeneous material)
 - Nuclear magnetic resonance (NMR), 167, 168f
 - Nuclear magnetic resonance imaging (NMRI), 167
 - method in polymer composite, 167–168
- O**
- Oil palm fiber-reinforced polyester composite (OPFRP), 70
 - Onset of nonlinearity, 163
 - Open-hole tension test (OHT test), 97–100, 99t, 100f
 - ANOVA for, 103
 - experimental results, 101t
- P**
- PAM actuators. *See* Pneumatic artificial muscle actuators (PAM actuators)
 - Partially stabilized zirconia (PSZ), 172–173, 172f
 - Particle-reinforced polymer composites, 159, 173–174
 - Particulate composites. *See also* Hybrid composites
 - practical problems of particulate composites manufacturing, 118
 - preparation of particulate composites and testing, 112
 - effect of specimen thickness, edge crack on, 115–118
 - tensile and impact properties, 113–115
 - Particulate natural fiber materials,
 - discarding, 111–112
 - babul tree, 111
 - balharshah teak, 112
 - neem tree, 112
 - subabul tree, 111
 - teak tree, 111
 - PBO. *See* Poly(paraphenylene benzobisoxazole) (PBO)
 - PBO-FRCM systems, 207–208
 - Pectin, 79
 - PEEK. *See* Poly(ether ether ketone) (PEEK)

- PHAE. *See* Poly hydroxyl amino ether (PHAE)
- Phenomenological-model, 13–15
- PLA. *See* Polylactic acid (PLA)
- Plastic deformation accumulation, 174–176
- Pneumatic artificial muscle actuators (PAM actuators), 193–194
- Point stress failure criteria, 88, 90
- Poly hydroxyl amino ether (PHAE), 4–5
- Polydopamine and GO-CFRP (PDA&GO-CFRP), 174
- Polyether ether ketone (PEEK), 12–13, 73, 238–239
- Polylactic acid (PLA), 66–70
- Polymer composites, 51. *See also* Hybrid composites
- detection of cracks and microcracks using NDT method, 166–172
 - energy absorption capability, 52
 - failure mechanisms, 52–53, 54t–56t
 - formation of cracks and microcracks in, 158–159
 - fracture mechanics in, 159–166
 - fracture toughness for, 172–178
 - strain rate, 53–58
 - effect on failure mechanisms and energy absorption, 58–73
- Polymer-matrix composite processing, 3–10
- CNT paper-based nanocomposites, 7–9
 - graphene paper-based nanocomposites, 9–10
 - melting process, 6–7, 6f
 - in situ polymerization process, 5–6, 5f
 - solution processing, 3–5, 3f
 - properties of solvents, 4t
- Polyparaphenylene benzobisoxazole (PBO), 199–200
- Polypropylene (PP), 62, 70, 73
- Polytetrafluoroethylene (PTFE), 238–239
- Polyurethane polymer (PU polymer), 181–182
- Polyurethane SMP, 189
- Poplar wood flour (PWF), 239–240, 243
- Positive or negative hybrid effect, 81
- PP. *See* Polypropylene (PP)
- Prediction delamination growth, methods for, 13–20
- CZMs, 18–19
 - extended finite element method, 19–20
 - fracture-mechanics based methods, 17–18
 - stress/strain-based methods, 15–17
- Pressure filtration, 7
- Progressive failure modes, 52–53
- Progressive-damage model, 13–15
- PSZ. *See* Partially stabilized zirconia (PSZ)
- PTFE. *See* Polytetrafluoroethylene (PTFE)
- PU polymer. *See* Polyurethane polymer (PU polymer)
- PWF. *See* Poplar wood flour (PWF)
- ## Q
- Quasi-static compression test, 57, 57f
- ## R
- RA. *See* Rise angle (RA)
- Radiofrequency (RF), 167
- RC. *See* Reinforced concrete (RC)
- Reciprocating sliding wear test, 238–239
- Recycled polycarbonate composite (RPC), 239–240
- Red mud, 231
- Reinforced concrete (RC), 200
- Reinforcement
- material, 231
 - of SMP, 184–187
 - of SMPC, 181–182
- Response surface methodology (RSM), 104–105
- RF. *See* Radiofrequency (RF)
- Ring sliding wear tester, 238–239, 239f
- Rise angle (RA), 233–235
- RPC. *See* Recycled polycarbonate composite (RPC)
- RSM. *See* Response surface methodology (RSM)
- ## S
- Scanning electron microscopy (SEM), 116f, 149, 235–236
- SEA. *See* Specific energy absorption (SEA)
- SEBS. *See* Styrene ethylene butylene styrene (SEBS)
- Seismic risk mitigation, 208
- Self-loosening of bolted joint, 87–88
- SEM. *See* Scanning electron microscopy (SEM)
- SERR. *See* Strain energy release rate (SERR)

- SGF. *See* Short glass fibers (SGF)
- SGF/Maleated styrene ethylene butylene styrene/PP hybrids (SGF/SEBS-g-MA/PP hybrids), 73
- Shape memory alloy (SMA), 181–182, 236–237
- Shape memory ceramic (SMC), 182–183
- Shape memory materials (SMM), 182–183
- Shape memory polymer (SMP), 181–183
- applications, 189–194
 - comparison of properties, 186t, 188t
 - history, 183–184
 - nichrome embedded into, 192f
 - reinforcement, 184–187
 - shape recovery of morphing wing with, 191f
 - wing-shaped Vetirex, 191f
- Shape memory polymer composites (SMPC), 181–182
- applications, 189–194
- Shape memory polyurethane (SMPU), 184
- Shear-compression tests, 212–213
- Shearing failure strength, 83–84
- Sheep—wool reinforced epoxy composites
- ANOVA for OHT test, 103
 - fabrication of laminated composite, 99f
 - failure analysis and optimal toughness design
 - experimental design, 98–99
 - materials and method, 98
 - OHT test, 99–100, 99t, 100f
 - linear regressions, 105–106
 - main effect plots, 101–103, 102f
 - RSM, 104–105
- Short glass fibers (SGF), 73
- Short glass fibers/Styrene ethylene butylene styrene/polypropylene hybrids (SGF/SEBS/PP hybrids), 73
- SIF. *See* Stress intensity factor (SIF)
- Single-side strengthening, 211–212
- with passing-through SFRP ties, 211–212
- Sisal/banana fiber hybrid polyester composite, 240–241
- Skin activation system, 191–192
- SMA. *See* Shape memory alloy (SMA)
- SMC. *See* Shape memory ceramic (SMC)
- SMM. *See* Shape memory materials (SMM)
- SMP. *See* Shape memory polymer (SMP)
- SMPC. *See* Shape memory polymer composites (SMPC)
- SMPU. *See* Shape memory polyurethane (SMPU)
- SN. *See* Stress vs. number of cycles to failure (SN)
- Soft clamping, 202–203
- Solution-centered technique, 9–10
- Specific energy absorption (SEA), 52
- Spin-coating method, 7, 8f
- SRG. *See* Steel Reinforced Grout (SRG)
- Steel Reinforced Grout (SRG), 199–200
- Strain energy release rate (SERR), 1–2, 12, 15, 17
- Strain rate, 53–57
- effect on failure mechanisms and energy absorption, 58–73
 - natural fiber-reinforced thermoset composites, 66
 - natural fibers reinforced thermoplastic composites, 66–70
 - synthetic fibers reinforced thermoplastic composites, 62–66
 - synthetic fibers reinforced thermoset composites, 58–62
 - thermoplastic-based hybrid composites, 73
 - thermoset-based hybrid composites, 70–73
 - test methods to determine strain rate effect, 57–58
 - impact test, 58
 - quasi-static compression test, 57, 57f
- Stress criteria, 44–45
- outside principal axes, 45
 - in principal axes, 44–45, 45f
- Stress intensity factor (SIF), 1–2, 15, 17
- Stress vs. number of cycles to failure (SN), 13–15
- Stress/strain-based methods, 15–17
- Stress–strain curves of FRCM specimens, 204–205, 204f
- Structural retrofitting, 199–201
- Styrene ethylene butylene styrene (SEBS), 73
- Subabul particulate fiber-reinforced epoxy tensile test composite thickness, 127t
- Subabul tree, 111

Synthetic fiber, 229–231
 mechanical properties of, 139t
 reinforced thermoplastic composites, 62–66
 research works on energy absorption and failure mode, 63t–65t
 reinforced thermoset composites, 58–62
 research works on energy absorption and failure mode, 59t–60t
 Synthetic nanofillers, 73
 Synthetic/natural hybridization, 80
 Synthetic/synthetic hybridization, 80

T

T700–12K carbon fiber, 231
 Taguchi analysis, 241
 Taguchi design, 97–98
 Taguchi L16 orthogonal array, 241
 Taguchi orthogonal array, 241
 TCB. *See* Trichloro benzene (TCB)
 TDE-86 epoxy resin, 230
 Teak particulate fiber-reinforced epoxy
 tensile test composite thickness, 126t
 Teak tree, 111
 Tectonagrandis, 111
 TEM. *See* Transmission electron microscopy (TEM)
 Tensile
 behavior, 201–202, 208
 of hybrid composites, 236–237
 failure analysis, 149–153
 strength, 236–237
 ANOVA, 103t
 effect plot, 102f
 experimental validation, 105t
 surface plot, 104f
 test, 194
 analysis, 143–147
 testing method, 140–141
 Tension, 90
 Textile Reinforced Concrete (TRC),
 199–200
 Textile Reinforced Mortar (TRM), 199–200
 Thermal effect, 83–84
 Thermal transition temperatures, 182–183
 Thermoplastic (TP), 73
 hybrid composites, 73
 research works on energy absorption and failure mode, 74t–75t
 polymers, 6–7

Thermoset-based hybrid composites, 70–73
 research works on energy absorption and failure mode, 71t–72t
 Thermosetting matrix, 30
 Three-point bending test, 165–166, 166f,
 237–238
 Time delay steered array, 170
 TP. *See* Thermoplastic (TP)
 Transition phase, 91
 Transmission electron microscopy (TEM),
 5–6
 Transverse cracking, 32
 Transverse tensile, unidirectional composite
 submitted to, 35–36
 TRC. *See* Textile Reinforced Concrete (TRC)
 Trichloro benzene (TCB), 8–9
 TRM. *See* Textile Reinforced Mortar (TRM)
 Tsai-hill criteria, 46
 Tuff stone masonry walls (TSM walls),
 200–201
 Two-way shape memory effect, 182–183

U

UAV. *See* Unmanned aerial vehicle (UAV)
 Ultimate limit state (ULS), 214–215
 Ultrasonic method in polymer composite,
 169–170
 Unidirectional composite
 mechanisms of rupture and damage,
 32
 failure of fiber, 33f
 longitudinal fracture of matrix, 33f
 rupture modes of matrix associated fiber
 rupture, 34f
 transverse fracture of matrix, 32f
 submitted to longitudinal tensile, 33–35
 failure propagation in case of weak
 adherence fiber-matrix, 36f
 mechanical characteristics of synthetic
 fibers, 35t
 rupture propagation in case of high
 adhesion, 35f
 stress-strain curves, 34f
 submitted to transverse tensile, 35–36
 failure of unidirectional composite, 36f
 Uniform lateral loading, 219–220
 Universal testing machine, 236–237
 Unmanned aerial vehicle (UAV), 189

Unreinforced masonry walls (URM walls), 200–201
Urethane, 183–184
URM walls. *See* Unreinforced masonry walls (URM walls)

V

Vacuum filtration, 7, 9–10
Variable stiffness element, 189
Variable sweep morphing wing, 192–193
Variable-camber wing concept, 190
Virtual-crack-closure-technique (VCCT), 17
Visual observation, 163
Volumetric energy absorption capability, 52

W

W/D. *See* Width-to-diameter ratio (W/D)
Waste Silk Fiber (WSF), 239–240, 243
Wear behavior
 of hybrid composites, 238–239
 of industrial waste-filled hybrid composites, 241
Weibull distribution, 235–236, 235f
WFRPC. *See* Wool fiber–reinforced polymer composites (WFRPC)
Width-to-diameter ratio (W/D), 83–84

Wood particulate composites. *See also* Hybrid composites
 discarded particulate natural fiber materials, 111–112
 practical problems of particulate composites manufacturing, 118
 preparation of particulate composites and testing, 112
 effect of specimen thickness, edge crack on particulate composites, 115–118
 tensile and impact properties of particulate composites, 113–115
Wool fiber–reinforced polymer composites (WFRPC), 98
Woven glass fibers, 141f
Woven jute, 142f
Woven kenaf, 141f, 149
WSF. *See* Waste Silk Fiber (WSF)

X

X-radiography method in polymer composite, 169
X-ray tomography, 236
XFEM. *See* Extended finite element method (XFEM)

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This five-volume set of titles focuses on advanced characterization techniques used for the analysis of composite materials developed from natural fiber/biomass, synthetic fibers, and a combination of these materials used as fillers and reinforcements to enhance materials performance and utilization in automotive, aerospace, construction, and building components.

The books provide detailed information on failure analysis, mechanical and physical properties, structural health monitoring, durability and life prediction, modeling of damage processes of natural fiber, synthetic fibers, and natural/natural, and natural/synthetic fibers hybrid composites. They will act as a detailed reference resource to encourage future research in natural fiber and hybrid composite materials, which is much in demand due to the need for more sustainable, recyclable, and eco-friendly composites in a broad range of different application fields.

Written by leading experts in the field and covering composite materials developed from different natural fibers and their hybridization with synthetic fibers, the chapters provide cutting-edge up-to-date research on the characterization, analysis and modeling of composite materials.

This book covers key aspects of fracture and failure in natural/synthetic fiber-reinforced polymer-based composite materials. These range from crack propagation to crack growth, notch-size effect to damage-tolerant design. The book describes a broad range of techniques and strategies for compositional and failure analysis of polymeric materials and products. It also illustrates the application of analytical methods for solving commonly encountered problems in the composite manufacturing industry.

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