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Foreword

THIS COMPILATION OF Selected Technical Papers, STP1607, *100 Years of E04 Development of Metallographic Standards*, contains peer-reviewed papers that were presented at a symposium held November 15–17, 2017, in Atlanta, Georgia, USA. The symposium was sponsored by ASTM International Committee E04 on Metallography.

Symposium Chair and STP Editor:

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Overview

ASTM Committee E04 on Metallography was created in 1916. On May 8–10, 1991, E04 celebrated its 75th anniversary with a conference in Atlantic City, NJ, resulting in publication of STP1165, *Metallography: Past, Present and Future*. On November 15–17, 2017, E04 celebrated its 100th anniversary at its fall meeting in Atlanta, GA. These 100 years have seen a vast growth in the science and technology of metallography, and E04 has continued to create new standards and update older standards as technology continues to grow and improve.

This new STP contains seven articles on the use and improvement of ASTM standards. Several years of experiments led to the development of an improved method to assess the degree of banding segregation in hot- or cold-worked steels, led by our Russian advisor, Alexander Kazakov, and his wife and students, and a vast improvement to E1268. Two other papers discuss how surface preparation can alter the measurement of retained austenite in steels and how proper preparation is required to assess and characterize thermal barrier coatings. Other talks were related to extending the knowledge on specific topics of concern to metallographers, such as improving the use of stereology when making quantitative measurements of microstructure or examining the errors and uncertainty that exist when performing such measurements. Two talks covered topics related to micro-indentation hardness tests: avoiding very low-test loads that yield diagonal lengths of less than 21 μm where normal variability in measuring the indents can create substantial errors, and the use of the control chart method in E2554 to show when a hardness tester has encountered problems that yield bad data and also allow for a simple determination of the uncertainty of the test data.

The new STP also contains a number of interesting papers on the use of metallographic techniques to better understand and control problems and quality. One paper discusses the influence of intermetallic particles on the quality of aluminum-to-steel friction welds, while a second paper covers the analysis and detection of intermetallic phases on the toughness and corrosion resistance of duplex stainless steels. Another author discusses the mechanical behavior and fractography of a hot-stamped steel as a function of strain rate while another author discusses the effect of strain rate on the properties and behavior of a dual-phase steel. Efforts to develop deep learning methods to assist in the interpretation and measurement of microstructures is a new topic of considerable interest, and authors from Germany, led by Dominik Britz, present an interesting study of the use of LePera's etchant in

color metallography to improve the use of this technology in evaluating the microstructure of low-carbon steels.

We hope that our readers will enjoy reading these papers and benefit from the knowledge they provide.

George F. Vander Voort
Symposium Chairman and STP Editor

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Alexander Kazakov,¹ George F. Vander Voort,²
Daniil Kiselev,¹ and Elena Kazakova¹

ASTM E1268: From Improvement to the New *Standard Practice for Assessing the Degree of Banding or Orientation of Microstructures* by Automatic Image Analysis

Citation

Kazakov, A., Vander Voort, G. F., Kiselev, D., and Kazakova, E., "ASTM E1268: From Improvement to the New *Standard Practice for Assessing the Degree of Banding or Orientation of Microstructures* by Automatic Image Analysis," *100 Years of E04 Development of Metallographic Standards*, ASTM STP1607, G. F. Vander Voort, Ed., ASTM International, West Conshohocken, PA, 2019, pp. 1–11, <http://dx.doi.org/10.1520/STP160720170206>³

ABSTRACT

It has been discovered through experiments that using directed secants is the only technique that is correct for estimation of the degree of banding segregation covered in ASTM E1268, *Standard Practice for Assessing the Degree of Banding or Orientation of Microstructures*. Unfortunately, none of the criteria chosen in this standard can adequately characterize banding; therefore, it is described verbally. Anisotropy and the degree of orientation used in this standard characterize only the elongation of the separate features of the structure but cannot assess the tendency of these features (pearlite, bainite, carbides, and so on) to be combined into bands. Using the directed secants method, we have proposed a criterion that adequately estimates the degree of microstructural banding for a wide range of steels and alloys, including all the structures that are listed in Annex A1 of ASTM E1268. This criterion is the ratio of

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the standard deviation of the volume fraction of a “second phase” on the secants that are parallel to the deformation direction to the standard deviation of the volume fraction of a “second phase” on the secants that are perpendicular to the deformation direction. Considering that the banding according to the new criterion and the anisotropy according to ASTM E1268 are estimated by the same technique, the introduction of the new criterion to ASTM E1268 will be simple. A draft of these improvements has been developed and successfully balloted. Further improvement of ASTM E1268 can be attributed to the use of an image analyzer. Therefore, the principles of the standard practice for assessing the degree of banding or orientation of microstructures by automatic image analysis have been formulated.

Keywords

microstructures, orientation, banding rate, standard practice, automatic image analysis

Introduction and Historical Background

The idea of a quantitative description of a banded segregated structure was formulated by Sarkis A. Saltykov in the 1960s, and it was published in his book on stereometric metallography in 1970 [1]. Analyzing the structure of a hypoeutectoid steel, represented in Fig. 1a, he came to the conclusion that

here the anisotropy of both the volume fraction and pearlite grains size takes place on lines parallel to the orientation axis the pearlite volume fraction varies from zero to almost 100 %, and the grain sizes along the axis significantly exceed the transverse dimensions of the pearlite grains The average number of intersections of the secants directed perpendicular to the axis with the boundary surfaces will be greater than the average number of intersections of secant lines parallel to the axis. Therefore, for this structure, the degree of linear orientation of the boundary surfaces can serve as an index of the anisotropic inhomogeneity (p. 352) [1].

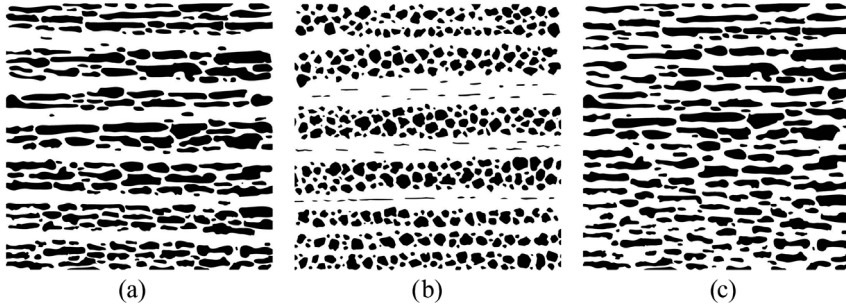
So, Saltykov has introduced an index of the anisotropic inhomogeneity that is an anisotropy index (AI) used in ASTM E1268, *Standard Practice for Assessing the Degree of Banding or Orientation of Microstructures* [2].

Further, continuing to talk about the banded arrangement of equiaxial grains of pearlite in the hypoeutectoid steel (Fig. 1b), Saltykov notes that

... it is easy to verify that the fraction of pearlite varies within narrow limits on lines perpendicular to the orientation axis, while on lines parallel to the axis it will vary from zero (when the line passes through the ferrite bands) close to 100 % (when the line passes through the pearlite grains) (p. 353)[1].

Based on these observations, he proposes to evaluate the banded structure as follows:

FIG. 1 The structures for analysis of stereological ratios for banding evaluation by the method of directed secants: (a) $AI = 3.47$, $BR = 5.81$, $BR(N) = 1.83$; (b) $AI = 1.21$, $BR = 4.24$, $BR(N) = 3.98$; and (c) $AI = 3.47$, $BR = 3.48$, $BR(N) = 1.08$.
AI = anisotropy index; BR = banding rate.



Therefore, the index of the anisotropic inhomogeneity of such a structure can be the ratio of the mean square deviations of pearlite content or the average number of intersections determined separately on transverse and on longitudinal lines or secant lines (p. 353) [1].

Note that Saltykov has proposed to estimate the banding by two criteria, but only one of them works equally well for any structures [3,4]; we will discuss the latter criterion here.

Finally, Saltykov determined the content of pearlite on 100 transverse and 100 longitudinal lines by the linear method on the structure represented in Fig. 1b.

The degree of anisotropic inhomogeneity of the pearlite content can be estimated by the ratio of mean square deviations $5.23/16.80 = 0.3$. For an isotropic structure, the value of this ratio is obviously 1, and an increase in the degree of anisotropy is accompanied by a decrease in the ratio of the mean square deviations determined for transverse and longitudinal lines separately (p. 353) [1].

The Stereological Criteria from Saltykov's Book

Therefore, the concepts of quantitative assessment of anisotropic inhomogeneity or the banding rate (BR) were proposed and illustrated by simple calculations in Saltykov's book nearly 50 years ago [1]. These criteria may be written as the expressions that are calculated by the method of directed secants using the image analyzer [3–5].

1. $AI = \frac{\bar{N}_{\perp}}{\bar{N}_{\parallel}}$ is the average number of particles crossed by secants that are perpendicular to the deformation direction divided by the same number but parallel to the deformation direction.

This is Saltykov's "index of the anisotropic inhomogeneity," which is used in ASTM E1268 [2] as the AI. This index well describes anisotropy of separate structural features for example, pearlite grains elongated along a deformation direction so that it is possible to distinguish an isomorphic structure from an oriented one.

2. $BR(N) = \frac{\sigma_{NL\parallel}}{\sigma_{NL\perp}}$ is the standard deviation of the number of particles crossed by secants that are parallel to the deformation direction divided by the same number but perpendicular to the deformation direction.

This is Saltykov's second "index of the anisotropic inhomogeneity." As we have shown earlier [3,4], it works as a banding criterion only on structures with equiaxial particles of the second phase, combined into bands, and does not allow the comparison of structures with different morphologies of the second phase.

3. $BR = \frac{\sigma_{LL\parallel}}{\sigma_{LL\perp}}$ is the standard deviation of the linear fraction of particles on secants that are parallel to the deformation direction divided by the same number but perpendicular to the deformation direction.

This is an inversion of Saltykov's third "index of anisotropic inhomogeneity," which is called the BR [4]. We have inverted Saltykov's criterion so that it would increase with an increase in the degree of banding of the structure. As was stated before, this is a universal banding criterion that works equally well with any morphology of the second phase combined into bands.

Let us compare the numerical values of the criteria for the three structures. The first and second structures have been taken from Saltykov's book (Fig. 1a and b), and the third one (Fig. 1c) is obtained from the first one by redistribution of the second phase in the ferrite matrix in such a way that the banding of the initial structure decreases significantly while the anisotropy of the separate particles of the second phase remains the same. Comparing these structures, it can be seen that the BR of these two microstructures decreases from 5.81 to 3.48, but the AI is still the same. So, the AI does not describe the degree of banding.

The criterion $BR(N) = \frac{\sigma_{NL\parallel}}{\sigma_{NL\perp}}$ gives good results in the evaluation of banding if particles forming the bands are equiaxed (Fig. 1b). In the case of elongated particles forming the bands (Fig. 1a), the number of intersections on secants along as well as across the deformation direction fluctuates insignificantly; thus, the sensitivity of $BR(N)$ for the banding estimation for such structures is worse. For example, from the first structure with elongated particles to the second structure with equiaxed particles, the BR drops a little (from 5.81 to 4.24), but $BR(N)$ grows more than twice. The third structure, according to $BR(N) = 1.08$, is isotropic, but that is not true.

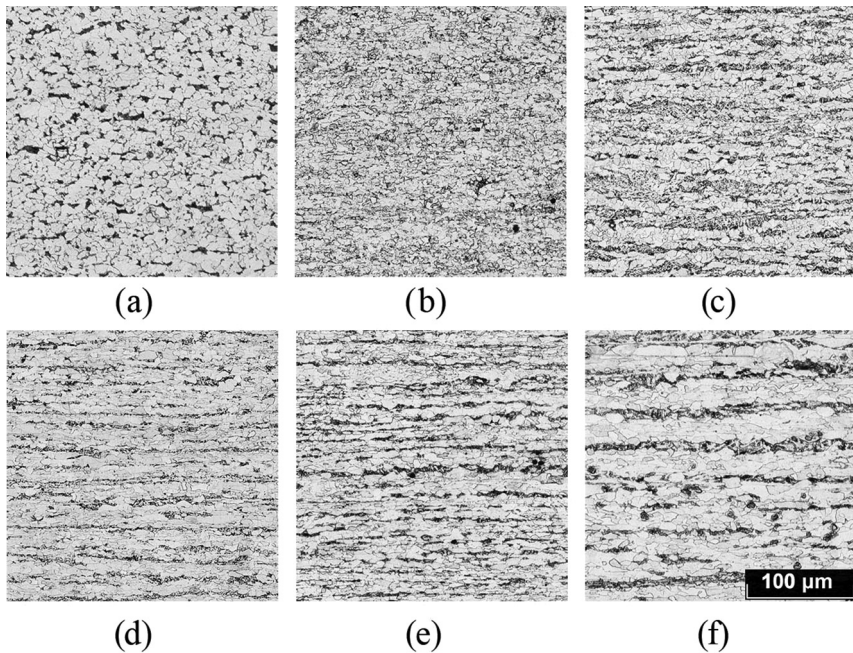
So, only $BR = \frac{\sigma_{LL\parallel}}{\sigma_{LL\perp}}$, based on measurements of the second phase fraction on directed secants along and across the deformation direction, will characterize structural banding for any morphology of particles that formed the bands. Indeed, the BR decreases regularly from the first structure ($BR = 5.81$) to the third ($BR = 3.48$).

For the evaluation of BR, the same method of directed secants is used that evaluates all other parameters of the structure, including AI, described in the current version of the ASTM E1268 [2].

Applicability of the BR

The applicability of the BR was illustrated on the test structures (Fig. 2). These structures were selected on the principle of increasing the number of the bands and their continuity in accordance with the description of the banding grade (BG) known from GOST 5640 [6]. The chart for banding estimation consists of six grades. The BG raises from Grade 0 for an isotropic, nonbanded ferrite pearlite structure (Fig. 2a) to Grade 1 for a fully oriented structure without well defined bands (Fig. 2b) to Grade 2 for structures with one or two solid and several broken bands (Fig. 2c) to Grade 3 for alternation of ferrite and bainite bands (Fig. 2d) and up to Grades 4 and 5 for corresponding uniform (Fig. 2e) and nonuniform (Fig. 2f) alternation, respectively, of ferrite and bainite bands.

FIG. 2 Microstructures to test applicability of the BR and AI for the different banding grades (BGs): (a) BG = 0, AI = 1.03, BR = 1.22; (b) BG = 1, AI = 1.17, BR = 1.71; (c) BG = 2, AI = 1.22, BR = 2.52; (d) BG = 3, AI = 1.3, BR = 3.96; (e) BG = 4, AI = 1.77, BR = 4.75; and (f) BG = 5, AI = 1.7, BR = 5.91. All images are at the same magnification, and the magnification bar in Fig. 2f is the same for all the images.



So, for the BG from zero up to five (0, 1, 2, 3, 4, 5), the BR regularly increases: 1.22, 1.71, 2.52, 3.96, 4.75, 5.91, correspondingly; but the AI does not reflect these changes (Fig. 2).

Description of the Main Improvements of the ASTM E1268 Draft

Due to the introduction of a new BR, new measurements have to be made. Therefore, Fig. 1 of ASTM E1268 [2] is supported with the new figures (c) and (d) (Fig. 3a and b, respectively) with an illustration of the length of feature interceptions (L) measurement. The note has been updated also: “Note 1 The test grid lines have been shown oriented perpendicular (A, C) to the deformation axis and parallel (B, D) to the deformation axis. The counts for $N_{\perp}$, $N_{\parallel}$, $P_{\perp}$, $P_{\parallel}$, $L_{\perp}$ and $L_{\parallel}$ are shown for the counts made from top to bottom (A, C) or from left to right (B, D).”

We have to update ASTM E1268, Section 3.3, with the new measured and calculated parameters necessary for the estimation of BR:

$L_{\parallel}$ is the summarized length of feature interceptions by a single test line parallel to the deformation direction;

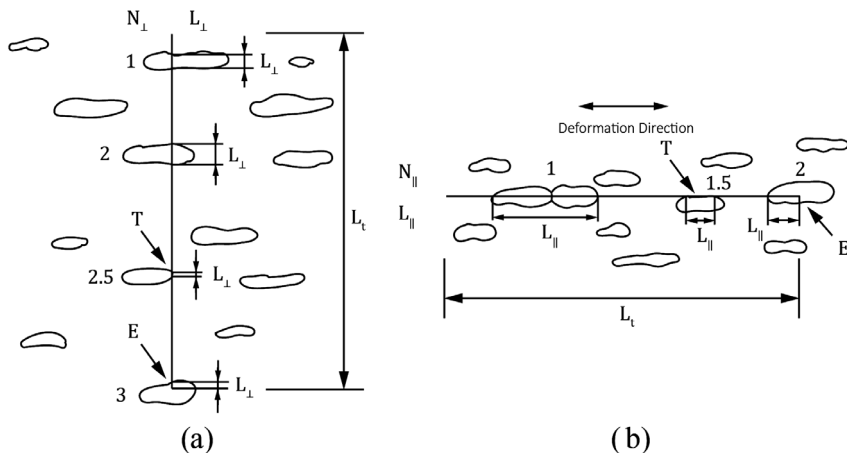
$L_{\perp}$ is the summarized length of feature interceptions by a single test line perpendicular to the deformation direction;

L_t is the full length of the test grid line;

$L_{L\parallel}$ is the linear fraction of a feature on a single test line parallel to the deformation direction:

$$L_{L\parallel} = \frac{L_{\parallel}}{L_t};$$

FIG. 3 The measurement scheme for the calculation of BR perpendicular (a) and parallel (b) to the deformation direction (Fig. 1c and d of ASTM E1268).



$L_{L\perp}$ is the linear fraction of a feature on a single test line perpendicular to the deformation direction:

$$L_{L\perp} = \frac{L_{\perp}}{L_t};$$

m is the number of the test lines parallel to the deformation direction;

k is the number of the test lines perpendicular to the deformation direction;

$\sigma_{L_{L\parallel}}$ is the standard deviation of the linear fraction of a feature for all test lines that are parallel to the deformation direction on a single field of view:

$$\sigma_{L_{L\parallel}} = \sqrt{\frac{\sum_{i=1}^m (L_{L\parallel i} - \overline{L_{L\parallel}})^2}{m-1}};$$

$\sigma_{L_{L\perp}}$ is the standard deviation of the linear fraction of a feature for all test lines that are perpendicular to the deformation direction on a single field of view:

$$\sigma_{L_{L\perp}} = \sqrt{\frac{\sum_{i=1}^k (L_{L\perp i} - \overline{L_{L\perp}})^2}{k-1}};$$

and BR is the banding rate in a single field of view:

$$BR = \frac{\sigma_{L_{L\parallel}}}{\sigma_{L_{L\perp}}}$$

As follows from the aforementioned parameters, each interception is measured and all interceptions for a single test line are summarized. For each test line, the linear fraction of a feature is measured. Further, the standard deviation of the linear fraction of a feature perpendicular and parallel to the deformation direction and the BR in a single field of view are calculated. Finally, the average value BR for all measured fields of view is calculated:

$$\overline{BR} = \frac{\sum BR}{n}.$$

The AI, like all other criteria, is calculated on each field of view, and the average value is calculated over all measured fields; so, instead of $AI = \frac{\overline{N_{L\perp}}}{\overline{N_{L\parallel}}} = \frac{\overline{P_{L\perp}}}{\overline{P_{L\parallel}}}$ we have to calculate: $AI = \frac{N_{L\perp}}{N_{L\parallel}}$ and $\overline{AI} = \frac{\sum AI}{n}$.

In ASTM E1268, Section 4.1, it is necessary to update: “The degree of micro structural banding or orientation is described *quantitatively* using metallographic specimens aligned parallel to the deformation direction of the product.”

The following additions have to be included in Section 10:

10.3.5 Measurement of AI for every single analyzed field of view, divide $N_{L\perp}$ by $N_{L\parallel}$.

10.3.6 Measurement of BR for every single analyzed field of view, the following calculations have to be made:

10.3.6.1 Measure $L_{\parallel}$ summarize lengths of feature interceptions for a single test line parallel to the deformation direction.

10.3.6.2 Calculate linear fraction $L_{L\parallel}$ divide $L_{\parallel}$ by L_t .

Repeat Steps 10.3.6.1 and 10.3.6.2 for every test line on a field of view and store each individual value ($L_{L\parallel i}$).

10.3.6.3 Calculate $\sigma_{L_{L\parallel}}$ standard deviation for $L_{L\parallel}$ using the formula:

$\sqrt{\frac{\sum_{i=1}^m (L_{L\parallel i} - \overline{L_{L\parallel}})^2}{m-1}}$, where: $\overline{L_{L\parallel}}$ is the mean linear fraction, and m is the number of test lines.

10.3.6.4 Measure $L_{\perp}$ summarize lengths of feature interceptions for a single test line perpendicular to the deformation direction.

10.3.6.5 Calculate linear fraction $L_{L\perp}$ divide $L_{\perp}$ by L_t .

Repeat Steps 10.3.6.4 and 10.3.6.5 for every test line on a field of view and store each individual value $L_{L\perp i}$.

10.3.6.6 Calculate $\sigma_{L_{L\perp}}$ standard deviation for $L_{L\perp}$ using the formula: $\sqrt{\frac{\sum_{i=1}^m (L_{L\perp i} - \overline{L_{L\perp}})^2}{k-1}}$, where $\overline{L_{L\perp}}$ is the mean linear fraction, and k is the number of test lines.

10.3.6.7 Calculate BR for current field of view divide $\sigma_{L_{L\parallel}}$ by $\sigma_{L_{L\perp}}$.

Note that for automatic image analysis performed by a computer, we take every pixel line or pixel row for a test line. This approach provides a wide statistical basis for each calculation of the standard deviation.

In Section 11, the calculation results will be added by a new average value of $\overline{BR}$ as well as an average value $\overline{AI}$ calculated the new way. Since the AI is measured for every single field of view and the mean value is calculated later, the old text of Section 11.6 for AI calculation should be removed.

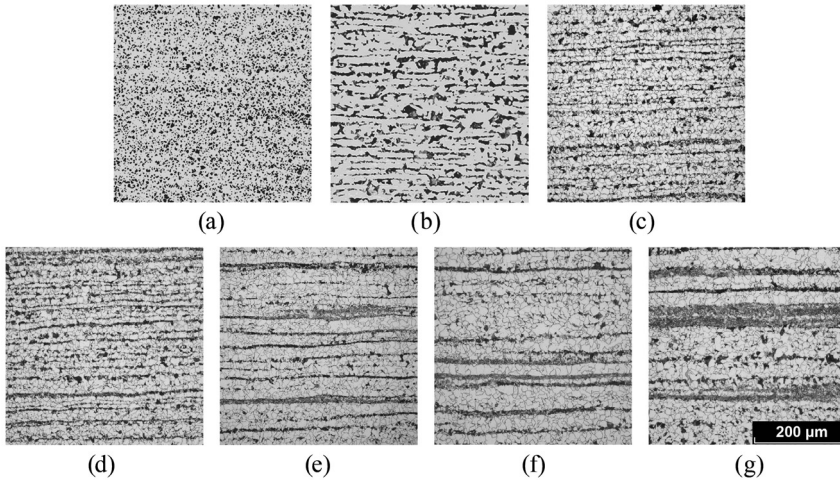
Sections 13.9 and 13.10 have to be deleted because they call for verbal description and contain reasons why it is impossible to quantify banding. Now that it has been shown that AI determines whether the microstructure is oriented parallel to the deformation direction and BR determines whether the microstructure is really banded and what the degree of banding is, these sections are not required.

So, the summary of the ASTM E1268 improvements is as follows:

- New figures to clarify the BR measurement
- New symbols, formulas, and descriptions (BR, linear fractions)
- Revised AI calculation (for every field of view)
- New procedure for the new BR measurement and calculation
- Changes to test report content (BR has been added)
- Changes in text (wording, paragraphs, page references, and so on)
- Microstructures with BR estimated for the old and the new examples for Annex A1.

A few new examples of microstructures for Annex A1 with BR and AI estimated are shown in Fig. 4: the AI (AI = 0.99) made it possible to identify the structure in Fig. 4a as isomorphous; however, only BR describes the steadily increasing degree of banding for structures from Fig. 4b g: (b) 2.57, (c) 3.15, (d) 3.83, (e) 4.01, (f) 5.00, and (g) 7.42.

FIG. 4 The examples of new microstructures for Annex A1. BR = (a) 1.43, (b) 2.57, (c) 3.15, (d) 3.83, (e) 4.01, (f) 5.00, and (g) 7.42. AI = (a) 0.99, (b) 1.80, (c) 1.21, (d) 1.32, (e) 1.25, (f) 1.14, and (g) 1.69. All images are at the same magnification, and the magnification bar in [Fig. 4g](#) is the same for all the images.



Thus, after the introduction of the new BR and the previously described minor changes in the text, the name of the standard ASTM [E1268](#) will match its purpose and content. With the help of the updated ASTM [E1268](#), it will be possible to give up the “verbal description of the nature of the banding” and go to the quantitative description of the banding not only to “to establish whether the microstructure is purely oriented towards the deformation direction or is actually banded” but also to assess the degree of this banding for different microstructures.

Note that correct use of automatic image analysis methods requires well defined microstructures. The specimens must be properly prepared, and the etchant must distinctly reveal the microstructural features. The choice of the best etchant to use is an important decision. It is also important to identify the microstructural constituents in the bands, and this usually must be done at a higher magnification than is used to measure the BR.

The New Standard Practice for Assessing the Degree of Banding or Orientation of Microstructures by Automatic Image Analysis

Further improvement of ASTM [E1268](#) is difficult because it contains many aspects based on the outdated hardware and software capabilities of those instrumental procedures that were relevant at the time of the first edition of the standard in 1999 and

were not updated later. Today, when image analysis is widely used in the industry, it seems that the time has come to create a new standard based entirely on modern methods of quantitative metallography implemented with these analyzers. Moreover, today, the microindentation hardness studies of ASTM E1268 are performed by motorized microhardness testers equipped with image analyzers, so it seems logical to create a new standard based on modern hardware software instrumentation procedures.

Attributes of the new standard could be the following:

- Assessment is based on automatic image analysis measurement only.
- The method of directed secants is used for orientation or banding estimation.
- The AI is used to establish whether the microstructure is random or oriented parallel to the deformation direction.
- The BR is used to establish whether the microstructure is banded and to estimate the degree of banding (number of the bands and their continuity).
- The operator carries out the measurements on a chosen area and repeats the procedure until the statistics become acceptable or reach a given number of analyzed fields of view.
- Banding revealed by metallographic methods can be characterized additionally by microindentation hardness measurement across the bands to estimate segregation levels.
- A test report is generated with AI and BR, corresponding statistical data, and also the microindentation hardness measurement across the bands to estimate the segregation levels.
- The microstructural constituents in the banded structure should be identified and reported.

Conclusions

1. Ideas for quantitative assessment of banding were proposed and illustrated by simple calculations in Saltykov's book nearly 50 years ago, but unfortunately, it was never translated into English.
2. None of the criteria chosen in ASTM E1268 can adequately characterize banding; therefore, it is described verbally.
3. Anisotropy is used to characterize only the elongation of the separate features of the structure, but it cannot assess the tendency of these features (pearlite, bainite, carbides, and so on) to be combined into bands.
4. A BR that adequately estimates the degree of microstructural banding for a wide range of steel and alloy microstructures, including all the structures that are currently listed in Annex A1 of ASTM E1268, was proposed.
5. The BR and the AI, according to ASTM E1268, are estimated by the same technique; therefore, introduction of the new criterion to ASTM E1268 will be simple. A draft of these improvements was presented and discussed.
6. Attributes of new a standard practice for assessing the degree of banding or orientation of microstructures by automatic image analysis were proposed.

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The Effect of Surface Preparation on Retained Austenite Measurement

Citation

Yu, H., Lu, Y., Xu, Y., and Sisson, R. D., Jr., "The Effect of Surface Preparation on Retained Austenite Measurement," *100 Years of E04 Development of Metallographic Standards*, ASTM STP1607, G. F. Vander Voort, Ed., ASTM International, West Conshohocken, PA, 2019, pp. 12–20, <http://dx.doi.org/10.1520/STP160720170210>²

ABSTRACT

The effect of surface finish on the evaluated content of retained austenite on quenched AISI 52100 steel ($R_c = 62$) by X-ray diffraction (XRD) was investigated. The sample surfaces were prepared with different surface finishes under different load and vibratory polishing with colloidal silica suspension prior to XRD testing. The surface morphology of samples was observed by scanning electron microscope, and surface roughness was evaluated. The measured content of austenite increased with the surface roughness resulting from grinding surfaces with different surface finishes. The coarse surface grinding can deform the surface and transform some of the retained austenite into martensite. The deformation layer became thinner with finer grit. The thickness of the deformation layer can be as high as 20 μm due to coarse grinding and can be less than 7 μm due to fine polishing. The deformation layer would have a huge influence on the measurement of retained austenite due to low analyzed depth of XRD. In order to accurately evaluate the content of the retained austenite in the sample, minimizing the depth of the deformation layer should be ensured, either by fine polishing or electropolishing.

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Keywords

retained austenite, XRD, surface roughness, surface finish, scanning electron microscope, ASTM E975-13

Introduction

Retained austenite is found in the microstructure of heat treated high strength steels that have medium (0.4 wt.%) or higher carbon contents due to incomplete transformation during quenching [1]. The excessive retained austenite can have a detrimental effect on the mechanical properties of the steel. Properties such as fatigue strength, toughness, hardness, and yield strength are influenced by retained austenite. Also, the performance of the steel may be affected by transformation of retained austenite to martensite during service [1]. Retained austenite may decompose if the part works in very low temperatures, and it will cause dimensional instability. Accuracy of the measurement of retained austenite is very important. X ray diffraction (XRD) measurement is one of the most effective ways to measure the weight percentage of retained austenite. Unfortunately, the retained austenite measurement by XRD is affected by the surface finishing. The surface preparation can have an effect on the damage layer [2], and in this work, the effect of surface finishing on retained austenite measurement was studied.

EXPERIMENTAL PROCEDURE

Material

Experimental AISI 52100 steel was purchased from Peterson Steel Co. (Worcester, MA). As a type of high carbon and chromium containing steel, AISI 52100 steel is widely used in the bearing industry. This type of steel can obtain either martensitic or bainitic microstructures by heat treatment [3,4].

Thermal Process

All the AISI 52100 steel samples were austenitized at 850°C for 1 h and air cooled for normalizing treatment. Normalized samples were heat treated at 1,000°C in air and held for 1 h. After austenitizing, the samples were directly quenched in 20°C water within 5 s. The samples were covered by high temperature protective coating from Advanced Technical Products Supply Co. (Cincinnati, OH) to prevent decarburization and oxidation effect during austenitizing.

Cutting

To minimize the surface damage and thermal effects by cutting, wet cutting was utilized on specimens by saw (Mark V Laboratory CS600 specimen saw [East Granby, CT]: aluminum oxide with thickness of 0.15 in.) rather than dry abrasive cutting.

Mounting

To avoid a tempering effect, cold mounting was conducted on the samples. The whole part was cold mounted by epoxy resin. The maximum mounting temperature was 50°C.

Grinding and Polishing

A Buehler (Lake Bluff, IL) Automet 250 Polisher/Grinder was used to conduct wet grinding and polishing on the mounted samples. The loading force was 10N and the plate speed was 250 rpm. The initial grinding was 120 grit followed by 320, 1,200 grit silicon carbide (SiC) paper and vibratory polishing. The grinding time for each procedure was 90 s. A Buehler Vibromet 2 Vibratory Polisher with colloidal silica suspension (0.05 μm with 9.8 pH) as the final polishing step. The vibratory polishing time was 8 h.

Test Methods

Chemical composition, measured by an optical emission spectrometer on the Spectromax (Mahwah, NJ) are listed in Table 1. Lattice parameter measurement and phase identification were conducted by a Malvern Pananalytical (Westborough, MA) Empyrean 2 X Ray Diffractometer with Cr $K\alpha$ radiation at 30kV and 55mA. High ScorePlus (Malvern Pananalytical, Westborough, MA) was used to proceed Rietveld refinement to identify phases and their percentage. A JEOL JSM 7000f scanning electron microscope (JEOL USA, Inc. Peabody, MA) with 20kV was used to evaluate the surface morphology. Surface roughness was analyzed using an Olympus laser microscope with MountainsMap[®] Premium 7.1 software (Besançon, France).

RESULTS AND DISCUSSION

Grinding Effect on Accuracy of Retained Austenite Measurement

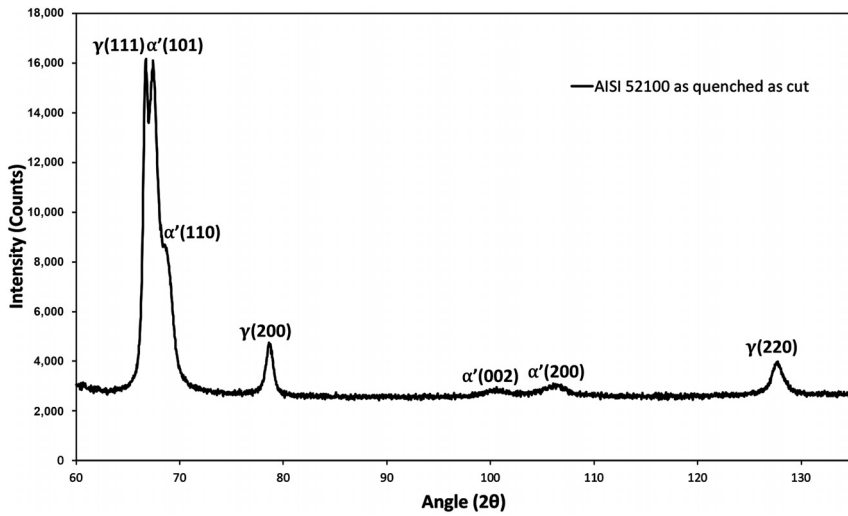
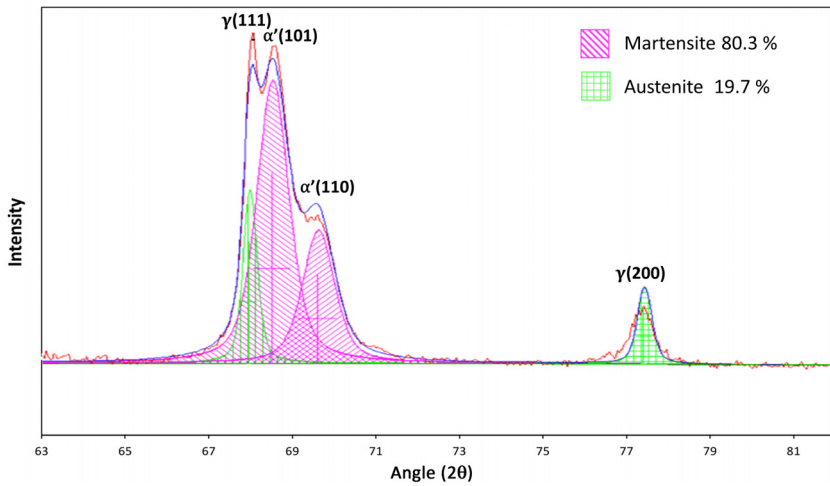
The XRD pattern of an as quenched sample is shown in Fig. 1. It can be seen that a crystal structure of body centered tetragonal is present as expected for as quenched martensite, the tetragonality c/a ratio of which is around 1.04.

As shown in Fig. 2, the Rietveld refinement method was used to analyze the phase percentage of austenite and martensite [5,6]. Martensite is a body centered tetragonal structure with $I4/mmm$ space group. Austenite is a face centered cubic structure with $Fm3m$ space group. After comparison between the experimental XRD pattern and the simulated XRD pattern based on the structure model, sample parameters, and instruments parameters, least square regression would be utilized to get the best fit. XRD patterns of samples ground with 120 grit, 320 grit, 1,200 grit, and vibratory polishing are shown in Fig. 3. It can be seen that the areas of retained austenite (111) peak and (200) peak increased with increasing grit number, and the peak areas in the polished specimen are maximum. The retained austenite

TABLE 1 OES result of AISI 52100 steel and AISI 4140 steel in wt.%.

Specimen	C	Si	Mn	P	S	Cr	Ni	Mo	Cu
AISI 52100	1.27	0.244	0.397	0.0095	0.0058	1.60	0.053	0.031	0.078

Note: OES optical emission spectrometer; C carbon; Si silicon; Mn manganese; P phosphorus; S sulfur; Cr chromium; Ni nickel; Mo molybdenum; Cu copper.

FIG. 1 XRD pattern of as quenched AISI 52100 steel as cut.**FIG. 2** Rietveld refinement method used to calculate the content of retained austenite.

content was calculated by the Rietveld refinement method, and the results are listed in [Table 2](#).

Furthermore, from [Table 2](#), it can be seen that the content of retained austenite is as low as 2.9 % with 120 grit grinding. With finer grinding, the content of retained austenite increases gradually. And the content of retained austenite is as

FIG. 3 XRD pattern of as quenched AISI 52100 steel with different surface finishing.

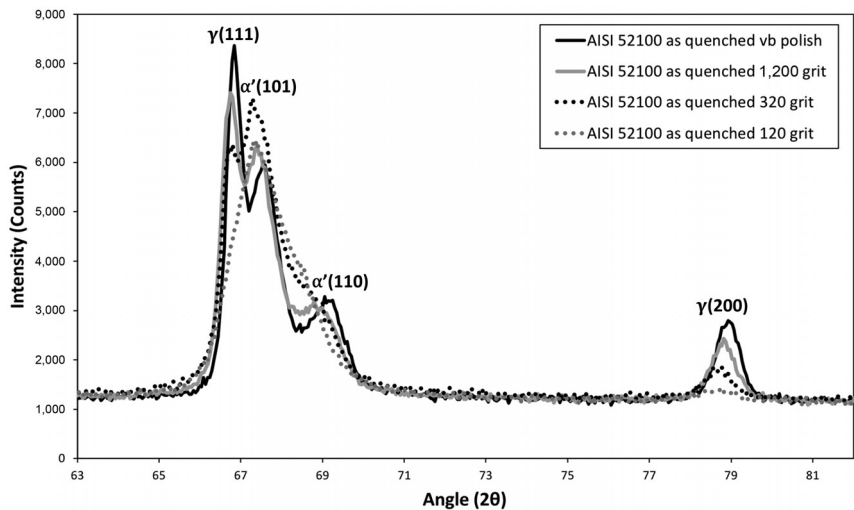


TABLE 2 The XRD result of retained austenite content with different surface finishes.

Surface Finishing Method	Content of Retained Austenite
AISI 52100 as quenched, as cut	19.7 %
AISI 52100 as quenched, grit 120	2.9 %
AISI 52100 as quenched, grit 320	13.3 %
AISI 52100 as quenched, grit 1,200	28.8 %
AISI 52100 as quenched as vb polishing	34.6 %

high as 34.6 % with vibratory polishing. It is suggested that the measured content of retained austenite will have a large difference when different surface preparations are chosen.

Force Effect on Retained Austenite Measurement

A Buehler Automet 250 Polisher/Grinder can provide as high as 40N force during grinding and polishing. XRD patterns of specimens ground with 320 grit under a grinding force between 10N and 40N are shown in Fig. 4, and no significant difference was observed. The same results were obtained with 120 grit and 1,200 grit.

Incident Beam to Prove the Existence of Deformation Layer

As shown in Fig. 5, incident beam diffraction was performed on a sample with 120 grit grinding. There is no austenite peak when the incident angle is 10°. The penetration depth for this condition is around 7 μm. It is suggested that in the

FIG. 4 XRD pattern of as quenched AISI 52100 steel, 320 grit grinding with different force.

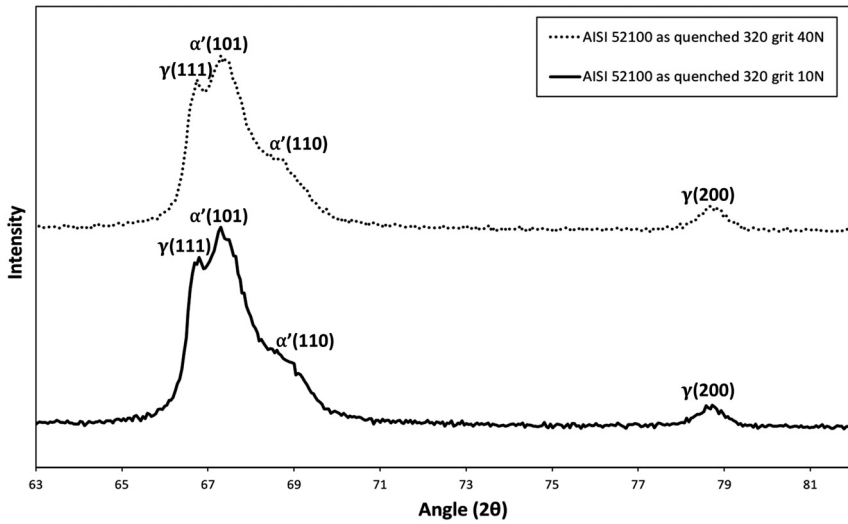
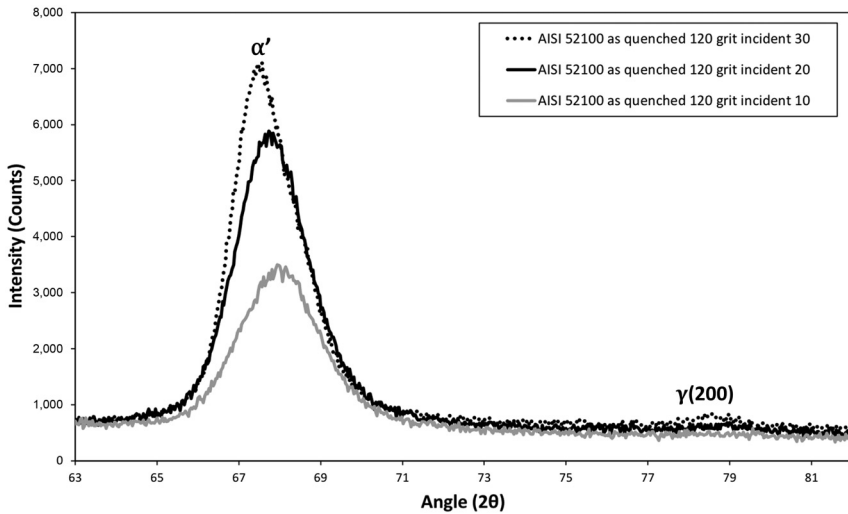


FIG. 5 XRD pattern of as quenched AISI 52100 steel, 320 grit with different incident angle.



deformation layer all the austenite transferred into martensite. It is noted that no peak splitting occurred on XRD patterns of martensite induced by grinding, meaning the $c/a = 1$ for the induced martensite. It is suggested that the induced

martensite in a deformed layer has a body centered cubic structure different from the quenched martensite of body centered tetragonal structure. When the incident angle expands to 30°, an austenite peak appears. The penetration depth for this condition is around 20 μm .

Surface Morphology of Surface under Different Grinding Conditions

Scanning electron microscope observations on the surface morphology are shown in Fig. 6. It can be seen that the grooves become finer and shallower with increasing grit number.

Surface Roughness Measurement

Surface morphology measurement was conducted by an OLS4100 Olympus three dimensional laser microscope (Olympus Scientific Solutions Americas Inc., Waltham, MA) with three by three stitching of the images, and the examined area was 1.8 mm by 1.8 mm. To analyze the results, three dimensional images were generated by MountainsMap[®] Premium 7.1 software. For a specific specimen, ten random profiles were extracted from the measured topography layer. Hybrid parameters

FIG. 6 Surface morphology of (a) as quenched AISI 52100 steel, 120 grit; (b) as quenched AISI 52100 steel, 320 grit; (c) as quenched AISI 52100 steel, 1200 grit; and (d) as quenched AISI 52100 steel, vibratory polishing.

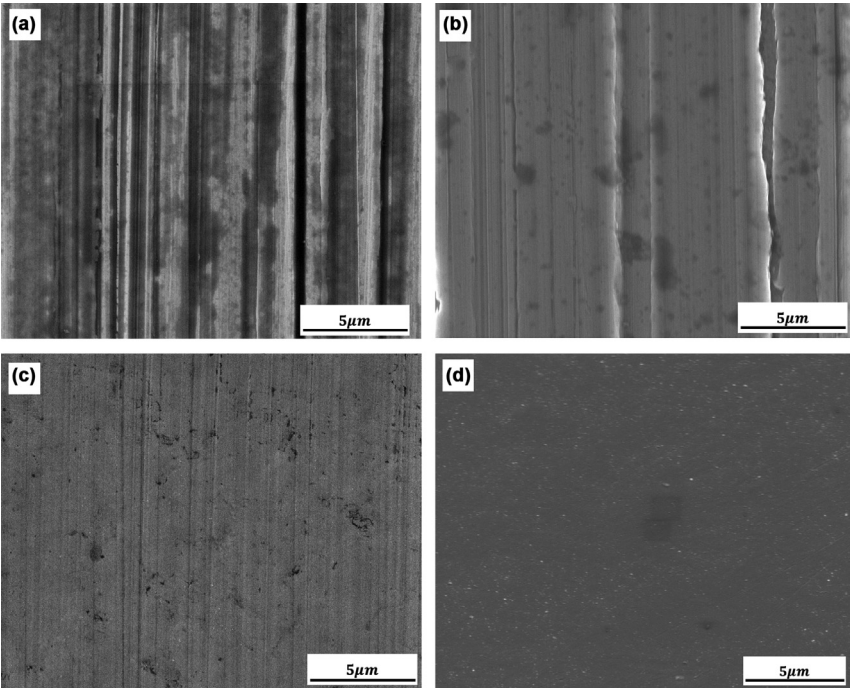
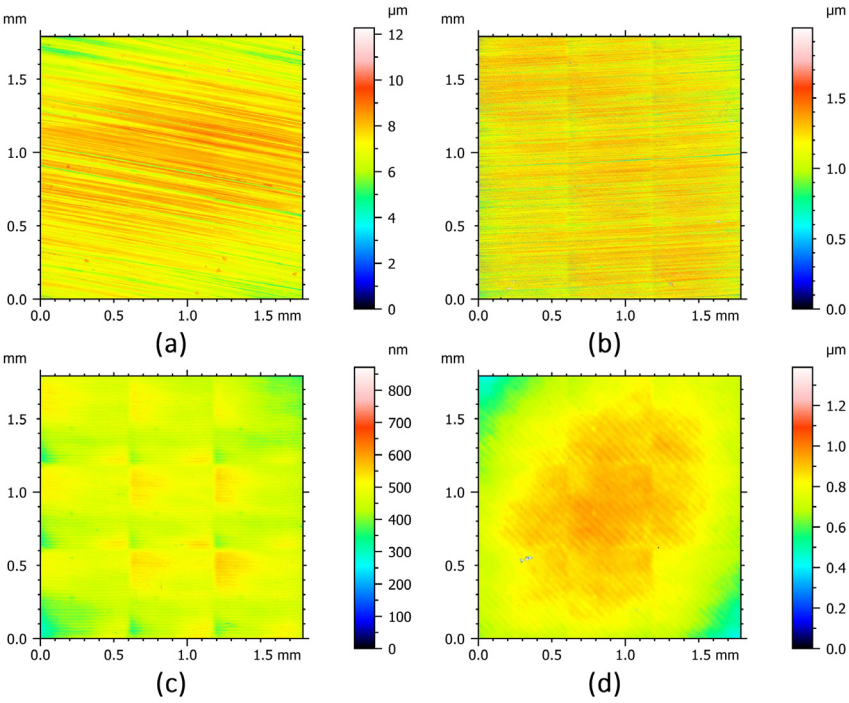


FIG. 7 Topography images of (a) as quenched AISI 52100 steel, 120 grit; (b) as quenched AISI 52100 steel, 320 grit; (c) as quenched AISI 52100 steel, 1200 grit; and (d) as quenched AISI 52100 steel, vibratory polishing.



(Sdq and Sdr), and the average surface roughness (Ra value) were generated from the software.

The surface morphology images are shown in Fig. 7. Based on the MountainsMap[®] Premium 7.1 software, the average surface roughness (Ra values) of the specimens with different surface finishes are listed in Table 3. Apart from the two dimensional surface roughness (Ra value) parameter, the three dimensional hybrid parameters were studied

TABLE 3 Surface roughness (Ra values) and hybrid parameters (three dimensional surface parameters: Sdq and Sdr) of the specimens with different surface finishes.

Specimen	Ra (nm)	Sdq	Sdr (%)
120 grit	393.4	0.849	19.4
320 grit	93.74	0.439	8.13
1200 grit	10.718	0.0427	0.0893
vb polishing	9.73	0.0392	0.0603

Note: vb vibratory

to analyze the surface morphologies. The resulting hybrid parameters (Sdq and Sdr) are also listed in [Table 1](#). The root mean square gradient (Sdq) is the root mean square value of the surface slope within the sampling area, and the surface area ratio (Sdr) expresses the increment of the interfacial surface area relative to the area of the projected plane [7–9].

Conclusion

The measured content of austenite of the specimens increased as the surface roughness decreased, resulting from grinding surfaces with different surface finishes. Coarse surface grinding can deform the surface and transform some of the retained austenite into martensite. The deformation layer became thinner with finer grit. The thickness of the deformation layer can be as high as 20 μm due to coarse grinding and can be less than 7 μm due to fine polishing. The deformation layer would have a huge influence on the measurement of retained austenite due to low analyzed depth of XRD. In order to evaluate accurately the content of retained austenite, a minimum depth of deformation layer should be ensured, either by fine polishing or electropolishing.

ACKNOWLEDGMENTS

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Metallographic Preparation and Characterization of Thermal Barrier Coatings

Citation

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ABSTRACT

Characterization of thermal sprayed thermal barrier coatings (TBCs) continues to challenge metallographers due to the lack of standardized preparation and analysis methods. Despite the past literature on this topic, ASTM E04 has yet to include a preparation method specific for TBCs. The present study aims to bring this topic to the forefront and reinvigorate the standardization in this area of metallography. Each step of metallographic preparation will be discussed to highlight its importance in revealing the true microstructure of a TBC. Modern metallographic characterization techniques will be utilized to identify a preferred preparation method for adoption as standard practice.

Keywords

thermal barrier coatings, metallographic preparation, sectioning, mounting, polishing, porosity, characterization, image analysis

Introduction

Thermal barrier coatings (TBCs) play a vital role in the successful usage of gas turbine components in modern aerospace and industrial engines. These ceramic layers are commonly applied to hot section components to allow for higher firing

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temperatures and thus improved engine efficiency. The primary goal of TBCs is to act as thermal insulation for metallic parts that are internally cooled by air or, more recently, steam. The resultant effect is reduced substrate metal temperature that allows operation of these components at service temperatures that can exceed their melting ranges [1].

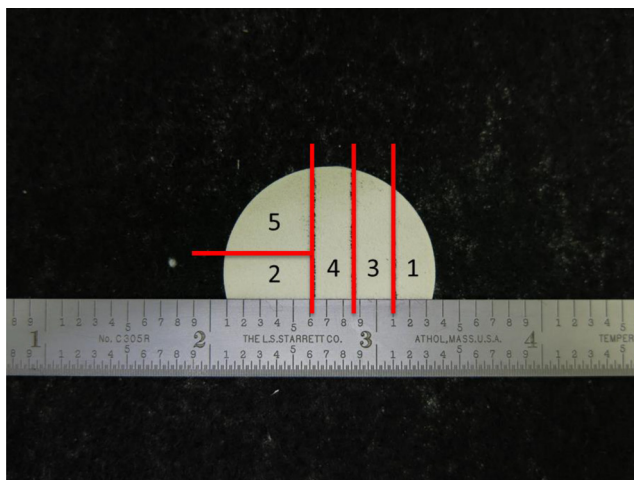
TBCs commonly are applied by two coating methods: electron beam physical vapor deposition and atmospheric plasma spray (APS). Metallographic evaluation of coatings applied by the latter thermal spray technique is an ongoing challenge faced by metallographers in the gas turbine industry. Despite the amount of literature covering this topic over the past 20 or more years [2–7], modern techniques for metallographic specimen preparation and evaluation of thermal sprayed TBCs have not proliferated the industry as some might expect. Currently, there are three key ASTM standards under jurisdiction of the E04 Committee associated with the metallographic preparation and evaluation of thermal spray coatings. These include ASTM E1920 03(2014), *Standard Guide for Metallographic Preparation of Thermal Sprayed Coatings* [8], ASTM E2109 01(2014), *Standard Test Methods for Determining Area Percentage Porosity in Thermal Sprayed Coatings* [9], and ASTM E1245 03(2016), *Standard Practice for Determining the Inclusion or Second Phase Constituent Content of Metals by Automatic Image Analysis* [10]. Until these standards gain more widespread acceptance by end users, original equipment manufacturers, and repair facilities alike, accurate metallographic characterization of thermal sprayed TBCs will be prevented by common preparation and evaluation pitfalls.

Description of Metallographic Specimen Preparation

The specimen selected for evaluation in the present work was a nickel based super alloy coupon coated with a multilayer thermal barrier coating system. The bond coat consisted of two layers; the first layer was an MCrAlY type coating applied by high velocity oxy fuel thermal spray, and the second layer was a similar MCrAlY type coating applied by APS. The top coat was a yttria stabilized zirconia TBC applied by APS. The coupon is shown in Fig. 1 and was sectioned along the red lines.

Sectioning was performed such that compressive forces were applied by the cut off wheel on the thermal spray coating. This sectioning technique is known to reduce delamination of thermal spray coatings [6,8]. The cut between Sample 2 and Sample 5 represented the aggressive sectioning method in the present work and was made using a general use tabletop abrasive cutoff saw with an aluminum oxide cutoff wheel. The wheel was designed for cutting high hardness materials and was 250 mm in diameter and 1.5 mm thick. The rotational speed and feed rate for the aggressive cut were 3,420 rpm and 0.1 mm per second, respectively. All other cuts were made using a precision tabletop cutoff saw with an aluminum oxide cutoff wheel designed for cutting high hardness materials. The precision cutoff wheel was 150 mm in diameter and 0.5 mm thick. The rotational speed and feed rate for all precision cuts were 4,700 rpm and 0.025 mm per second, respectively. Following sectioning and prior to

FIG. 1 Nickel based superalloy coupon coated with a TBC system was sectioned as shown by the red lines.



mounting, each metallographic specimen was soaked in ethanol and allowed to air dry to remove contamination from cutting fluids.

Samples 1, 2, 3, and 5 were mounted in epoxy using 25 mL of resin and 3 mL of hardener per sample as prescribed by the manufacturer's instructions. A fluorescent dye was mixed into the epoxy prior to mounting to aid in subsequent metallographic characterization. Samples 1, 2, and 5 were mounted in a vacuum impregnation machine, while Sample 3 was simply cold mounted. Prior to introducing the epoxy mixture during vacuum impregnation, the chamber was evacuated to a pressure of 0.10 bar and held for 10 min. to ensure adequate withdrawal of atmosphere from the porosity in the TBC layer. The epoxy mixture was then introduced into the mold cups, and the pressure was returned to atmospheric level to drive the epoxy into the TBC. The samples set overnight before proceeding to the grinding and polishing steps. Sample 4 was hot compression mounted in 30 mL of phenolic resin according to the recommended settings of the manufacturer.

The grinding and polishing steps were accomplished using a semiautomatic preparation machine. Samples 1, 2, 3, and 4 were ground and polished according to a method adapted from the equipment manufacturer for dense and thick ceramic plasma spray coatings. This method was similar to the diamond grinding method defined in ASTM E1920. Table 1 outlines the specific parameters for each step of this preparation method. Planar grinding was accomplished in two steps with 180 grit and 220 grit silicon carbide (SiC). Fine grinding also included two steps; the first utilized a composite disc with a 9 μm diamond suspension and the second a hard woven cloth with a 6 μm diamond suspension. Two rough polishing steps were performed the first on a hard woven cloth with a 3 μm diamond suspension and

TABLE 1 The grinding and polishing method recommended by the preparation equipment manufacturer for brittle or hard thermal spray coatings (or both) was used for Samples 1–4. This method is adapted from Table 4 in ASTM E1920 for diamond grinding.

Surface	Coolant/ Lubricant	Abrasive Size, Type	Time, min	Force, N	Surface Speed, rpm	Relative Rotation
SiC foil	Water	180 grit	Planar grinding, 0.75	40	300	Complementary
SiC foil	Water	220 grit	Planar grinding, 0.75	40	300	Complementary
Composite disc	Water based suspension	9 μm, diamond	Fine grinding, 6	30	300	Complementary
Hard Woven cloth	Water based suspension	6 μm, diamond	Fine grinding, 5	35	300	Complementary
Hard Woven cloth	Water based suspension	3 μm, diamond	Rough polishing, 4	35	300	Complementary
Low Nap cloth	Water based suspension	1 μm, diamond	Rough polishing, 5	35	300	Complementary
Synthetic suede		0.04 μm, colloidal silica	Final polishing, 1	10	300	Contrary

the second on a low nap cloth with a 1 μm diamond suspension. Final polishing was performed on a synthetic suede surface with a 0.04 μm colloidal silica and used contrary rotation of the polishing disc and sample head.

Sample 5 was ground and polished according to Table 2. The preparation method for Sample 5 was adapted from the SiC abrasive grinding method from ASTM E1920. As can be seen in Table 2, planar grinding was identical to that performed for Samples 1, 2, 3, and 4. Fine grinding was accomplished with SiC fixed abrasive foils

TABLE 2 The grinding and polishing method recommended by the preparation equipment for brittle or hard thermal spray coatings (or both) was used for Sample 5. This method is adapted from Table 3 in ASTM E1920 for SiC abrasive paper grinding.

Surface	Coolant/ Lubricant	Abrasive Size, Type	Time, min	Force, N	Surface Speed, rpm	Relative Rotation
SiC foil	Water	180 grit	Planar grinding, 0.75	40	300	Complementary
SiC foil	Water	220 grit	Planar grinding, 0.75	40	300	Complementary
SiC foil	Water	320 grit	Fine grinding, 0.75	30	300	Complementary
SiC foil	Water	500 grit	Fine grinding, 0.75	30	300	Complementary
SiC foil	Water	800 grit	Fine grinding, 0.75	30	300	Complementary
SiC foil	Water	1,200 grit	Fine grinding, 0.75	30	300	Complementary
Hard woven cloth	Water based suspension	3 μm, diamond	Rough polishing, 4	35	300	Complementary
Synthetic suede		0.04 μm, colloidal silica	Final polishing, 1	10	300	Contrary

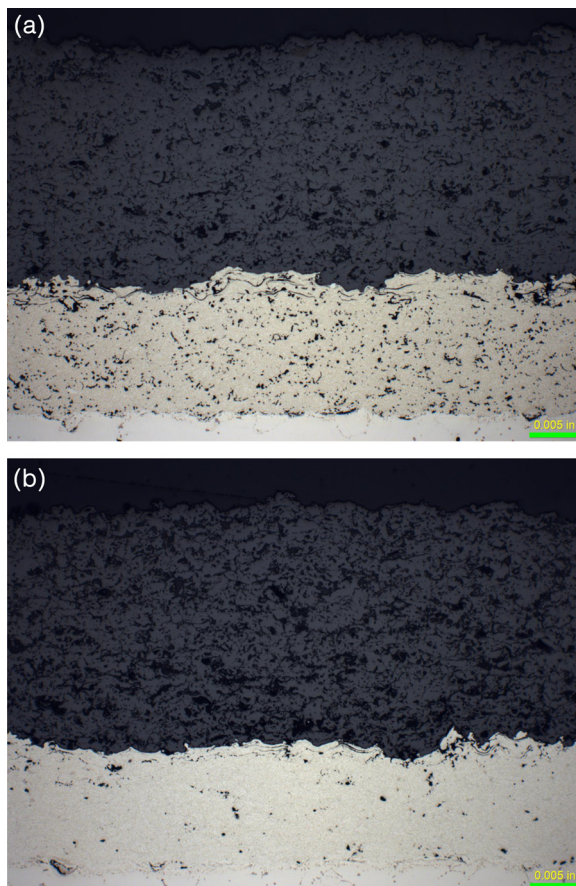
with successively finer grit sizes. Rough polishing was performed with a 3 μm diamond suspension on a hard woven cloth. Final polishing was identical to that performed on Samples 1, 2, 3, and 4.

Discussion of Results

SECTIONING

Representative photomicrographs of the metallographic specimens that were sectioned with different equipment are shown in Fig. 2. No cracks or delamination were observed within the TBC or at the bond coat top coat interface of either

FIG. 2 Comparison of thermal barrier coating microstructures of (a) Sample 1 that was sectioned by a precision cutoff saw and (b) Sample 2 that was sectioned by a more aggressive tabletop cutoff saw. Both specimens were ground and polished using the same technique.



Sample 1 or Sample 2, as can be seen in [Fig. 2a](#) and [b](#), respectively. However, a qualitative comparison of the two TBC microstructures revealed variations in the apparent level of porosity. This observation indicated that the recommended grinding and polishing procedure adapted from the diamond grinding method of ASTM [E1920](#) did not remove all damage caused by the more aggressive sectioning of Sample 2. It should also be noted that the bond coat microstructures varied significantly due to differences in sectioning methods. The more aggressive cutoff saw caused smearing of the metallic bond coat of Sample 2, thus reducing the amount of apparent porosity with respect to Sample 1.

MOUNTING

Three different mounting techniques were used to evaluate the effect on the resultant TBC microstructures. As described earlier in the metallographic preparation section, the techniques included vacuum impregnation epoxy cold mounting, epoxy cold mounting, and hot compression mounting.

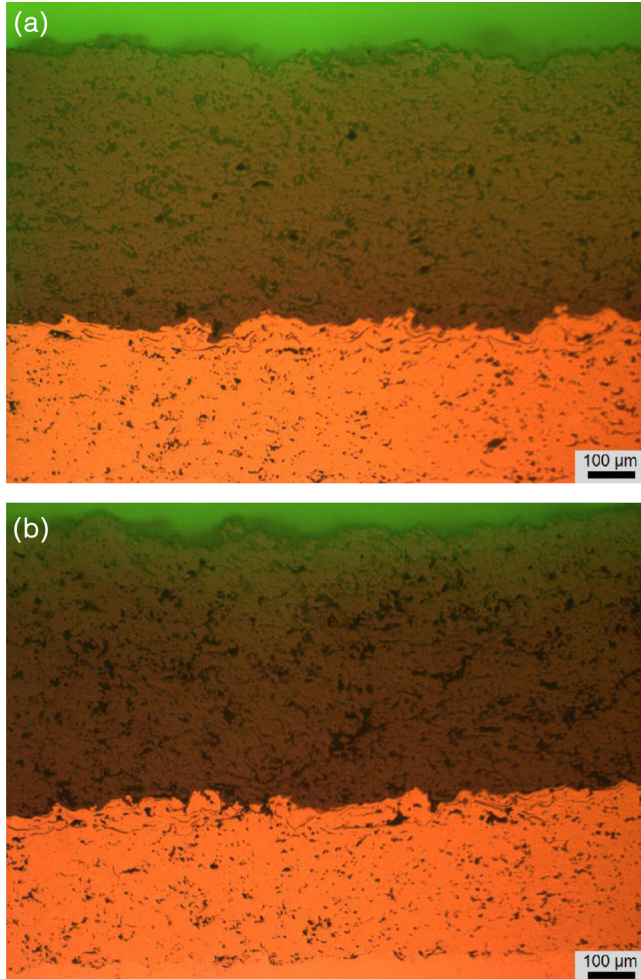
Representative photomicrographs are presented of Sample 1 ([Fig. 3a](#)), which was vacuum impregnated with epoxy and fluorescent dye, and Sample 3 ([Fig. 3b](#)), which was cold mounted with epoxy and fluorescent dye. Sample 3 did not undergo vacuum impregnation and, as expected, the epoxy mixture did not adequately penetrate the TBC layer. The fluorescent dye was only visible near the surface of the TBC as evidenced by the green hue. This finding was previously reported by Puerta [\[2\]](#) and Sauer [\[7\]](#). Sample 1 did exhibit complete infiltration of epoxy in the TBC following vacuum impregnation. The fluorescent dye was detectable at the bond coat top coat interface, as shown in [Fig. 3a](#). This observation contradicts the claim made by Sauer, Leonhardt, and Geary [\[5\]](#) that noted complete infiltration of the TBC was not possible with vacuum impregnation alone, but rather vacuum and pressure impregnation were required to achieve total infiltration of thick TBCs. Despite these different findings, the authors agree with the literature [\[2,5,7\]](#) that complete penetration of the brittle ceramic TBC layer by epoxy is necessary to support the coating during subsequent preparation steps.

Representative photomicrographs are shown of Sample 1 ([Fig. 4a](#)) and Sample 4 ([Fig. 4b](#)), which were vacuum impregnated with epoxy and fluorescent dye and hot compression mounted with phenolic resin, respectively. The supporting effect of the epoxy in Sample 1 is highlighted by the presence of severe pull out in Sample 4. Due to the compressive forces applied during hot mounting, the brittle TBC was more susceptible to pull out defects during subsequent grinding and polishing. Despite the use of the recommended method from ASTM [E1920](#), the mechanically induced damage during hot mounting could not be removed. Sauer, Leonhardt, and Geary [\[5\]](#) had similar results following preparation of a hot mounted TBC.

GRINDING AND POLISHING

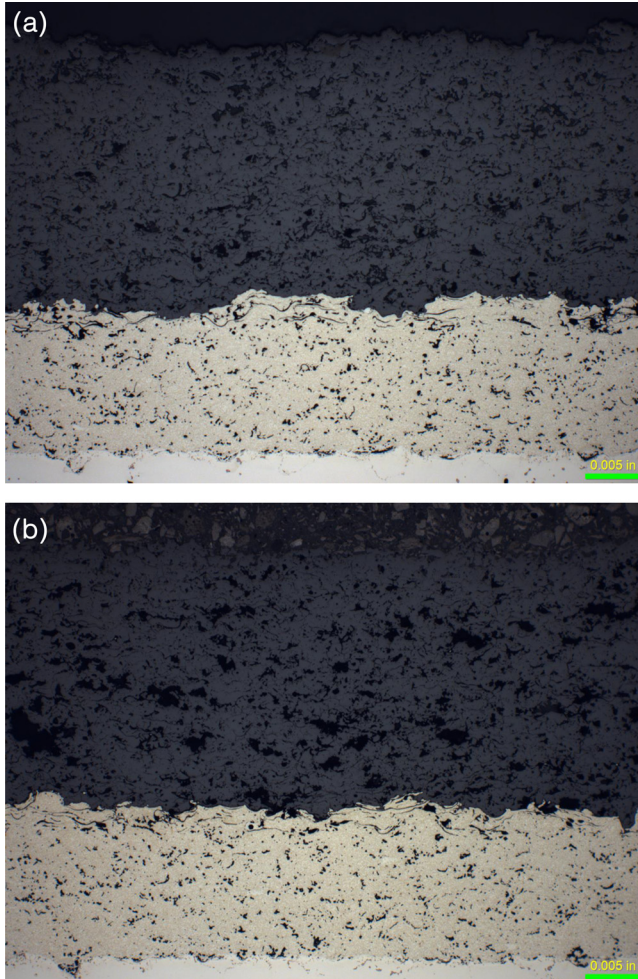
Samples 1 and 5 were ground and polished with different methods to compare the effect of these methods. Sample 1 was ground and polished following a procedure

FIG. 3 Comparison of thermal barrier coating microstructures of (a) Sample 1 that was vacuum impregnation mounted with epoxy and fluorescent dye and (b) Sample 3 that was cold mounted with epoxy and fluorescent dye. Both specimens were polished using the same technique.



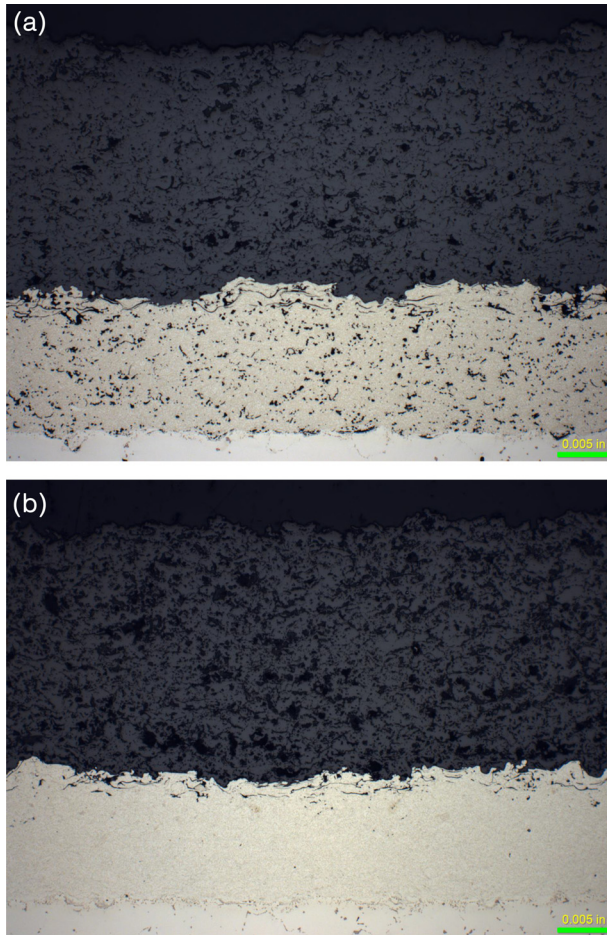
adapted from ASTM E1920 for diamond grinding, and Sample 5 followed a procedure adapted from ASTM E1920 for SiC abrasive paper grinding. Based on the representative photomicrographs shown in Fig. 5, it was clear that the two methods revealed different TBC microstructures. The TBC in Sample 1 (Fig. 5a) exhibited sharp edges around the features of interest, including porosity, interlamellar splats, and pull out. Sample 5 (Fig. 5b) differed due to edge rounding, exaggerated pull out, and the presence of damage from coarse grinding that was not removed by

FIG. 4 Comparison of thermal barrier coating microstructures of (a) Sample 1 that was vacuum impregnation mounted with epoxy and fluorescent dye and (b) Sample 4 that was hot compression mounted. Both specimens were polished using the same technique.



subsequent polishing steps. These observations indicated that the diamond grinding method is preferable for revealing the true microstructure of TBCs. Although not the topic of this work, it should be noted that the SiC abrasive grinding resulted in a drastically different bond coat microstructure compared to the diamond grinding method. The use of fixed abrasives caused the metallic bond coat to smear, which hid the true microstructure. The one step of rough polishing with 3 μm diamond suspension was unable to remove the smearing damage.

FIG. 5 Comparison of thermal barrier coating microstructures of (a) Sample 1 that was prepared by the diamond grinding method, and (b) Sample 5 that was prepared by the silicon carbide abrasive grinding method. Both methods were adapted from ASTM E1920 with recommendations from the preparation equipment manufacturer.



FLUORESCENCE MICROSCOPY

The use of fluorescent dye additives in conjunction with vacuum impregnation epoxy cold mounting has been shown to assist in metallographic characterization of thermal spray coatings [11]. However, despite its existence and successful implementation by some metallographers [2,5], fluorescent microscopy is not widely used. In the preparation of the current work, the authors attempted to acquire the necessary microscope filter to excite the commercially available fluorescent dye. This proved to be a challenge because this filter was no longer manufactured by the

former sole supplier. As a result, the authors contacted the dye manufacturer and were graciously granted use of their facilities to perform fluorescent microscopy.

Representative photomicrographs of Sample 1 taken in fluorescence mode are shown in Fig. 6. Minor brightness and contrast adjustments were made to the as captured image shown in Fig. 6a to enable accurate characterization of the porosity in the TBC. The processed photomicrograph is shown in Fig. 6b.

The necessity for postprocessing highlights the challenges that can accompany any new microscopy technique. As this was the first attempt at fluorescence microscopy for the authors, it is expected that the as captured photomicrographs can be improved and thus reduce the amount of postprocessing required prior to image analysis.

FIG. 6 Representative fluorescence microscopy photomicrographs of Sample 1 are shown in (a) the as captured condition, and (b) the postprocessed condition.

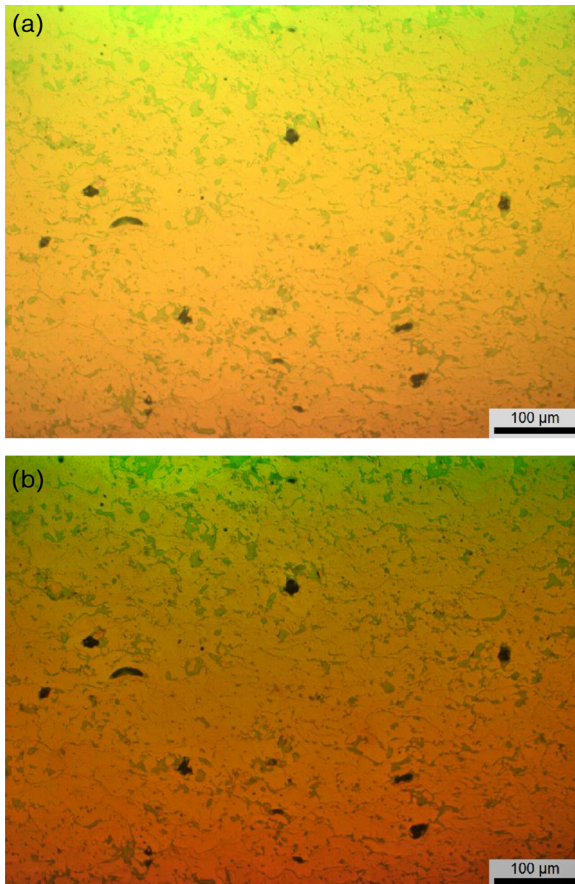
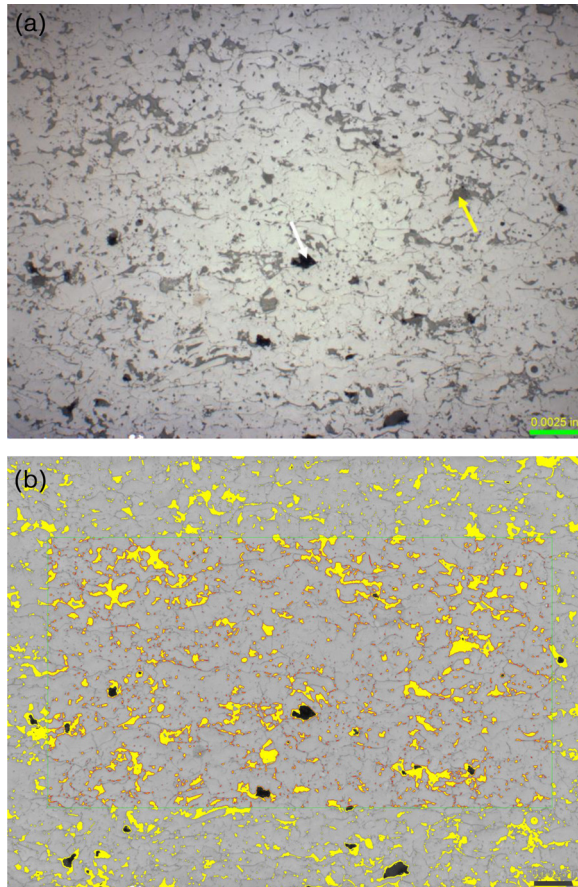


IMAGE ANALYSIS

Metallographic characterization of the ground and polished samples was conducted to quantify the porosity area percentage in the TBC layer. This characterization was supported by use of semiautomated image analysis software. First, photomicrographs from seven nonoverlapping fields of view were captured per sample. A background correction was applied to each photomicrograph to improve lighting uniformity across the field of view. Next, the processed photomicrographs underwent image analysis using the thresholding technique to segment the porosity in the ceramic TBC.

In the case of the vacuum impregnated samples, the porosity was quantified as that area that contained evidence of epoxy infiltration, while areas characterized as preparation induced pull out were discounted. [Fig. 7](#) shows a representative field of

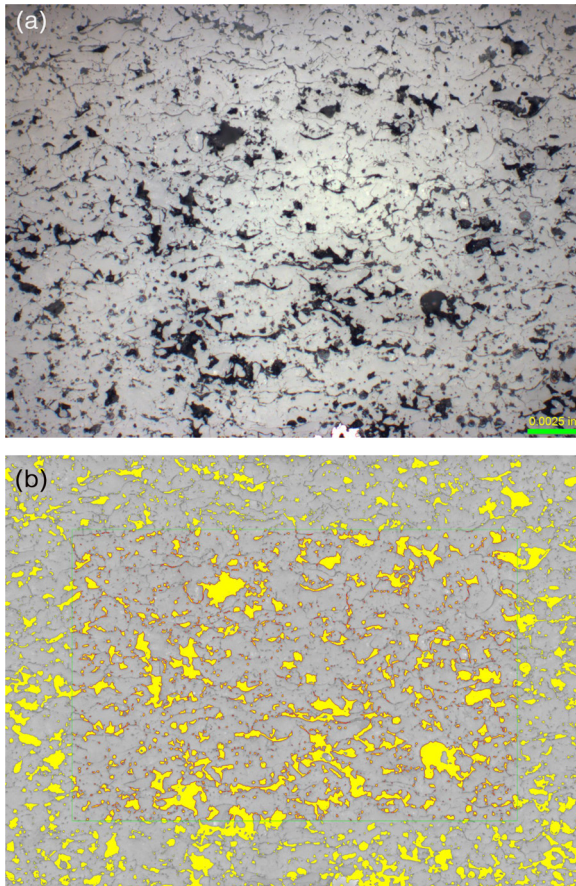
FIG. 7 Representative field of view of the TBC in Sample 1 showing (a) the as captured photomicrograph and (b) the post image analysis photomicrograph with porosity colored yellow.



view of Sample 1 before and after image analysis. Epoxy infiltrated porosity is identified by the yellow arrow and pull out by the white arrow in Fig. 7a. The photomicrograph following image analysis is shown in Fig. 7b with the porosity colored yellow. It could be argued that the lack of epoxy infiltration was due to closed porosity. However, the presence of epoxy touching the majority of these pores indicated that they are primarily pull out of the TBC ceramic. It should also be noted that a minor misinterpretation of true porosity as pull out or vice versa is within the error of the thresholding technique used for image analysis.

Sample 3, which was cold mounted in epoxy without vacuum impregnation, is shown in Fig. 8. The lack of epoxy infiltration made discernment of inherent

FIG. 8 Representative field of view of the TBC in Sample 3 showing (a) the as captured photomicrograph and (b) the post image analysis photomicrograph with porosity or pull out (or both) colored yellow.



coating porosity versus preparation induced pull out impossible. Therefore, all voids in the TBC were quantified as porosity, which artificially elevated the area percentage.

Image analysis was also conducted on photomicrographs captured using fluorescence microscopy. A representative field of view from Sample 1 is shown after postprocessing (Fig. 9a) and after image analysis (Fig 9b). Nonuniform lighting across the field of view is evident in the post image analysis photomicrograph. This required selection of a smaller analysis area to reduce thresholding artifacts near the perimeter of the analysis area.

FIG. 9 Representative field of view of the TBC in Sample 1 captured using fluorescence microscopy showing (a) the post processed photomicrograph and (b) the post image analysis photomicrograph with porosity colored yellow.

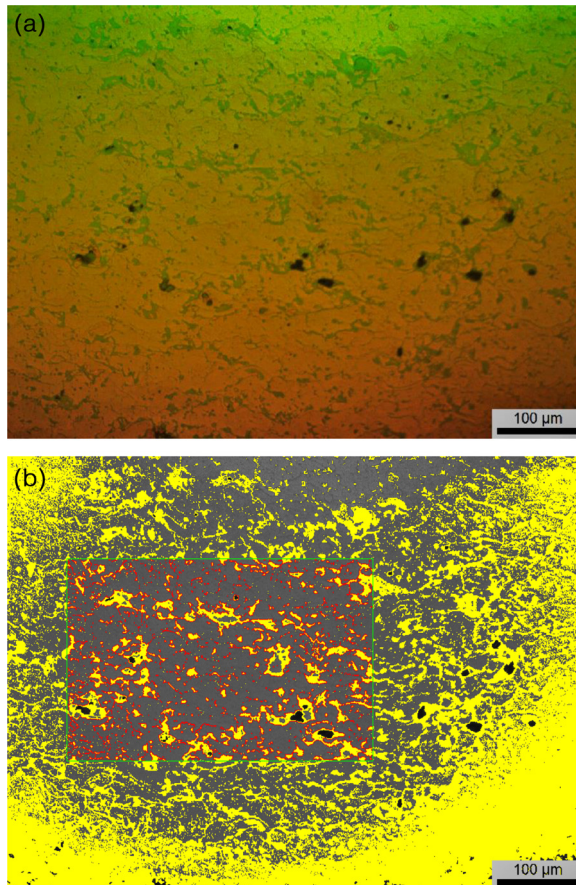


TABLE 3 Area percentage porosity results for Samples 1 through 5. All values reported as area percentages.

Field	1	2	3	4	5
1	13.82	15.49	17.61	15.79	23.98
2	9.99	17.71	15.17	17.94	21.06
3	11.76	16.58	18.00	18.15	21.35
4	9.44	14.16	17.28	24.18	21.94
5	14.06	15.84	17.94	32.01	25.37
6	10.28	17.13	17.04	20.92	22.15
7	9.86	18.39	15.91	20.98	24.50
Average	11.32	16.47	16.99	21.42	22.91
Std. Dev.	1.94	1.44	1.07	5.40	1.69

The area percentage porosity results are presented in [Table 3](#) for Samples 1 through 5. The reported results were obtained through analysis of bright light optical photomicrographs to maintain consistency across all samples. Sample 1 exhibited the least amount of porosity area percentage due to the lack of artifacts caused by improper preparation. All other samples exhibited increased porosity levels caused by a departure from the recommended preparation method Sample 2 due to aggressive sectioning, Sample 3 due to lack of epoxy infiltration in TBC, Sample 4 due to hot compression mounting, and Sample 5 due to fixed abrasive grinding and polishing.

Conclusions

The present work reiterates many of the conclusions found in the literature on metallographic preparation and characterization of thermal sprayed coatings. Based on the analysis performed by the authors, it is abundantly clear that each step of specimen preparation is crucial to reveal the true coating microstructure and, in turn, accurate coating characterization.

Thermal barrier coatings are particularly challenging due to the brittle nature of a thick, ceramic layer applied by thermal spray. For accurate characterization of the true microstructure of a TBC, the authors conclude the following based on the present study:

- Sectioning should be accomplished with a precision cutoff saw and thin abrasive cutoff wheel, when possible. If a more aggressive sectioning method is required due to part geometry, the operator must ensure sufficient material is removed during grinding to remove coating damage from sectioning.
- Mounting should be accomplished by vacuum impregnation epoxy cold mounting to provide support to the brittle TBC layer. Use of a fluorescent dye additive in the epoxy can facilitate subsequent image analysis.

- Grinding and polishing should be performed in accordance with the procedure outlined in [Table 1](#), which was adapted from the diamond grinding method in ASTM [E1920](#). Fixed abrasive grinding can develop artifacts in the coating microstructure that can only be removed with significant rough polishing using diamond suspensions.

Adoption of these recommended preparation methods will require support from key players in the thermal spray industry and end users of products with thermal barrier coatings. To facilitate usage of a standardized preparation method for TBCs, ASTM International E04 Committee on Metallography may consider revision of ASTM [E1920](#) to include a recommended method for this coating type.

ACKNOWLEDGMENTS

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Utility of Stereology for Quantitative Metallography

Citation

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ABSTRACT

Metallographers typically characterize microstructure observed in two-dimensional (2D) metallographic sections through the three-dimensional (3D) material microstructure, although 3D microstructure governs material properties. Stereology utilizes statistics and stochastic geometry to relate important properties of 3D microstructure to the measurements that can be performed in 2D metallographic sections. Furthermore, stereology provides statistical techniques for unbiased sampling and characterization of microstructure observed in 2D metallographic sections. Consequently, stereology is a useful tool for metallographers. Two important stereological techniques that are useful for quantitative metallography are reviewed in this paper.

Keywords

stereology, quantitative metallography, number density, surface area, anisotropy

Introduction and Background

Process structure properties relationships are of central importance in materials science and engineering. Microstructure is an important aspect of material structure. Consequently, quantitative characterization, modeling, and simulations of microstructural geometry are of importance. Material microstructures are usually

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three dimensional (3D); therefore, geometric attributes of 3D microstructure are of relevance in process structure properties relationships. Consequently, quantitative characterization of 3D microstructures is of core interest.

Although 3D microstructural parameters are of fundamental importance, it is convenient to observe microstructures in two dimensional (2D) metallographic planes/sections through the 3D microstructure (a standard metallography practice). Microstructure observed in a 2D metallographic section consists of intersections of the features in the 3D microstructure with the sectioning plane. As a result, in a metallographic plane, volumes (for example, particles, grains, and so on) in a 3D microstructure that appear as areas and surfaces (for example, grain boundaries) in a 3D microstructure appear as lines. Clearly, a 2D metallographic section cannot contain all the information concerning the 3D microstructural geometry. Nonetheless, several important geometric parameters of 3D microstructures can be estimated from the measurements performed in 2D metallographic sections without involving any assumptions using stereological techniques. Stereology involves applications of statistics and stochastic geometry [1,2] to determine which attributes of 3D microstructure can be estimated from measurements in 2D metallographic sections without making any assumptions and how to perform the required measurements in the 2D metallographic sections to estimate accessible 3D microstructural parameters [3–8].

For all practical purposes, material microstructures are of infinite extent. Therefore, it is not possible to measure geometric parameters of each feature in a microstructure to arrive at the mean values (or distributions) of such populations. Furthermore, microstructures are stochastic in nature; no two fields of view in the same microstructure are identical. Consequently, it is imperative to use unbiased statistical sampling techniques for unbiased estimation of 2D and 3D microstructural parameters. Stereology enables unbiased estimation of microstructural parameters together with the associated statistical sampling error.

Despite geometric complexities of microstructures, stereological procedures are fairly simple to use in practice. Numerous geometric parameters of 3D microstructures can be estimated in an unbiased manner from the counting measurements performed in 2D metallographic sections, without involving any assumptions concerning microstructural geometry. Stereological methods usually involve statistical sampling of 3D microstructure using lower dimensional test probes like points, straight lines (or curves), and planes (or surface). In most cases, the procedure simply consists of counting the number of times the test probe intersects the microstructural features of interest; the mean value of such counts is uniquely related to specific geometric attributes of the 3D microstructure through simple stereological equations.

Basic principles of classical stereology are covered in numerous books [3–8]. Nevertheless, during the past couple of decades or so, there has been substantial progress in the development of new design based stereological techniques for efficient characterization of microstructures [9–18] and techniques for unbiased

estimation of 2D and 3D number density of microstructural features [19 22]. In addition, techniques have been developed for detailed statistical representation of microstructures using radial distribution functions [23], coordination number distributions [24], correlation functions [25], contact distributions [26], and lineal path probability distributions [27]. Techniques have also been developed for estimation of bivariate and trivariate size shape orientation distributions of particles/inclusions in 3D microstructures [28 30] and for microstructure simulations [31 33]. It is beyond the scope of this contribution to present all of these techniques in detail. Consequently, the focus of the paper is on unbiased and efficient stereological techniques for estimation of number density of microstructural features in 2D and 3D microstructures and unbiased and efficient estimation of total surface area of interfaces/grain boundaries per unit volume, particularly in anisotropic 3D microstructures. Both of these microstructural parameters are of significant interest in metallography and in ASTM standards for quantitative metallographic techniques.

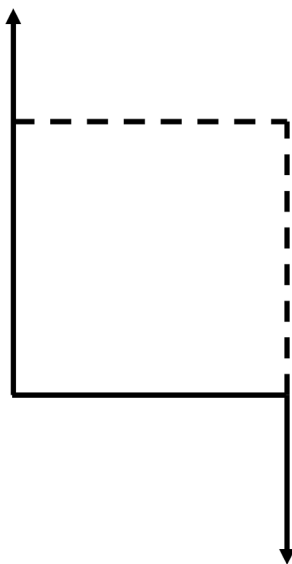
Estimation of Number Density of Microstructural Features

NUMBER DENSITY IN 2D METALLOGRAPHIC SECTIONS

The average number of microstructural features of interest (inclusions, grains, and so on) per unit area of metallographic plane is a frequently measured quantitative metallographic parameter. Unfortunately, the techniques used by metallographers for estimation of number density of features in a metallographic plane are biased; they lead to a systematic error of unknown magnitude. The bias arises due to an “edge” effect. This edge effect and unbiased stereological procedure that eliminates the edge effect are described as follows.

A microstructural field of view observed in a microscope typically contains a small fraction of the total area of the metallographic plane because metallographic observations and measurements have to be performed at sufficiently high magnification to detect and clearly resolve the features of interest. Consequently, in each field of view, there are always some features of interest that are partly in/partly out. How can we account for such partly in/partly out features when the number of features in that field of view/measurement frame are counted? Not including such features in the number count leads to a bias that results in an underestimation of number density; including them in the number count also leads to a bias that leads to an overestimation; counting each partly in/partly out feature as one half also leads to a bias that varies from one microstructure to another, and therefore, is unknown. A statistically correct and geometrically general solution to this edge effect problem was given by Gundersen [19], which involves the use of an unbiased counting/measurement frame. Fig. 1 depicts such an unbiased counting/measurement frame. It is a counting frame of area A_f that has two forbidden edges (two solid lines in Fig. 1) and two permissible edges (two broken lines in Fig. 1). The unbiased counting

FIG.1 Unbiased counting/measurement frame for estimation of number density in a metallographic plane [19]. The solid lines are the forbidden edges, and the broken lines are the permissible edges.



frame is superimposed on a microstructural field of view/micrograph. The counting procedure is as follows:

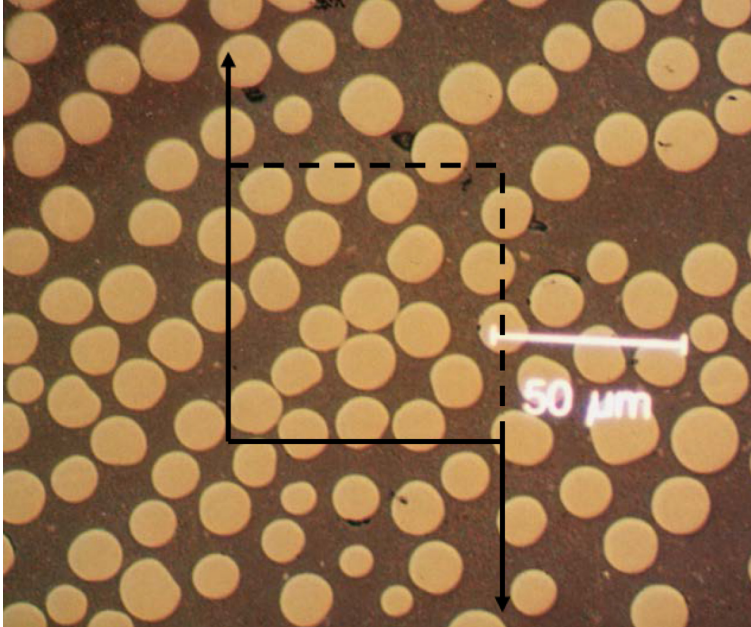
1. Count every feature that is completely contained in the unbiased measurement frame and, therefore, does not intersect any edge of the counting frame.
2. Do *not* count features that intersect one or both forbidden edges (solid lines).
3. Count every feature that intersects the permissible edge(s) that is, broken lines provided they do *not* also intersect any forbidden edge (solid line).
4. The average value of the number of features counted in this manner divided by the area of the unbiased measurement frame A_f is an unbiased estimator of the 2D number density in the observed metallographic plane.
5. The statistical sampling error in the estimated number density can be estimated using standard statistical procedures [3,4].

This procedure for unbiased counting of the number of features in a metallographic section is illustrated in [Fig. 2](#).

ESTIMATION OF NUMBER DENSITY IN 3D MICROSTRUCTURE

Three dimensional number density (i.e., number per unit *volume*) of microstructural features (inclusions, grains, voids, and so on) is an important microstructural parameter of interest in process microstructure properties studies. Unfortunately, in general, the number density of the features in a 3D microstructure cannot be

FIG. 2 Microstructure of uniaxial aligned fiber composite. There are ten fibers completely inside the counting frame; there are seven fibers that intersect one or both forbidden edges (and, therefore, are not to be included in the number count); and there are six fibers that intersect one or both permissible edges and do not intersect any forbidden edge (and, therefore, are to be included in the number count). Effectively there are 16 fibers in the measurement frame having an area $3,953 \mu\text{m}^2$. Thus, the local value of the number of fibers per unit area is equal to 4.05×10^{-3} per μm^2 [8].



estimated from any measurements performed on independent random 2D metallographic sections, unless the particles/grains have a known simple convex shape [2–6]. Sterio [20] has developed a 3D stereological sampling probe, a disector, for unbiased estimation of the number density of features of any arbitrary shapes and sizes in a 3D microstructure. A disector consists of two parallel metallographic planes (two serial sections) that are separated by a known distance t . The distance t between the disector planes must be less than one fifth of the average size of the features of interest. Observations on *both* disector planes are necessary for estimation of 3D number density. An unbiased counting frame of area A_f is placed in the first disector plane, and the features effectively present in that frame are noted. These features are followed in the second plane. Out of the features sampled by the unbiased counting frame in the first disector plane, those that are *not* present in the second plane, Q^- , are counted. The average value of the quantity $[Q^-]/[A_f t]$ is an

unbiased estimator of the number of features per unit volume (i.e., number density) N_V in the 3D microstructure. Alternately, an unbiased counting frame can be placed in the second metallographic plane of the disector, and the features sampled by the unbiased frame that are *not* present in the first plane, Q^+ , may be counted in exactly the same way. In practice, it is efficient to count both Q^+ and Q^- and use the following equation for an unbiased estimation of number density, N_V [20].

$$N_V = [Q^+ + Q^-] / [2A_f t] \quad (1)$$

The disector estimation procedure is illustrated in Fig. 3.

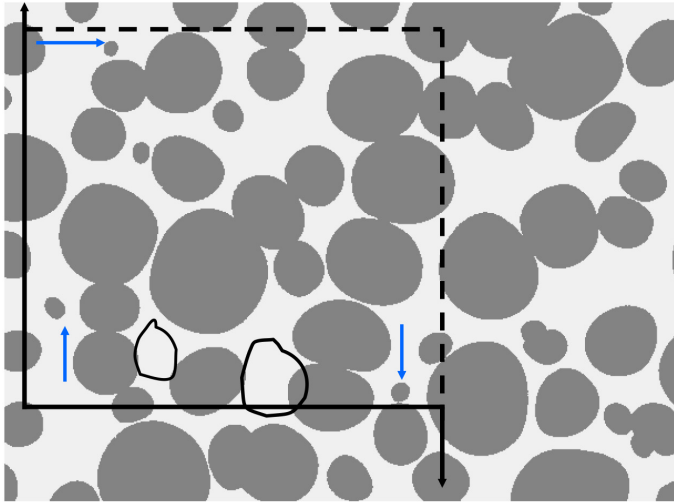
In opaque material microstructures, practical application of the disector probe requires storing of digital image of a microstructural window (field of view) in the first disector plane, polishing away small *known* thickness t of the metallographic specimen, etching to reveal microstructure, and storage of the microstructural window in the second disector plane exactly below the window captured in the first plane, which is followed by the stereological measurements described in the previous paragraph. In this process, almost all effort is spent on the steps such as polishing and etching of disector planes, identification of the same microstructural region in the two sectioning planes, alignment of the images in the two planes, and measurement of thickness (i.e., distance between disector planes t) of the material removed. After all the tedious metallographic work, a very small numbers of particle counts are obtained. The number density estimated from such a small statistical sample (although unbiased) has a large sampling error. Therefore, to decrease the sampling error, it is necessary to repeat the procedure on (typically) 25 to 50 different disectors, which requires physical sectioning of a large number of metallographic planes and involves tedious metallographic work. A practical solution to this problem utilizes a combination of the disector principle and modern digital image processing techniques, resulting in a very efficient sampling technique, a large area disector (LAD), which is described in the next subsection [21].

LARGE AREA DISECTOR (LAD)

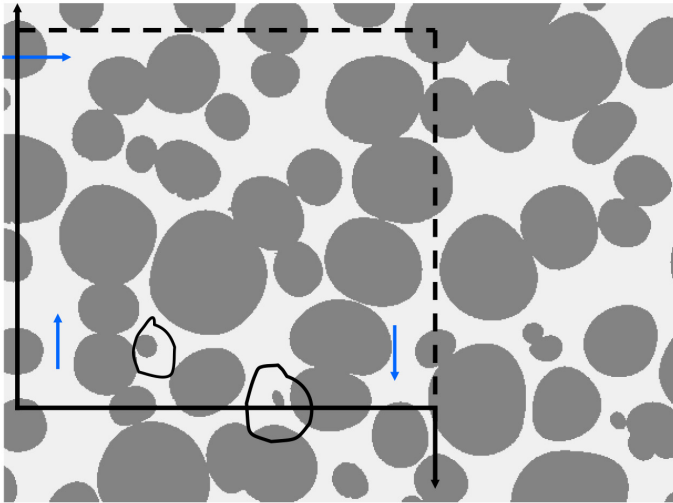
For the same effort, the disector counts can be increased by expanding the area of the disector planes by observing the microstructure at a lower magnification. This is often not acceptable due to loss of resolution that can lead to significant measurement errors. It is possible to obtain a large microstructural area *at a high resolution* by creating a “seamless montage” of a very large number of contiguous microstructural fields grabbed at a high resolution and “stitching them together” using digital image processing [34]. Number density can be then efficiently estimated as follows [21]:

1. Identify a region in the first disector plane, covering about 25 to 100 microstructural fields, and place microhardness indents at its boundaries.
2. Create a seamless “montage” covering all the microstructural fields in this region at sufficiently high resolution (magnification) such that all features of interest are clearly resolved using the “montage” technique described by Louis and Gokhale [34].

FIG. 3 An example of an application of the disector method. In (a), the three particles at the arrows that are sampled by the unbiased counting frame in the first plane are not present in the second disector plane segment shown in (b). Thus, in this case, $Q = 3$. On the other hand, two particles encircled in (b) are not present in (a). However, one of these particles intersects the forbidden edge and, consequently, is not effectively present in the counting frame in (b). Therefore, $Q^+ = 1$.



(a)



(b)

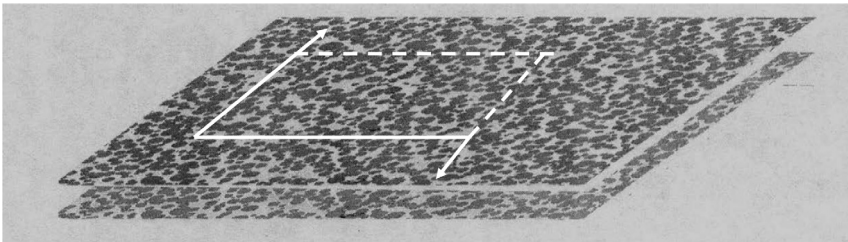
3. Remove a small amount of material by polishing and identify the same region in the second disector plane by using the positions of the microhardness indents for reference.
4. Create the seamless montage of all the microstructural fields of the same region in the second disector plane, and align the two montages using the microhardness indents as the reference [21]. The two montages then constitute the LAD (Fig. 4).
5. Measure the disector thickness t from the decrease in the sizes of microhardness indents.
6. Place a large unbiased counting frame in the first LAD plane and note the features that can be effectively assigned to that frame using the unbiased counting procedure given by Sterio [20].
7. Out of the features sampled by the first plane, identify those absent in the second plane (i.e., Q^-) or vice versa (i.e., Q^+), as in Fig. 3, and estimate the number density using Eq 1.

In the uniform random microstructures, efficient and reliable estimate of 3D number density of microstructural features of interest can be obtained by systematically sampling with three LADs at three different systematic random locations [21].

Efficient and Unbiased Estimation of Surface Area per Unit Volume

The total area of microstructural surfaces such as grain boundaries per unit microstructural volume is the ratio of the sum of the areas of all the internal surfaces of interest contained in a specimen divided by the volume of the specimen, and it is denoted by the symbol S_V . In two microstructures having the same volume fraction of a phase, S_V is higher in the microstructure having a finer

FIG. 4 Two planes of LAD, each of which is a seamless montage of a large number of contiguous microstructural fields grabbed at a high resolution. A large area of the unbiased counting frame is overlaid on the first LAD plane [21].



dispersion of that phase. Therefore, S_V can be used to characterize how fine (or coarse) a microstructure is. In polycrystalline single phase materials, mean intercept grain size $\langle\lambda\rangle$ is related to total surface area of the grain boundaries per unit volume S_V by the following geometrically general and assumption free general stereological equation [3,4].

$$\langle\lambda\rangle = 2/S_V \quad (2)$$

In Eq 2, S_V is also a useful parameter for monitoring evolution of microstructure during the processes such as particle coarsening and grain growth, where the driving force is reduction in the total area of the microstructural interfaces.

Estimation of S_V involves statistical sampling of the 3D microstructure by *test lines*. The number of intersections of the test lines with the surfaces of interest is counted. The *population average* value of the *number* of intersections between the test lines and the surfaces of interest per unit test line length, $\langle P_L \rangle$, is related to the total surface area per unit volume, S_V , through the following stereological equation given by Smith and Guttman [35] and Saltykov [5].

$$S_V = 2\langle P_L \rangle \quad (3)$$

Eq 3 is geometrically general; it is applicable to microstructural surfaces of any arbitrary geometry. In practice, the test lines are placed in a metallographic plane to perform the intersection counting.

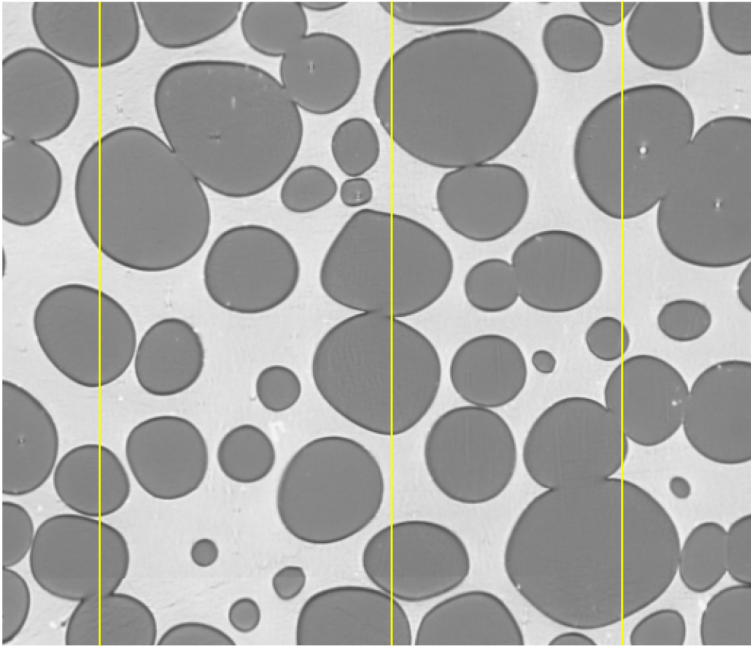
ESTIMATION OF S_V IN ISOTROPIC MICROSTRUCTURES

In an isotropic uniform random microstructure, grain boundaries/interfaces have uniform random angular orientations. Consequently, the number of intersections between the test lines and the grain boundaries/interfaces of interest does not vary systematically with the angular orientation of the test lines. Therefore, in an isotropic uniform random microstructure, it is not necessary to perform the intersection counts using test lines or metallographic planes of different angular orientations (or both); the measurements in a metallographic plane of any one convenient angular orientation using test lines of any one convenient angular orientation can yield a reliable estimate of the population average value of the intersection count $\langle P_L \rangle$. An example of P_L measurements in an isotropic microstructure is given in Fig. 5. In such microstructures, the measurements on about 50 systematic random microstructural fields in just one metallographic plane can yield a reliable estimate of S_V . Even when the measurements are performed manually, this does not take more than about half an hour. Thus, unbiased and precise stereological estimation of S_V in the isotropic microstructures is straightforward and efficient.

ESTIMATION OF S_V IN ANISOTROPIC MICROSTRUCTURES

In an anisotropic microstructure, grain boundaries/interfaces have preferred orientations. Consequently, the intersection count P_L *systematically* varies with the test

FIG. 5 Intersection counting illustrated in an isotropic microstructure of a liquid phase sintered iron nickel tungsten (Fe Ni W) alloy depicting W grains. There are three test lines overlaid on the micrograph. After dividing by magnification, the length of each test line is 0.1 mm. There are 27 intersections between the test lines and boundaries between the W grains and matrix, so $P_L = 27/0.3 = 90$ per mm.



line orientation. Fig. 6 depicts an anisotropic microstructure of a cold rolled iron specimen. In such a microstructure, a horizontal test line is expected to intersect significantly less number of grain boundaries as compared to a vertical test line of the same length. In anisotropic microstructures, as P_L varies systematically with orientation of the test lines, it is critical to perform intersection counting using test lines of numerous random angular orientations placed in numerous metallographic planes having random angular orientations to obtain a reliable unbiased estimate of the population average $\langle P_L \rangle$, which makes the procedure very arduous. Further, in such a sampling process, it is not known beforehand on how many different orientations the measurement needs to be performed on test lines and test planes for a reliable estimate of S_V because the answer depends on the nature and extent of the anisotropy. An efficient practical solution to this problem has been developed using design based stereology and a vertical section sampling technique [9] described here.

A convenient reference direction is chosen in the 3D space, which is called the vertical axis. Any plane that contains the vertical axis is called a *vertical plane* (Fig. 7 and Fig. 8). Baddeley, Gundersen, and Cruz Orive [9] have shown that the set of

FIG. 6 Anisotropic microstructure of cold rolled iron. A horizontal test line is expected to intersect grain boundaries less frequently as compared to a vertical test line of the same length.

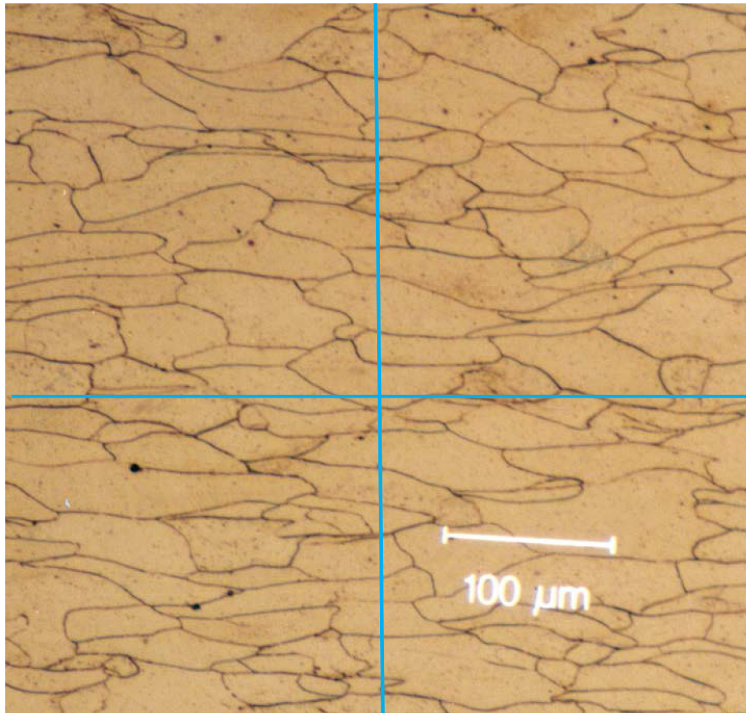


FIG. 7 Illustration of vertical axis and vertical planes [10].

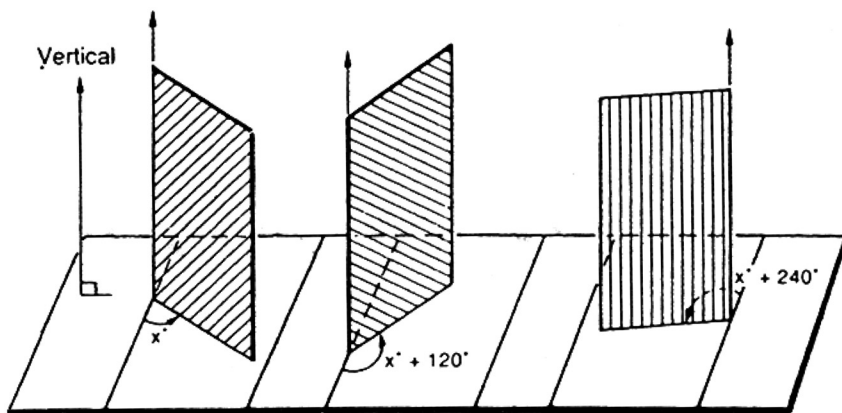
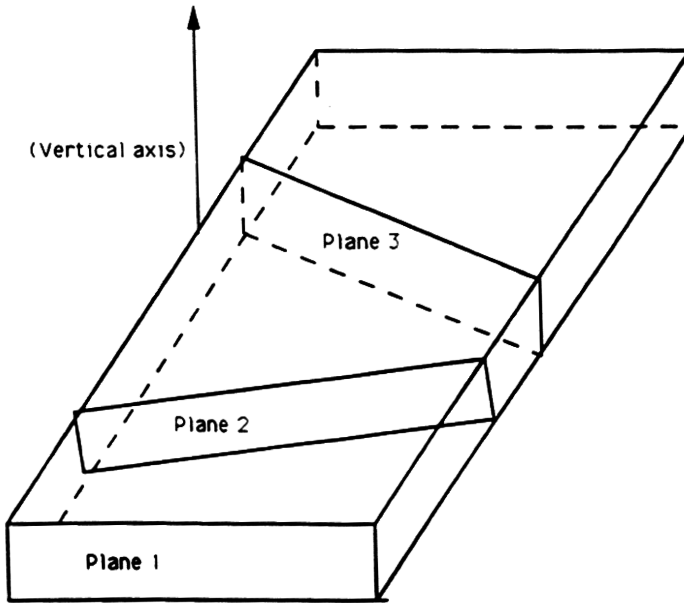


FIG. 8 Another illustration of the choice of vertical axis and corresponding vertical planes [9].



vertical planes belonging to any chosen vertical axis contains test lines of all possible angular orientations in the 3D space. Therefore, for estimation of S_V , it is sufficient to perform the intersection counts using the test lines in any one set of vertical metallographic planes (i.e., there is no need to perform the measurements in the metallographic planes that are inclined with respect to the chosen vertical axis). Nonetheless, the frequency of the angular orientations of the lines in the vertical planes is not equal to that in the 3D space; therefore, it is essential to correct for this bias when the intersection counts are performed only in the vertical planes. Baddeley, Gundersen, and Cruz Orive [9] showed that this bias can be eliminated if the intersection counts are performed using *cycloid shape* test lines (Fig. 9) that are oriented such that the cycloid minor axis is *parallel* to the vertical axis. In such a case, Eq 4 [9] can be used for an unbiased estimation of S_V .

$$S_V = 2\langle [P_L]^{cyc} \rangle \quad (4)$$

The term $\langle [P_L]^{cyc} \rangle$ is the population mean value of the number of intersections between the cycloid shape test lines and the microstructural surfaces of interest per unit of test line length when the cycloids are placed in the vertical sections with their minor axis oriented parallel to the vertical axis. Fig. 10 illustrates such intersection counting using cycloid test lines.

FIG. 9 Cycloid shape test line. AB is the minor axis of the cycloid, which should be parallel to the vertical axis as shown. Note that the length of the cycloid (i.e., test line length) is equal to two times the length of its minor axis AB [9].

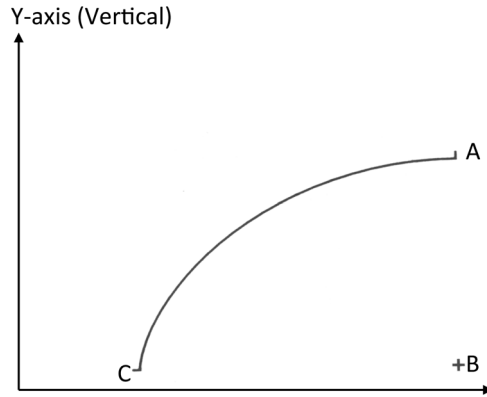
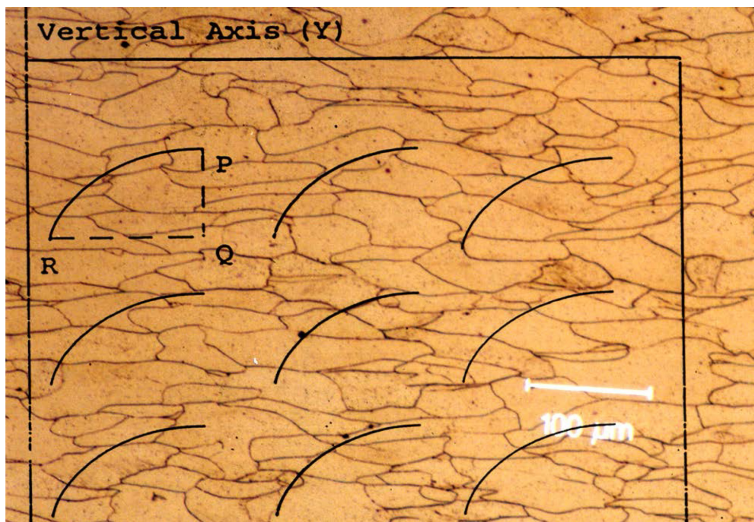


FIG. 10 A grid of nine cycloids of the same orientation is overlaid on a field of view in one plane of a trisector. The minor axis (PQ) is parallel to the vertical axis. There are a total of 45 intersections between nine cycloids and the grain boundaries. The total length of nine cycloids is 1.8 mm when normalized by magnification. Therefore, the number of intersections per unit of length is equal to 25 per mm.



The vertical sections methodology given by Baddeley, Gundersen, and Cruz Orive [9] is efficient. However, the number of vertical plane orientations that need to be sampled for a reliable estimation of S_V is not known beforehand because the answer depends on the nature and extent of the anisotropy (which is usually unknown). This problem was analyzed by Gokhale and Drury [10] using theoretical analysis and computer simulations. It has been shown that the measurements on vertical planes of (at the most) *three* orientations are sufficient for an estimation of S_V in a microstructure of any arbitrary anisotropy, if the sampling and the intersection counts are performed using a trisector probe (which consists of three vertical planes) using the following procedure [10]:

1. The vertical axis should be chosen such that it is *not* parallel to most of the surfaces of interest. For example, in a cold rolled plate or sheet, the thickness direction can serve as such a vertical axis.
2. The first vertical plane containing the chosen vertical axis is selected randomly.
3. The second and the third vertical planes of the *trisector* are chosen such that they are at an angle of 120° to the first vertical plane and to one another. This yields a set of three vertical planes that are set mutually at angle of 120° and contain a common direction that is the chosen vertical axis (Fig. 7). The three planes of the trisector need not be at the same location in the microstructure and, to optimize the efficiency, they should not be at the same location.
4. A set of cycloid shape test lines is placed on each vertical plane of the trisector, and the plane is sampled in a systematic manner. The cycloid minor axis must be parallel to the vertical axis.
5. The number of intersections between the cycloid test lines and the microstructural surfaces of interest are counted, and the average number of intersections per unit test line length are calculated (see Fig. 10 for an illustration of the counting procedure).

The total surface area per unit volume S_V can be then estimated using the following stereological equation [10].

$$S_V = 2[P_L]_3 \quad (5)$$

In Eq 5, $[P_L]_3$ is the mean value of the number of intersections of the microstructural surfaces with the cycloid shape test lines in the three trisector planes per unit of test line length. Note that Eqs 3–5 are formally identical; they differ only in the sampling procedures used for the intersection counting. The trisector technique has been used in several studies for estimation of S_V in anisotropic microstructures [11,36].

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Error and Uncertainty in Metallographic Measurement

Citation

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ABSTRACT

Technical standards are increasingly moving toward a requirement to understand and account for uncertainties in measurement. The need to quantify uncertainty, and to express it together with the measurement result, has been a focus for international standards for some time. In practice, this has led to a great deal of difficulty in some applications as to how uncertainty is assessed and how much uncertainty there really is from any part of the apparatus and process. Simultaneously, the competitive nature of business is driving a need for tighter manufacturing tolerances in order to improve technical performance or efficiencies. The consequences of errors in measurement are potentially more severe, and there is an associated need to measure with greater accuracy. New technologies can accommodate this need in many circumstances, but for a wide cross section of established businesses, it may not be financially viable to reinvent the process, and refining measurement accuracy is a preferable approach. Mathematical analysis can quantify uncertainty but does not always address how it may be improved. Understanding potential sources of error, and as importantly their relative contribution, can be essential to addressing the need to improve both reliability and reproducibility of results. The objective of this paper is to separate and examine individual sources of error in a number of typical measurements. We will focus on measurements of real microstructural features using optical microscopy and digital imaging. Areas examined will include

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metallographic preparation, microscope components and settings, camera selection and settings, software considerations, and some human factors. For each potential source of error, the relative contribution to total error will be assessed. Where possible, specific recommendations for best practice will be outlined.

Keywords

metallography, measurement, uncertainty, imaging, image analysis, microstructure, sample preparation

Introduction

The adoption of the idea of “uncertainty” has been slow to be adopted in many industries this is perhaps understandable given the length of explanation required to cover all aspects of the general guide 134 pages in the general guide to uncertainty measurement published by the Joint Committee for Guides in Metrology [1]. However, the reason for its introduction is to pursue a better understanding of the causes of differences in measurement, as well as to promote the consideration of causes of uncertainty. The basic principles are quite straightforward, and when we apply them to daily practice in the workplace as well as to our approach to development of standards we can be better positioned to understand and control the measurements that we make to reduce uncertainty and measure more precisely.

In most fields of scientific and industrial research, we are constantly striving to improve the performance of the products and the materials from which they are made. This can sometimes be achieved through breakthrough technologies, but a significant proportion of progress is through incremental improvement, driven by enhanced understanding of the performance characteristics and properties of the material. This can lead both to the refinement of key properties and to reducing margins built into the design. For example, a soda can may be designed with a certain thickness because the material and structural characteristics are sufficient to prevent the can from rupturing in use. The thickness of material in the design will include a margin of safety dependent on factors such as the variability of the material, the manufacturing process, the conditions of use, the acceptable failure rate, and so on. These factors, along with unaccounted factors that were *not* considered during design, will each have their respective uncertainties. If we can control these various contributions to uncertainty, we can reduce the required margin built into the design. In this example, the benefit might be to optimize the amount of material used to make the can and, therefore, the manufacturing cost. This can be achieved through material improvement and better control of manufacturing but it can also be achieved by improving measurement confidence.

Mathematical analysis can quantify uncertainty but does not always address how it may be improved. Understanding potential sources of error and, as importantly, their relative contribution, can be essential to addressing the need to improve both reliability and reproducibility of results.

The implementation of uncertainty into standards generally has focused on providing a method to characterize equipment used and the built in process associated with it. Merging this concept with existing standards has some challenges, typically in translating the mathematical concepts to practical and usable methods. It is not the purpose of this paper to address these challenges but instead to approach the subject from the other direction how the user can look at components of uncertainty in their own methodologies and control them more effectively.

Definitions of Error and Uncertainty

It is helpful to start off with some definitions. The following are from Joint Committee for Guides in Metrology's guide to uncertainty measurement [1]:

uncertainty (of measurement): parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

measurand: A quantity intended to be measured

We may quickly realize that the definition of the measurand is important to improving certainty. Using the soda can example, if we wish to improve the measurement of thickness we could consider all the things that may affect the measurement. Typically, we would think of the measurement device as controlling accuracy, but other factors such as the location and number of measurements as well as alignment of the measurement device and environmental factors such as temperature may also affect the measured value. If we list and control those factors, we may reduce the uncertainty in our result. In this example, we would redefine the measurand from "measure the average thickness" to a more detailed instruction to control the spacing, temperature, and alignment. By suitably controlling the definition of the measurand, and addressing sources of uncertainty, we improve accuracy, repeatability, and reproducibility.

Assessing Sources of Error in Metallography

Measurements in metallography can present a more complex challenge. The standards aim to define the measurand, but the condition of the item being measured (the polished specimen), as well as the equipment used to do the measuring, can be relatively uncontrolled and each factor can add to uncertainty.

There is a significant body of work that has examined sources of error in common metallographic measurements. For example, optically measured hardness tests such as Vickers and Knoop have been shown to be sensitive to magnification, operator perception, and specimen preparation, especially at lower loads [2–6], which has led to recommendations on minimum indent size and preparation level [7]. Another area where there has been considerable work on understanding and controlling measurement error is grain size analysis [8]. Studies such as these have been invaluable in quantifying total error and in improving measurement reproducibility in given circumstances. However, there is an opportunity in these and other

metallographic measurements to go further. If we isolate and examine individual contributing factors to measurement error, we can address each of them separately and appropriately to reduce total error, developing a better understanding of contributing factors, and their relative importance to the measurement result, and effectively reducing uncertainty and allowing improvement in both repeatability and reproducibility. As metallography involves multistep processes in the creation of the measurand, and both sample and measurement requirements can be so varied, it makes sense to address factors associated with the measuring system first—the microscope, image capture, and analysis devices.

The objective of this work is to separate and examine some of the individual sources of uncertainty that are typical of measurements in light optical microscope examination of metallographic samples. These factors would each be a potential contributor to an uncertainty calculation. We will investigate the relative impact of a variety of measurement factors individually, to characterize the most significant factors. Future work will look at these factors in different scenarios, to examine how they change with different samples, and their combined effects.

Experimental Method

To demonstrate the effect of varying a range of parameters on the microscope and imaging camera, a relatively simple sample was selected, a carbon fiber reinforced polymer (CFRP). This is a product consisting of layers of woven mats of carbon fiber encapsulated in resin. The properties of the material depend on the lay up of the fiber, the quantity of fiber relative to the resin, and the size and type of voids within the component. In the samples used, there is one readily recognizable harder phase (the fiber) surrounded by the softer polymer matrix. The effect of variables will be more readily recognized on a sample of this type.

To demonstrate effects on a more complex scenario, a tungsten carbide cobalt (WC Co) thermal spray coating has been used. This type of coating typically is applied to improve wear properties of a component. The coating is a moderately complex structure; the size and type of voids as well as the size and shape of features within the coating such as unmelted particles, WC size, phase area, oxides, interface features, and cracks are all important to quality control.

Prior to looking at the microscope and imaging parameters, we should mention some fundamental considerations for sample preparation—ensuring there is no preparation induced damage to the structure, that real structural features are retained, and ensuring that the sample is flat. These aspects will be discussed briefly because isolating these factors from our investigation of the microscope and image analysis functions is important.

Preparation Factors—Unrecovered Damage

It is well understood that the process of metallographic specimen preparation aims at each step to remove preparation damage induced in prior steps, such that the

true structure can be analyzed. However, recognizing and removing damage is not always straightforward. This may be due to complex structures in the specimen, components that are particularly sensitive to damage from the preparation process, or from scenarios where defects can be more complex, such as failure analysis or research and development, and the structure and properties may be less well known. This is far too wide a subject for this paper, but an example helps to illustrate the problem.

Fig. 1 shows porosity measurements on a WC Co spray coating at different stages of preparation [9]. The earlier stages of preparation used, which included grinding with silicon carbide paper and polishing with a 9 μm diamond on a nap less cloth, have been omitted from the graph as the surface damage makes estimation of porosity impossible. Fig. 1 shows the last three stages of preparation, interrupted at regular intervals in order to measure the apparent porosity. It can be clearly seen that using sufficient time at each stage is important to getting an accurate result. It has also been shown that the application of the preparation method can be more significant than the choice of preparation method [9].

In this example, the apparent porosity has been reduced by a factor of three in just the last three stages of the preparation route used. Fig. 2 demonstrates what a 50 % reduction in apparent porosity looks like. An operator would need to have knowledge of the material and observe the specimen during preparation in order to conclude that the final measurement was accurate.

Clearly, the amount of preparation induced damage in this sample has a significant effect on measurement results. The correct metallographic recipe depends on the material being prepared, but a good understanding of the characteristics of the material being examined, along with appropriate inspection, can help us identify good preparation.

In this sample, prior to the thermal spray process, the surface of the substrate material is roughened by grit blasting to promote mechanical adhesion. The condition of any embedded particles remaining at the interface, the clarity of

FIG. 1 Change in apparent porosity during preparation for a WC Co thermal spray coating.

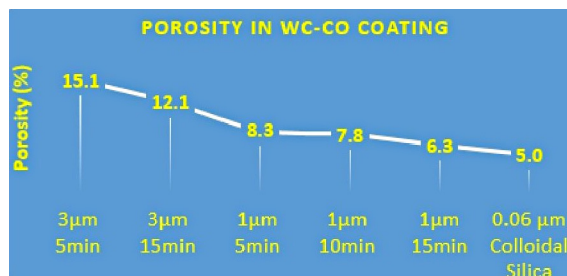
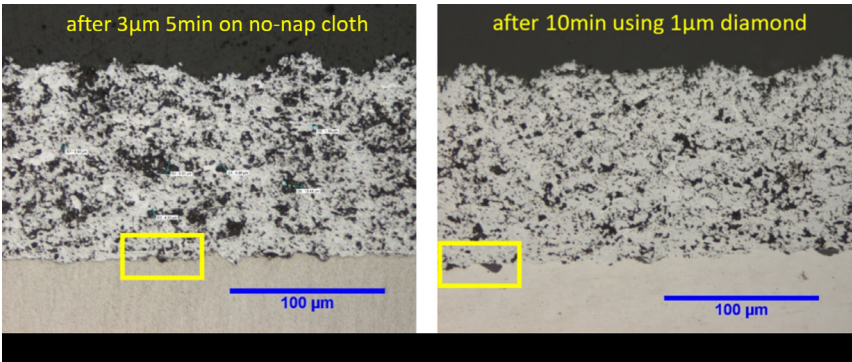


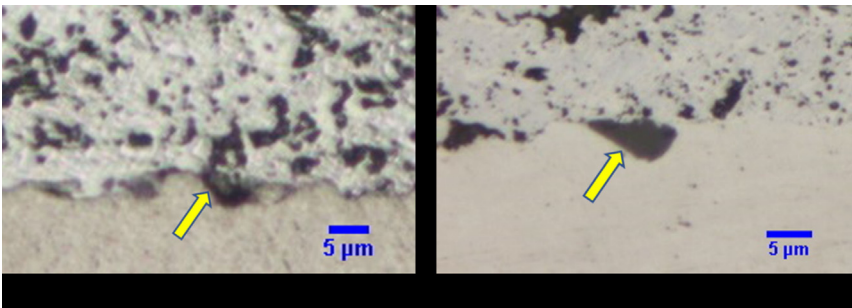
FIG. 2 WC Co samples during diamond preparation stages: (a) has 15 % porosity (b) has 8 % porosity.



microstructural features, and definition of boundaries are examples of areas we can examine to give an indication of effective preparation.

Fig. 3 shows digitally magnified areas from the images in **Fig. 2**. **Fig. 3a** shows a poorly prepared specimen with significant remaining damage. The embedded grit particle is fractured (arrow); the boundary between the coating (top) and the substrate (bottom) is unclear, indicating surface deformation. The definition of detail on the coating is also poor again due to surface deformation. Contrast this with **Fig. 3b**, showing the same type of sample boundary at the same magnification. This specimen has a clearly defined boundary between coating (top) and substrate (bottom), as well as an embedded grit particle with no evidence of damage or missing parts (arrow).

FIG. 3 Same specimen as **Fig. 2** digitally magnified to show detail. (a) Shows partially recovered damage and (b) shows greatly improved features, with no gross deformation or fracture.



Preparation Factors—Polishing Relief

A common mistake in metallography is to extend polishing times (most often on “soft” or napped cloths) to remove visible damage remaining at the end of a preparation route. Extended polishing on soft surfaces can lead to polishing relief—areas of the same specimen polish at different rates, and a height difference is generated. As light optical microscopy relies on reflection to produce contrast in the image, the sloped areas caused by polishing relief will appear darker or even as a black line. For unsupported areas and edges, this can inhibit accurate measurement. In the example in [Fig. 4](#), an overestimate of porosity size is likely due to the rounding at the edges.

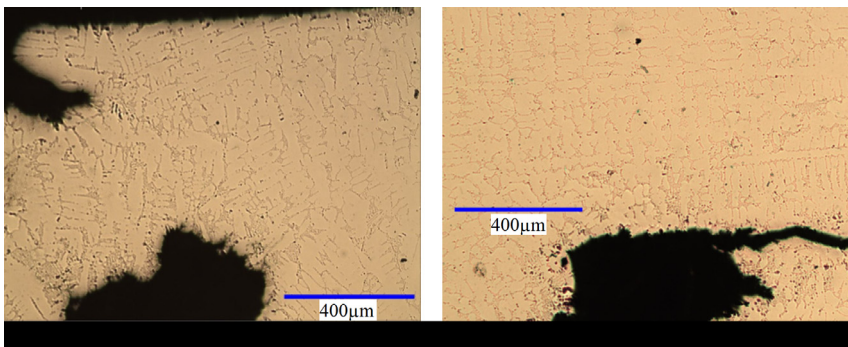
It is therefore good practice to minimize polishing time on soft surfaces, and the correct action would be to ensure that the penultimate stage of preparation:

- (i) Keeps the specimen flat, typically by using a harder/flatter preparation surface.
- (ii) Allows for a short polishing step—the difference in abrasive size should not be excessive so that remaining damage, such as scratches, can be removed in a short time—typically 1 to 2 min.

Microscope and Camera Settings

By using a single field of view on a single sample and varying one parameter at a time, we are able to isolate the effect of that parameter on a measurement. This was done for a variety of camera settings, and the percentage change in apparent fiber content for a CFRP sample was measured using grayscale based thresholding with visual confirmation of upper and lower threshold values. All tests were done with a single operator, and all conditions that were not being changed were held constant, where possible. A high quality inverted compound microscope was used, along

FIG. 4 The effect of (a) over polishing versus (b) good preparation on apparent porosity in a nickel superalloy.



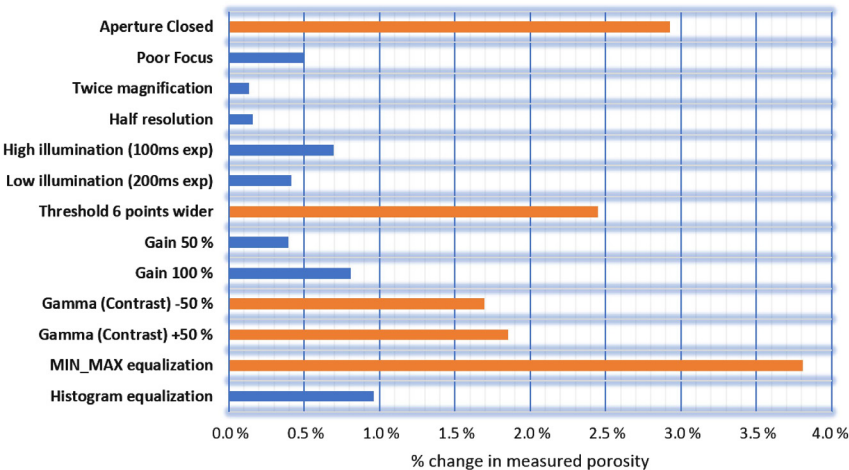
with a digital charge coupled device microscopy camera with a 0.5 in. chip. Commercial digital imaging software for metallography was used for capture, digital transformations, and image analysis. To maintain best comparability among images, the overall brightness was controlled in every case, such that the maximum pixel brightness was just below saturation.

The graph in Fig. 5 shows the overall results of each of the factors investigated expressed as absolute value percentage variance from a baseline measurement. The factors below 1 % variance from the baseline value represent variability that is not clearly distinguishable from the inherent sample variability (i.e., that found from repeated measurements on the same sample with no changes in microscope or analysis setup). Some of the factors that typically would draw more attention such as camera resolution and poor focus had a relatively small effect. Other factors that are often overlooked or used with little consideration had a much larger effect on measurements.

The samples used in this study were prepared to a high quality finish, retaining excellent flatness and free of surface damage. On a sample with no significant rounding between phases, the change of illumination does not significantly shift the point at which the boundary appears to be. However, if the sample was rounded from overpolishing, or from etching, we would expect a greater effect on measurement error. Similarly, gain and histogram equalization both effect the point at which thresholding works and would be expected to have a greater influence on measurement error when the samples are not flat.

The digital resolution was varied from a 3.1 MP captured image to a 1.6 MP captured image with the same field of view. At high magnifications, the resolution

FIG. 5 Comparative effects of microscope and imaging functions (percent change in measured porosity).



of the image is controlled primarily by optical resolution, and the reduction in digital resolution is less significant [10]. The consequence of poor digital resolution is much greater at lower optical magnifications, lower digital resolutions, or for smaller objects [6]. In this case, the out of focus image has the effect of reducing effective resolution but did not significantly change measured values. Similarly, the optical magnification in this example did not introduce a significant change in error because the change in resolution was small compared with the feature size being measured.

The largest variations (outside the 95 % confidence interval from the tests for baseline value) are represented by the factors with greater than 1 % variability from the baseline value shown in Fig. 5 and will be discussed in more detail. Looking more closely at these high impact criteria can help us to improve measurement certainty the most.

Microscope Factors—Aperture Diaphragm

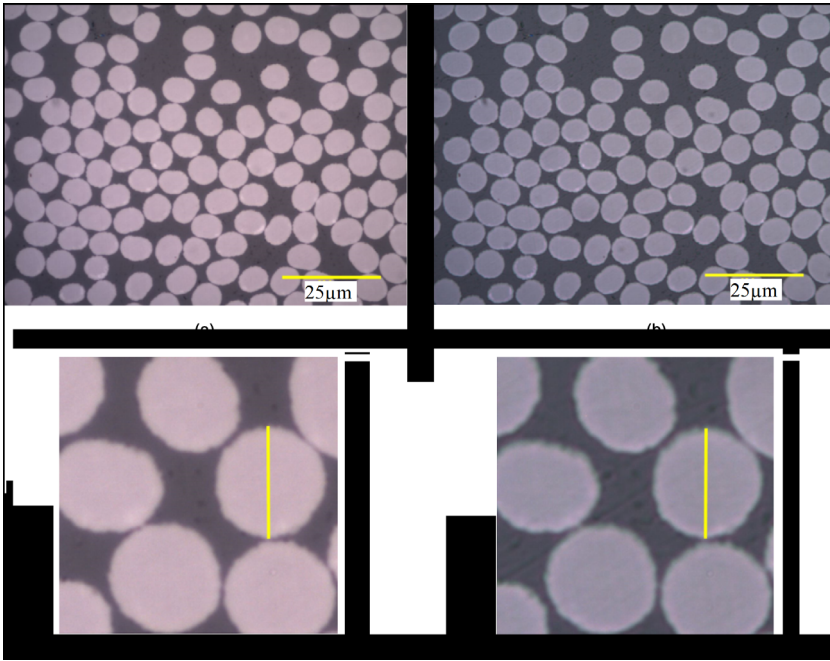
Almost all compound microscopes have an aperture diaphragm. This device modifies the size of an aperture in the light path of the microscope, and controls the amount of light passing through, as well as effecting focal depth, resolution, and contrast. Many operators are not aware of the correct use of this component, but it can have a significant effect on measurement results. When resolution is reduced, darker areas appear to “grow” and thresholding (defining the boundary between light and dark) becomes more difficult. Fig. 6 shows the same field of view on a CFRP specimen with the aperture fully open (a) and closed (b). In Fig. 6a, the overall appearance of the image is light, and we can see the individual fibers clearly. Fig. 6b looks very similar at first glance, but when we digitally enhance these images, there are very visible differences in contrast and resolution. Fine scratches from 0.3 μm alumina polishing can be seen in the polymer matrix in Fig. 6d. The interface between the fiber (white) and matrix (gray) also appears thicker and less distinct. When we take a measurement from these images (representative vertical lines shown on image), we find that the linear distance shown in Fig. 6d is 4 % shorter than the line shown in Fig. 6c even though the measurement itself appears the same. This is attributable to the difference in resolution from the change in aperture setting.

The recommendation for best practice is thankfully straightforward. The aperture diaphragm generally should be open when making measurements, to maximize optical resolution. Of course, if the sample is not well prepared and has rounding, polishing relief, or other topographic effects, we may lose the ability to focus consistently on the specimen. Other important criteria to optimize resolution are centered and even lighting as well as a good maintenance/cleaning schedule.

Image Processing Factors—Thresholding

Thresholding is a common process by which we identify different areas of an image, to enable digitized measurements. The principle is straightforward a typical

FIG. 6 CFRP specimen at 50 $\times$ objective magnification: (a) aperture open; (b) aperture closed; (c) digitally magnified area from (a); (d) digitally magnified area from (b).

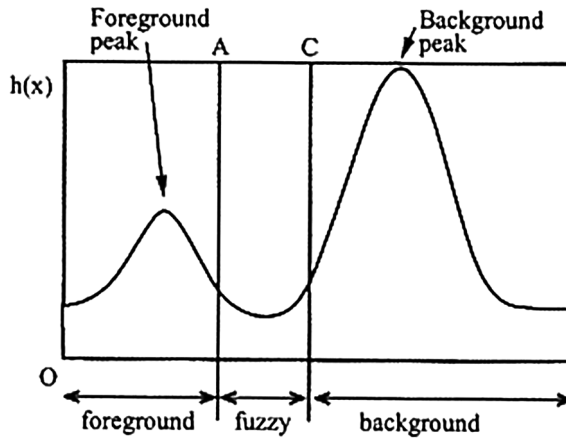


grayscale image has 256 gray levels. If we plot out a diagram of gray levels against number of pixels, we can identify peaks. When we use software to help us identify a phase under the microscope, we are setting “threshold” points to characterize the areas of the image. In the example shown in Fig. 7, two regions can be clearly defined—the region from 0 up to line A and the region from line C to maximum. The region between A and C appears fuzzy, and it may not be easy to define exactly where a *single* line separating the two peaks should be placed.

When we have poor resolution, or edge rounding, the “fuzzy” area tends to be larger, and the minimum point may be skewed, due to the uneven effects of intensity distribution. This can, therefore, give us more variability in results (greater uncertainty), as well as a shift in mean measured value (error). It is also easy to see from this example how selection of the threshold value is critical to accurate and reproducible measurement and is a major contributor to error (Fig. 5).

Image processing algorithms, and often automatic thresholding tools, can vary greatly in their success in finding this threshold point. In the CFRP example used, the “fuzzy” area was quite small because the material was well prepared with minimal edge rounding and good contrast between phases. This helps to keep the thresholding more reproducible, and automated systems are more likely to work

FIG. 7 Representative image of intensity histogram, from Devi [11].



effectively. It is also strongly recommended to maintain a consistent image brightness among specimens.

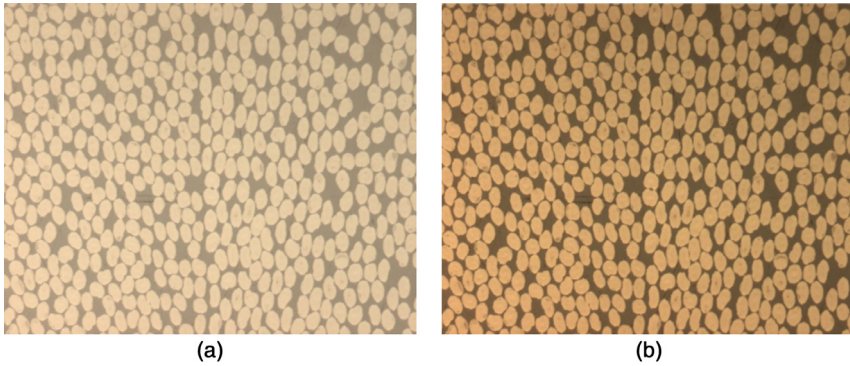
When the “fuzzy” area is large, it is tempting to use image processing to improve the contrast of the image. Image processing is not new and has been successfully used in many applications for decades. However, as technology has advanced, the use of image manipulation algorithms has become familiar—for instance, in photo editing software freely available with just about every “smart” device. There is a perception that image “enhancement” is beneficial—and, indeed, in many applications outside metallography this is the case. However, when we are taking precise measurements from images, it is important to appreciate that some of these functions may significantly change our results.

Image Processing Factors—Image Contrast

Gamma adjustment is a tool available in many image processing applications. Essentially, it modifies the distribution of intensity in the histogram. Our eyes perceive image contrast more effectively at low levels than at high, and so when we look at modified images, such as Fig. 8, we tend to “prefer” the look of image (b). In fact, this modification changes the distribution function and consequently the apparent thresholding point, and our measurement changes.

Histogram equalization is another method of modifying contrast in an image. In this case, the intent is to take an image with a poor range of contrast levels and expand it across the available range. In our example, the effect of this process was not severe—but it has just as much potential to modify measurements as the gamma adjustment. This principle is true for many types of image enhancement, and it is recommended to avoid these whenever possible.

FIG. 8 CFRP surface (a) as captured and (b) after enhancing with Gamma contrast adjustment.



Discussion and Conclusions

The microscope settings and image analysis factors examined in this work were being applied to a sample that was flat, well prepared, and presented a clear contrast between phases. This has allowed the individual effects of the variables used in the analysis to be isolated and their contribution to error in measurement to be estimated.

Error in measurement associated with each factor will depend on the sample being examined. Contrast level between phases, size of feature, the type of measurement, numerical aperture, and lighting are all examples of factors that will modify the errors discussed. The tendency to cause an undermeasurement or overmeasurement will also depend on whether a light phase (e.g., fiber in CFRP) or dark phase (e.g., porosity) is being examined. A study of combined effects is therefore heavily dependent on the specimen being examined.

For the example used, variation in measurement of up to 3.8 % was introduced by individual factors. The value of good practice in the use of the light optical microscope, and restraint in the use of digital processing, can avoid much of this potential error.

Most of the principles discussed in this paper are relatively straightforward, and it should be a simple matter to understand and control these in the laboratory. There are some key recommendations that will help minimize error and uncertainty in light optical analysis of metallographic specimens.

- Keep specimens flat throughout preparation and minimize polishing relief.
- Ensure the microscope has high quality lighting (Köhler illumination) and lenses (particularly in the numerical aperture rating).
- Optimize resolution of the microscope using good maintenance and adjusting correctly.

- Avoid changing the captured image. Create contrast with the specimen and microscope and not through digital enhancement.
- Recognize and account for sources of error where possible.
- Understand the tools and processes being used to avoid unnecessary error.

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Avoid Microindentation Hardness Testing at Low Loads!

Citation

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ABSTRACT

For many years, ASTM E384, *Standard Test Method for Microindentation Hardness of Materials*, has stated that the load range for microindentation hardness testing with both Knoop and Vickers indenters is 1 gf to 1,000 gf. But, is it realistic to consider using very low-test forces when the indents are measured with a light optical microscope? ASTM E384 also states that tests that produce a Knoop long diagonal or a Vickers mean diagonal of less than 20 μm should be avoided as the precision in measuring such small indents is poor. The standard recommends using loads no lower than 25 gf. This article shows that the Knoop test exhibits better measurement precision at loads of 200 gf and below because the long diagonal is 2.7 times greater in length than the Vickers mean diagonal length for the same specimen tested at identical loads. Knoop, however, does not produce constant hardness values as the load changes. Most microindentation hardness testers provide the user with a 10 $\times$ objective used to find the area of interest for testing and one measurement objective, with 40 $\times$ magnification being the most common. A few testers have offered 50 $\times$ or 60 $\times$ objectives to measure the indents. It is rare to find a tester with a multiple objective turret. If one can use a 100 $\times$ objective with a numerical aperture of 0.95—while obtaining adequate image contrast—indents as small as 14.7 μm in length could be measured. But the challenge is to obtain acceptable image contrast at 1,000 $\times$ magnification so that the indent tips can be clearly seen. Realistically, a minimum diagonal length of 20 μm is a more realistic limit. These

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two ASTM standards should eliminate recommending the use of test loads less than 50 gf for Vickers and less than 20 gf for Knoop.

Keywords

Knoop, Vickers, microindentation hardness test, test loads (forces), precision, reproducibility

Introduction

ASTM E384, *Standard Test Method for Microindentation Hardness of Materials* [1], has stated for many years that tests with Knoop and Vickers indenters can be performed over the load (force) range from 1 gf to 1,000 gf. But, is it wise to recommend testing at loads below 25 gf, or perhaps even below 50 gf? ASTM E92, *Standard Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials* [2], has been revised and reinstated after being withdrawn in 2010. Table 3 in the 2016 revision lists objectives, their numerical aperture, and the smallest indent that can be measured with precision, assuming that the image contrast at the highest magnifications listed is adequate to see the indent tips. The recommended minimum diagonal length that can be measured with precision is 14.5 μm using a 100 $\times$ objective with a 0.95 numerical aperture (NA). But, how many microindentation systems offer objectives greater than 40 $\times$ or 50 $\times$? For a 40 $\times$ objective, with an NA of 0.65, the minimum recommended diagonal length to measure is 21 μm . If the NA of the 40 $\times$ objective is 0.55, the minimum recommended diagonal length to measure is 25 μm . If the system has a 50 $\times$ objective, which is less common, and an NA of 0.65, the recommended minimum diagonal length to measure is still 21 μm .

Vickers tests at 25 gf or above usually give essentially the same hardness numbers if you can increase the magnification as the indents become smaller [3,4]. If the diagonal length is less than 20 μm , indirect verifications using a certified test block are not recommended in ASTM E92. ASTM E384 states that the precision in measuring indents less than 20 μm in length is poor, and the data are suspect. Tests that produce indents less than 20 μm in length should be avoided. Yet, both ASTM E92 and ASTM E384 state that the low end of the test load range is 1 gf. This is inconsistent.

Experimental Data

Vickers hardness (HV) and Knoop hardness (HK) were calculated using diagonal values for HV and HK ranges from 100 to 1,000, which are typical for steels from the purest iron available to the surface hardness of a nitrided steel using 1, 10, 15, 20, 25, and 50 gf test forces. Fig. 1 shows a plot of HV versus mean diagonal length using these six test forces for the Vickers scale, and Fig. 2 shows similar data for the HK scale. Note that even at a 50 gf load, only a tiny fraction of the lowest steel hardness range has mean Vickers diagonals greater than or equal to 20 μm .

Of course, a steel specimen tested with both Knoop and Vickers indenters will not have the exact same HK and HV number. ASTM E140, *Standard Hardness*

FIG. 1 Vickers mean diagonal length versus HV for the six test forces.

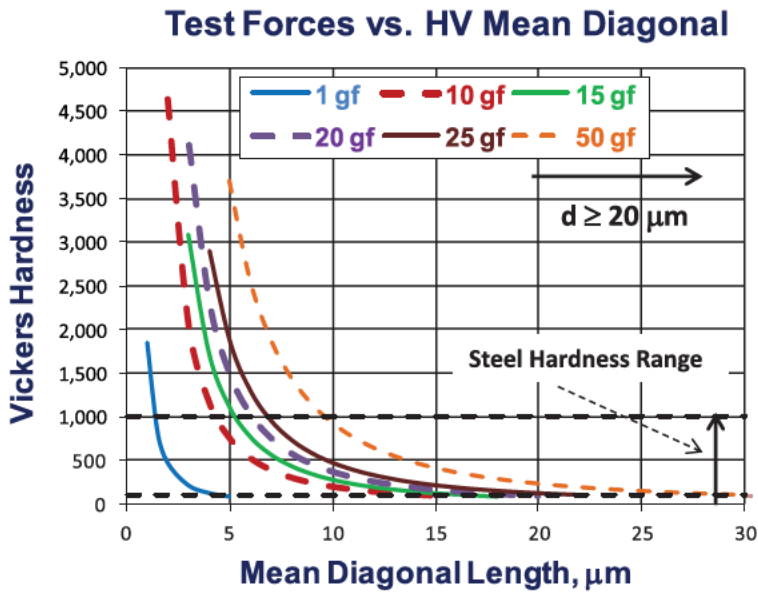
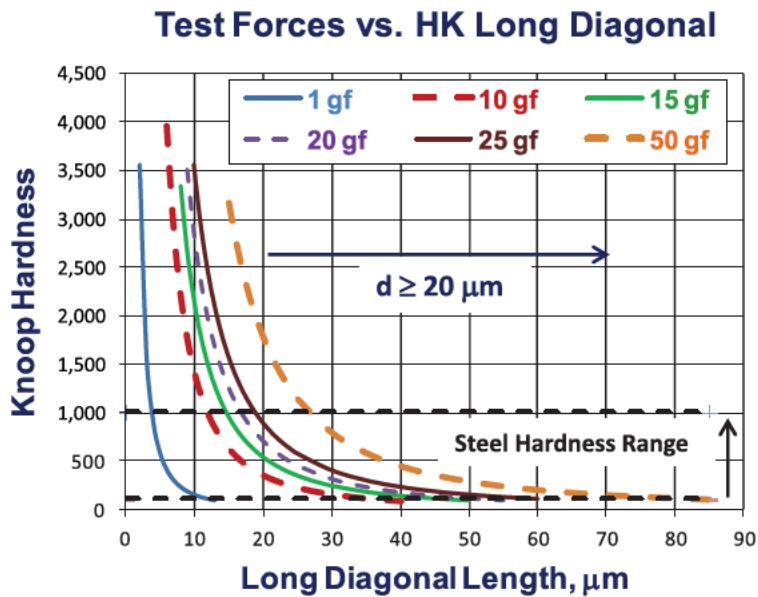


FIG. 2 Knoop long diagonal length versus HK for the six test forces.



Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness [5], shows a 20 unit difference in HK versus HV for steel with a 68 Rockwell hardness (HRC) number; and we know that HK will rise for a given specimen as the test force drops below 300 gf, with the rise increasing as the test force decreases and the specimen hardness increases. HV is higher than HK at 67 HRC and 68 HRC, while HK is greater from 20 HRC to 66 HRC. At 20 HRC, there is a 13 unit difference between the HK (251) and HV (238). From 55 HRB to 100 HRB, the HK number is also greater than the HV number. At 55 HRB, the HV is 100 while the HK is 112. At 100 HRB, the HV is 240, while the HK is 251. ASTM E140 does not state a test force for Vickers because HV is statistically constant from 25 gf to macro loads. ASTM E140 (and ASTM A370, *Standard Test Methods and Definitions for Mechanical Testing of Steel Products* [6], where the conversions were first published) states that the test force for the Knoop conversions is 500 gf and “greater” (ill defined and incorrect). So the data shown for the Knoop test using the six test forces do not take into account that the numbers will increase as the test force decreases for the same specimen. Nevertheless, the comparisons are reasonably valid.

Because the Knoop long diagonal is 2.7 times greater in length than the mean Vickers diagonal, Fig. 3, for the same test force and same hardness, Knoop diagonals greater than or equal to 20 μm cover a wider range of steel hardness than the Vickers test at all test loads, as shown in Fig. 4 and Fig. 5. Note that when using a 50 gf load, the entire steel range, from 100 HK to 1,000 HK, yielded long diagonals greater than or equal to 20 μm . When a 25 gf test load is used, steels from 100 HK to 889 HK (which covers all steels made) exhibited long diagonal lengths greater than or equal to 20 μm . When a 20 gf load is used, steels from 100 HK to 711.5 HK

FIG. 3 Relationship between the long Knoop diagonal to the mean Vickers diagonal as a function of the equivalent HRC hardness. The mean ratio is 2.724.

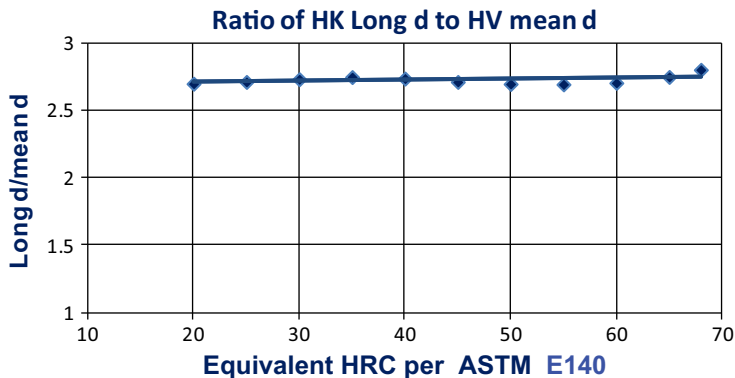


FIG. 4 Test force versus diagonal length for steels with 100 to 1,000 HV or HK.

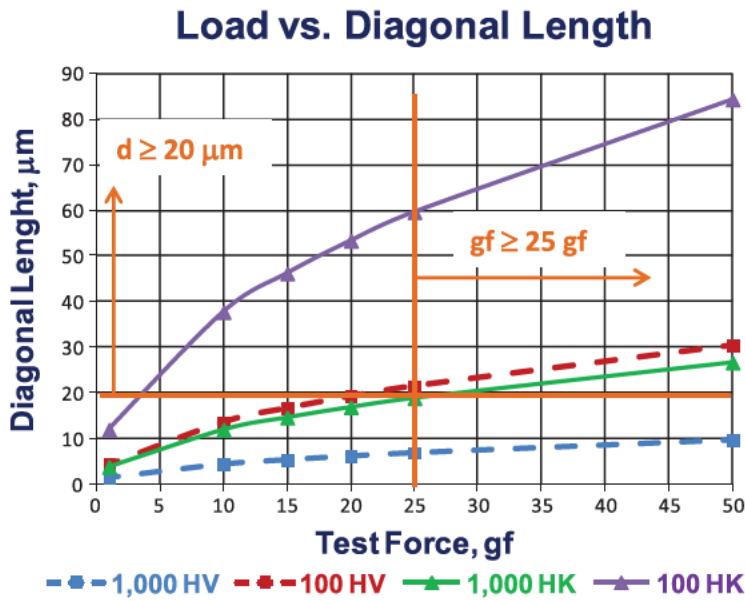
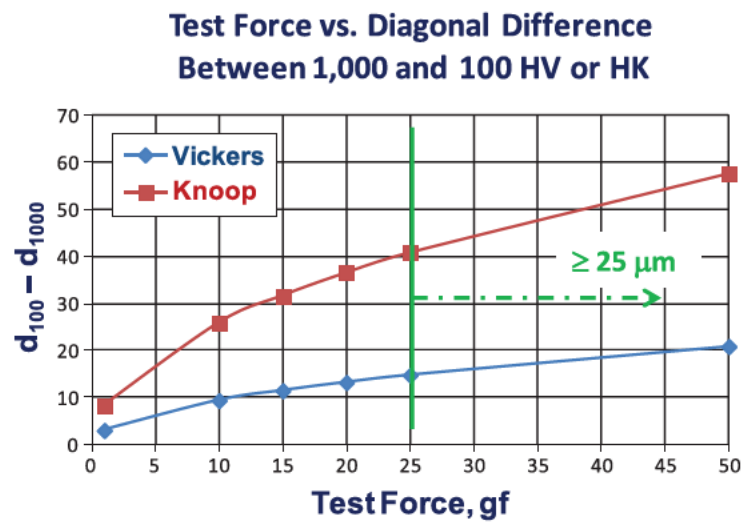


FIG. 5 Difference in diagonal lengths for 100 versus 1,000 HV or HK hardness numbers as a function of test force.



exhibited long diagonal lengths greater than or equal to 20 μm . In contrast, when a 50 gf load is used with the Vickers indenter, steels from only 100 HV to 231.8 HV will have mean diagonals greater than or equal to 20 μm , while for a 25 gf load, steels from only 100 HV to 115.9 HV will have mean diagonals greater than or equal to 20 μm . Going to higher applied forces, with a 100 gf test load, steels from 100 HV to 463.6 HV will have mean diagonals greater than or equal to 20 μm , and for a 200 gf load, steels from 100 HV to 927.2 HV will have mean diagonals greater than or equal to 20 μm in length. This seriously restricts the utilization of the Vickers test at low test loads.

Fig. 4 and **Fig. 5** show plots of the six test forces used versus the mean Vickers diagonal length or the long Knoop diagonal for the 100 to 1,000 hardness number range for steels. Again, it is obvious that the Knoop indenter has greater validity for tests in this hardness range, but there is really no value in recommending use of test forces less than 25 gf for either test, and especially not for test forces less than 20 gf. **Fig. 5** further illustrates the better sensitivity of the Knoop compared to the Vickers test at loads greater than or equal to 25 gf. For test forces above 25 gf, Knoop has a wider variation in diagonal length and thus is better able to discriminate minor hardness differences with better precision at 25 gf or 50 gf test loads than the Vickers test can. **Table 1** lists the mean diagonal length or long diagonal length for the Vickers and Knoop tests at 100 HV or HK and 1,000 HV or HK for loads of 1 gf to 50 gf.

In general, most metallographers cannot repeatedly measure indent diagonals in tests performed with a substantial time break between measurements with a precision better than about $\pm 0.5 \mu\text{m}$. So, if we calculate the HV or HK as a function of test load and diagonal length, where we determine the hardness difference between $-0.5 \mu\text{m}$ versus $+0.5 \mu\text{m}$ of a given diagonal length, we can see how this degree of imprecision in the diagonal measurement will affect the reported hardness value. **Fig. 6** shows a plot of the mean diagonal length versus the difference in HV for $-0.5 \mu\text{m}$ and $+0.5 \mu\text{m}$ as a function of test force. **Fig. 7** shows a similar plot for HK. **Fig. 6** shows a strong detrimental error in HV for mean diagonals less than 20 μm for loads less than or equal to 50 gf. In comparison, **Fig. 7** shows less imprecision at all test forces and diagonal lengths, compared to **Fig. 6** for the Vickers test, at test

TABLE 1 Range (d_{100} $d_{1,000}$) of steel hardness in HV and HK.

	1 gf	10 gf	15 gf	20 gf	25 gf	50 gf
1,000 HK	3.772	11.929	14.609	16.87	18.861	26.673
100 HK	11.929	37.72	46.199	53.346	59.643	84.348
Δd	8.157	25.791	31.59	36.476	40.782	57.675
1,000 HV	1.362	4.306	5.274	6.09	6.809	9.629
100 HV	4.306	13.618	16.678	19.258	21.53	30.45
Δd	2.944	9.312	11.404	13.168	14.721	20.812

FIG. 6 Influence of a $\pm 0.5\text{ }\mu\text{m}$ measurement imprecision in measuring the mean Vickers diagonals upon the possible variation in HV as a function of test force and mean diagonal length.

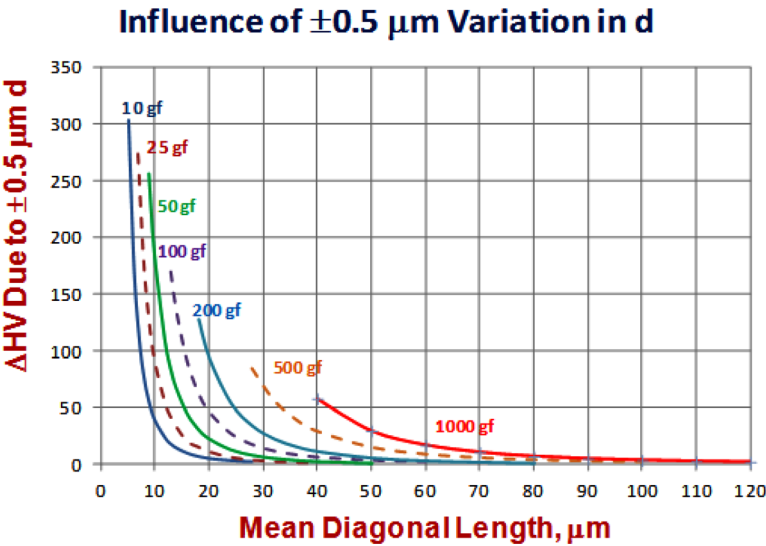
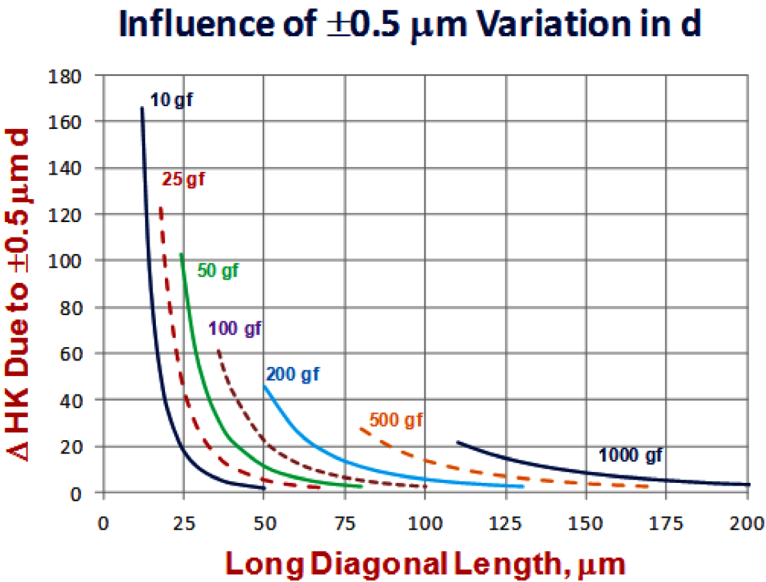


FIG. 7 Influence of a $\pm 0.5\text{ }\mu\text{m}$ measurement imprecision in measuring the long Knoop diagonal upon the possible variation in HK as a function of test force and mean diagonal length.



forces less than 100 gf. This work shows the need for correlations among HK as a function of test forces less than 500 gf so that the greater precision in measuring Knoop indents at test forces less than 100 gf can be better utilized. Presently, the only correlation between Knoop and other test scales is available only for a 500 gf (“and greater”) test force.

Conclusions

These calculations very clearly demonstrate that microindentation hardness tests at loads less than 25 gf yield indents that are too small to measure with a light optical microscope at 400 $\times$ or 500 $\times$. At 1,000 $\times$, it is possible to measure somewhat smaller indents than 20 μ m. However, at 1,000 $\times$, it is difficult to get strong image contrast in bright field illumination to see where the indent tips are, which strongly affects measurement precision. The Knoop indenter produces a long diagonal that is about 2.7 times greater than the mean Vickers diagonal for the same steel hardness and test load. This makes it possible to cover the full range from 100 HK to 1,000 HK with a 50 gf test load as the long diagonal will be greater than or equal to 20 μ m in length. For a 25 gf test load, specimens from 100 HK to 889 HK (which actually covers all steels made) will exhibit long diagonals greater than or equal to 20 μ m in length. For the Vickers test, test loads above 200 gf are required to cover the range from 100 HV to 1,000 HV, producing mean diagonals greater than or equal to 20 μ m in length.

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Using the Control Chart Approach to Evaluate Hardness Tester Performance

Citation

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ABSTRACT

The control chart data analysis approach is an ideal method to evaluate the quality of test data using a specific tester, such as a microindentation hardness tester, over a period of usage time with the same certified test block and indenter. The method described in ASTM E2554, *Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques*, was used for this work. This analysis is done by plotting a means and a standard deviation control chart of the weekly or periodic verification data obtained with a certified hardness test block at a specific test force and a specific hardness level. The method is first illustrated using a Knoop hardness test block certified at a test force of 500 gf and with a long diagonal length of 116.18 μm (527.1 HK). From this data, one can easily calculate the uncertainty of the measurements. The second example is for a 300-gf Knoop test block with a certified long diagonal length of 154.5 μm (178.8 HK). Different problems were encountered during the use of each test block, which are readily detected using the control chart method plots.

Keywords

control charts, microindentation hardness, hardness test blocks, Knoop, Vickers, Rockwell

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Introduction

The control chart method described in ASTM E2554, *Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques* [1], and discussed by Neil Ullman [2] is an ideal procedure for evaluating the performance of hardness testers as it will detect any abnormalities that may occur with usage time. The control chart concept was first developed by Walter Shewhart in 1931 to define the state of statistical control and to detect random or special problems. In 1933, ASTM Committee E1 produced *ASTM Manual on Presentation of Data*, ASTM STP15 [3]. This was supplemented in 1935 with "Supplement A Presentation $\pm$ Limits of Uncertainty of an Observed Average," the first use of the term "uncertainty" in statistical analysis of test data. Today, additional information on control charts is provided by ASTM E2587, *Standard Practice for Use of Control Charts in Statistical Process Control* [4]. Either a range chart or a standard deviation chart can be prepared. If the variations observed appear to be random, an estimate of repeatability can be calculated. A means control chart can also be prepared to detect variations among time periods. If the control charts demonstrate that statistical control is present, the uncertainty can be assumed to be approximately equal to the repeatability standard deviation.

To prepare a standard deviation control chart, list the weekly (or periodic) verification indent length values sequentially with time and calculate the standard deviation for each set of three (or five) indent measurements made on each test block for each weekly or periodic verification test. Then, plot the standard deviation of the weekly verification data, after each verification test is performed, according to the time sequence of the weekly verifications. The means control chart is prepared by plotting the mean value of each of the three (or five) indent measurements made for each weekly verification test. The three or five verification indents are made within a short time period, generally less than 10 min., not over an extended time period. Plot the mean of each weekly verification test according to the time sequence of the weekly verifications after each verification test is performed.

It is recommended that when a new machine is installed, a new indenter is utilized, or a new test block is obtained, the weekly verification be performed each day for about 20 to 25 consecutive working days. Verification results after each test should be calculated and plotted as described and illustrated as follows. This daily verification data will be used to establish the upper and lower control limits. After that period, a weekly or periodic verification procedure is adequate unless the indenter must be physically removed from the machine and replaced by another, or repaired and reinserted. After each verification test, plot the results so that any anomalous performance can be immediately detected.

The mean standard deviation is computed by dividing the sum of the 20 or 25 (or more) standard deviations of each weekly verification by the number of tests, p , as shown in Eq 1:

$$\text{Mean } s = \Sigma s/p \quad (1)$$

The upper control limit for the standard deviation values is determined as (Eq 2):

$$\text{UCLs} = B_4 * \text{Mean } s \quad (2)$$

where B_4 for three measurements is 2.568 and B_4 for five measurements is 2.089 (see Table 2 in ASTM E2554, for values of B_4 for sample sizes from two to six). The lower control limit for two to five tests is zero (see Table 2 in ASTM E2554).

The means control chart is prepared by plotting the mean value of each weekly or periodic verification test as mentioned previously. The sample means are averaged for the first 20 to 25 (or more) means of each weekly (or daily, as preferred) verification test. The grand mean of the 20 to 25 initial weekly verification means is calculated. The upper control limit (UCL) and lower control limit (LCL) for the means control chart are calculated by Eq 3 and Eq 4, respectively:

$$\text{UCL} = \text{GrandMean} + (A_3 * \text{Mean } s) \quad (3)$$

$$\text{LCL} = \text{GrandMean} - (A_3 * \text{Mean } s) \quad (4)$$

Table 2 in ASTM E2554 lists A_3 values for two to six tests; A_3 for three tests is 1.954 and A_3 for five tests is 1.427.

Example of the Method

Weekly or periodic verification test data were obtained for a Knoop hardness test block where the certified long diagonal length at 500 gf was 116.18 μm (the mean of five means of five indents; it would be better to calculate the mean of the 25 indents used to certify the test block, but this data was not available) with a standard deviation of 0.130384 (based on the five mean values of five sets of five indents). The mean HK 500 gf was 527.1. Fig. 1 shows the standard deviation control chart using five indents for each weekly verification test for the first 48 tests. After that, the test method standard was changed recommending only three indents per verification. The first 48 weekly verification tests were used to calculate a mean s and the UCL (the LCL = 0). Then, the standard deviations for the next 148 sequential weekly verification tests (three indents per test) before and 128 verification tests (also three indents per test) after the indenter was reground are shown.

Fig. 2 shows a similar plot; but this is for all of the 324 verification test results using a grand average for the standard deviation, 0.3638, which is less than for the first 48 verification test values but slightly greater than the remaining 276 verification tests using three indents per verification. At least nine verification test results exceeded the upper control limit for all 324 tests. The value of plotting the standard deviations according to the variations in the machine operation and the number of indents per verification (Fig. 1) is obvious.

The means control chart was plotted and is shown in Fig. 3a-c. The mean for the first 48 tests using five indents was 114.99 μm (538.05 HK); for the 148 tests before the regrind, the mean was 115.32 μm (534.96 HK); and, for the 128 tests

FIG. 1 Standard deviation (SD) control chart for the Knoop test block (500 gf certified diagonal of 116.18 μm) for the first 48 weekly verification tests (five indents per test) and 148 weekly verification tests (three indents per test) before and 128 weekly verification tests after the indenter was reground. The mean standard deviation of the first 48 tests was 0.5409; for the next 148, it was 0.3448; and, for the 128 after the regrind, it was 0.3312. Note that five indents per verification test yielded a larger standard deviation than three indents per verification, and the upper control limit for five indents was greater than for three indents before or after regrinding the indenter. Note also that five verification test results using three indents exceeded the upper control limits for the tests using three indents.

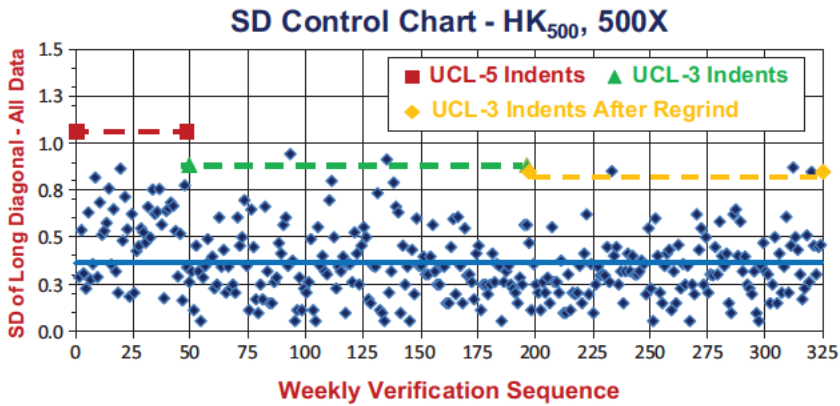


FIG. 2 Standard deviation control chart based upon all 324 verification test results. The grand mean standard deviation is 0.3638 and the UCL is 0.7698. Nine data points are above the UCL.

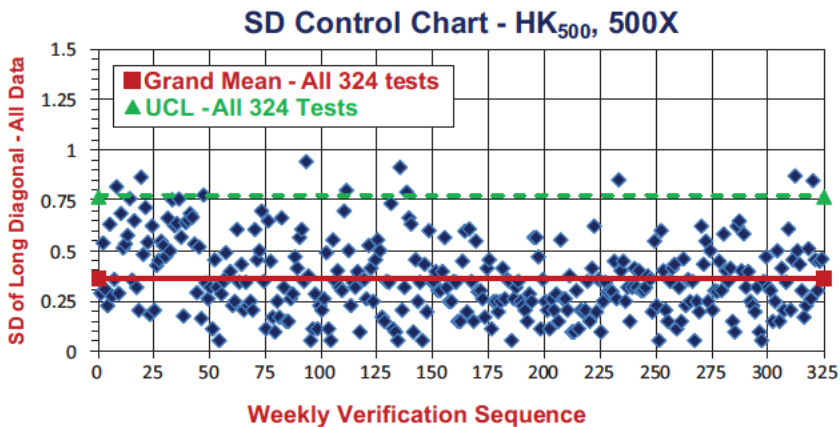
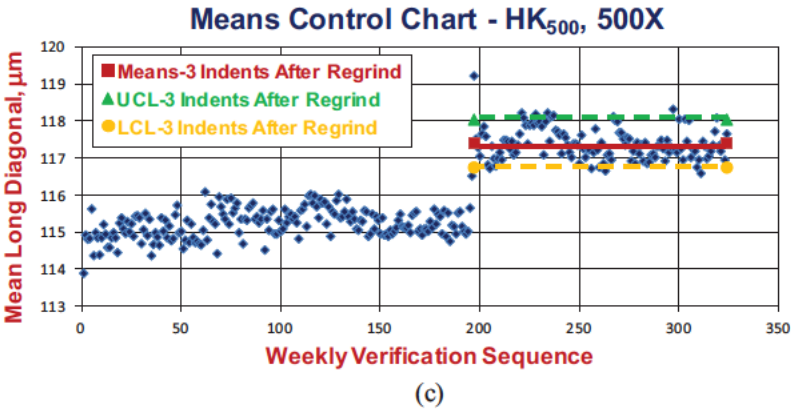
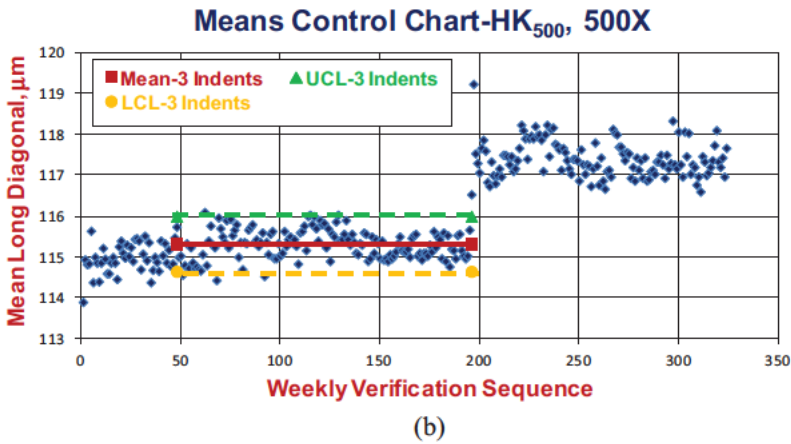
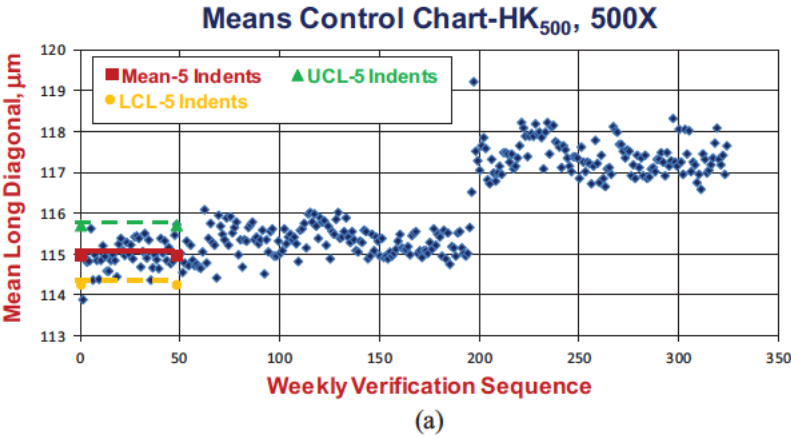


FIG. 3 Control charts for the mean indent length of the first 48 tests (a) using five indents per test; (b) for the next 148 tests using three indents per verification test; and (c) for the 128 verification tests after the indenter was reground, also using three indents per verification test.



after the regrind, the grand mean was $117.41\text{ }\mu\text{m}$ (516.13 HK). The UCL for the first 48 weekly verifications was $115.72\text{ }\mu\text{m}$ and the LCL was $114.26\text{ }\mu\text{m}$. The UCL for the 148 tests using three indents per verification was $116.0\text{ }\mu\text{m}$ and the LCL was $114.65\text{ }\mu\text{m}$. After the regrind, the UCL for the next 128 verification tests was $118.05\text{ }\mu\text{m}$ and the LCL was $116.76\text{ }\mu\text{m}$. This shows that the regrind had a pronounced influence on the test block results. The standard deviation control chart shows no difference before and after the regrind in comparison.

Note that the mean values using five indents per test, in the first 48 verification tests, yielded a slightly lower mean value and slightly lower UCL and LCL values compared to using only three indents per verification test. The data in this study show that using five indents per verification is better from a statistical viewpoint than using three indents per verification test. The last test before the regrind (No. 196) yielded a noticeably higher long diagonal measurement of $116.53\text{ }\mu\text{m}$ (523.9 HK), above the UCL for the first 48 ($115.72\text{ }\mu\text{m}$) and above the UCL value for the next 148 ($116.0\text{ }\mu\text{m}$). After the regrind, the very first verification test yielded a substantially higher value, $119.23\text{ }\mu\text{m}$ (500.5 HK), than before or after. Hence, the means control chart was very effective in showing the need for a regrind and the subsequent influence of the regrind of the indenter upon its test data.

The plots in Fig. 3 of the means control charts for the Knoop test block (500 gf certified diagonal of $116.18\text{ }\mu\text{m}$) for the mean values of the first 48 weekly verification tests (five indents per verification), then 148 verification tests (three indents per verification) before the indenter was reground, and then 128 weekly verification tests (three indents per verification) after it was reground vividly revealed the need for the regrind (Test 196) and the effect of the regrind upon the subsequent indenter test results. The grand mean of the first 48 weekly verification tests was $114.99\text{ }\mu\text{m}$ (538.05 HK); for the 148 weekly verification tests before the regrind the grand mean was $115.32\text{ }\mu\text{m}$ (534.96 HK); and for the 128 weekly verification tests after the regrind, the grand mean was $117.41\text{ }\mu\text{m}$ (516.13 HK). For the first 48 weekly verification tests, the UCL was $115.72\text{ }\mu\text{m}$ while the LCL was $114.26\text{ }\mu\text{m}$. For the next 148 verification tests the UCL was $116.0\text{ }\mu\text{m}$ and the LCL was $114.64\text{ }\mu\text{m}$ not too different. But, after the regrind, the UCL was $118.05\text{ }\mu\text{m}$ and the LCL was $116.76\text{ }\mu\text{m}$ a substantial increase. Note that the weekly verification means test data after the indenter was reground were all above the UCLs before the regrind, indicating a change in the indenter's performance.

As a follow up, Fig. 4 shows all of the mean diagonal values of the verification tests ignoring the different conditions of the verification test and the regrinding of the indenter. The mean value of all the data and the UCL and LCL of all of the tests are almost meaningless compared to the plots in Fig. 3.

Fig. 5 shows a control chart of verification data means for a different micro indentation hardness tester and a different Knoop test block where five indents were made for each verification from February 19, 1983, to October 6, 1998 (verifications were done monthly at that time per ASTM E384, *Standard Test Method for Microindentation Hardness of Materials* [5]). The certified long diagonal was

FIG. 4 Plot of the mean values of all 324 verification tests, ignoring the difference in the number of indents per verification and the regrinding of the indenter. The grand mean is 116.1 μm , the UCL is 116.62 μm , and the LCL is 115.57 μm . Most of the data before the regrind is below the LCL and almost all of the data after the regrind are above the UCL.

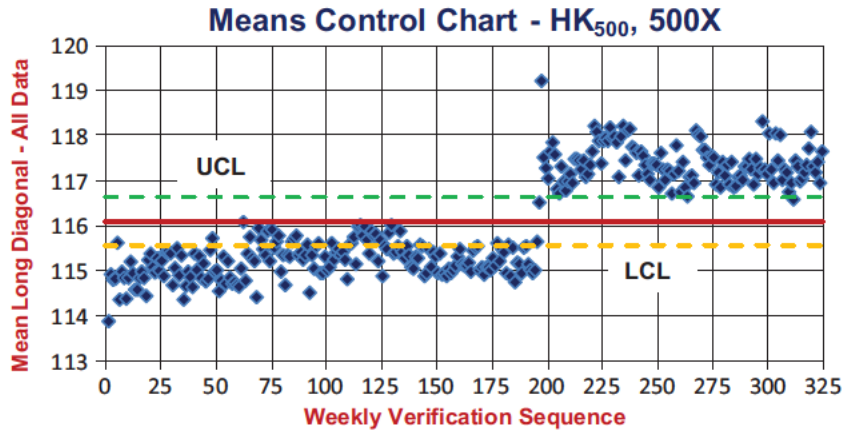
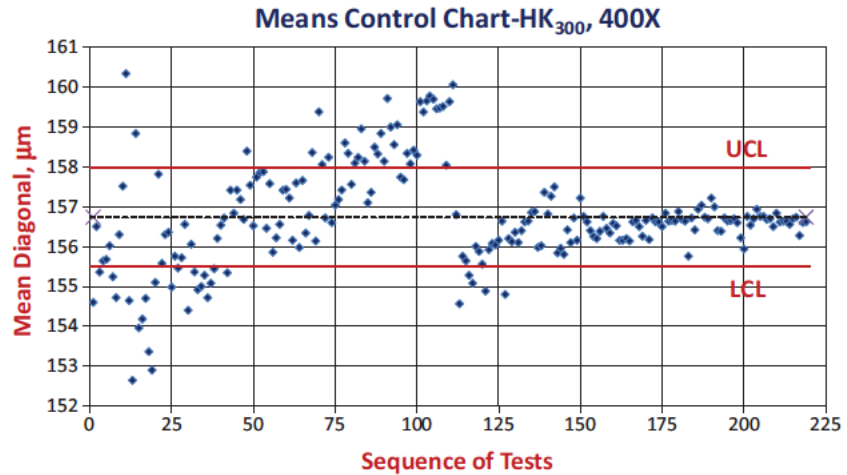


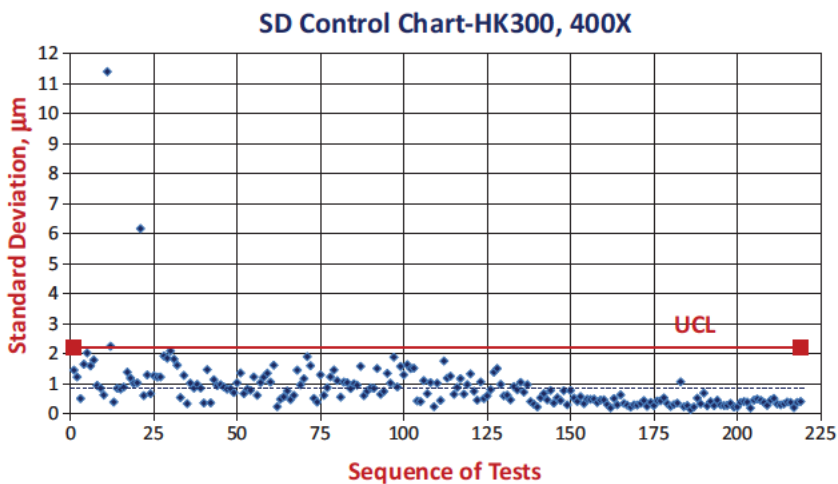
FIG. 5 Plot of the mean values of 219 verifications of a Knoop test block (using five indents per verification test). The certified long diagonal length for the block was 154.5 μm . The grand mean of the long diagonal measurements for the 219 verifications was 156.3 μm ; the UCL was 157.97 μm and the LCL was 155.5 μm .



154.5 μm using a 300 gf load and measurements at 400 $\times$ magnification. A total of 219 verification tests were done during this time period with this test block and the same tester and indenter but with four different operators. Note that the first 112 verifications exhibited a high degree of variability while the next 25 exhibited less variability, and the last 82 verifications exhibited the least variability. The first operator (Tests 1 to 112) was new to microindentation hardness testing, and the tester was brand new. The next two operators (for Tests 113 to 130 and 131 to 137) were more experienced, while the fourth operator (for Tests 138 to 219) was highly experienced. After his second test, he leveled the tester and cleaned the indenter, helping to produce more consistent results. While the certified long diagonal for the block was 154.5 μm , the grand mean for the long diagonal for the 219 verifications was 156.34 μm (1.84 μm greater). The UCL for the mean long diagonal was 157.97 μm while the LCL was 155.5 μm . Hence, the LCL was 1.0 μm above the certified mean long diagonal length.

Fig. 6 shows the standard deviation control chart for the 219 verifications made on this test block. The grand mean of the standard deviation is 0.86765 μm and the UCL is 2.228125 μm . Note the two values that are well above the UCL. Verification No. 11 had a standard deviation of 11.4089 μm while No. 21 had a standard deviation of 6.1731 μm . The mean standard deviation for the first 112 verifications was 1.199312 μm , and for the final 107 verifications it was 0.52049 μm . The maximum and minimum standard deviations for the first 112 verifications were 11.4089 μm and 0.250998 μm , respectively, and for the last 107 verifications the maximum and

FIG. 6 Plot of the standard deviation control chart of each of the 219 verification tests where the mean of all standard deviations was 0.86765 μm and the UCL was 2.2281 μm .



minimum standard deviations were 1.516575 μm and 0.151658 μm , respectively, which shows a vast difference between the two verification time periods. The change in performance coincided with a change in the operators of the hardness tester from verifications 113 to 137. These two new operators were also relatively inexperienced. But, with verifications 138 through 219, a highly skilled operator performed the verifications and the variability was much lower for these tests, as shown in Fig. 5 and Fig. 6. It must be noted that only a few of the 219 verification test results were close to the certified value of 154.5 μm . The accuracy of the block certification is questionable and may be a problem as well.

Conclusions

The control chart method defined in ASTM E2554 is a straightforward, excellent method for examining the weekly or periodic verification data to determine if the microindentation hardness tester is performing properly or being used correctly. The data suggest that the use of five indents per verification is better statistically than using three indents per verification. These test results discovered that the 196th verification test in the first example produced a statistically longer diagonal measurement than previously encountered. Of course, when the user did this test, they immediately noticed that the mean diagonals of the three indents were greater than observed before because the same person did all of these tests. But, if a variety of people were doing the verifications, this might not have been as obvious. However, if the control chart method was being used, it would be immediately obvious that the 196th verification result was unusual and required examination of the indenter tip. Consequently, the indenter was sent out and reground. But, after the regrind, the control chart method noted a distinct shift in the verification test results. From the control chart data one can easily calculate the uncertainty of the test data. The second example described showed how unskilled operators can produce erratic data while a properly trained operator using the same tester and test block for verifications 138 to 219 was able to get much better results. The precision of the certification needed to be independently verified because the certified long diagonal value was well below most of the verification results, regardless of which operator did the tests.

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Intermetallic Effects in Aluminum-To-Steel Friction Stir Weld

Citation

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ABSTRACT

In this study, the friction stir welding process was employed in lap joining of aluminum alloy 6061-T6 and dual phase 500 steel. The novelty of this study was the focus on the measurement of the intermetallic compounds present in the weld interface. Weld temperature and heat input were recorded and validated against the finite element analysis model using COMSOL Multiphysics® software. The measurement of the experimental temperature showed a good correlation with the thermal simulation. There was an increase in the intermetallic compound formed in the weld interface as the heat input and weld temperature were increased. Destructive testing showed that the bend test angle was improved with less quantity of the intermetallic produced by controlling the weld temperature. With other parameters kept constant, an increase in weld travel speed reduced the propensity of the intermetallic formation that increased the toughness of the welded joint.

Keywords

friction stir welding, intermetallics, aluminum alloy 6061, dual phase 500, mechanical test

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Introduction

Friction stir welding (FSW) is a solid phase welding process that permits a broad range of parts and geometries to be welded. The process is efficient, reliable, and involves the interaction of a nonconsumable rotating tool with the workpiece, which creates a welded joint through frictional heating and plastic deformation at temperatures below the melting point of the alloy being joined [1–3]. FSW has therefore become a unique technique that is well suited for joining many hard to weld metals, especially high strength aluminum alloys and some dissimilar metals. The lower peak temperature is an advantage, especially while joining dissimilar metals and alloys, as it limits the brittle intermetallic growth [4–6]. In addition, distortion and residual stress that cause weld failures and reduction in mechanical properties are reduced in FSW because there is no melting of the base material. FSW of dissimilar alloys, particularly aluminum (Al), has several benefits, which include fewer intermetallics precipitated at the joint interface. However, FSW of dissimilar metals (Al and steel) can be challenging because a higher concentration of iron (Fe) allows for more favorable conditions for intermetallic formation at lower temperatures. While there has been some significant work on dissimilar welds, most studies on FSW of aluminum and steel lack a detailed report on the detrimental effect of intermetallic precipitates at the joint interface. In the literature [7–10], most researchers only refer to the intermetallic precipitates as deleterious and unpredictable compounds that develop in an unusual manner in the weld matrix.

Intermetallics are ceramic phases that form in alloys. These intermetallics are hard and brittle compounds that tend to form at the weld interface of dissimilar metals. The intermetallics are closely related to the weld joint strength and fracture behavior, which is of high importance, especially in the automotive industry. Welds that have a high volume of intermetallics would be brittle with extreme hardness value and have little or no material toughness, an important property for energy absorption of a member in automobiles. At the weld interface, depending on the material mixing, a single layer or mix stir zone of intermetallics can form. The fracture toughness of the weld tends to decrease with the mix zone of an irregular layer of intermetallics, which allows for growth of intermetallics [5]. But, there is a lack of adequate destructive testing of the weld reported in the literature that describes the detrimental effect of the high volume of intermetallics on the material toughness. These intermetallics are undesirable compounds that promote brittle failure and corrosion susceptibility in the weld.

The distribution of intermetallics follows the tool rotation and material flow and thus is difficult to quantify. As the tool rotates and traverses at a preset rotational speed (rpm) and travel speed (mm/min), frictional heat is generated that brings the material to plasticization. The plasticized material is extruded and transported from the advancing to the retreating side of the tool. Depending on the heat and weld temperature (a function of downforce, rotation, and travel speed), intermetallics of different sizes and thicknesses are formed. During the material flow,

these intermetallics swirl around; hence, they are very difficult to quantify. This combination of factors has placed a huge constraint on many researchers due to the complexity involved in mapping out the area of intermetallics. The optimization of process parameters for weld temperature and heat input in order to provide a proper material mixing via tool design also requires much attention to improve productivity.

As a result, the present study was intended to fill some holes in the knowledge base. The driving force for this study was that automotive manufacturers currently are interested in improving the fuel economy and performance of automobiles [10]. As a result, reducing the weight of vehicles becomes an area of interest. Lightweight metals such as Al alloys or combinations of an Al alloy and a steel alloy to replace the heavier steel alloy parts in the structural members of the vehicles is beneficial; however, there are problems associated with dissimilar joining processes because the low solubility of iron in aluminum causes formation of intermetallic compounds at the weld interface, which significantly affects the joint performance. In the automotive industry, some structural parts operate under different service conditions. Therefore, dissimilar metal welded joints are required to fulfill some structural requirements.

In the automotive and in other industries, several methods are being implemented to join aluminum with steel [11,13]. This includes the use of adhesive bonding, mechanical riveting of parts, clinching, metal inert gas brazing, and a hybrid method that involves the combination of any two joining processes. Although adhesive joining is still in use, the strength of the joints produced is not as high when compared to FSW. These adhesives are used in conjunction with spot welding to improve crash safety performance and assembly time. However, the technique does not provide adequate strength when compared to FSW. The use of rivets and flow drill screws in automobiles (such as at a Mercedes Benz® plant) provides a joint with high strength, although the screws can also cause damage to the material to be joined. Riveting increases the static and fatigue strength of the joints [11]. However, the cost and suitability of the rivets can be a challenge. This also adds extra weight to the material being joined. A self piercing rivet is popularly used in the automotive industry, involving the use of a punch and die. The material is clamped, and the rivet is inserted using the tool. The FSW process, a relatively new technology, produces low weld distortion and greater weld strength when compared to the fusion welding process, adhesive bonding, and riveting method.

Welding aluminum and steel has proven difficult with conventional fusion welding processes because of the differences in thermal and mechanical properties (a 900°C difference in the melting point). Additionally, the brittle intermetallic formation that deteriorates the mechanical properties of the joint is highly inevitable while joining these two dissimilar metals. It is expected that FSW, which is a solid state process, will reduce the formation of intermetallic compounds by adequately controlling the process parameters, thereby improving mechanical properties. Several efforts have been geared toward achieving FSW of aluminum and steel

(both lap and butt); however, the formation of aluminum and iron rich intermetallic compounds at the interface has always been the case. A survey carried out by Movahedi et al. [4], reports the effect of annealing treatment on the joint strength of the Al 5083 and St 12 (cold rolled steel sheet) friction stir lap weld. The observation made from their study includes an increase in joint strength at 350°C compared to 300°C. The increased temperature was known to potentially cause intermetallic growth. However, a thin layer of the intermetallic enhanced the strength of the welded joint. Chen and Yazdanian [5] also conducted a study of microstructures in the interface region and the strength of friction stir lap of Al to steel and Al to titanium (Ti) welds (aluminum alloy [AA] 6060 T5, mild steel and Ti 6Al 4V). In their study, the intermetallic formation, by penetrating into the steel, was concluded to improve joint strength; however, forming a single layer instead of a mixed stir zone gave an increase in fracture strength.

Additionally, Kimapong and Watanabe [6] reported that intermetallic compounds were not observed at the interface between the steel and aluminum alloy. However, some intermetallic compounds were seen in the upper region of the weld where the temperature was higher due to the additional heat generated by the rotating tool shoulder, which caused weld deterioration. Kimapong and Watanabe [10] also experimented with joining Al 5083 and SS400 martensitic stainless steels, using a lap joint configuration in FSW with aluminum on top. The variation of welding parameters was found to influence the joint properties by decreasing the shear load [15] as a result of the intermetallic formation at the interface. Patterson [9] also conducted a study on the microstructural characterization of the FSW Al steel joints (6061 T6 aluminum armor plate alloy and a homogeneous armor plate steel alloy). While the study provided microstructural and mechanical analysis, there was no measurement of intermetallic thickness and volume fraction to explain the increase or decrease in weld strength and fracture behavior of the welded samples. In the study conducted by Mena et al. [4], an innovative related process was employed in the characterization of aluminum to steel welds (Al 6061 and dual phase [DP] 600). The aluminum sheet melted at the interface, thereby controlling the intermetallic formation to 2 μm to 3 μm at a higher travel speed of 700 mm/min and a constant 2,000 rpm. They also added that the intermetallic formation was dependent on the thermomechanical cycle. Therefore, a higher travel speed will produce a shorter cycle, thereby reducing the thickness of the intermetallic layer. Shen et al. [8] investigated the role of welding parameters on the interfacial bonding in the dissimilar FSW of steel and aluminum (AA 5754 to DP 600). Observations made from this weld include plastic deformation of the steel as a result of the low travel speed. It allowed weld softening and extrusion of steel into the aluminum matrix, which assisted in increasing the weld strength. The mixture of aluminum and iron was determined at the weld interface with $\text{Fe}_4\text{Al}_{13}$ phases formed. Their work suggested an iron rich intermetallic, after observing many steel fragment distributions in the interface between aluminum and steel.

Therefore, the novelty of this work addresses the gaps found in the literature. This includes the advancement of the existing knowledge in the FSW of aluminum and steel by providing an evaluation of the volume fraction of intermetallics at the aluminum and steel weld interface and correlation with the weld mechanical strength.

Objectives

The objective of this project was to develop a process and model to link intermetallic growth to weld heat input and mechanical performance. The driving force for this study was that automotive manufacturers currently are interested in improving the fuel economy and performance of the automobiles. Finite element analysis (FEA) modeling was employed to calculate the pin temperature using surface temperature measurements, and the volume fraction of the Al Fe intermetallics was to be related to the weld properties.

Methodology

For this study, a lap joint configuration with aluminum on the top and steel underneath was considered, and this allowed the use of a combination of a separate tool steel shoulder and tungsten rhenium tool pin. AA 6061 T6, with the dimensions 200 mm by 100 mm by 2.2 mm, was considered for this study. [Table 1](#) and [Table 2](#) highlight the alloy composition for the aluminum and steel, respectively.

The T6 condition is age hardened material [11] that is widely used in automotive and marine industries. The dimension of the steel is 200 mm by 100 mm by 1.6 mm. Since the part of the tool that plunges into the steel was the tool pin, 75 wt.% of tungsten and 25 wt.% of rhenium were chosen. The H13 tool steel capable of welding and plasticizing aluminum was used as the tool shoulder because it only makes contact with the aluminum top sheet. As a result, a fixed tool shoulder was employed with varying tool geometries for material mixing and improved

TABLE 1 AA 6061 T6 composition.

Element	Mg	Si	Fe	Cu	Cr	Zn	Ti	Mn	Others	Al
% Weight	1.00	0.6	0.35	0.275	0.195	0.125	0.075	0.075	0.075	97.23

Note: Mg magnesium; Si silicon; Cu copper; Cr chromium; Zn zinc; Mn manganese.

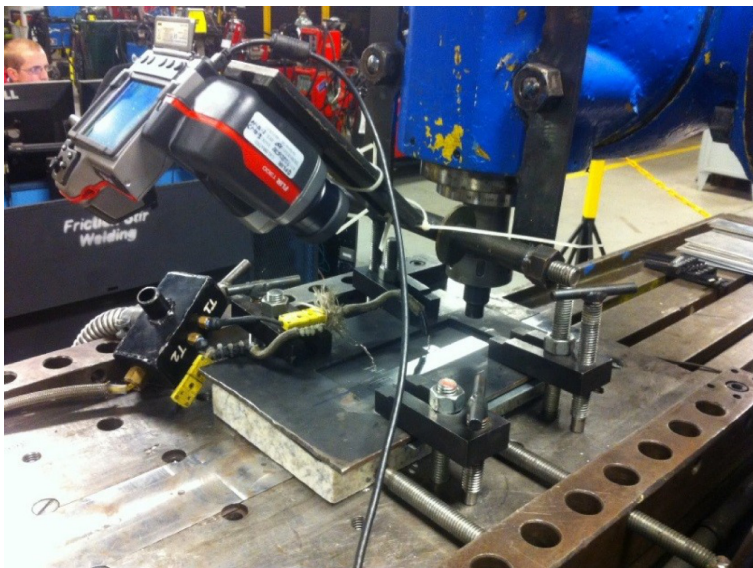
TABLE 2 DP 500 composition.

Element	C	Si	Mn	Al	P	S	Ti+Nb	Fe
≤ % Weight	0.10	0.50	1.00	0.015	0.025	0.020	0.10	Bal

Note: DP 500 is defined by the mechanical properties not composition; the values are approximate. C carbon; P phosphorus; S sulfur; Nb niobium.

dynamic to static volume ratio. Typically, surface temperatures were recorded using a thermal camera and thermocouple. Owing to the difference in the melting points of aluminum and steel, weld temperature monitoring assisted in obtaining a process parameter combination that prevented the melting of aluminum. To verify the actual temperatures, K type thermocouples were implanted at the interface between aluminum and steel. This was achieved by drilling a hole through the aluminum sheet at a given distance of 10 mm from the edge of the tool shoulder (Fig. 1). A T300 thermal camera (Flir Systems, Wilsonville, OR) was calibrated using a Gleeble[®] machine (Dynamic Systems, Poestenkill, NY) to ensure that an accurate temperature reading was measured at all times. The calibration was achieved by contact method, with thermocouples at a controlled temperature, and a noncontact method was used to find the camera emissivity. The camera was mounted and focused at the weld with initial parameters set, including room temperature, reflectivity, emissivity, and temperature range (200°C to 1200°C). The thermal camera and thermocouple temperature readings from each weld were recorded primarily when the travel speed and revolutions per minute were varied. Before welding started, the top surface of the aluminum sheet, excluding the weld zone (tool path), was lightly sprayed with flat black paint and allowed to dry completely to allow consistent optical temperature measurement. Before spraying with the flat black paint, the tool path was covered, thereby preventing the paint from being mixed into the weld. Fig. 1 is the setup adopted that incorporates the thermal camera for surface

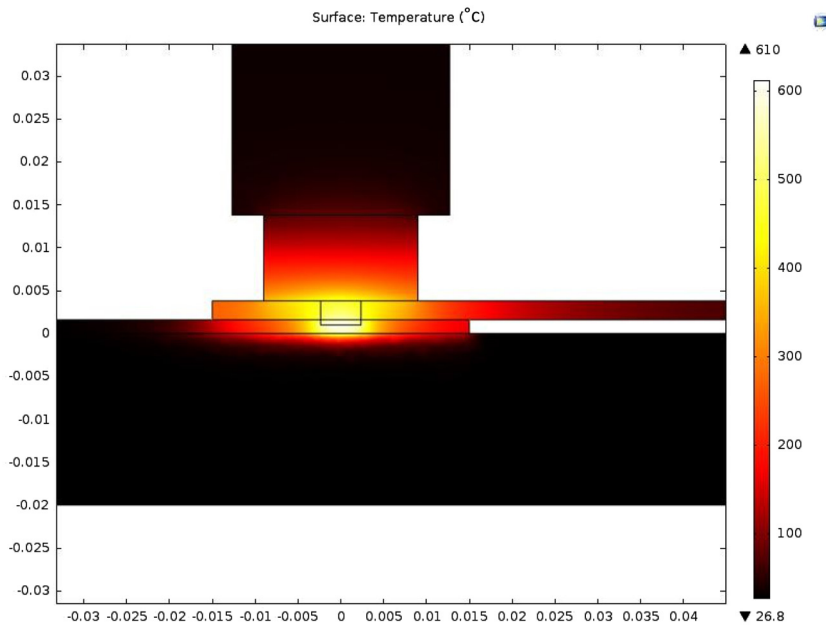
FIG. 1 Experimental setup.



temperature reading as the tool traverses to provide a correlation with the intermetallic volume fraction in the weld.

The prediction and validation of the temperature data was done with a steady state FEA model using Multiphysics[®] software (Svante Littmarch and Farhad Saeidi, Royal Institute of Technology, Stockholm, Sweden). The approach adopted for the COMSOL Multiphysics model is a moving coordinate system fixed at the tool axis, thereby making the heat transfer problem a stationary convection conduction. For simplification, the assumption for coordinate transformation is that the plates are infinitely long, which does not account for plate edges. Also, the model does not include mass flow. After the model was built, heat transfer in solid was applied to the model, considering the inflow, outflow, and surface to ambient radiation. For each heat transfer condition, boundaries and domains were carefully selected before computing the model. The boundaries include tool (shoulder and pin), aluminum, and steel, backing granite, heat flux, and ambient air. Material properties were added for aluminum, dual phase steel, tool steel, and the tungsten rhenium tool pin for the model to be solved. Free tetrahedral meshing was done to break the model into smaller nodes and for faster convergence. The model was computed and post processed for surface temperature, pin temperature, and weld heat input (Fig. 2). With a cutline at 10 mm from the tool shoulder, weld surface temperatures were measured. Aluminum and steel were cut to measure into

FIG. 2 COMSOL surface temperature plot of weld (1,000 rpm and 150 mm/min).



100 mm by 200 mm sized samples. With the aluminum on top and steel at the bottom, an overlap of 40 mm was used. The high melting temperature of the tungsten rhenium tool pin made it capable of plunging into the lower melting temperature dual phase 500 steel, while the steel tool shoulder generated heat from friction with the aluminum sheet placed on the steel.

In order to optimize weld quality and equally have a lesser amount of the intermetallic, the initial process parameters (rpm and travel speed) were adjusted by having two rpm settings while the travel speed was varied from 250 mm/min to 437 mm/min. Table 3 describes the test conditions adopted for the experiment.

The hypothesis was that increasing the travel speed enhances the productivity by reducing the process time, at the same time minimizing the chance of intermetallic formation. By increasing the travel speed of the tool, the process time was reduced, which is an advantage from an industrial viewpoint for profit maximization as a longer time increases production cost. The process parameters were also adjusted to promote material mixing between aluminum and steel for improved strength. Heat input in any welding process is a measure of energy per unit length (kJ/in). The calculation of weld heat input was carried out using COMSOL Multi physics after the weld temperature had been validated. Heat input calculation in FSW is limited to the area under the tool shoulder and pin. Therefore, the power generated by the shoulder was approximated by integrating the pressure underneath the tool shoulder and multiplied by the coefficient of kinetic friction across a velocity vector field on the tool shoulder. Eq 1 illustrates the method of heat input calculation where p_s is the power generated by the tool shoulder, $\frac{F_n}{A_s}$ is the pressure, μ_k is the kinetic friction, $v(x, y)$ is velocity field, and A is the area under the tool.

$$p_s = \int \frac{F_n}{A_s} \mu_k v(x, y) dA \quad (1)$$

Assuming that the surrounding material around the pin was in a plastic state, power generated by the pin was therefore calculated by coupling the coefficient of friction with the yield stress of the material for a total heat input of the tool pin.

TABLE 3 Experimental test conditions.

Run Order	Tool Rotational Speed (rpm)	Travel Speed (mm/min)
1	1,000	150
2	1,000	250
3	1,000	350
4	1,000	437
5	1,250	437
6	1,250	312
7	1,250	375
8	1,000	100

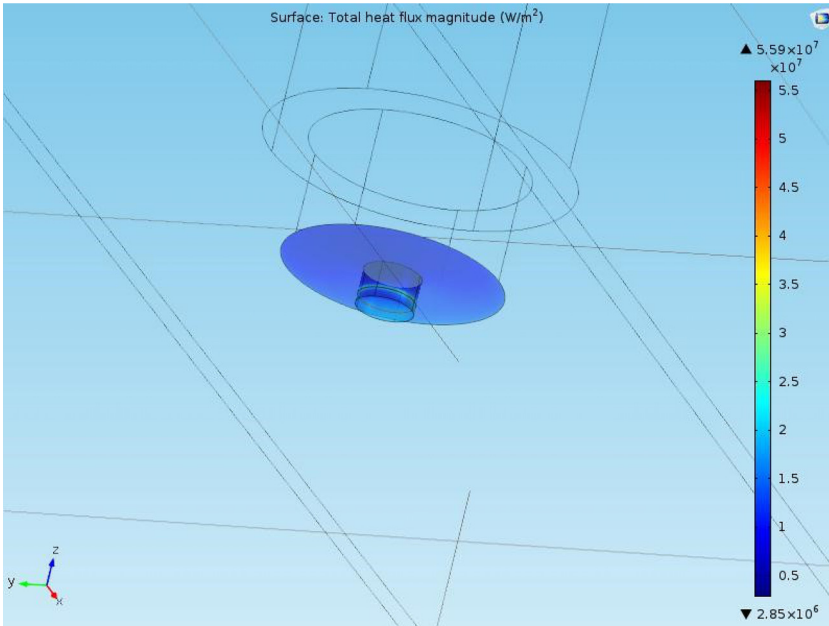
Eq 2 illustrates the calculation method adopted as guided by the COMSOL development team; p_p , σ_y , ω , T , and r_p represent the power generated by the tool pin, yield strength, angular velocity, temperature, and tool pin radius, respectively. The yield strength for each material was calculated separately as a function of temperature based on empirical data.

$$p_p = \int \frac{\mu}{\sqrt{3(1 + \mu^2)}} (r_p \omega) \sigma_y(T(x, y)) dA \quad (2)$$

At varied process parameters, the heat input was measured, and the values obtained were correlated with the intermetallic volume fraction present in the respective welds. Using COMSOL Multiphysics, surface integration was performed on the heat flux magnitude produced under the tool shoulder and tool pin (Fig. 3). The integration gave power (Watts), which was then divided by the travel speed (Eq 3) and heat input was then obtained. This method of extrapolating heat input is a novel approach of calculating heat input from the frictional heat generated when compared to integrating the area under the temperature time plot obtained from the thermocouple.

$$\text{Heat input (kJ/in)} = \frac{\text{Power (W)}}{\text{Travel speed (in/sec)}} \quad (3)$$

FIG. 3 Heat flux magnitude under the tool shoulder using COMSOL.



Determination of the intermetallic volume fraction at the weld interface between aluminum and steel was made possible by measuring the cross section area of the weld interface and the intermetallic area. The intermetallics are in a swirl shape that dispersed into the weld matrix; as a result, it poses some difficulties while measuring its quantity. In order to measure the intermetallic region, scanning electron microscope/energy dispersive spectroscopy (SEM/EDS) was used as a method to identify the presence and location of the intermetallics in the weld matrix. The images of the weld interface were taken using a backscattered electron detector from which the weld interface cross section and intermetallic areas were measured using imaging software (Motic, Hong Kong). It is noteworthy that two separate images at the same magnification were taken and combined to have a complete weld nugget with good contrast selection and visibility of the intermetallics in the matrix. With the Motic imaging software, the imported weld images with intermetallics were identified and analyzed. The Motic software tool is capable of measuring area and length of the irregular shape of the intermetallics and weld cross section. The formed intermetallic compounds were marked on the assembled micrographs, and the area was measured. The percentage of the intermetallic compound was calculated by dividing the intermetallic area by the weld interface cross sectional area. It is important to mention that this method only gives an approximation of the intermetallics' volume fraction.

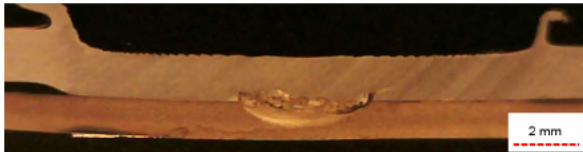
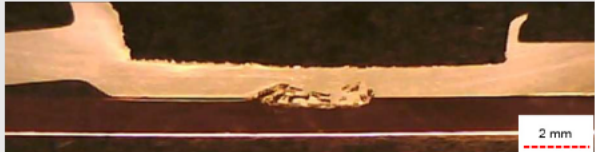
Results and Discussion

The optimized welds were made with an increasing travel speed at a fixed rotational speed (1,000 rpm). The final welds' process window also includes 1,250 rpm for improved weld strength. [Table 4](#) presents macrographs of the successful welds using 1,000 rpm and 1,250 rpm.

Micrographs of welds (1, 3, and 5) are illustrated in [Figs. 4–6](#). The micrographs revealed material mixing and dispersion of steel pieces into the matrix. Under the weld interface ([Fig. 4a](#) and [f](#)), there was a dynamic recrystallization because the steel underwent heavy plastic deformation. At a 250 mm/min travel speed, a successful weld was obtained, although the quantification of the intermetallic would assist in finding a correlation with the weld temperature (a function of rpm and travel speed).

In [Fig. 5](#), there was also mixing between steel and aluminum. A hook formation was observed on both sides of the welds ([Fig. 5c](#) and [e](#)), which apparently serves as an interlock for increased weld strength. Grain refinement was also observed in the steel side of the weld ([Fig. 5d–f](#)). The size of the grains in the weld nugget was smaller than the base metal as a result of the decrease in the deformation rate by the tool pin in the thermomechanically affected zone and heat affected zone. Steel fragments were broken into the weld nugget, similar to the observations made by Shen et al. [8] and Jiang and Kovacevic [14]. It should be noted that there was no visible void observed in the weld nugget. A similar weld micrograph was obtained by Shen et al. [8] with a combination of 1,800 rpm and 45 mm/min.

TABLE 4 Welds macrograph for further analysis.

Parameters	Macrograph
1,000 rpm/250 mm/min	
1,000 rpm/350 mm/min	
1,000 rpm/437 mm/min	
1,250 rpm/375 mm/min	
1,250 rpm/437 mm/min	

A combination of 1,250 rpm and 437 mm/min process parameters was used to enhance material softening for tool pin penetration. There was an increase in the amount of steel extrusion, particularly on the advancing and retreating sides of the weld (Fig. 6a and b). The advancing side and part of the weld nugget had a good mixture of steel and aluminum at the weld interface (Fig. 6c and d). Similar to Fig. 5, a hook formation was also observed on the advancing side with more refined grains that could be a result of heat input difference (Fig. 6f).

Weld Surface and Tool-Pin Temperature Measurements

Table 5 summarizes measured weld temperatures at 10 mm off to the side from the tool shoulder. The temperatures were recorded with a thermal camera and

FIG. 4 Micrograph of weld (1,000 rpm and 250 mm/min).

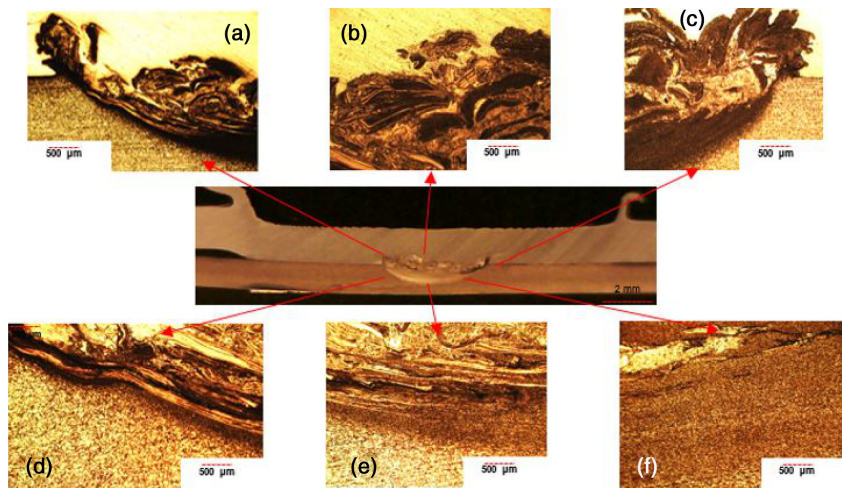
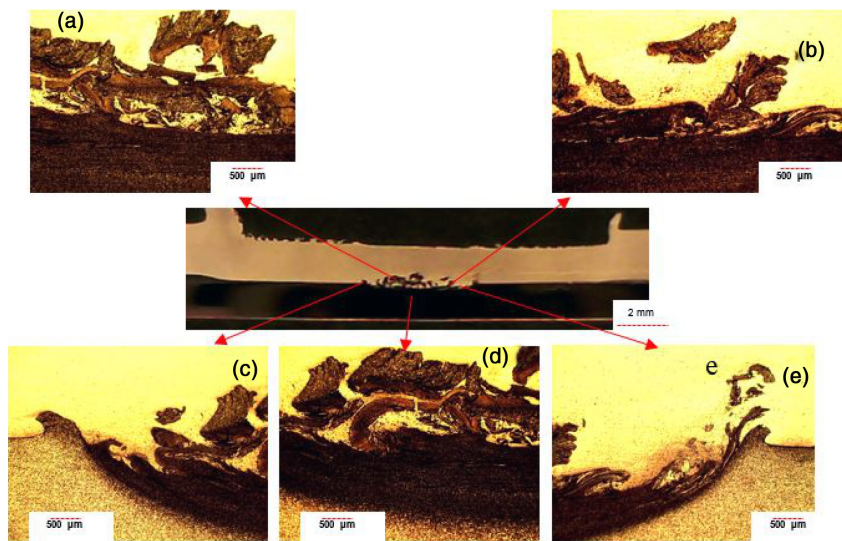


FIG. 5 Micrograph of weld (1,000 rpm and 437 mm/min).



thermocouple and compared to the FEA model using COMSOL Multiphysics. Increases in travel speed reduced the thermomechanical cycle and the processing time of the weld. With a lower travel speed and fixed rpm, more heat and higher temperatures were introduced into the weld. From [Table 5](#), the measured surface

FIG. 6 Micrograph of weld (1,250 rpm and 437 mm/min).

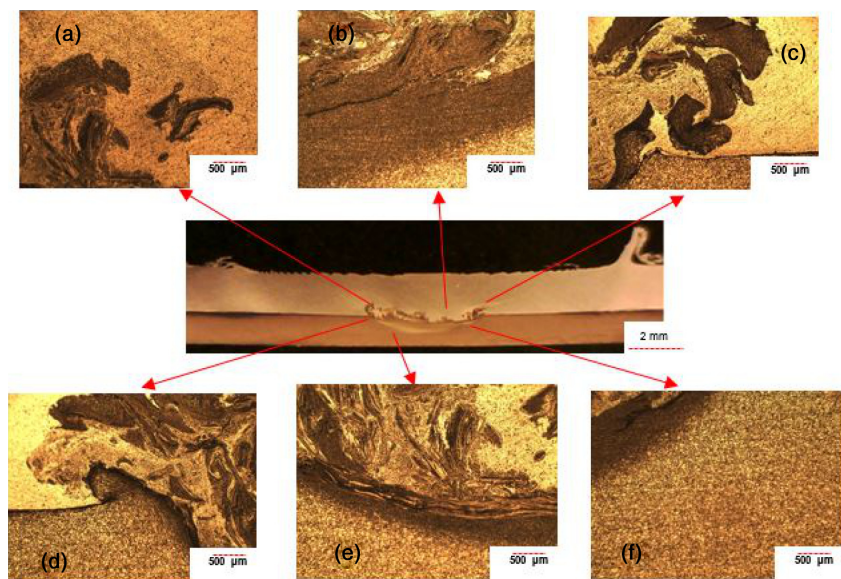


TABLE 5 The correlation between the actual surface and pin predicted temperature from COMSOL Multiphysics.

Weld No.	Rotational Speed (rpm)	Travel Speed (mm/min)	Thermocouple Temperature (10 mm from Tool Shoulder) (°C)	Thermal Camera Temperature (10 mm from Tool Shoulder) (°C)	COMSOL Temperature (°C)	Predicted Tool-Pin Temperature (°C)
1	1,000	150	264	241	250	790
2	1,000	250	256	236	235	750
3	1,000	350	210	202	210	660
4	1,000	437	185	213	213	610
5	1,250	437	195	182	190	690
6	1,250	312	220	218	218	740
7	1,250	375	213	183	211	730
8	1,000	100	281	270	270	860

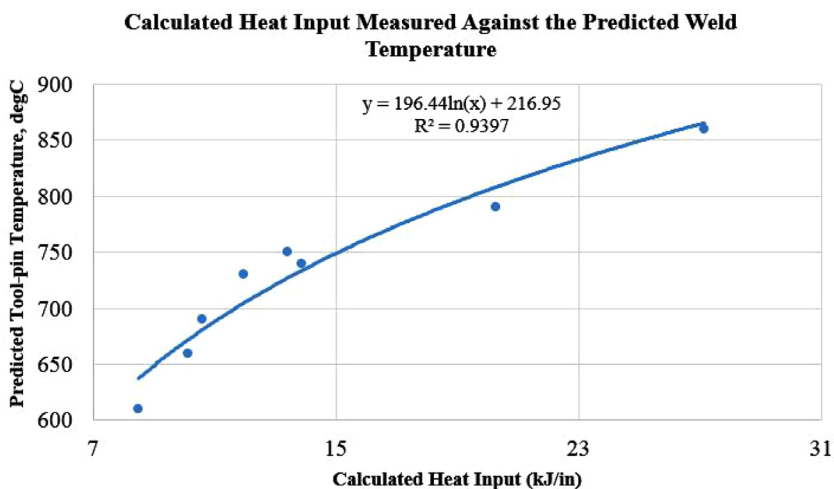
temperatures correlated well with a standard deviation of 5 % from COMSOL Multiphysics. The surface temperature measured away from the tool shoulder corresponded to the pin temperatures measured at the hot spot, where the tool pin was in contact with the DP 500 steel sheet. The increase in travel speed while the rpm is kept constant resulted in a decrease in weld temperature. This observation from the temperature measurement in the weld could be correlated to the study

conducted by Jiang and Kovacevic [14], where thermocouples were placed at 5 mm and 2 mm from the interface between the 6061 T6 and AISI 1018 steel, while the tool was rotated at 914 rpm and a 137.5 mm/min travel speed. From their study, maximum temperatures in aluminum and steel were 491°C and 613°C, respectively. A similar observation was reported by Shen et al. [8] in a study conducted on the interface bonding in different welds between AA 5754 aluminum and DP 600 steel. In their study, the peak temperature measured at the tool periphery was 424.8°C, where 1,800 rpm and 45 mm/min process parameters were employed. Table 5 shows the weld surface temperature measured at 10 mm from the tool shoulder and the corresponding tool pin temperature predicted at the various process parameters.

Calculation of Heat Input

The heat input was calculated from the welds made at all travel speeds. This was done to establish heat generation at different weld temperatures. The weld made with the lowest travel speed (100 mm/min) had the highest heat input. These heat inputs (10 to 27 kJ/in) compared well with low heat inputs used in fusion welding [16]. The calculated heat inputs from the COMSOL Multiphysics were compared to the tool pin temperatures. With a lower travel speed and fixed rpms, more heat and higher temperatures were introduced into the weld. Fig. 7 shows the heat input calculated from COMSOL Multiphysics and plotted against the weld temperature. The predicted temperatures show a correlation with heat input calculated with a proximity of 0.95 to the fitted regression line. As the weld temperature increases, the heat input also increases.

FIG. 7 Relationship between calculated heat input and weld temperature at the tool pin.



Intermetallic Quantification and the Predicted Weld Temperature

The following analysis of the intermetallics is based on the weld temperature that correlates to the joint strength obtained from the bend and fracture strength. The weld made with a 100 mm/min travel speed showed a large amount of intermetallics in the weld matrix. As a result of the slower speed, there was likely more time for intermetallics to form, which promoted the brittle fracture of the weld during the bend test. From the SEM image (Fig. 8), the heat of the weld was more on the retreating side, where a higher volume of intermetallics was found. The intermetallics were estimated to be iron rich, which showed a darker gray color as opposed to aluminum (which was a dark color), while the steel was gray.

As a result of the slow travel speed, there appears to have been high temperature concentrated on the retreating side, which assisted in plasticizing the steel. This was obvious as the tool pin touched the DP 500 and aluminum swirled into the region, causing a mixed zone. The downside of the high temperature is the formation of intermetallics at the weld interface in the weld nugget and the advancing side as shown in Fig. 9 and Fig. 10. The slower travel speed increased the weld temperature as a result of the increased material processing time, giving rise to the formation of brittle and swirling intermetallics in the steel region.

Intermetallic Volume Fraction

The SEM image of the whole nugget was taken separately at a higher magnification and combined, ensuring that no part was missing. In order to measure the intermetallic zone, SEM/EDS was used as an optical method to identify the presence and location of the intermetallics in the weld matrix. The images of the weld interface were taken using a backscattered electron detector from which the weld interface and intermetallic areas were measured using the Motic imaging software. The formed intermetallic compounds were marked on the assembled optical micrographs (Fig. 11); the area was measured, and the percentage of the intermetallic compound was calculated by dividing the intermetallic area by the weld interface area.

Fig. 12 displays the relationship of the formed intermetallics with the weld temperature. The weld made with a 100 mm/min travel speed showed a large amount of intermetallics in the weld matrix. This implies that more than one quarter of the weld nugget was composed of intermetallics, a similar observation to that made by Ravikumar, Seshagiri, and Ranjan [17] where a weld made at 1,000 rpm and 110 mm/min resulted in a severe defect. The slower travel speed increased the weld temperature as a result of the increased material processing time, giving rise to the formation of a brittle and swirling intermetallic in the steel region. From the 350 mm/min to 437 mm/min travel speed for 1,000 rpm and 1,250 rpm, respectively, there was a linear decrease in the percentage of the intermetallic. This linear decrease was similarly recorded from the weld temperature.

FIG. 8 Iron rich intermetallics on the retreating side of weld (1,000 rpm and 100 mm/min).

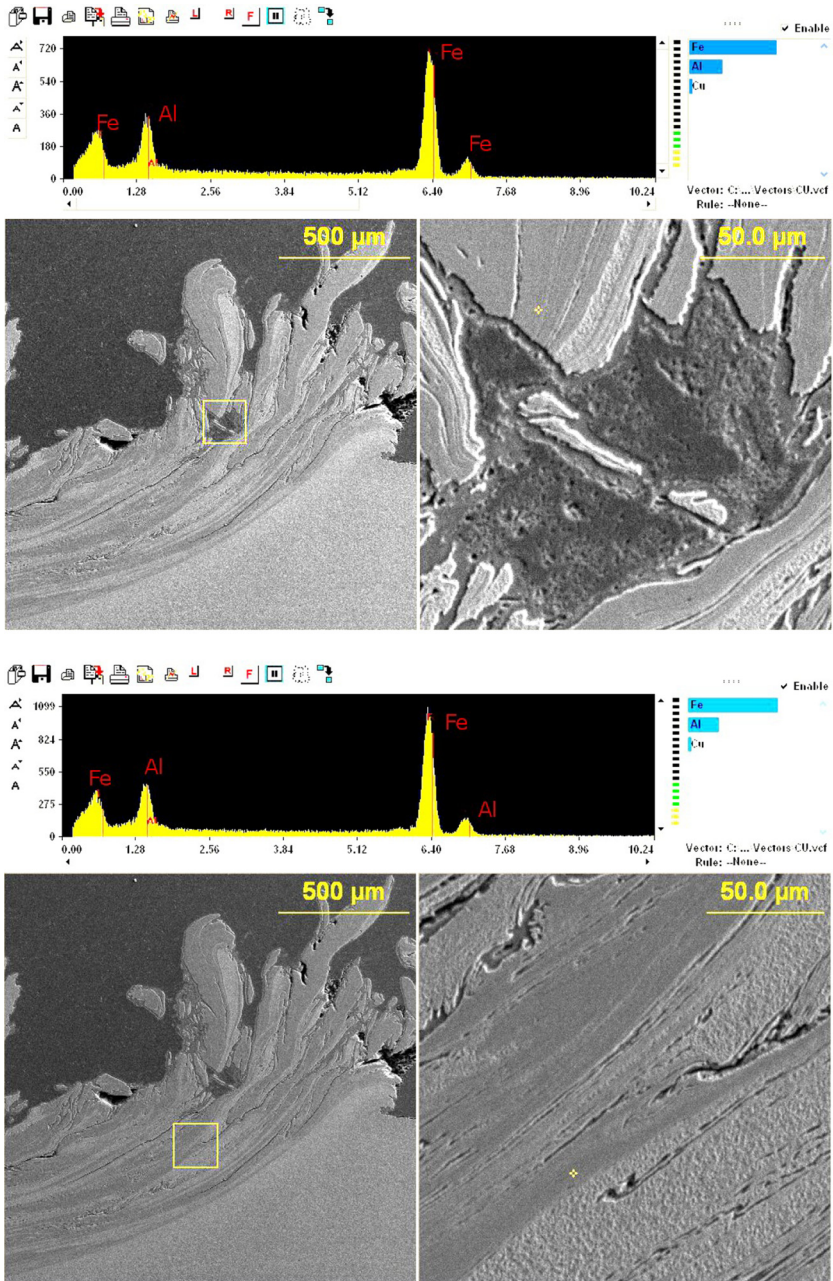


FIG. 9 Intermetallics in the weld nugget (1,000 rpm and 100 mm/min).

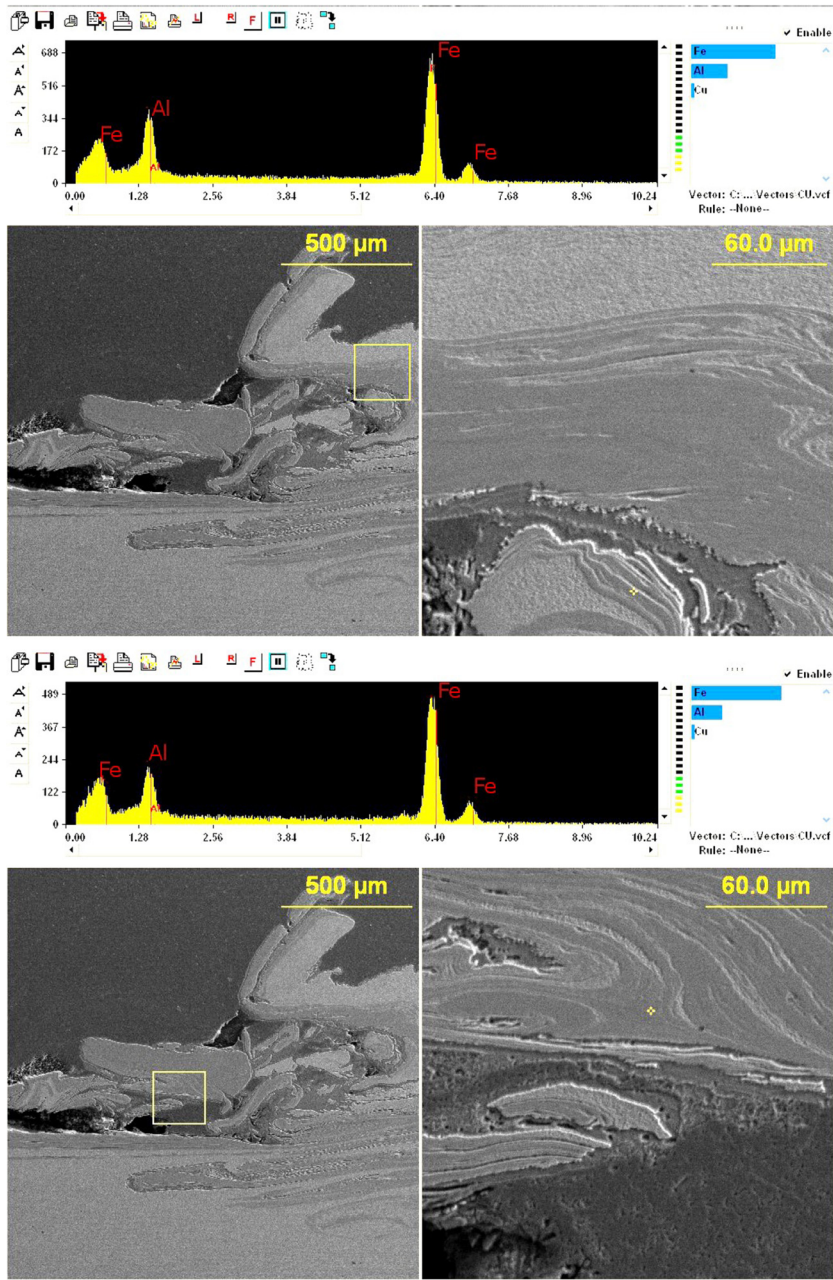


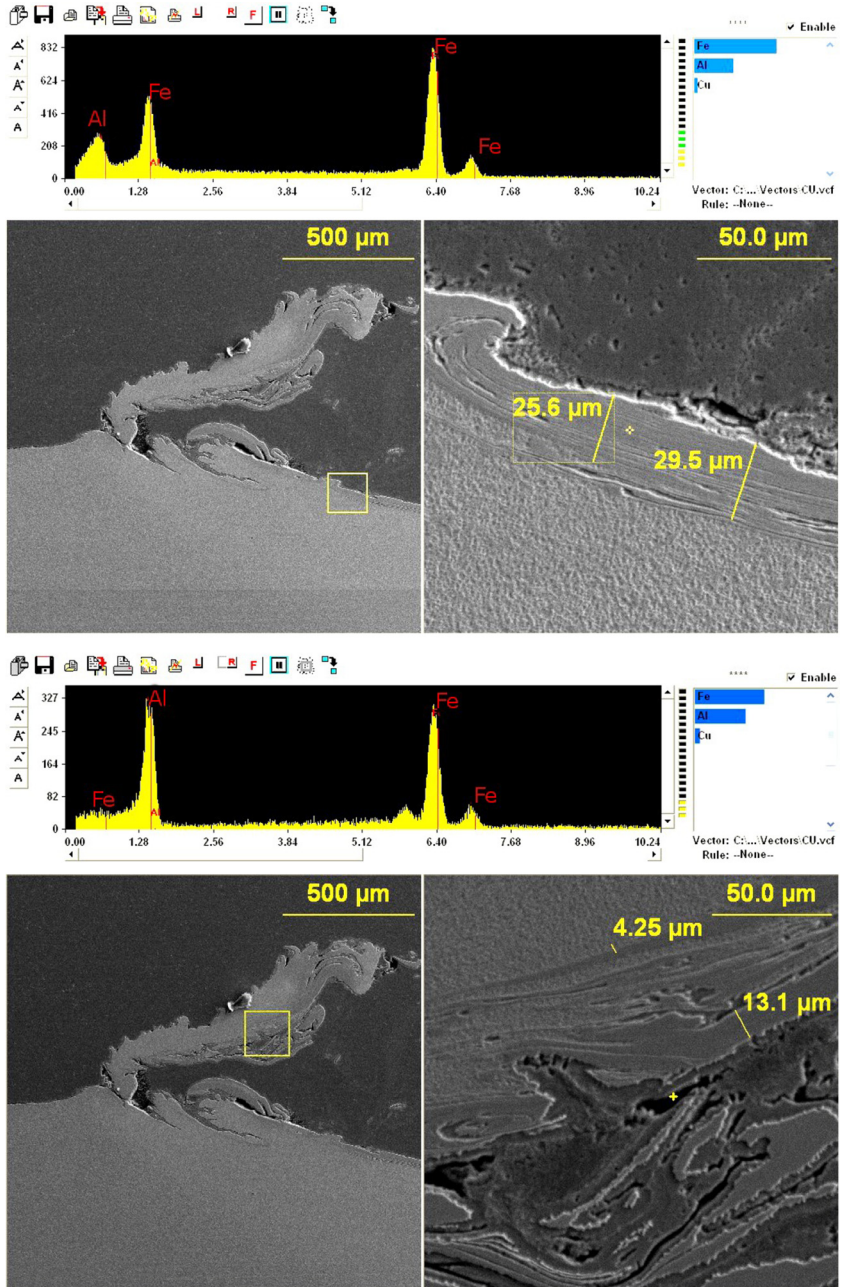
FIG. 10 Intermetallics in the weld nugget (1,000 rpm and 100 mm/min).

FIG. 11 Typical SEM image for the intermetallic volume fraction measurement.

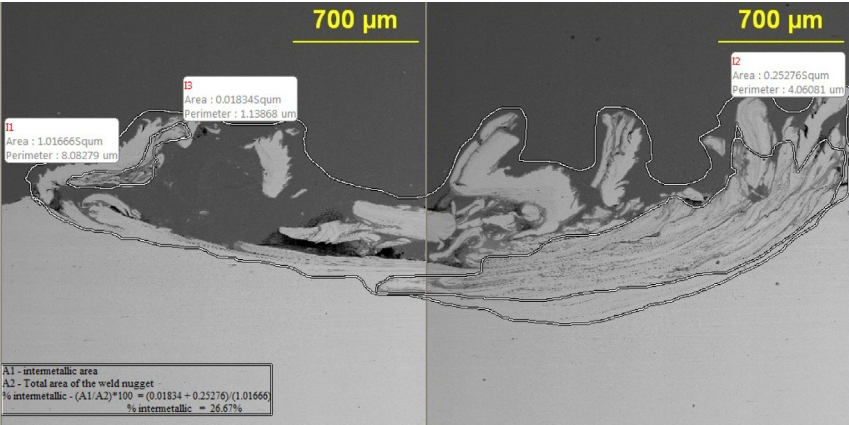
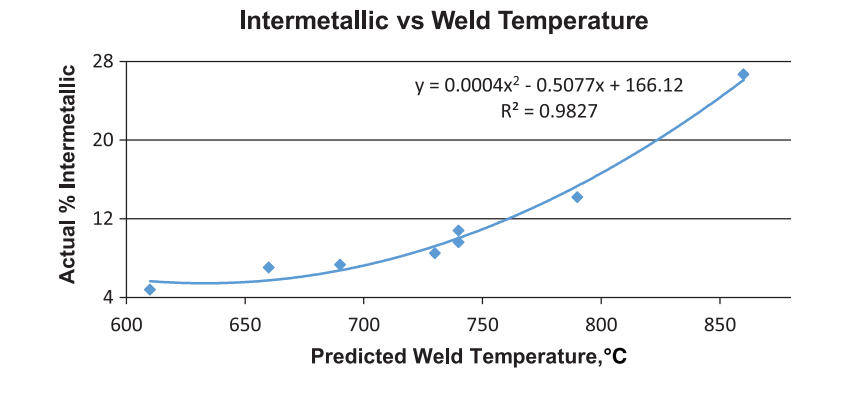
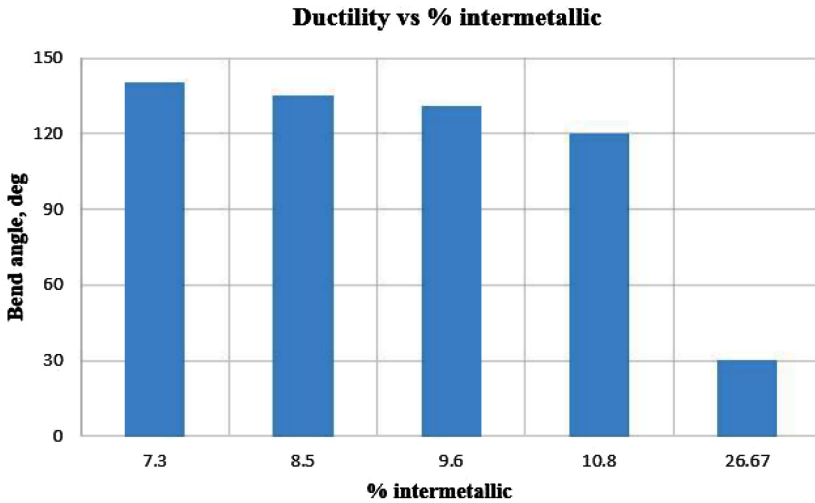


FIG. 12 Percentage of intermetallics versus predicted tool pin temperature.



With an increase in the tool pin temperature, the percentage of intermetallics also increases. From Fig. 13, there are two shelf behaviors from the percentage of intermetallics and heat input relationship. Below 14 kJ/in, the percentage of intermetallic behavior was found to be similar as the heat input increased from 8.5 to 14 kJ/in for a 350 mm/min to 437 mm/min travel speed at 1,000 rpm and 1,250 rpm. Above 14 kJ/in, there was a sudden increase in the percentage of intermetallics as the heat input increased. This cutoff in heat input at approximately 11 kJ/in above which the percentage of intermetallics suddenly increased was similarly observed from the relationship between the weld temperature and bend angle. The trend in the result was found interesting as the weld temperature is closely related to the intermetallic volume fraction. The relationship between the actual percentage of intermetallics and the bend angle was plotted in Fig. 13. The high bend angle was

FIG. 13 Relationship between bend angle and percentage of intermetallics.

a good and beneficial predictor, particularly in an automotive application where energy absorption of the structure is an important factor. The formation and distribution of intermetallics at the weld interface affects the ductility of the weld and its dependency on the variability of weld parameters. The percentage of intermetallics decreases with a decrease in the weld temperature and heat input, while the bend angle increases with a decrease in the percentage of intermetallics and weld temperature. Welds made with a low travel speed (1,000 rpm and 100 mm/min) had a high heat input and weld temperature [17], resulting in a higher volume fraction of intermetallics. The bend angle measured was 30°, with no ductility in the weld. With an increase in travel speed, there was less time for intermetallics to form as the weld temperature and heat input decreased.

At the 1,000 rpm and 150 mm/min combination, there was heat distributed in the weld, which promoted material mixing. However, a brittle failure was recorded that could be a result of the intermetallics in the weld matrix. A bend angle of 120° was measured with partial ductile failure when a process parameter of 1,000 rpm and 250 mm/min was employed. With 1,250 rpm and 312 mm/min, a bend angle of 131° was measured. A similar observation was made with the weld at a 437 mm/min travel speed, although with an increase in the bend angle of approximately 140°. There was no surface crack in the weld that implied adequate material mixing. From the bend test analysis, it could be said that a high heat input promoted the formation of intermetallics. This caused an early fracture of the weld with a low bend angle. Fig. 13 is the plot that shows the relationship between the actual percentage of intermetallics and the bend angle. The percentage of intermetallics decreases with a decrease in the weld temperature, while the bend angle increases with a decrease in the percentage of

intermetallics and the weld temperature. There were two shelves obtained from the plot; the first shelf had a linear decrease in the bend angle as the travel speed increased from 250 mm/min to 437 mm/min. This observation was similar to the trend in the weld temperature and heat input in Fig. 12. It could be inferred that a FSW has a good ductility when the percentage of intermetallics is equal to or less than 11 %. The second shelf had a sudden drop in the ductility from 100 mm/min to 250 mm/min as the percentage of intermetallics increased above the threshold (11 %). The increase in rpm provided heat to the weld for plasticization and material mixing [18], while the increase in travel speed assisted in reducing the formation of intermetallics as similarly postulated by Shen et al. and Das, Ghosh, and Pal et al. [8,19].

Conclusions

Friction stir lap welding of the AA 6061 T6 and DP 500 steel were successfully made using a combination of a tool steel shoulder and tungsten rhenium tool pin. From the microstructural standpoint, the final welds had no voids that could increase their sensitivity to cracking in the welded joint. The weld transition zone consisted of steel penetration into the aluminum matrix, which is beneficial in improving interfacial and fracture strength.

With the increase in travel speed, the weld temperature was reduced to minimize the growth of brittle intermetallics that could increase its susceptibility to brittle failure. Specifically, good correlations were found between the increased strength and reduced ductility of dissimilar aluminum to steel welds with an increased volume fraction of Fe Al intermetallic in the FSW weld nugget. On the other hand, lower heat inputs and associated higher travel speeds were able to reduce the volume of intermetallics; therefore, the cost optimization via increased productivity goes hand in hand with improved weld properties.

The decrease in the weld temperature and heat input resulted in welds with a lower amount of intermetallics. The research demonstrated that a decrease in the weld heat can be used to promote ductile weld properties in the FSW of aluminum to steel. Higher ductility was found among the welds made at a higher travel speed with a lower volume fraction of intermetallics. These findings should help the automotive industry further improve their dissimilar part designs, while improving productivity, properties, and fuel efficiency due to the overall light weighting effect.

While significant work has been done to achieve the stated objectives of this study, there are other aspects of this study that require further investigation. These include:

- A design of experiment can be established with a statistically significant number of welds with different alloy and material thickness for repeatability tests in the area of high percentages of intermetallics.
- X ray diffraction of the weld matrix could be done to identify the chemical composition of the intermetallic present in relation to the percentage of Al in the matrix. The mass balance of Al and Fe in the matrix dictates the type of intermetallics formed.

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Mechanical Behavior and Quantitative Fractographic Characterization of Hot-Stamped Usibor[®] 1500 Steel as a Function of Strain Rate

Citation

Mishra, K., Sharma, S. M., Gokhale, A. M., Thadhani, N., and Bhat, S. P., "Mechanical Behavior and Quantitative Fractographic Characterization of Hot-Stamped Usibor[®] 1500 Steel as a Function of Strain Rate," *100 Years of E04 Development of Metallographic Standards, ASTM STP1607*, G. F. Vander Voort, Ed., ASTM International, West Conshohocken, PA, 2019, pp. 107–116, <http://dx.doi.org/10.1520/STP160720170231>⁴

ABSTRACT

Effects of strain rate on mechanical behavior and fractographic features are elucidated in Usibor[®] 1500 hot-stamped at extreme conditions. Uniaxial tensile tests have been performed at strain rates ranging from 10^{-4} to 10^3 per second. It is observed that the ductility and ultimate tensile strength are higher at the highest strain rate as compared to the lowest strain rate studied. Unbiased quantitative fractography has been applied to characterize the fracture surfaces after tensile tests. Numerous quantitative fractographic attributes including area fractions of the fractures surfaces generated by different fracture micromechanisms and number density of dimples in the dimpled fracture regions have been estimated

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⁴ASTM Symposium on *100 Years of E04 Development of Metallographic Standards* on November 15–17, 2017 in Atlanta, GA, USA.

from the quantitative fractographic data. These data reveal that ductile dimpled fracture is the dominant fracture micromechanism at all strain rates. Nonetheless, the extent of the fracture surface generated by ductile dimpled fracture increases with increase in the strain rate. Therefore, increase in the strain rate facilitates dimpled ductile fracture or increases resistance to brittle fracture (or both).

Keywords

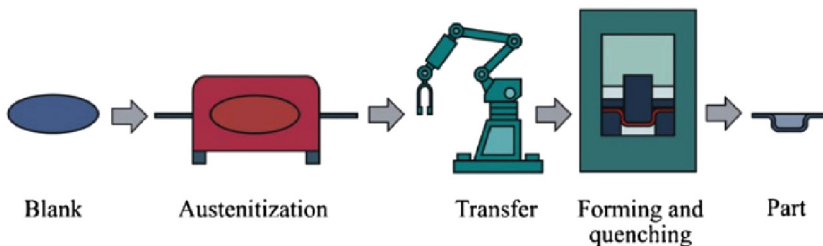
hot-stamping, high strain rate deformation, AHSS steels, stereology, quantitative fractography

Introduction

Increasing demand for reduced vehicle weight and passenger safety has led to interest in thinner gauge, lightweight, high strength components for automotive structures [1]. Development of advanced high strength sheet steels and innovations in the hot stamping process have enabled cost effective commercial production of such automotive components. While there are several variants of the hot stamping process, most high strength hot stamped steel components are manufactured using direct hot stamping [2]. In this process, sheet steel is heated above its austenitization temperature. The austenitized steel is then simultaneously formed and quenched inside a water cooled die, resulting in a near net shape part as illustrated in Fig. 1 [2]. The microstructure of the hot stamped component depends on many factors, including the steel chemistry, the cooling rate, the contact pressure, and the transfer time. To obtain predominantly martensitic microstructure (and consequently, high strength), 22MnB5 steel grade (or its variant) is commonly used for such applications leading to tensile strength levels on the order of 1500 MPa (and elongation of approximately 5 %) in the components. Such high strength levels are attractive for complex automotive structural parts like B pillar and side rails [3].

During a car crash, steel body parts can experience deformation at strain rates as high as 1,000 per second. Consequently, it is of interest to understand the effects of strain rate on mechanical response of high strength sheet steels. There have been a few investigations on the effects of strain rate on mechanical response of martensitic hot stamped sheet steel [4]. Nonetheless, there has been no quantitative

FIG. 1 Direct hot stamping process [2].



investigation of how strain rate affects fracture micromechanisms in martensitic hot stamped sheet steel components. Accordingly, the objective of this contribution is to quantitatively characterize the effects of strain rate on the fracture surface morphology and fracture micromechanisms in a martensitic hot stamped Usibor[®] 1500, using unbiased quantitative fractography.

Experimental Work

MATERIALS AND PROCESSING

Commercially produced 1.5 mm thick sheets of Usibor[®] 1500 were used in this study. Nominal chemical composition of the steel is given in Table 1.

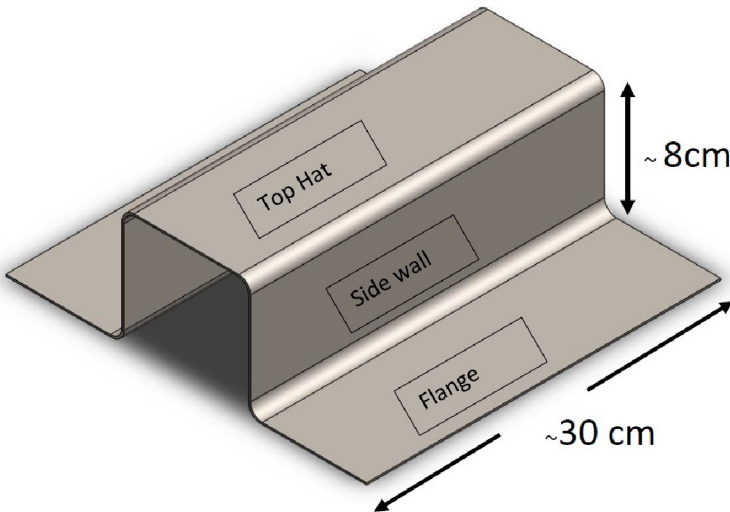
Hot stamping experiments were performed at ArcelorMittal Global R&D, East Chicago, IL. Usibor[®] 1500 steel blanks were austenitized at 980°C for 30 min., and then simultaneously deformed and quenched in a closed water cooled die to produce hat sections having the geometry shown in Fig. 2. Note that these are *not* typical austenitization process parameters used in the commercial practice of

TABLE 1 Nominal chemical composition (in wt.%) of Usibor[®] 1500 steel.

C	Mn	Si	Cr	Ti	Al	N	P	S	B
0.23	1.25	0.27	0.02	0.04	0.04	0.004	0.01	0.002	30 Ppm

Note: C carbon; Mn manganese; Si silicon Cr chromium; Ti titanium; Al aluminum; N nickel; P phosphorus; and S sulfur.

FIG. 2 Geometry of hot stamped hat section [5].



hot stamping of Usibor[®] 1500 steel and were selected to illustrate the effects of strain rate at extreme (abuse) conditions.

MECHANICAL PROPERTIES AND MICROSTRUCTURE CHARACTERIZATION

Tensile test specimens were machined with gauge along the long length from the top hat region (Fig. 2). In this study, tensile tests at all strain rates were performed with the same specimen dimensions and geometry (Fig. 3) because mechanical properties, especially the elongation to failure (ductility), are sensitive to specimen geometry. Uniaxial tensile tests up to a strain rate of one per second were carried out on an MTS servohydraulic machine (MTS Systems Corporation, Eden Prairie, MN). The tests at a strain rate of 10^3 per second were performed using a split Hopkinson bar setup at REL Laboratory (Calumet, MI).

Metallographic specimens were prepared from the cross section using procedures described in detail by Mishra [5]. The polished specimens were etched for 15 s in a 2 % nital solution to reveal the microstructure depicted in Fig. 4.

QUANTITATIVE FRACTOGRAPHY

Fracture surfaces were observed in a Hitachi SU8010 scanning electron microscope (SEM). All fractographic observations and measurements were performed at the same magnification, in the middle one third region of the fracture surface, to avoid any edge effects. Area fractions of ductile dimpled fracture regions and brittle fracture regions (pullouts as well as featureless facets) were estimated using systematic uniform random sampling and well known quantitative fractographic techniques [6,7]. Number density of the features in the fracture surfaces was estimated using unbiased frame [8], and average dimple size was calculated from the estimated number density of the dimples in the dimpled fracture regions. Ten different fields of view were used for quantitative measurements. Each field of view had measurements at four different locations.

Results and Discussion

Fig. 5 depicts uniaxial engineering stress strain curves of hot stamped Usibor[®] 1500 at the highest and lowest strain rates studied and illustrates the strain rate

FIG. 3 Tensile test specimen geometry (all dimensions are in millimeters).

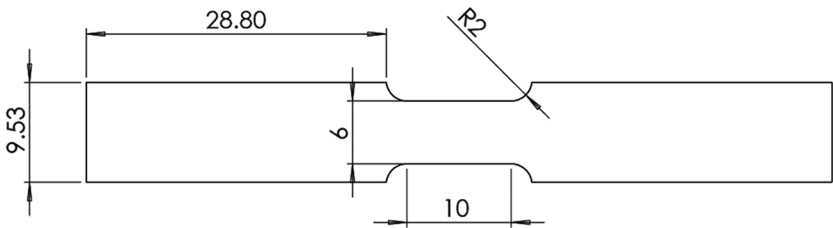


FIG. 4 Representative SEM micrograph of hot stamped Usibor® 1500, depicting martensite platelets.

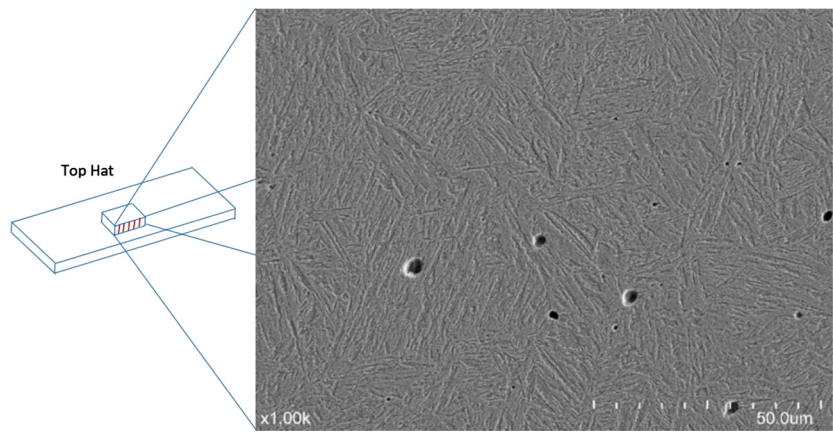
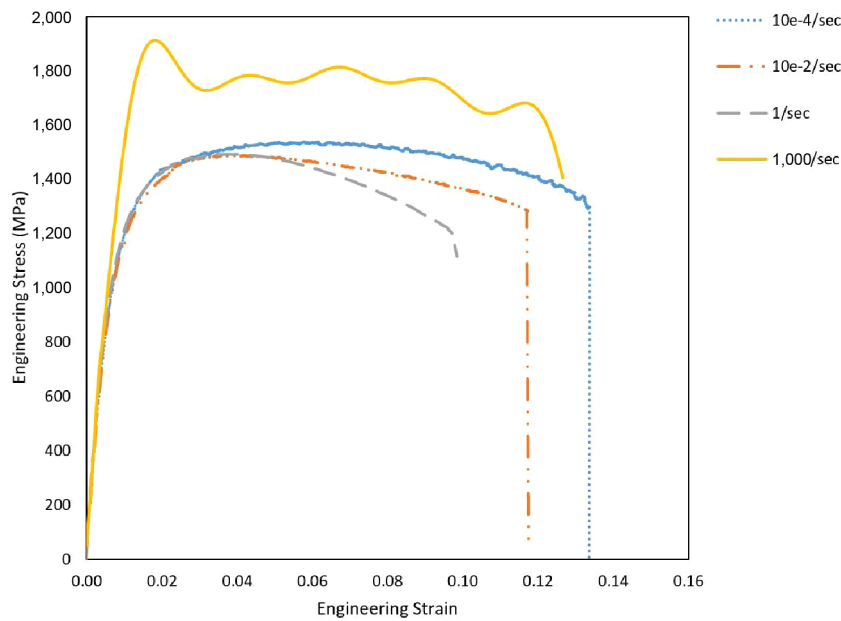


FIG. 5 Engineering stress strain curves. In the test at the strain rate of 10^{-3} per second, data acquisition was terminated before fracture.



dependence of the mechanical behavior of the steel. One tensile test was conducted at each strain rate. Data acquisition in tests at $10^3/s$ had to be terminated before the final fracture; therefore, the fracture ductility was measured by comparing the gage length before and after the test for all strain rates. Ultimate tensile stress was calculated using the peak load.

Fig. 6 and 7 depict strain rate dependence of ultimate tensile strength (UTS) and fracture ductility. Due to inconsistency in failure of strain gauge from test to test, ductility was measured using gage length extensions in the specimen. Both UTS and ductility are higher at the highest strain rate ($10^3/s$) as compared to their values at the lowest strain rate studied ($10^{-4}/s$). Thus, the tensile properties are better at high strain rates ($10^3/s$) as compared to the quasistatic properties usually reported in the literature, although intermediate strain rate data do not follow the trend.

FIG. 6 Strain rate dependence of UTS represented by plot of UTS versus log (strain rate).

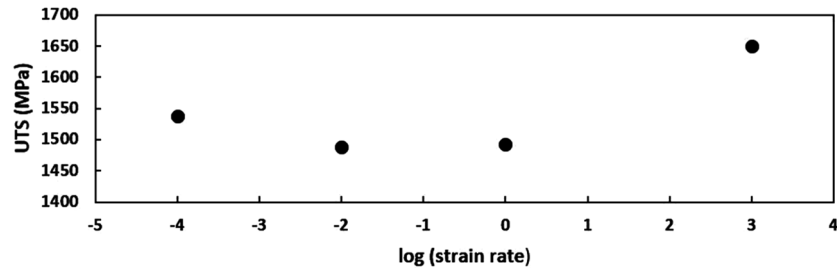


FIG. 7 Strain rate dependence of fracture ductility. Ductility was measured from change in gauge length of specimens.

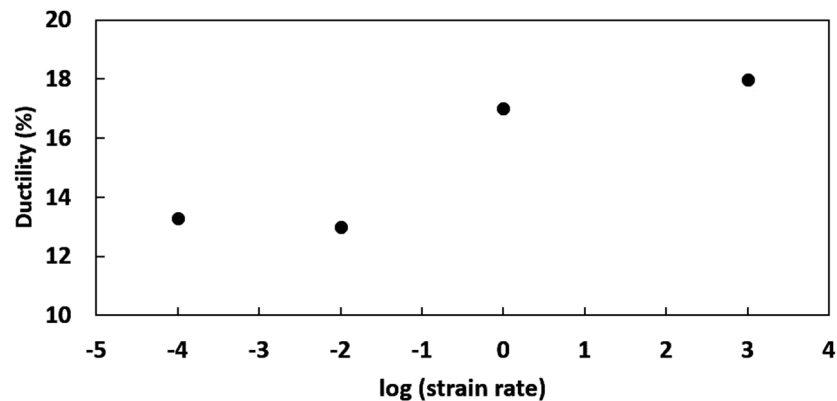


Fig. 8 and 9 depict SEM fractographs following the uniaxial tensile tests at the lowest (10^{-4} /s) and the highest (10^3 /s) strain rates. In both cases, a significant extent of fracture surfaces is generated by ductile dimple features. However, the

FIG. 8 SEM fractograph at 10^{-4} per second strain rate.

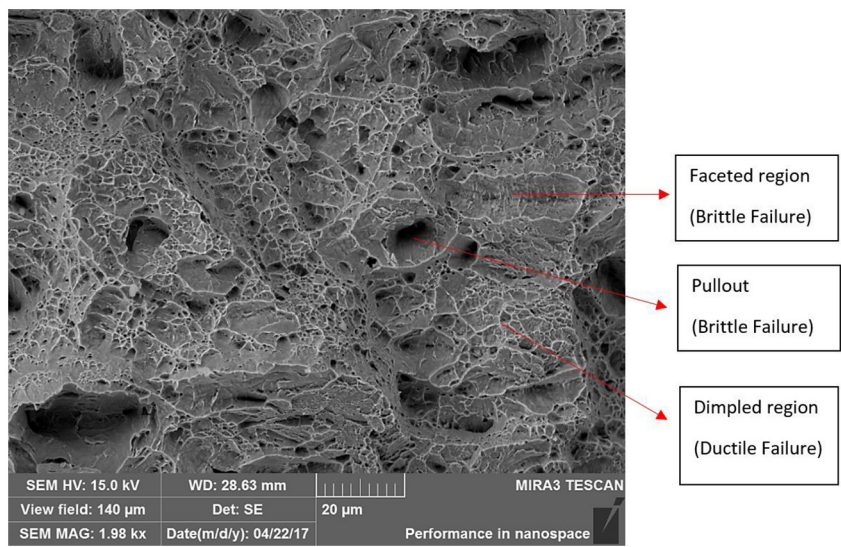
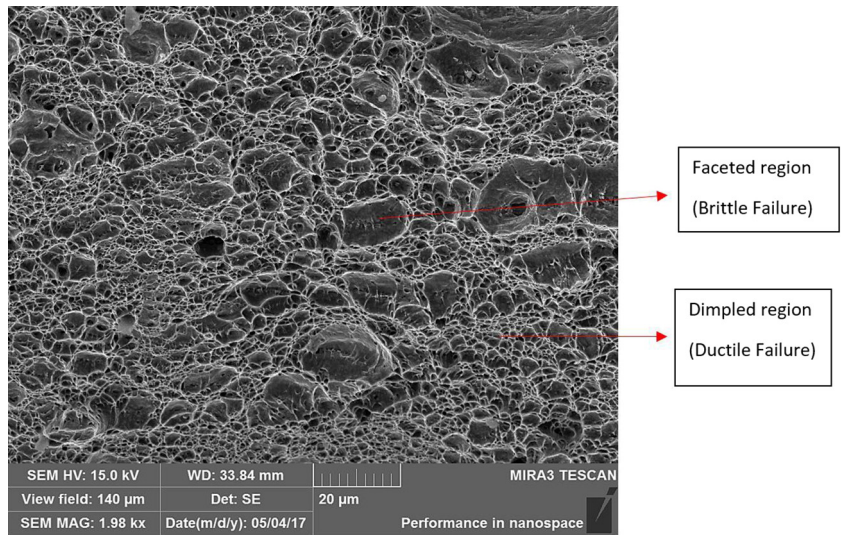
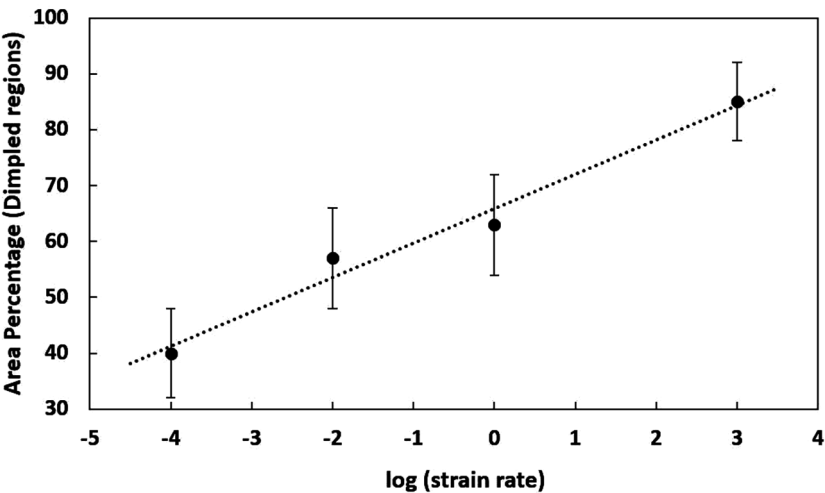


FIG. 9 SEM fractograph at 10^3 per second strain rate.



extent of dimpled fracture is substantially higher in the specimen tested at 10^3 per second as compared to that tested at 10^{-4} per second strain rate. Unbiased quantitative fractographic data indicate that the morphological features represented in the final fracture surface are strain rate sensitive in this steel. For example, Fig. 10 shows the variation of the area percentage of the fracture surface generated by ductile dimples as a function of the strain rate. It is interesting to note that the area percentage of the ductile fracture increases monotonically with the logarithm of the strain rate, implying that higher strain rate promotes ductile fracture or increases resistance to brittle fracture or both. Quantitative fractographic data also revealed that the number density of the dimples in the ductile fracture regions and the average dimple size are not very sensitive to the strain rate [5]. More detailed analysis indicates that while the intrinsic nature of the operative fracture mechanisms do not vary significantly with the strain rate, the contributions of different fracture mechanisms in the generation of the fracture surface vary significantly with the strain rate for this steel. It is interesting to note that other investigators using different test methods (for example, double edged notched tensile, stacked Charpy, and so on) have indicated that extreme hot stamping process conditions similar to that used in this investigation may lead to reduced toughness in the part [9 11]. Thus different loading conditions may change the extent of ductile dimple versus brittle fracture processes in the hot stamped steel at high strain rates.

FIG. 10 Plot of area percentage of dimpled region versus log (strain rate).



Summary and Conclusions

Effects of strain rate on the mechanical behavior and fracture mechanisms in hot stamped martensitic Usibor® 1500 sheet steel are quantitatively characterized. The engineering stress strain relationship is sensitive to the strain rate. UTS and ductility are higher at the highest strain rate as compared to the lowest strain rate studied. Significant extent of the fracture surface is generated by ductile dimple fracture mechanism at all strain rates. Nonetheless, the extent of the fracture surface generated by ductile dimple fracture mechanism monotonically increases with the increase in the strain rate. Therefore, a higher strain rate promotes ductile fracture.

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Analysis of an Unusual Failure in a Brazed Tube Assembly

Citation

Capo, J. C., Bagnall, C., and Moorhead, W. J., "Analysis of an Unusual Failure in a Brazed Tube Assembly," *100 Years of E04 Development of Metallographic Standards, ASTM STP1607*, G. F. Vander Voort, Ed., ASTM International, West Conshohocken, PA, 2019, pp. 117–129, <http://dx.doi.org/10.1520/STP160720170218>³

ABSTRACT

A tube assembly with a through-wall fracture at a brazed bracket joint was investigated to determine the cause for component failure. The product was made from 1008 cold-rolled steel sheet, shaped into 12.7-mm diameter tubing and seam welded axially. The material was manufactured and shipped as a straight tube. During component manufacture, an approximately 650-mm length of the tubing was bent at two locations to form an "S"-shaped component. Four different mounting brackets were then brazed onto it. The fully assembled part fractured adjacent to one of the brackets, within the "S"-bend of the tube assembly, prior to entering service. Failure analysis revealed that premature fracture was related to excessive infusion of copper into the tube wall along grain boundaries. Metallography of tube sections demonstrated exposure of the joint to excessively high temperature, sufficient to cause partial dissolution of some grains. Scanning electron microscopy and energy dispersive spectroscopy revealed that the braze metal appears to be a copper-manganese (Cu-Mn) base alloy. The widespread intergranular dispersion of braze metal in the tube wall adjacent to the brazed bracket indicated excessive temperature exposure and liquid metal embrittlement. Possible causes for the anomalies observed at the bracket braze failure site are discussed.

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³ASTM Symposium on *100 Years of E04 Development of Metallographic Standards* on November 15–17, 2017 in Atlanta, GA, USA.

Keywords

Cu-Mn braze alloy, liquid metal embrittlement, grain boundaries, failure analysis

Introduction

The subject tube assemblies are mass produced and have an end use as auxiliary equipment in a small engine application. Dimensions of the tubing are a 14.00 mm outer diameter (OD) by a 0.99 mm wall thickness. The tube is fabricated from aluminum killed drawing quality (AKDQ) 1008 carbon steel. The strip stock is shaped into a tube and joined with an autogenous seam weld. Initially, the parts are manufactured as straight tubing and then cut to smaller lengths before delivery to the customer. Tube sections are bent at two different locations to form an “S” shaped part. Four different mounting brackets are then furnace brazed onto the tube. Details of the brazing operation are not known. The braze metal is assumed to be in wire or thin foil form and located at each of the four brackets, which would be held in place in a tooling fixture. Batches of one or more parts would then be exposed in a braze furnace.

A combination of macrophotography, fractography, metallography, scanning electron microscopy and microindentation hardness evaluation was performed on samples from the fracture area adjacent to one bracket. The data collected were then reviewed for development of a potential mode of failure.

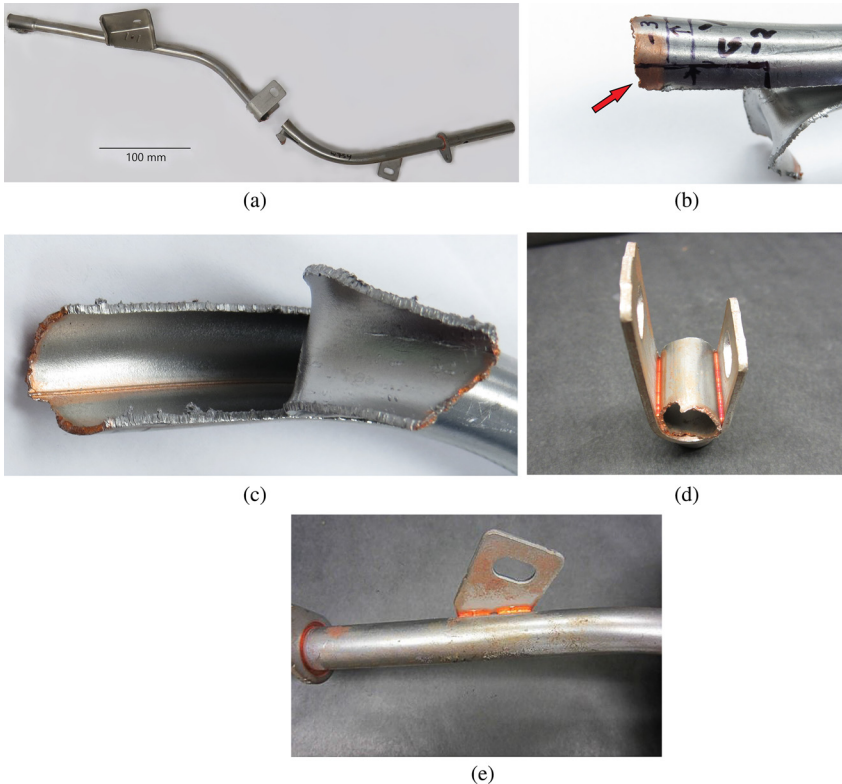
Macrophotography, Visual Examination, and Sectioning

Macrophotographs of the failed part are provided in [Fig. 1a](#). The part fractured adjacent to one of the brazed mounting brackets. This area was sectioned to yield both transverse and longitudinal samples for fractographic and metallographic evaluations. The transverse sample was used to study dominant features of the fracture face, using stereomicroscopy. Mounted sections were prepared to examine microstructure and to determine hardness of the seam weld, as compared to the base metal. The longitudinal sample was used to determine crack propagation mode and condition of the microstructure adjacent to the crack.

A dominant copper color was observed on the tubing in the vicinity of the fracture surface and around all the braze joints ([Fig. 1b–e](#)). Its intensity was brightest on, and adjacent to, the fracture face. In some locations, this surface coloration extended up to 6.5 mm away from the fracture. The vivid color, in sharp contrast to the steel tube surface, migrated along the seam weld on the inner diameter (ID) surface with decreasing intensity as it progressed away from the fracture for a distance of more than 25 mm.

A second tube was included in the study; this sample consisted of a straight piece of tubing in the as delivered condition (i.e., the final condition just prior to

FIG. 1 Macro views of the fractured brazed component and details of braze flow at fracture site: (a) As received failed tube assembly. (b) Fracture surface marked for sectioning—note location of longitudinal section (arrow). (c) Braze alloy migration from fracture along seam weld—ID view of (b). (d) Bright, Cu colored braze alloy at fracture site. (e) Braze appearance at two other brackets without apparent damage.

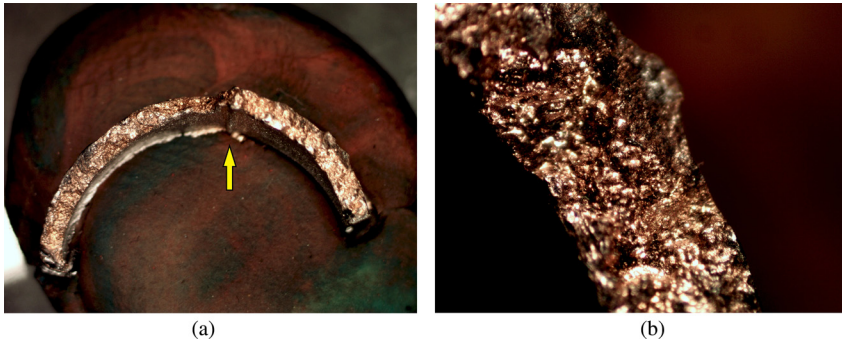


bending and brazing operations). This straight tube was sectioned transversely to compare the seam weld microstructure to that observed on the failed tube assembly.

Fractography

One half of the fractured tube was examined using stereomicroscopy ([Fig. 2](#)). The fracture surface, located adjacent to the side of a brazed bracket as shown in [Fig. 1](#), exhibited a copper color. Features appeared as smoothed over facets, indicating that the surface was coated with a foreign material. The fracture was predominantly

FIG. 2 Macrophotograph of a 180° arc of the tube fracture. (a): Step in fracture is at seam weld (arrow), 7×. Details of the fracture features appear to be obscured by a coating in (b): Section of RHS of fracture surface, 40×.



planar but not perpendicular to the tube axis. A step was formed in the fracture plane as it crossed the tube seam weld.

Metallographic Analysis and Hardness Testing

FRACTURE FACE EVALUATION

A longitudinal section that contained the fracture face at one end was cut from the tube (Fig. 1b) and mounted in cross section to examine the full through wall thickness. The sample was prepared using a hot compression thermosetting resin for the mount and polished to a 0.05 μm finish, using colloidal silica for the final stage. Samples were examined in unetched and etched conditions. Examples of microstructural appearance are illustrated in Fig. 3. It was evident that copper braze material was present along most of the fracture face and that copper had also penetrated deeper into the steel tubing along the grain boundaries. On the OD surface, up to 5 mm away from the fracture face, copper was observed at random locations. The longitudinal section also revealed braze metal penetration along grain boundaries in these areas. The degree of copper contamination present was excessive. In some areas adjacent to the fracture, copper braze metal was observed in pools with steel grains “floating” as islands within it.

Change in Seam Weld Microstructure and Hardness: Thermal History Evaluation

Metallographic examination of the seam weld microstructure was performed on the fractured tube assembly and on a straight tube section from the as delivered material. This study provided a comparison of microstructural changes introduced during the brazing cycle for the fractured tube assembly. Weld structures are compared in Fig. 4.

FIG. 3 Unetched (a, c, e) and etched (b, d, f) areas taken at the fracture end of a longitudinal sample cut from the tube wall. Large accumulations of Cu colored braze metal at the fracture are evident. Braze metal has also penetrated the microstructure away from the fracture. (a) Tube OD (top) and fracture face, 200 $\times$. (b) Etched detail of top of fracture in (a), 400 $\times$. (c) Top (OD) surface, away from fracture, 1000 $\times$. (d) Etched detail of area shown in (b), 1000 $\times$. (e) Bottom/ID corner: Heavy accumulation of Cu; note Cu on ID surface, 200 $\times$. (f) Enlarged view of bottom/ID corner after etching, 400 $\times$.

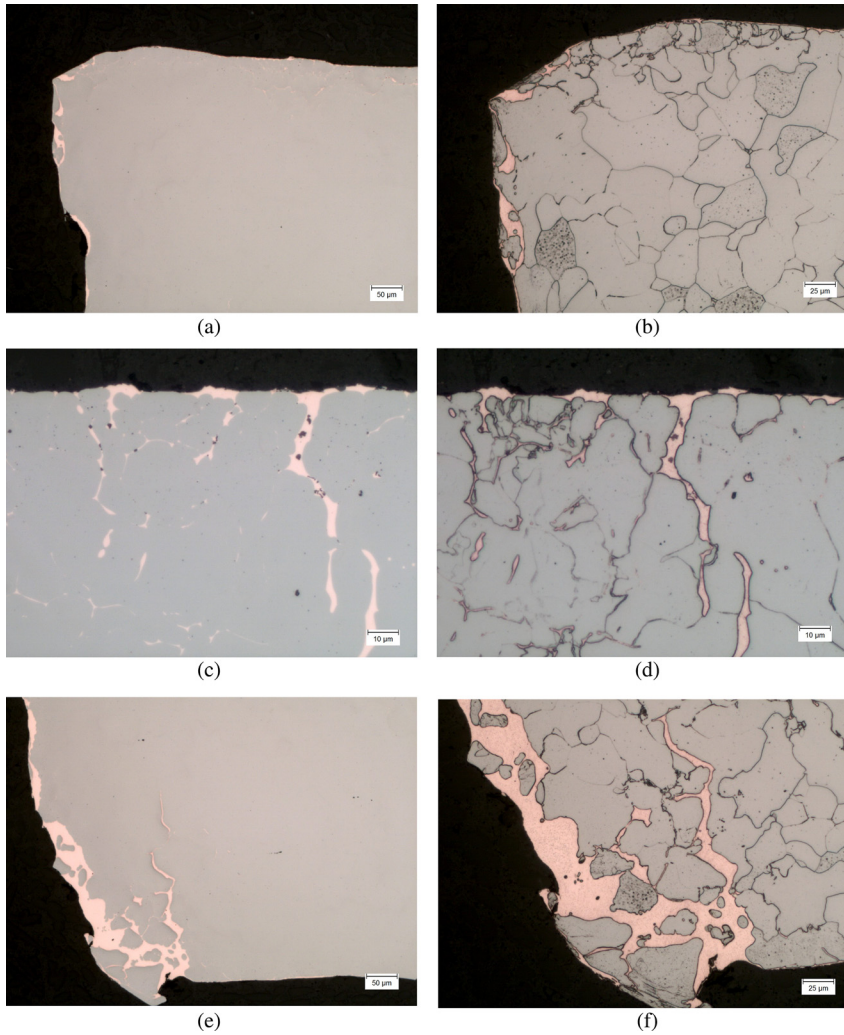
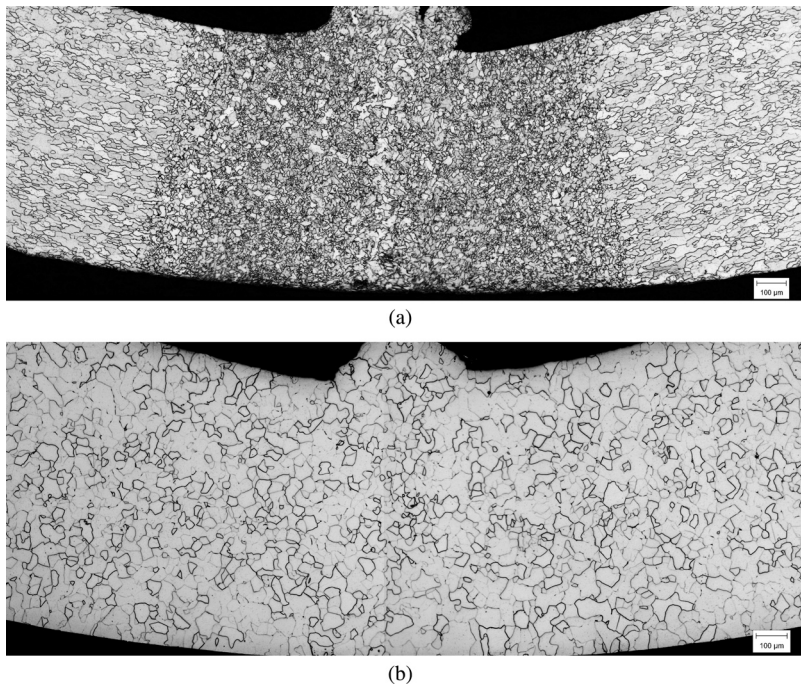


FIG. 4 Metallographic examination of the seam weld (a) as received, before brazing, and (b) in vicinity of fracture after brazing.



The condition of the seam weld before brazing is presented in [Fig. 4a](#). The upset flash on the ID side of the autogenous weld is clearly visible. Also visible is the heat affected zone (HAZ) from the welding operation, which spans nearly 1 mm on either side of the weld fusion line. Outside of the HAZ, the base metal has a grain structure consistent with rolled product.

After brazing, [Fig. 4b](#), microstructural changes are significant. AKDQ 1008 steel is a plain, low carbon product and is essentially single phase. Thermal exposure of this bracket during the braze cycle was sufficient to recrystallize the tube and weld areas. If not for the existence of the weld upset flash on the tube ID, it would be nearly impossible to identify the weld area. Any microstructural indications of a prior weld in this part are no longer visible. Even the rolled grain structure in the base metal has been recrystallized and now has an equiaxed appearance. Some grain growth has also occurred.

Microindentation hardness measurements were made in the base metal and weld center regions of tube weld joint samples before and after brazing. Results are provided in [Table 1](#). As expected, in the prebrazing condition, the weld has a much higher hardness than the base metal due to rapid cooling achieved after welding. After the braze cycle, there is virtually no difference between the weld area and the tube. Results are summarized in [Table 1](#).

TABLE 1 Microindentation hardness measurements from the base metal and seam weld locations, before and after the braze cycle.

Location	Before Braze Cycle	After Braze Cycle
	HV/HRB Converted*	HV/HRB Converted*
Base Metal	112/63	121/68
Center of Axial Weld	210/95	112/63

Note: Values shown are averages of three measurements in each location.

*HV Vickers hardness value, HRB Rockwell 'B' hardness value.

SCANNING ELECTRON MICROSCOPY

The tube fracture, at a brazed bracket, and the mounted and polished longitudinal section were examined in a scanning electron microscope (SEM). Evaluation details of the two samples are presented here.

The Fracture at the Brazed Bracket

The bracket, braze metal, and tube geometry at the fracture plane are shown in Fig. 5. The energy dispersive spectroscopy (EDS) analysis of the braze metal, obtained from an area between the bracket and tube (Fig. 5b), is tabulated in Fig. 5c. The data reveal significant dilution from iron (Fe). Presence of Mn indicates the braze metal is a Cu Mn base alloy, in view of the fact that neither the tube nor the bracket contain Mn additions of more than 0.5 wt.%.

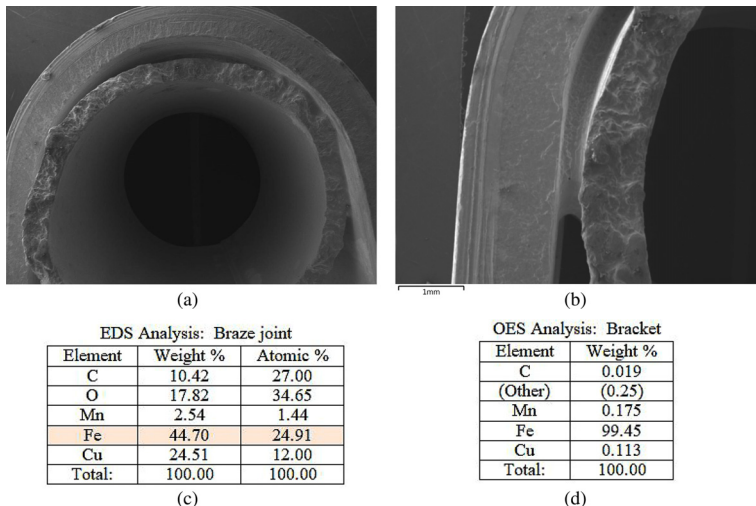
FIG. 5 View of tube fracture at end of brazed bracket. (a) Bracket/tube assembly at fracture plane, approximately 20 $\times$. (b) Braze joint, 50 $\times$. (c) EDS analysis of braze metal in braze joint. (d) OES analysis of bracket. (Note: EDS data are semiquantitative, standardless analyses.)

Fig. 6 provides examples of the appearance and composition of the braze alloy covering the fracture surface. The backscattered electron (BSE) images shown in **Fig. 6a** and **b** reveal predominant bright areas of Cu rich braze alloy, together with dark dendritic like patches of Cu Mn compound. Fe is present in both phases. This is attributed to Fe dilution into the braze rather than pickup from the tube alloy below the braze metal.

From the SEM/EDS evidence in **Fig. 5** and **Fig. 6**, it would appear that a Cu Mn base alloy was employed for brazing, and that a combination of high temperature and moderate cooling rate (i.e., not quenched) resulted in Cu Fe Mn dendrite formation on the fracture surface. Appearance of the braze alloy features clearly indicate that solidification took place in a vacuum environment (low oxygen levels in EDS data) on a free surface within the cracked or fractured tube wall.

The Mounted Longitudinal Section of the Tube Wall

Fig. 7 illustrates the appearance of the polished tube wall section in the SEM. The widespread distribution of Cu throughout the microstructure in the vicinity of the fracture is highlighted in **Fig. 7a**. One finding from the EDS data, away from the fracture surface, was that concentration of Cu, especially in remote grain bound aries, increases to such an extent that Mn was not measurable by EDS (detection limit is approximately 0.2 wt. %).

FIG. 6 Braze alloy covering tube surface. Analysis of bright and dark areas (BSE

images) indicate braze is a Cu Mn base alloy. (a) Braze metal covering faceted fracture, 300x. (b) Detail of braze metal on fracture, 1.5kx. (c) EDS analysis of bright Cu rich area in (b). (d) EDS analysis of dark phase in (b).

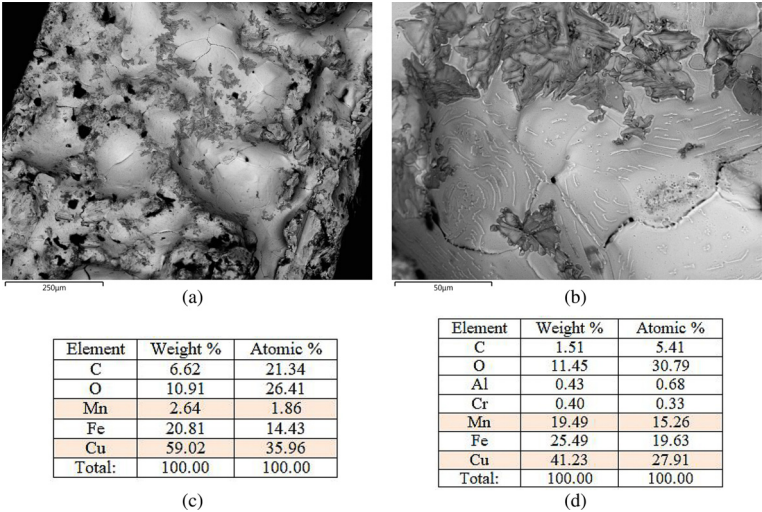
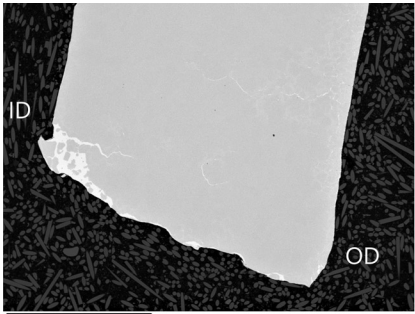


FIG. 7 SEM and EDS data obtained from mounted and polished longitudinal section of tube wall. (a) Fracture section at lower end of sample: Bright phase is Cu, 200x. (b) EDS detail of braze metal near ID. (c) Remote intergranular crack, 2.0kx. (d) EDS: Almost pure Cu at crack tip. (e) OD of tube away from fracture: Braze metal attack, 500x. (f) EDS analysis of tube alloy and OES composition of 1008 steel (for reference).

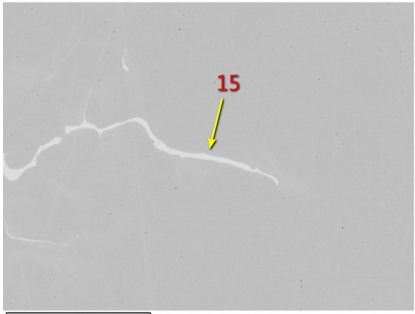


(a)

Analysis from large braze alloy area near ID:

Element	Weight %	Atomic %
C	2.33	11.09
Fe	8.80	9.00
Cu	88.87	79.91
Total:	100.00	100.00

(b)



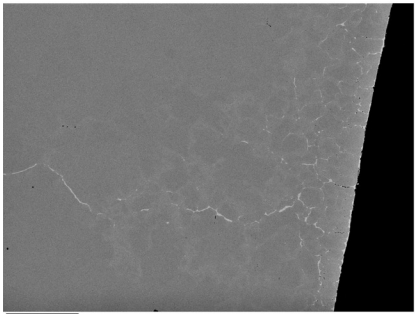
(c)

Location 15 in (c):

Element	Weight %	Atomic %
*C	2.33	11.14
Fe	3.50	3.61
Cu	94.17	85.25
Total:	100.00	100.00

*Carbon is from surface contamination.

(d)



(e)

Element	Weight %	Atomic %
*C	1.75	7.66
Fe	98.25	92.34
Total:	100.00	100.00

*Carbon is from surface contamination.

Composition of 1008 steel (OES):

Element	Weight %
C	0.1 max
Mn	0.3-0.5
P	0.04 max
S	0.05 max
Fe	98.25
Total:	100.00

(f)

The intergranular decoration by Cu away from the fracture, shown in [Fig. 7e](#), has resulted from the braze alloy running along the OD surface from the bracket braze location. One prominent example of this attack is an intergranular

propagation across the field of view, parallel to the tube wall fracture and 600 μm to 700 μm away from it.

CAUSE OF FAILURE AND POTENTIAL CONTRIBUTING MECHANISMS

In many cases, failure analysis investigators are not always made aware of pertinent information regarding the product and its manufacturing process. However, constructive deductions can be made from the experimental data, and potential mechanisms contributing to the tube failure can be discussed.

Macrophotography

Condition of the brazed component appeared to be good, except for the single failure at one bracket in the middle of the “S” bend. There was no evidence of oxidation, either on the brazed joints or on the steel tube and brackets. Based on these observations, the most probable production method is vacuum or environmentally controlled furnace brazing. In this process, the tube is set in a tooling jig to hold the components in place. Braze alloy is applied to joint areas in wire or tape form. Brazing occurs as either a batch or continuous furnace operation. It is not clear how the braze joint became overheated, but some interruption in processing may have exposed this tube to the heat source for an extended period of time.

Metallography and Hardness Data

The complete recrystallization of weld areas and the tube wall demonstrate exposure to elevated temperature. Employing the knowledge that recrystallization temperature for most metals is in the range of 0.3 to 0.4 T_m , and for alloys it is closer to 0.5 T_m , we obtain an estimated recrystallization temperature for the 1008 steel (almost a pure metal) of approximately 700°C to 750°C. This estimate is a similar temperature range for common brazing scenarios. However, it should be noted that recrystallization is a function of time and temperature; the aforementioned calculation is based on the assumption of a nominal 1 h exposure.

Scanning Electron Microscopy and Energy Dispersive Spectroscopy

The EDS data of the braze metal and identification of Mn rich dendrites solidified on the tube fracture surface indicate that a Cu Mn alloy was used for brazing. This type of alloy has good high temperature strength, excellent wetting and gap filling properties, and does not require a flux. Certainly, the EDS data indicate a high percentage of Cu in the braze material. With this evidence, and based on data for the Cu/Mn/Co alloy composition, we can estimate the braze temperature to be in the range of 960°C to 1030°C. These brazing materials are popular not only for their attractive brazing properties but also because they offer nontoxic alternatives to the more expensive silver base alloys [1].

As a fraction of the absolute melting point of the tube material, this brazing temperature range is at, or about, 0.7 T_m . Excessive dwell time or overtemperature during brazing will certainly result in reaction with the host metal. Grain boundary areas will be a prime target for liquid metal attack.

PROBABLE FAILURE MODE

Furnace braze tooling is designed to accommodate the thermal behavior of the parts held in the tooling. In an ideal situation, tolerances between the tooling and the components are close to an interference fit at brazing temperature. The parts are thus held in position while the braze metal is in the liquid state. In this case, it is possible with the “S” shaped component that prolonged localized heating of the central bracket created tensile stress in the vicinity of the braze area. This stress then activated the process of liquid metal embrittlement (LME) and caused the tube wall to separate or to weaken the area to such an extent that the tube joint failed during subsequent handling.

The SEM images show that braze metal did not tear or fracture but solidified as a free surface on the tube fracture face. At the point of fracture, the initial crack rapidly developed into a near rupture situation. In the vicinity of this rupture, braze metal, running over the tube surface, set off more intergranular attacks in the microstructure. Numerous theories have been put forward to define the mechanism of LME [2–12]. Some claim it is due to wetting of the grain boundaries by a liquid metal phase [6,12], while others attribute the migration of the low melting point metal by vapor transport mechanisms [4]. Regardless of the underlying transport mechanism, key characteristics of LME are as follows [2,5]: For this type of intergranular attack, it is generally believed that the liquid metal, which must wet the surface of the host material to overcome the protective nature of a surface oxide, influences the binding energy between grains and results in crack propagation without preceding deformation ahead of the crack. The embrittling metal can travel down a grain boundary as vapor, thus creating crack propagation rates on the order of several meters per second. The presence of stress, induced in the component either by internal or external sources, is also required.

The widespread grain boundary attack near the OD surface (Fig. 7e) may have been the result of some remaining residual stress from the strip forming history prior to seam welding into a tube. The single large penetration of copper, along a crack parallel to the main fracture, may have been a competing crack development site that eventually was stopped by relief of stress when the main crack ran to completion across the entire tube cross section.

From the metallographic and SEM evidence, the most probable source of the embrittlement was caused by a processing anomaly. Brazing alone generally will not result in LME, especially when performed properly. Either excessive brazing temperatures or excessive brazing alloy, or both, were used in this tube joining application. Nearly pure copper was observed in the SEM/EDS investigation of a secondary crack tip located deep within the wall thickness. This may be due to processing temperatures that exceeded the normal brazing temperature associated with the braze alloy. Excessive braze alloy could be responsible for both the unusual appearance of the braze joints and the relatively large pools of copper present along, and around, the grain boundaries. This factor, in combination with temperature

exposure at the high end of the desired range, could account for the microstructures observed in the investigation.

Conclusions

The failure of the tube assembly was due to LME or, at the very least, copper induced cracking. This condition is caused by wetting and penetration of copper, a low melting point metal, into the steel tubing along its grain boundaries. This resulted in decreased strength and ductility in the final tube assembly. The source for the liquid copper was determined to be the braze alloy. Brazing alone should not cause LME to occur. Possible causes of failure include excessive dwell time or overtemperature associated with the braze cycle. The tube assembly apparently was subjected to abnormally high temperatures during, or after, the furnace brazing operation for the braze alloy chemistry used.

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A New Approach for Color Metallography: Through Controlled Conditions to Objective Microstructure Analysis of Low-Carbon Steels by LePera-Etching

Citation

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ABSTRACT

Although metallography is a very old discipline, its importance is growing steadily. High-performance materials need an accurate evaluation of the microstructure to guarantee the ever-tightening specifications in the range of properties and to control the production process. With the continuously growing demand of well-tailored microstructures, in most cases with smaller and more complex constituents of defined geometry, the requirements for reproducible metallography are also more and more challenging. In this contribution, we want to show how new trends in metallography can help scientists and engineers ensure reproducibility in their daily work with etching routines governed by controlled and monitored systems. The presented setup keeps all critical parameters during color-etching constant. Thus the reproducibility and homogeneity of LePera etching on low-alloyed steel can be guaranteed. Furthermore, a parameter study for LePera

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was conducted not only to show the application range of critical parameters such as temperature and time but also to find the best parameter setting for an automated phase-separation toward image analysis and microstructural classification. With the help of a focused ion beam, quantitative correlations of the etching mechanism can also be derived.

Keywords

color metallography, LOM, FIB, low-alloyed steels, LePera

Introduction

Steel is used in a broad field of applications due to its wide range of properties [1,2]. In addition to chemical composition, this range of properties is significantly influenced by the microstructure resulting from the manufacturing process [3]. In order to meet the ever more specific and demanding applications, alloys are constantly being further developed, resulting in increasingly complex microstructures. In the field of metallography, these have to be investigated in order to understand and optimize specific properties [4–6]. For this purpose, it is necessary to understand the etching processes used for revealing the microstructure. This knowledge will facilitate comprehension of the mechanisms that are taking place and allow them to be adapted to continued microstructural development.

In order to visualize the different phases and constituents in steels and to carry out a characterization, color etching is recommended because this technique produces a different coloration of the phases [7]. The color etching method was introduced in 1951 by Klemm using metabisulfites [8]. A saturated aqueous sodium thiosulfate solution with addition of potassium metabisulfite to contrast cementite and retained austenite in a ferritic matrix was used. Consequently, this etching solution was also used in many other fields of application [8].

Beraha also used metabisulfites in his work. In 1966, he published an etching process using potassium metabisulfite, which was used on stainless and heat resistant steels [9]. In 1967, he developed two etching solutions that enhanced the representation of chemical and physical inhomogeneities in iron, carbon, and alloy steels [10]. The first etchant was based on an aqueous 3 % potassium metabisulfite solution, which produced a sulfide film on the various structural components but did not attack carbides, nitrides, or chromium layers, for example.

One way of visualizing physical inhomogeneities, such as subgrain boundaries, was made possible by the use of potassium metabisulfite and sodium thiosulfate [10]. Beraha applied hydrochloric acid to decompose the potassium metabisulfite used to form a sulfide film on the ferritic, martensitic, and austenitic grains [11]. This led to clearly visible grain boundaries and a colored matrix. By developing further color etching solutions [12,13], Beraha demonstrated the important potential of these color contrasting methods and their wide field of application.

LePera also used sulfide films for his work. However, he used a picric acid based etching solution with aqueous sodium metabisulfite to reveal the different

phases in high strength dual phase steels. A detailed quantitative microstructural analysis was a problem due to the lack of contrast [14]. He emphasized that the specimen must not have a deformation layer on its surface. He explained how this can be removed by alternating polishing and etching with nital. The etching was based on a 1 % aqueous sodium bisulfite solution and 4 % alcoholic picric acid mixed in equal parts. The sample was inserted immediately after mixing the two components for 7 s to 12 s. The sample was then cleaned with ethanol, which produced a blue orange color on its surface. As a result, bainite appeared black, ferrite appeared light brown, and martensite appeared white. Etching time was extended if the obtained contrast was not sufficient.

LePera [15] showed the same etchant on high strength, chrome containing dual phase steels. The deformation layer was removed with picral instead of nital, and the final polishing was carried out with 0.05 μm of gamma aluminum oxide. The etching time was only 5 s to 6 s, after which the polished surface exhibited a shiny gold/orange to blue color. Depending on the composition of the sample (e. g., for samples with a chromium content of less than 0.1 wt.%), an insufficient contrast between the phases could occur. The contrast could be adjusted with a longer etching time. In addition, it was shown that the etchant was suitable for standard dual phase steels with a martensite content of up to 60 %; without using this etching technique, the contrast between martensite and ferrite is lost.

Girault, Jacques, and Harlet [16] investigated the metallographic etching methods for transformation induced plasticity (TRIP) steels, which contain the four phases ferrite, bainite, martensite, and retained austenite, in order to be able to study the individual phases by electron microscopy. The methods published to date did not allow adequate differentiation of the phases, but colors generated by LePera's etching method were effective. In the light microscope, ferrite appeared green blue, bainite was brown, and martensite as well as retained austenite appeared white. It was found that in some cases the ratio of the two etching components determined the coloring. This could be compensated for by adding more picric acid solution to the etching solution, when all phases appeared with a bluish contrast, and more sodium bisulfite solution, when the microstructure appeared brown. The addition was done within the framework of the quantities stated previously. In addition, the coloring was largely determined by the steel composition. This was strongly influenced by carbon and silicon content. With an increasing carbon content, the color of ferrite changed from blue to brown. At a silicon content of more than 1 wt.%, it was possible to differentiate ferrite and bainite.

Angeli, Kneissl, and Füreder [17] carried out model considerations on selective etching and contrasting of structure and color etchings on low alloyed dual phase and TRIP steels to gain further insight into the etching processes and the subsequent phase identification. In order to be able to estimate the dissolution behavior of the multiphase steels, potential differences were calculated for ferrite, bainite, martensite, and retained austenite, neglecting variations in alloying elements.

In addition to chemical composition, defects in the regular lattice structure, such as dislocations, also reduce the potential. As a result, the martensite phase was classified as less noble than austenite. To verify this theory, a nital etching was performed. This led to a slightly etched structure on the surface of the martensite. The austenite phase was unaffected. Bainite and ferrite were removed by the etching agent, with no removal of the carbides. In addition, an influence of grain orientation could be observed with ferrite because the [100] orientation was attacked less than the [111] orientation. In order to check the model considerations for anodic color etching as well, three etching processes were applied to TRIP steels. Anodic color etching according to Klemm and LePera, as well as cathodic color etching with molybdenum acid, were carried out. It was shown that LePera etching is only applicable as pure color etching on samples with a carbon content of about 0.6 wt.%.

Füreder et al. used Beraha and LePera etching methods to perform quantitative microstructural characterization on low alloyed dual phase steels [18]. They obtained coloration corresponding to other published results [14–17]. However, it was shown that the use of the Beraha etchant roughened the martensite and only a slight layer formation occurred, whereas ferrite did not experience any etching attack. In contrast, LePera etching revealed a layer on both ferrite and martensite, which was less developed than on the ferritic grains. Scanning electron microscope (SEM) examinations also revealed that layer formation does not, as commonly thought, occur in both color etching techniques. Metal attack was more pronounced in Beraha etching than in LePera etching. Therefore, it was assumed that the contrast due to Beraha etching was mainly caused by etching and that the contrast due to Lepera etching was caused in equal parts by removal and layer formation.

Shui et al. used a variety of etching techniques to quantify the bainite and martensite volume fractions of a molybdenum containing steel [19]. The samples used differed in the amount of bainite, which was 10 % and 45 %, respectively, and in the martensitic matrix. The success of the various etching techniques depended largely on the proportions of phases present, which is why the choice of etching agent should be based on this. The LePera etching was the only one that could cover all orientations of the ferrite in both structural states, as well as the widest range of bainite content. However, the contrast between bainite and martensite was not always sufficient for a differentiation. This problem could be solved by an additional etching with sodium metabisulfite, which increased the contrast. For high bainite contents, Klemm's etching solution and boiling alkaline sodium picrate solution provided the best contrast.

Marder and Benscoter showed that the result of quantitative microstructural analysis depends to a large extent on the etching method used. The volume fraction of the second phase in a dual phase steel is lower when using LePera etching solution than in producing a phase contrast with 1 % aqueous sodium metabisulfite solution because the LePera method also makes low carbon martensite and ferrite appear colored [20].

Bandoh, Matsumura, and Sakuma [21] developed an etching process consisting of sodium thiosulfate, sodium thiosulfate pentahydrate, picral, and nital to

characterize the structure of high strength steels. It turned out that there are a number of factors influencing the etching result. The most important factors were the choice of the embedding material, etching time, etching temperature, and the air humidity of the drying air. After polishing, the samples were air dried with a relative humidity of 45 % maximum. The various etching components were degassed using argon, and the sample was etched immediately after mixing the etching solution. The sample surface turned brown at first, but the coloring of the different phases followed after a few seconds. After cleaning the samples, they were dried with warm air containing a relative humidity of less than 40 %. This was necessary because the etching could not be fixed otherwise. As a result, the ferrite appeared blue, martensite and retained austenite yellow, and the bainite brown.

In her work, Pokorný showed the influence of surface condition on the etching effect of copper primary etchants on steel [22]. In addition to the mechanical influences resulting from sample preparation, the chemical influences were also a decisive factor in the etching success. These influences included a chemically adsorbed layer that formed during polishing. This did not have any influence on revealing microstructure such as produced by nital etching, for example because the layer was removed by the etching process. The color etchings used were less prone to removal from the sample material, which is why etching with sodium thiosulfate, for example, was sensitive to mechanical and chemical influences. The findings on the chemically adsorbed layer were examined in more detail by means of Auger electron spectroscopy and secondary ion mass spectroscopy, showing that the layer consisted of oxygen metal compounds and also sulfur or ammonium compounds. The decisive factor was whether the last polishing step took place in town or country air. The resulting increase in the surface chemical potential caused by the adsorbed elements suppressed the etching process. This phenomenon could be observed in copper etching as freshly polished samples produced poorer results than samples stored in air or vacuum for 20 h.

Britz et al. [23] demonstrated that the etching process according to Beraha [24] could be explained more precisely with the development of an in situ etching setup. Etching was performed on normalized dual phase steel samples using a potassium metabisulfite solution. In the first few seconds, the contrast of the second phase dominates, and the matrix was only slightly colored after one minute. With increasing etching time, the colors of the interference layer became clearer, and the entire color spectrum could be observed. By repeated performance of this test, it could be shown that the color etching according to Beraha could be carried out reproducibly and, thus, reproducible results could be achieved. For this, however, it was essential that constant test conditions prevailed. Furthermore, it was confirmed that crystallographic orientation had a significant influence on the coloring and that the same orientations are dyed in the same color.

From the literature review, it becomes clear that the method of color etching is still very important today and that there are a number of factors that affect the etching result. In addition, it has been shown that there has been no systematic

investigation of these influences and that the etching results are therefore not always satisfactory or reproducible. In order to enable a reproducible characterization of the increasingly complex structure formation, and for a better understanding, the reactions and factors influencing them must be investigated more closely. The aim of the present work is to carry out a systematic parameter study by establishing a suitable etching setup. With this equipment, the influences on etching according to LePera can be monitored to aid in the understanding of the partly competing mechanisms that take place. Through this approach, reproducible etching results can be obtained.

Experimental

This study was conducted on low carbon, thermomechanically rolled samples that were taken from the same sheet. The carbon equivalent was 0.42 % at a carbon content of 0.06 wt.%. The microstructure consisted of a ferritic matrix with a small amount of martensite/austenite (MA) and bainitic features with cementite agglomeration.

Before the samples were etched, a classical metallographic treatment consisting of grinding and polishing with steps of 6 μm , 3 μm , and 1 μm was applied. The final polishing step was carried out with gamma alumina solution according to LePera [15]. For a reproducible and homogeneous result, it is important that the samples are not mounted.

Finally, the samples were deoxidized for 1 min. in an ultrasonic bath using a 1 % citric acid solution to ensure the same starting condition for all samples. To prevent the samples from further oxidation, the samples had to be etched immediately after this step.

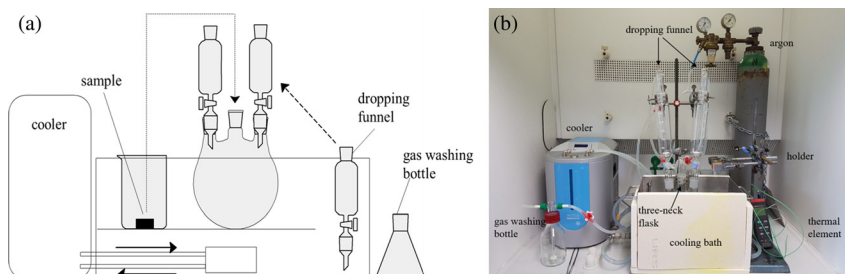
According to LePera [14], the etching used consisted of 4.6 % picral solution in ethanol and 1 % aqueous sodium metabisulfite solution, which were mixed in a ratio of 1:1 before etching.

It is of utmost importance that concentration of the picral solution is adjusted precisely. Therefore, the solution was prepared by the authors in a drying process taking into account the necessary safety precautions. For drying, small quantities of phlegmatized (stabilized) picric acid were weighed with the aid of a Teflon spatula and dried in an empty desiccator for at least two days using drying beads. The dry matter of the acid was then determined by weighing and immediately dissolved in ethanol. The desired concentration could thus be maintained by the amount of ethanol used and finally controlled by refractometry based on a generated calibration line.

In order to compare the etching results of the parameter study with one another, the parameters and influences found in preliminary tests were varied or kept constant in a controlled manner. The test setup shown in Fig. 1 was developed for this purpose.

To carry out etching at different temperatures, a temperature control bath was required, which facilitated temperature adjustment and equilibrium of the etching solutions. In addition, the released reaction heat was dissipated through the bath, which necessitated a relatively large filling volume. Preheating of the solutions was also carried out in this bath. These requirements were met by means of an

FIG. 1 Setup for the parameter study to keep all parameters constant. The main components are a cooler, a cooling/heating bath, and a closed system with argon connection. (a) Schematic representation of the setup. (b) Representation of the actual setup in the laboratory.



immersible platform, with adjustable height, and a circulating pump, which ensured good mixing of the fluid volume in the bath. The temperature was controlled by means of a recirculating cooler that fed the isolated water bath. To ensure a steady temperature, an additional circulating pump in combination with an integrated cooling coil was also installed. For etching treatments at temperatures higher than room temperature, a heating plate with temperature control was used. Argon was needed to keep environmental factors such as the atmosphere or humidity constant. Etching was therefore carried out under an argon atmosphere and with degassed etching solutions. The acid solutions were degassed in dropping funnels by passing argon through the gas washing bottle tops for at least 1 h. This step was carried out in the tempering bath in order to prepare the etching solutions at the desired temperature. In order to be able to carry out the entire etching process without oxygen, the ethanol, which was used to stop the etching process, was also degassed.

The three necked flask was placed in the temperature control bath, closed with two stoppers, and rinsed with argon via a cut off gas washing bottle attachment. The samples were also heated after preparation and deoxidation to prevent a drop in temperature at the sample surface. The temperatures of the samples and etching solutions were checked by means of thermocouples. When the desired temperature was reached, and after degassing, the dropping funnels were taken from the temperature control bath and placed on the three necked flask; 50 ml aliquots of each of the two LePera etch components were allowed to run into the three necked flask, and the sample was quickly immersed in the etchant. The etching solution was stirred several times during the etching period. The sample was then dipped into the degassed ethanol to stop the etching process, thoroughly cleaned, and dried (without further rinsing) with compressed air. Sample drying must be carried out continuously to avoid formation of undesired residues on the sample surface. These residues are harmful and interfere with the coloring.

Three different experimental series will be presented in the present work:

- Constant time ($t = 135$ s) and varying temperature (0°C to 60°C)
- Constant temperature ($T = 5^{\circ}\text{C}$) and varying longer etching times (40 s to 420 s)
- Constant temperature ($T = 40^{\circ}\text{C}$) and varying shorter etching times (20 s to 40 s)

These parameter ranges were determined from preliminary investigations.

Subsequent microscope evaluations of the etching results was carried out using both a light microscope and a laser scanning microscope (LSM). A dual beam microscope was used for high resolution microscopy and evaluation of the interference layer. The samples were sputter coated with gold palladium to permit a more precise measurement of the interference layer. In addition, a platinum protective layer was applied on the region of interest to protect the sample surface and to avoid rounding of the edges, which was required for the focused ion beam (FIB) cross sections. For the SEM images, an acceleration voltage of 5 kV and currents of 0.69 nA (for secondary electron detector) and 86 pA (for the in lens detector) were used. The FIB cross sections were carried out with an acceleration voltage of 30 kV and beam currents of 20 nA, 6.5 nA, 2.7 nA, and 0.9 nA. To characterize a representative microstructure, all micrographs and cross sections shown were recorded at one quarter of the sheet thickness.

The etching process was optimized for light microscopy because this method mainly was used for the later microstructural characterization. The necessary phase separation was carried out using image processing software. In addition, smoothing, sharpness, and binary image functions were used for image optimization within the software. The phase separation was achieved by gray value thresholding. No manual corrections were made.

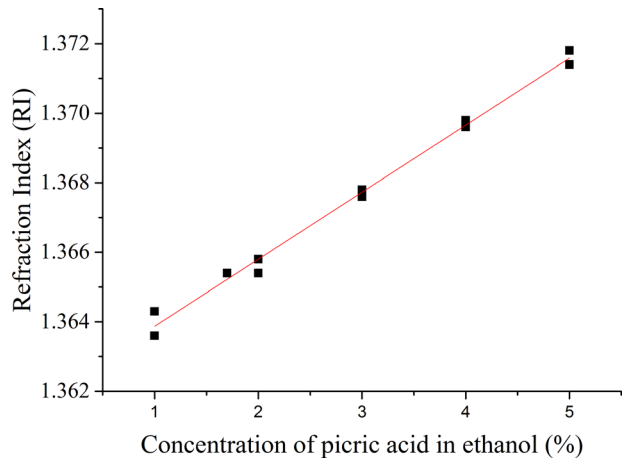
Results

As preliminary tests have shown, it was not possible to adjust the concentration of the picric solution by simply adding ethanol or using ready made solutions to ensure that a reproducible etching procedure was carried out. This problem was solved by drying the picric acid as described in the experimental section. Apart from the safety aspect, adjusting the concentration in this way was relatively time consuming but of utmost importance. This is why the obtained picric concentration was determined by refractometry.

The preparation of several defined concentrations by drying the picric acid and subsequent refractive index measurements showed that refractive index (RI) increases linearly with concentration (Fig. 2). RI could therefore be employed to verify the concentration of etching mixtures used in the trials described and illustrated here.

As the concentration of the etching solution has a major influence on the final result, we used the calibration curve to verify our concentration and, after preliminary tests, set it to 4.6 %.

FIG. 2 A plot of concentration versus refractive index (RI) of different alcohol solutions of picric acid; RI increases linearly with the concentration.



Based on the etching described in the experimental part according to LePera [14,15], a systematic parameter study was carried out using the developed etching setup (Fig. 1). By varying the parameters' temperature and time, reproducible etching results were obtained, which enabled subsequent reliable and reproducible phase differentiation and microstructure characterization. For this purpose, it was necessary to investigate the influence of the respective parameters on etching by keeping one of them constant.

First, the influence of temperature on LePera etching was investigated. For this purpose, picric acid with a reference concentration of 4.6 % was used. The etching time was also kept constant at 135 s. Fig. 3 shows examples obtained at selected temperatures.

According to Britz et al. [23], the resulting interference layer at 0°C only shows a slight coloring of the ferrite and indicates its orientation. Furthermore, the grain boundaries are clearly visible and the MAs appear white (Fig. 4). However, the bainitic constituent is not sufficiently contrasted, which makes it difficult to distinguish it clearly from the matrix.

By increasing the temperature to 5°C (Fig. 3a), the influence of orientation increases significantly, and the cementite agglomerations appear black. The grain boundaries remain clearly contrasted, which permits differentiation between ferrite and the cementite agglomerations. This etching result is comparable to etching at 20°C, but the orientation effect is less pronounced. This is shown in Fig. 3b. The bainitic grains remain in black contrasted and can easily be distinguished from the matrix, while the grain boundaries are no longer revealed as a prominent feature. The (white) MAs can easily be distinguished.

FIG. 3 Variation of temperature using LePera etchant for an etching time of 135 s. Samples etched at (a) 5°C, (b) 20°C, (c) 30°C, and at (d) 40°C.

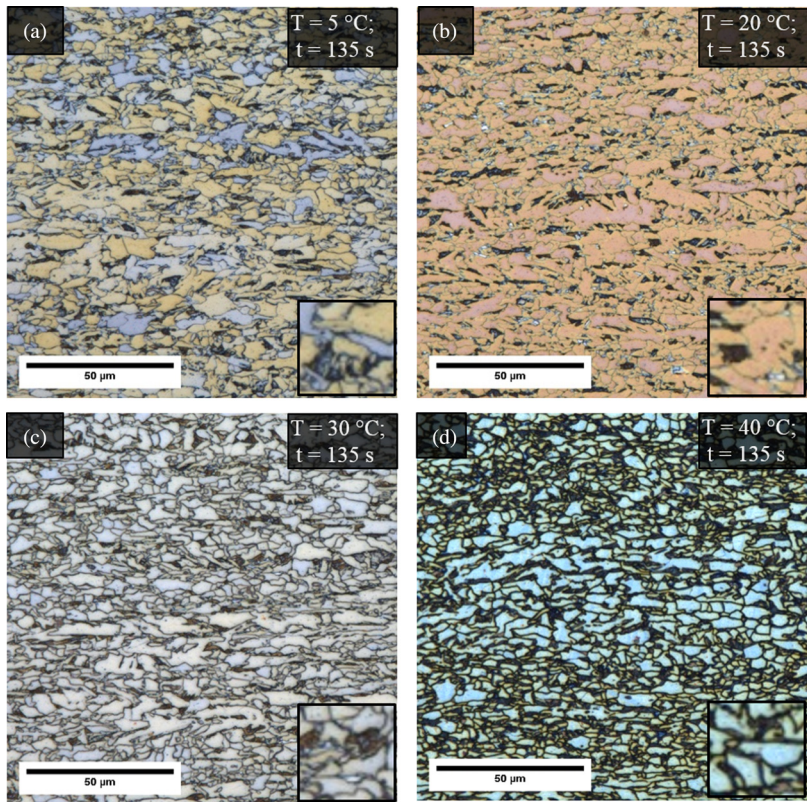
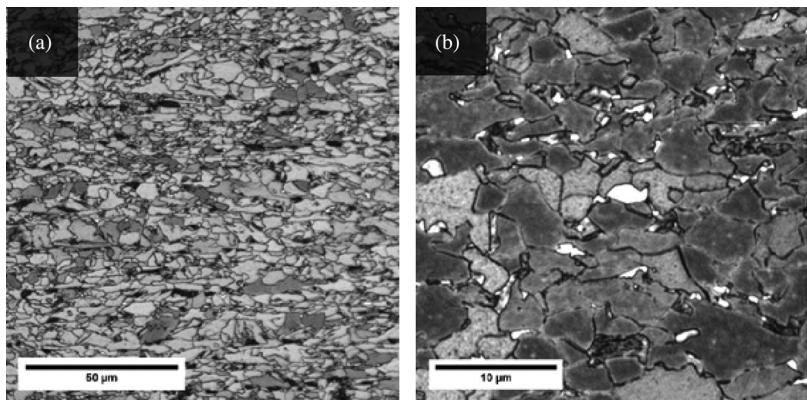


FIG. 4 Exemplary and representative representation of the MAs in the LSM at two different magnifications (a, b) etched at 0°C and for 135 s. The MAs appears brilliant white.



After etching at 30°C, the coloration is almost completely lost ([Fig. 3c](#)). This indicates completion of the first color order [23,25]. Grain boundaries are clearly shown, and the bainitic grains appear distinctly separated from the matrix. However, contrast of the MAs is almost completely lost.

At 40°C, the sample appears to be significantly overetched, as shown in [Fig. 3d](#). The grain boundaries and bainitic grains can hardly be distinguished from each other. There is little influence of orientation, and color gradients within the larger ferrite grains occur.

Contrast of the MAs depends to a large extent on the etching result obtained. Characterization of MAs in a light microscope color image is difficult. By using an LSM, the contrast of the MAs due to their reflectivity is much better ([Fig. 4](#)). A further advantage of the LSM is its higher resolution (up to 125 nm), so that there are also advantages for later phase separation and quantification.

In a further series of tests, the etching temperature is kept constant at 5°C and the etching time is systematically varied ([Fig. 5](#)). In the first series of measurements, the temperature variations provided an optimum etching result, with regard to contrast of the grain boundaries and the second phase, and showed a pronounced orientation contrast.

After an etching time of 40 s ([Fig. 5a](#)), the grain boundaries are clearly visible and a clear orientation contrast can be seen, which overlays the contrast of the MAs. The cementite arrangements appear to be relatively weakly contrasted.

An increase in the etching time to 180 s ([Fig. 5b](#)) produces a better contrast between the grain boundaries and the other constituents. A further increase of the etching time to 240 s results in overetching of the sample ([Fig. 5c](#)). The grain boundaries are barely visible, and there is a strong influence of grain orientation. The black bainitic grains appear to be clearly separated from the matrix.

An etching time of 420 s ([Fig. 5d](#)) leads to a loss of orientation contrast. A continuous blue coloration across the microstructure precludes clear phase separation. However, grain boundaries remain well contrasted. This is a sign of the completion of the first color order.

Samples etched at 5°C and in a time range of 120 s to 240 s are especially suitable for phase separation and gray scale thresholding. A good example of this is shown in [Fig. 6](#). With this preparation, quantification of the different constituents and measurement of grain size is facilitated.

The previous test series, illustrating etching changes produced by varying temperature, do not clearly show whether the results (at relatively high temperatures) are promising for a reproducible and reliable etching condition. These are essential requirements for true microstructure characterization. Therefore, in a further series of tests, the etching temperature was kept constant at 40°C, and the etching time was systematically varied. Results are presented in [Fig. 7](#).

[Fig. 7a](#) shows the result of sample exposure to LePera etchant at 40°C for 20 s. The microstructure displays a clear contrast between ferrite and MA (white areas). However, grain boundaries are relatively weak. An increase in the etching time to 40 s

FIG. 5 Variation of etching time using LePera etchant at 5°C. Samples etched for (a) 40 s, (b) 180 s, (c) 240 s, and (d) 420 s.

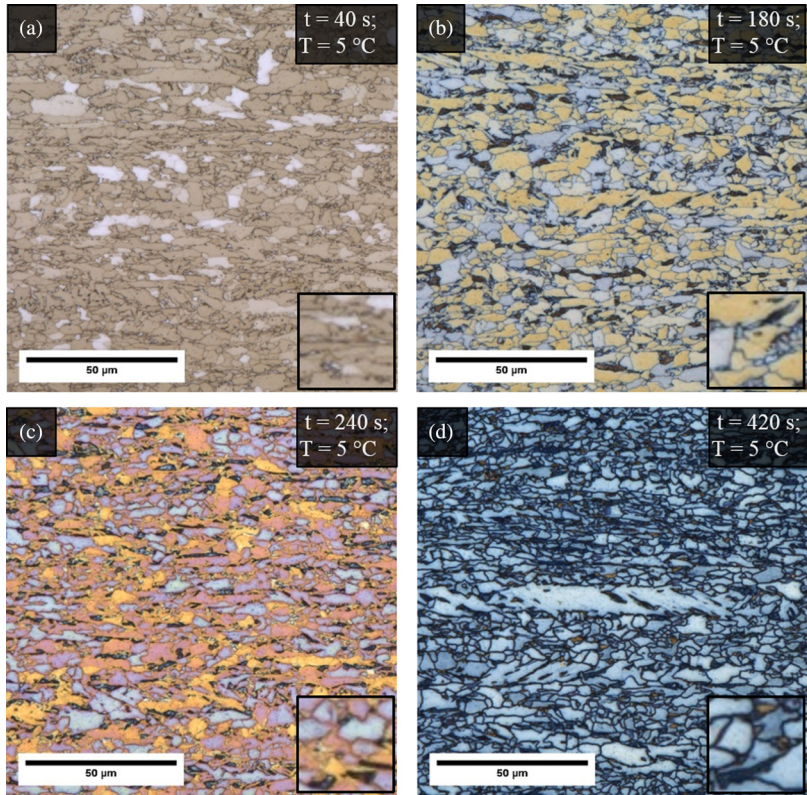


FIG. 6 Image of etched microstructure (a) at 5°C for 120 s and (b) gray scale thresholding.

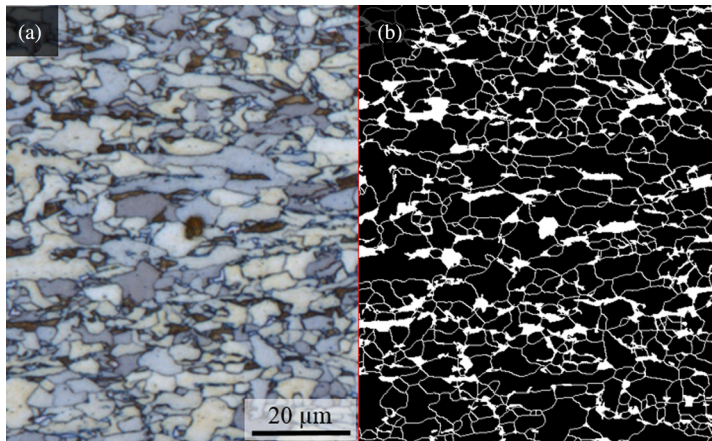
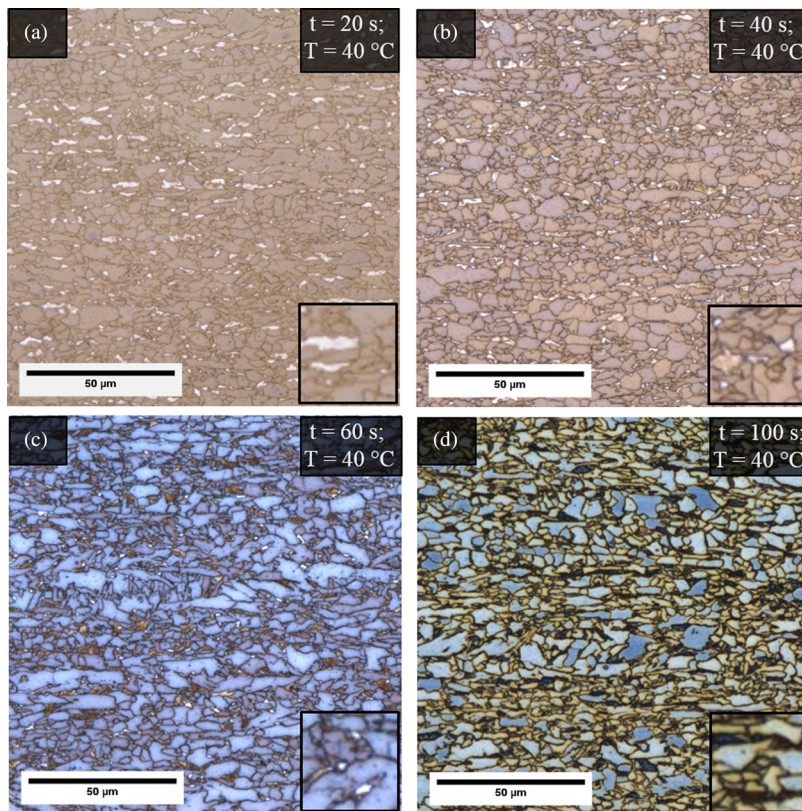


FIG. 7 Variation of etching time using LePera etchant at a temperature of 40°C. Samples etched for (a) 20 s, (b) 40 s, (c) 60 s, and (d) 100 s.



increases the influence of orientation, and the grain boundaries are more distinct (Fig. 7b). The MAs are still clearly contrasted, but bainite is difficult to distinguish from the ferritic matrix. By increasing the etching time to 60 s, the bainitic grains are recognizable as brown areas between the ferrite grains and the MA (Fig. 7c). A slight orientation effect is still visible, and the grain boundaries are clearly contrasted.

With an etching time of 100 s, the influence of orientation becomes even clearer as some ferrite grains differentiate themselves from the rather yellowish matrix by their blue coloring (Fig. 7d). Due to the strong overetching, the grain boundaries appear excessively wide and black. The bainitic phase, also black, becomes lost in this broad grain boundary zone. In addition, the MAs are no longer visible.

Appearance of the 100 s microstructure is similar to the result from etching for 120 s. The only differences are that the influence of orientation is no longer visible, and the matrix appears blue.

In order to be able to assess the coloring and etching attack obtained in these tests more precisely, FIB cross sectional investigations were carried out on all samples within the areas shown in Fig. 3, 5, and 7. In this way, the etching process could be investigated with regard to metal removal rate from ferrite grains. That is, the relative difference between metal removal from ferrite and the MA or carbide agglomerations (which are nobler than ferrite and therefore not attacked, or only mildly attacked) and the resulting interference layer thickness. Fig. 8 shows one example of an FIB cross section.

Fig. 8 shows the result of etching at 50°C for 135 s. The grain boundaries appear clearly and stand out. Particularly noticeable is the strong etching attack, which produces a distinct topography (Fig. 8b). It can be seen that the MAs stand above the matrix and show little or no etching attack. In Fig. 8c, the interference layer (marked in red) can be seen. For this temperature, a thin interference layer already covers the second phase.

Metal removal and layer thickness measurements made by the FIB cross sections show that both etching and interference layer formation follow a linear trend (Fig. 9). It should be noted that there is significant scatter associated with the measurements. Etch removal rate is strongly influenced by the number of Mas, and cementite arrangements is small. Furthermore, there is a rather strong influence of grain orientation, which can cause the layer thickness to vary by about 15 % to 25 %.

FIG. 8 Details of an FIB cross section, made from a sample etched at 50°C for 135 s. (a) A 52° tilted SEM image of the exact specimen location of the FIB cross section. Grain boundaries are clearly visible. The region of interest is covered by a protective layer of platinum (Pt). (b) Overview of the cross section. There is a distinct topography that has been created by the etching attack. The difference in height between the MAs/bainite and the ferrite is clearly visible. The interference layer can be recognized by the thin dark layer (marked in red). (c) Detailed view of the interference layer (marked in red) on top of the steel substrate. At this temperature, a layer on the second phase is already visible. To protect the surface from any damage, samples were sputtered with a very thin layer of gold (bright line above the interference layer). To determine the relative removal rate, the height between the elevated second phase and the ferrite is measured (yellow arrow).

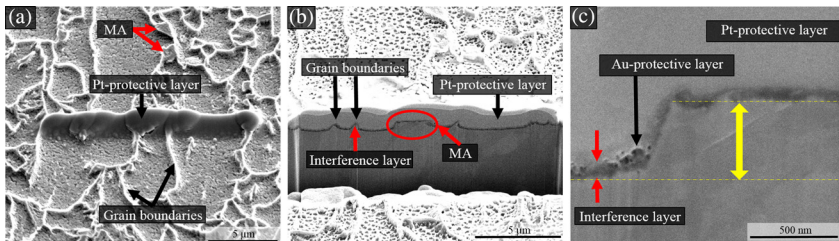
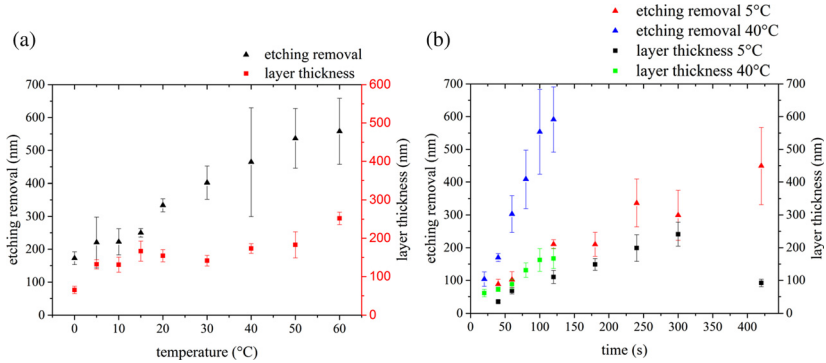


FIG. 9 Comparison of etching removal and layer thickness measurements for the series of measurements carried out. (a) Etching removal and layer thickness measurements for the first series after 135 s (variation of temperature). (b) Total etching removal and layer thickness measurements for the second and third measurement series (variation of time).



The interference layer thicknesses were measured only on the ferrite grains. The FIB cross sections in the parameter study show that longer etching times and higher etching temperatures also lead to the formation of interference layers on the cementite agglomerations.

Discussion

SOURCES OF ERROR

The aim of the present work is to establish a reproducible color etching technique using an etchant based on the LePera solution for low alloy, low carbon steels. General reported experience of the metallography community with the LePera solution as well as the preliminary tests in the presented study have arrived at similar conclusions. It has been clearly demonstrated that both the etching temperature and etching time have a significant influence on the appearance of the resulting microstructure.

Great importance has always been attached to sample preparation because all etching procedures require a good final polish on the sample that is free of artifacts and surface deformation. For etching with the LePera solution, it is now clear that concentration of the picric acid solution has a significant influence on the result and on reproducibility. In the literature referenced in the introduction, concentration is normally stated as 4 %, but it is never discussed further and apparently never measured. In addition, commercially acquired picric acid deviates considerably from the nominal concentration; according to one manufacturer, actual concentration is not checked. Etching results, which are not regarded as satisfactory in the literature, may therefore be due to use of a high or low picric acid concentration.

In the present study, an attempt was made to obtain a constant and defined picric acid concentration by drying the picric acid. The drying process used and the determination of the concentration by refractometry were suitable to ensure a constant concentration. It is important to keep the temperature constant for these measurements because RI is dependent on temperature [26]. However, it cannot be completely ruled out that a very small amount of water is still contained in the picric acid after drying, which leads to a deviation of the specified concentration or the listed calibration line. In order to achieve completely anhydrous picric acid crystals (see "Safety Precautions"), further drying processes must be used in order to create a statistically verified RI calibration line. One conceivable possibility for this would be drying under an inert gas atmosphere at a moderately higher temperature or measuring the residual water content with a corresponding measuring method.

Nevertheless, it is not recommended to dry the picric acid in the daily work routine because of safety issues. Prepared solutions of picric acid are commercially available and can be used instead of an accurately known concentration. The refractometer then only has to be used to determine the actual concentration.

In future work, the possibility of adjusting temperature or time to compensate for differences in the picric acid will be investigated.

Safety Precautions

In view of the fact that the experimental work described in this study has involved handling and drying picric acid to a degree far greater than involved with the normal laboratory preparation of picral, the authors consider it of great importance to point out the safety risks associated with picric acid. The material safety data sheet should be reviewed, and all safety precautions should be observed. Only persons who are trained in handling hazardous substances should work with picric acid.

PARAMETER STUDY

After a constant and known picric acid concentration is ensured, a systematic parameter study could be carried out with the etching apparatus described earlier and illustrated in Fig. 1.

In addition to temperature control, the etching setup (Fig. 1) also had to offer the capability of degassing the etching solutions and etching under an argon atmosphere in order to provide the essential requirements for the parameter study and to establish a reproducible etching technique. Temperature control of the etching solutions is carried out by dropping funnels in the thermal bath, whereby the temperature of the solutions can be set very precisely. However, the dropping funnels have to be removed from the bath for etching, which is why the heating or cooling process is interrupted and therefore influenced by the ambient temperature.

Depending on the desired etching temperature, the solution is either heated up or cooled down. This influence of ambient temperature can be minimized by maintaining a constant etching procedure, but it cannot be eliminated. To eliminate this influence altogether, it would be necessary to find a way to carry out the etching

process without removing the etching solutions from the thermal bath or to keep the ambient temperature constant in order to be able to adapt the etching process accordingly by coordinated overheating/undercooling of the etching solutions.

Degassing of the etching solutions is accomplished by passing argon through them, which physically removes any dissolved oxygen. This method of degassing is to be regarded as reproducible because this process step can always be carried out in the same way. However, it cannot be ruled out that a small amount of oxygen still remains in the etching solutions after the degassing treatment. In order to remove this residual oxygen or to reduce the oxygen content to a minimum, the “freeze pump thaw method” must be used. For this method, the two etching components of LePera etching solution either have to be mixed before degassing, which is different from the conventional procedure, or the two components have to go through this procedure separately, which results in an even higher workload. A comparison of this degassing method with the one used in the present work showed no change in the etching results obtained and in the appearance of the interference layer. Therefore, the degassing performed using argon alone can be considered sufficient.

However, it should be noted that the etching process is not completely oxygen free because samples transfer from the bath to the three neck flask or removal of the sample from the bath leads to an etching interruption and contact with ambient air. Etching in the three necked flask (which was rinsed with argon before etching) proceeded under an argon atmosphere. This makes it possible to keep the atmosphere constant during etching and to exclude parameters such as the composition of air or the degree of humidity. For more general use, the flask opening places a limit on sample size, but this was not an issue in the present work.

As an alternative test setup, etching can be carried out in a glove box. This also allows etching under a controlled atmosphere. Furthermore, drying the samples after etching can also take place in this environment. However, the need for a temperature control bath and, if necessary, etching solution degassing leads to additional complexity and degree of difficulty for the operator. The test setup used in this work thus offers the possibility of carrying out etching procedures under defined and well controlled conditions. Environmental influences are greatly reduced, and the most important parameters etching time, etching temperature, and acid concentration (not demonstrated in this work) can be adjusted.

In the systematic parameter study, the first series of measurements was carried out under a constant etching time of 135 s and variable temperatures. The results obtained show that for temperatures up to 30°C, the selected etching time leads to satisfactory etching results, depending on the objective. Time differs considerably from the times given in the literature, which are limited to a few seconds [14–16]. This can be due to the sample material used (which, depending on the microstructure, has a significant influence on the etching result) as well as to the picric acid concentration. Furthermore, it is evident that low temperatures lead to a very good contrast of the entire microstructure. A temperature of 5°C is recommended because the second phase and the matrix are well contrasted (Fig. 3). In addition,

the whole process is slower, which enables better etching control. Etching temperatures of 15°C or 20°C result in a loss of grain boundary contrast (Fig. 3d and e). This is caused by growth of an interference layer thickness on the ferrite grains, which compensates for the different removal depths produced by the etching.

In general, it should be noted that the etching times required for good results for a phase separation become shorter and shorter as the temperature rises. This makes controlled etching increasingly difficult also in terms of reproducibility even if the results at 40°C give very good results under the given conditions, which is also in accordance with Fig. 9b. Temperatures above 40°C are not recommended for low alloy steels because over etching is inevitable.

As a result of the good etching results at low temperatures, the second test series was carried out at a constant etching temperature of 5°C with variable etching time. Due to the short etching times of 40 s and 60 s, only a very thin interference layer was produced, which did not permit microstructure characterization. Results of the first series at 5°C and results of the second series are in agreement.

Developing of a consistent color etching process has resulted in the ability to contrast microstructures with good reproducibility and with clear phase definition.

Longer etching times initially lead to added contrast of the second phase with loss of grain boundaries. Subsequently, the influence of orientation is lost, and the second phase cannot be clearly distinguished from the grain boundaries.

In order to determine whether good phase contrast is possible at higher temperatures, a third measurement series was carried out at a constant temperature of 40°C. An etching time of 20 s clearly distinguished the matrix from the second phase without sharply contrasting the grain boundaries. An increase in the etching time to 40 s made the grain boundaries clearer. The MAs were not affected by the etching attack, so they still appeared white. The SEM evaluation showed that etching times of 60 s and longer led to removal of the anodic areas within the bainitic grain cementite clusters, which is why light scattering occurred and these areas appeared black in the light microscope. The coloring obtained in the present work deviated to some extent from the color reproductions and descriptions given in the literature. In the works of LePera [14,15], the ferrite is contrasted light brown, the bainite black, and the martensite white. The results of the parameter study showed that the ferritic matrix, depending on the etching parameters used, covered a large part of the color spectrum and that no phase could be assigned to a definite coloring for the given sample set. Only the contrast of the MAs, which appeared white, corresponded to the LePera literature [14,15]. This means that a direct comparison with the mostly high alloyed and high carbon steel samples as well as with samples with a different microstructure was not possible.

The results of etchings under a range of temperature show that, especially at lower temperatures, they are promising with regard to successful and reproducible microstructural characterization and phase quantification.

Because of the strong dependency between chemical composition and microstructure and the etching result, the presented work can give guidance on how to

adjust the parameters, but a general time, temperature, and concentration range for the etching solution cannot be given. To minimize the amount of work needed to define the optimum parameter settings for a given sample, a workflow as described by Britz et al. [23] with a flowcell in combination with the presented parameter study, is recommended.

INTERFERENCE LAYER THICKNESS MEASUREMENTS

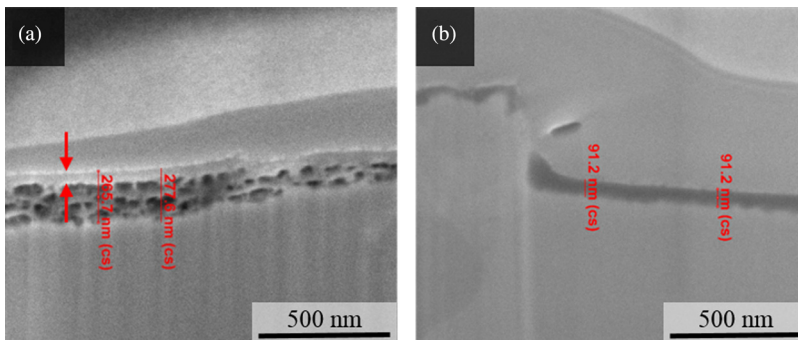
Layer thickness measurements made with FIB cross sections show that both etching depth and interference layer formation follow a linear trend (Fig. 9). It should be noted that the measurements exhibit significant scatter. Etch removal is more affected because the number of MAs and cementite arrangements is small. Furthermore, a rather strong influence of orientation can be shown, which causes the layer thickness to vary by about 15 % to 25 % and thus also leads to fluctuations in the measurements.

The interference layer thicknesses were measured only on the ferrite grains. The FIB cross sections in the parameter study show that longer etching times and higher etching temperatures also lead to the formation of interference layers on the cementite clusters. This finding is in agreement with the results published by Füreder [18], which show that the formation of layers on the nobler structural components can also occur.

It should be mentioned that some FIB cross sections and SEM images showed a high degree of porosity in the interference layer (Fig. 10).

In the literature reviewed in the introduction, this observation is not mentioned by other investigators. Previous works [23,27] show that the interference layers obtained by Beraha etching and Klemm etching appear very compact. The physically

FIG. 10 SEM image of two FIB cross sections of an interference layer after LePera etching. (a) Porous layer with a small dense layer (red arrows) on top after etching at 5°C for 300 s. (b) Very dense interference layer after etching at 5°C for 420 s.



dissolved oxygen present in the etching solutions was assumed to be a possible cause of the pores. The pores could not be reduced by degassing the etching solutions and by etching under an argon atmosphere. The degassing of the etching solutions is already mentioned in the work of Bandoh, Matsumura, and Sakuma [21] without any further explanation, such as a comparison with etching solutions that have not been degassed or FIB cross sectional investigations. Other possible causes for the pores in the interference layer of LePera etching may be due to the hydrogen released during etching, which is deposited in the interference layer. Furthermore, it is possible that hydrogen adsorbs on the sample surface before the etching process despite intensive and multistage sample preparation and etch solution purification. Despite this, pore formation occurs.

These causes for pore formation could possibly be resolved by expanding the etching structure by means of an ultrasonic finger, which removes the adsorbed hydrogen and strongly mixes the etching solution. The fact that an adsorbed layer can influence the etching result is demonstrated by Pokorný [22]. In this study, a layer of oxygen metal compounds and also sulfur and ammonium compounds, was investigated. Pokorný showed that this layer had the potential to suppress etching. Therefore, it cannot be ruled out that an adsorbed layer of hydrogen, or other elements, could have a significant effect on etching results.

Based on the second series of tests, which were carried out at a constant etching temperature of 5°C, it was found that the pores in the interference layer are covered by a dense layer (see arrows in Fig. 10a). This can be compared with the layer of Fig. 10b produced at the same temperature but for a different etching time (420 s). By measuring the dense layer fraction for the etching results from the second measurement series, it can be determined that this also develops linearly with the time. From this knowledge, it can be assumed that coloration results from the dense interference layer portion and the porosity influence the intensity of the color. Thus, porosity does not have a significant influence on the coloring obtained or on the etching result viewed in the light microscope.

It is not possible to determine clearly from previous findings on the etching mechanism to what extent removal and the formation of the interference layer act together. It is assumed that both processes occur simultaneously in equilibrium. First of all, the oxide layer, which varies in thickness depending on grain orientation and therefore contributes to the potential difference, is removed. Furthermore, the iron ions that are removed from the sample material then form the interference layer as a deposit by joining with sodium metabisulfite. The equilibrium balance of this process can be confirmed by etching with a higher concentration of sodium metabisulfite, which is responsible for the formation of the layer. An increase in concentration will lead to an increase in layer thickness.

Based on these findings, it can be concluded that a variation in the concentration of the components of the etching solution (consciously, or unconsciously due to missing measurement) can lead to significant changes in the contrast or coloration especially of the ferrite.

Furthermore, the sample center is slightly less contrasted than the sample corners and edges. The reason for this is probably some segregation within the sheet thickness or a stronger etching reaction in these areas because the iron ions are easier to dissolve and a better ion exchange can take place due to the higher dynamics of the liquid. Therefore, it is very important to take micrographs away from the edges of the sample.

Conclusions

The aim of the present work was to develop and demonstrate a reproducible color etching method based on the original work of LePera with a color etchant on low alloy, low carbon steels. This goal was achieved by development of an etching setup in which variables could be controlled and by different parameter studies. The etching setup allowed the environmental parameters to be kept constant as far as possible and the two key parameters of etching time and etching temperature to be varied. Refractometry was used to determine the exact concentration of picric acid used. This made it possible to rule out this variable as a possible cause of fluctuations in the etching results indicated in the literature. In the parameter study, the parameters mentioned previously finally were used to optimize the etching process in terms of reproducibility and etching results to enable quantitative microstructure analyses. The results obtained from this study showed that removal and interference layer formation take place in equilibrium and that these mechanisms follow a linear trend with respect to the etching time and etching temperature used.

The results of these experiments on low alloy steels, employing the developed etching setup for use with LePera's etchant, have demonstrated that it is possible to etch materials that react in a sensitive manner. These materials can be treated in a targeted and reproducible way to generate colored microstructures suitable for reliable phase identification and quantification.

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Effects of Strain Rate on the Mechanical Properties and Fracture Characteristics of a Dual Phase 980 Steel

Citation

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ABSTRACT

Dual phase (DP) steels are being increasingly used as structural components in the automotive industry owing to their combination of high strength and moderate ductility. Formability and crash resistance are important mechanical properties that expose the steel to high strain rates. Fracture characteristics of a DP steel at strain rates spanning seven orders of magnitude are investigated using quantitative fractography. The DP steel investigated in this study is comprised of a continuous matrix of martensite with interspersed ferrite islands. The data reveal that the flow stress, uniform strain, and ductility are substantially higher at the higher strain rates. Quantitative fractography, employed to understand the fracture characteristics, shows an increase in the extent of ductile fracture at high strain rates. In this presentation, the results demonstrating high strain rates facilitating dimpled ductile fracture and/or increased resistance to faceted fracture are discussed.

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Keywords

dual phase steels, advanced high strength sheet steels, fracture mechanism, stereology, quantitative fractography, high strain rates, quasistatic and dynamic testing

Introduction

The increase in the demand for fuel efficiency and vehicle weight reduction led to the development of advanced high strength sheet (AHSS) steels that have been used extensively in the automotive industry [1–3]. Cold rolled dual phase (DP) steels possessing a combination of high strength, good formability, continuous yielding, and high initial strain hardening were among the first class of AHSS steels developed to manufacture structural members of the automobile. The tensile strength of DP steels can be varied across a wide range from 400 MPa to 1400 MPa by tuning the processing conditions and the underlying microstructure. Hence, these steels have gained considerable technological importance [4,5].

Microstructure of DP steels is comprised of the ferrite phase (imparting ductility and toughness) and the martensite phase (imparting strength and hardness) with some trace amounts of bainite and retained austenite phases depending on the processing routes followed. The volume fractions, morphology, and spatial distributions of these phases govern the mechanical properties and fracture mechanisms of DP steels [5].

Extensive studies have been conducted in the past to understand the role of the morphology and distribution of different phases on the deformation and failure of DP steels [3,6–13]. Tensile tests coupled with detailed damage accumulation and propagation studies have revealed martensite cracking as the predominant mechanism that *initiates* failure, irrespective of the spatial distribution of the phases [6,7,14]. Additionally, decohesion at the ferrite/martensite interface is recognized as one of the main damage propagation mechanisms [15,16]. Post failure fractography has also been performed to identify different failure modes linked to the underlying microstructure with varying volume fractions of the phases present [6,8,10,17,18]. However, most of these qualitative fractography and deformation mechanism studies have been conducted at considerably low strain rates. DP steels are exposed to strain rates of the order of 10^{-2} – 10^2 /s during sheet metal forming operations, and strain rates of the order of 10^2 – 10^4 /s can be reached under an automotive crash condition. As the volume fraction of martensite is increased, the strain rate sensitivity of the steel is shown to reduce as martensite is comparatively strain rate insensitive [19,20]. A previous study conducted by Wang et al. [20] on the dynamic behavior of various grades of DP steels observed that with an increase in the volume fraction and connectivity of martensite, the fracture surfaces *qualitatively* show increased plasticity at higher strain rates depicted by the increased dimpled appearance. Although the deformation and constitutive modeling have been extensively studied with some qualitative fractography [16,19–23], there have been no quantitative

studies on fracture surfaces to study how microstructure and phase morphologies affect the strain rate dependence of fracture micromechanisms in DP steels.

Another aspect of the intermediate to high strain rate study is measuring and determining the mechanical response that is representative of the material behavior. In the past, extensive studies have been conducted using different geometries across different strain rate regimes due to the lack of standard sheet metal geometry for testing in the Hopkinson bar experiments [16,22,23]. Properties such as ductility are geometry dependent; hence, using different geometries across strain rates makes direct comparison difficult [24–26]. In the current study, the geometry of all samples tested was kept constant (one that is compatible with the Hopkinson bar) across all strain rates to ensure that there is no discrepancy inherent in the ductilities and therefore to enable direct comparison.

The current study aims to bridge the existing gap by subjecting a cold rolled dual phase steel (with a nominal tensile strength of 980 MPa under quasistatic strain rates) to strain rates spanning seven orders of magnitude and measuring the deformation and fracture response using a constant specimen geometry. The strain rate sensitivity of fracture characteristics is investigated by conducting detailed quantitative measurements on the fractured surfaces. To the best of the authors’ knowledge, no previous studies have been conducted to quantify the fracture surfaces and understand the variations in the amount of fracture regions generated by different underlying mechanisms (such as void coalition/shear and so on) as a function of strain rate in DP 980 steels.

Experimental Procedure

MATERIAL CHARACTERISTICS AND TESTING

Tensile deformation experiments were performed on cold rolled, commercial 2 mm thick DP 980 steel provided by ArcelorMittal (USA) and processed in a water quenched continuous anneal line. The nominal chemistry of the steel is provided in Table 1. Tensile tests were performed to characterize mechanical properties across strain rates ranging from 10^{-4} /s to 2.5×10^3 /s. All tests were performed at room temperature. Postmortem fracture characteristics are quantified using stereological techniques.

Metallographic specimens were cut along the three orthogonal planes of the starting sheet steel, mounted, and polished using standard metallography techniques. The polished metallographic samples were etched using 2 % nital to reveal the ferrite

TABLE 1 Nominal chemistry of cold rolled DP 980 steel (in wt. %).

C	Mn	Si	Mo	Ti	Al	Nb
0.09	2.02	0.62	0.06	0.02	0.03	0.01

Note: Mn manganese; Si silicon; Mo molybdenum; Ti titanium; Al aluminum; and Nb niobium.

grain boundaries and the general ferrite martensite microstructure and 10 % aqueous solution of sodium metabisulfite to predominantly etch the martensitic structure. The resulting microstructure was analyzed using both optical and scanning electron microscopy. Fig. 1 depicts the typical microstructure along the longitudinal section (containing the rolling and thickness directions) with ferrite being the darker phase and martensite being the lighter phase with relief. The volume fraction of martensite was estimated to be $68 \% \pm 2 \%$ using statistically unbiased methods [27].

Tensile tests were performed on the as received steel using servo hydraulic and Hopkinson bar systems. Tensile tests in the strain rate range of $10^{-4}/s$ to $1/s$ were conducted using the servo hydraulic system at Georgia Tech. The properties were measured using a piezoelectric load cell, precisely calibrated LVDT cylinder, and strain gauges.

The intermediate strain rate tests (greater than $500/s$) were conducted on the Hopkinson bar at REL, MI. The stress values were computed from the stress pulses recorded by the strain gauges on the incident and transmitted bars. As the current study focuses on the fracture and deformation, for further information on the computation of the stress, strain, and strain rate from the pulses, the reader is referred to other detailed documents [28,29]. All high strain rate samples were speckled with a random pattern to record the deformation map and strain using digital image correlation (DIC) with the help of high speed digital cameras. In certain cases, the DIC failed and the final ductility was measured using the high speed camera images just before the fractured samples separated. Fig. 2 shows an example of the high speed camera images corresponding to (a) the undeformed specimen before the test began and image (b) used to compute the maximum elongation along the gage length beyond which clear necking is observed. This also corresponded to the highest stress when the mechanical instability leading to higher stresses (at low strains) are ignored; image (c) was used to compute the fracture elongation, and image (d) shows the two

FIG.1 SEM image of the longitudinal section of DP 980 steel showing continuous martensitic phase as the matrix with uniformly dispersed ferrite islands.

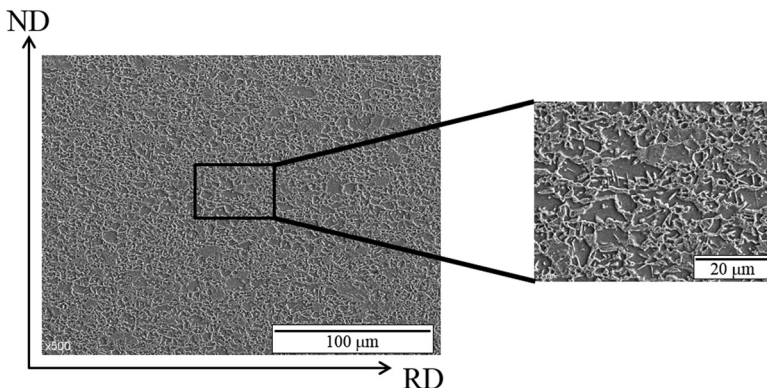
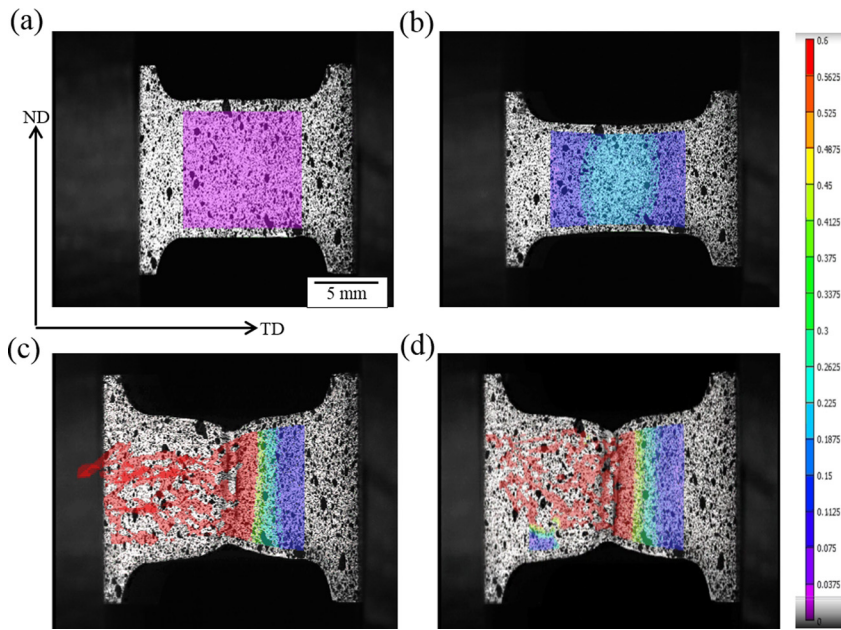


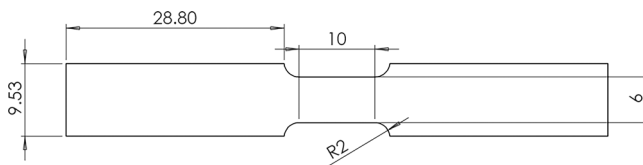
FIG. 2 High speed camera images showing the specimen corresponding to (a) no strain, undeformed specimen; (b) maximum elongation along gage length, onset of necking; (c) fracture elongation; and (d) beginning of separation after fracture at 2,500/s strain rate.



halves of the specimen fractured and separated. A strain map showing the extent of local strain at each point on the gage section is overlaid on the images. To maintain consistency, the stresses corresponding to the measured uniform strain (at quasistatic strain rates) and maximum elongation along the gage length (at high strain rates) are defined as the ultimate tensile strengths.

The geometry used for all the tests was compatible with the Hopkinson bar setup in this study and is shown in [Fig. 3](#). All servo hydraulic test properties were

FIG. 3 Tensile test geometry used for all servo hydraulic and Hopkinson bar experiments. All dimensions are in millimeters.



averaged over two samples. Only one specimen was tested at each strain rate in the Hopkinson bar. All specimens were precisely machined using a water jet to prevent excessive heating of the specimens. The gage sections of the samples were polished and deburred to remove any notches created during machining to ensure uniform deformation and prevent premature fracture. The direction of loading was along the transverse direction of the sheet steel.

QUANTITATIVE FRACTOGRAPHY

The influence of microstructure on the mechanical properties was better understood by carrying out detailed quantitative fractography of the failed specimens. The tensile fracture surfaces of the broken samples from the different strain rates were analyzed using a TESCAN (Georgia Tech) scanning electron microscope (SEM). The central region (one third) of each fracture surface was characterized. For this purpose, ten SEM fractographic images, each 140 μm by 140 μm in size, were grabbed in a systematic random manner for subsequent statistically unbiased quantitative characterization for each of the specimens investigated. All fracture surfaces exhibited ductile features consisting of dimples and regions comprised of secondary cracks, pullouts, and featureless areas hereafter called facets. Fig. 4a and b illustrate examples of nominal fractographic features observed across all samples.

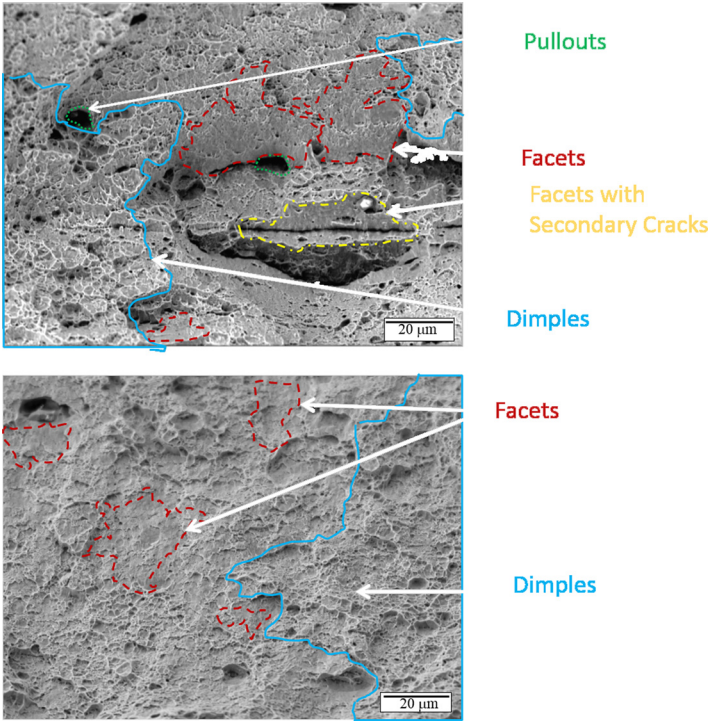
The area fractions of all classified features were estimated using statistically unbiased stereological techniques [30]. The area fractions of the different regions were quantified using point counting; the number densities were estimated using unbiased counting frames. The average dimple size was estimated by assuming that the dimples are projected as circles in the SEM image. Details of the derivation and estimation procedures are described elsewhere [27,30–35]. All features were estimated manually. Regions apart from the dimpled areas were further classified into flat featureless regions called facets, large round pullouts, and secondary parallel cracks called facets with secondary cracks. Since the quantitative study revealed that only about 15 % of the fracture surface on average was occupied by the pullouts and the secondary cracks, the area fractions of these features are not discussed in detail in the current study. The primary focus is on the variation of the fracture mechanism of DP 980 steel based on the dimple ductile and flat facet regions because the strain rates are varied.

Results and Discussions

MEASURED TENSILE PROPERTIES AT VARYING STRAIN RATES

Fig. 5 shows representative engineering stress strain curves summarizing the mechanical properties of the DP 980 steel under five strain rates spanning the range of $10^{-4}/\text{s}$ to $2.5 \times 10^3/\text{s}$. Continuous yielding with strain hardening was observed at the quasistatic strain rates (i.e., up to 1/s). At the strain rates of 500/s and 2,500/s, the jaggedness in high stresses associated with small strains is neglected. The jaggedness is believed to be attributed to mechanical instability arising from a

FIG. 4 (a) SEM image showing nominal fracture surface at 1/s strain rate characterized by ductile dimples (), secondary cracks (. . .), pullouts (.....), and flat featureless facets (). (b) Different field of view showing the areas characterized by facets and dimples.



nonconstant, still stabilizing strain rate as concluded and supported by previous studies [36,37]. The stress strain curves at the higher strain rates are extrapolated (using dashed lines) to the measured ductility values because the DIC failed at very large strains. The *shape* of the extrapolated curve is an approximation; however, the ductility values indicated are the measured values.

Fig. 6a–d shows the variation of the ultimate tensile strength, ductility, and uniform elongation and postuniform elongation as a function of the logarithm of the strain rate. All the measured properties are seen to increase at the higher strain rates. These results compare well with the previous studies conducted on similar DP grades [19,20].

Additionally, as shown in Fig. 6d, the postuniform elongation (difference between the total and uniform elongation) also increases substantially at strain rates of 500/s and greater and nearly triples when compared between the lowest and highest strain rate. Previous studies have shown increased localized deformation,

FIG. 5 Engineering stress strain curves for DP 980 steel across five strain rates.

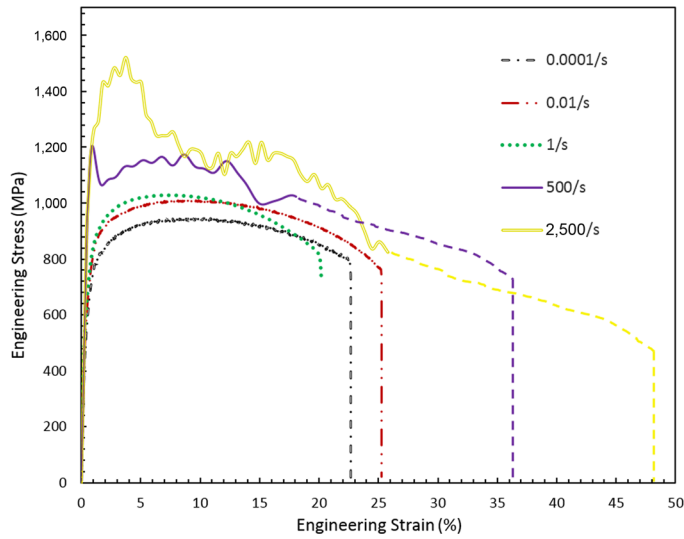
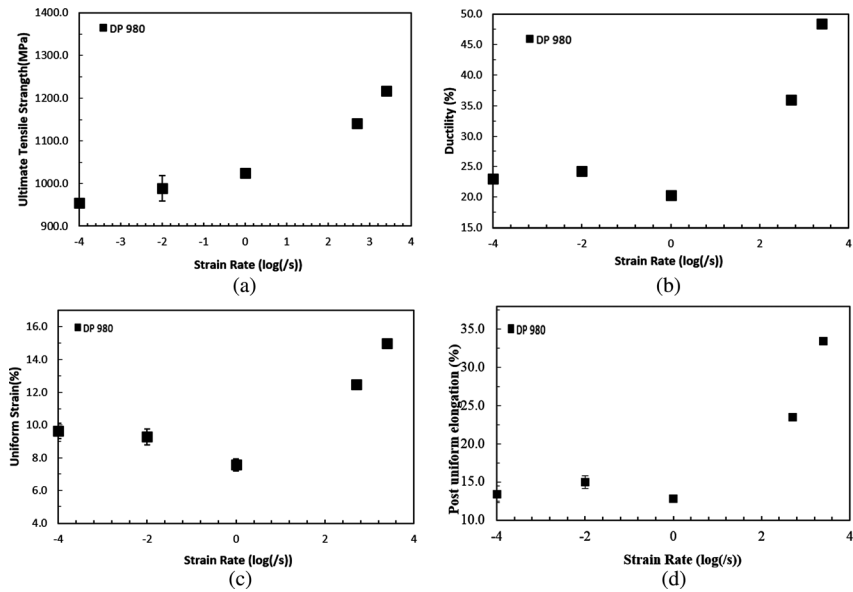


FIG. 6 Summary plots showing variation of (a) ultimate tensile stress (MPa), (b) ductility (%), (c) uniform elongation (%), and (d) post uniform elongation (%) as a function of strain rate.

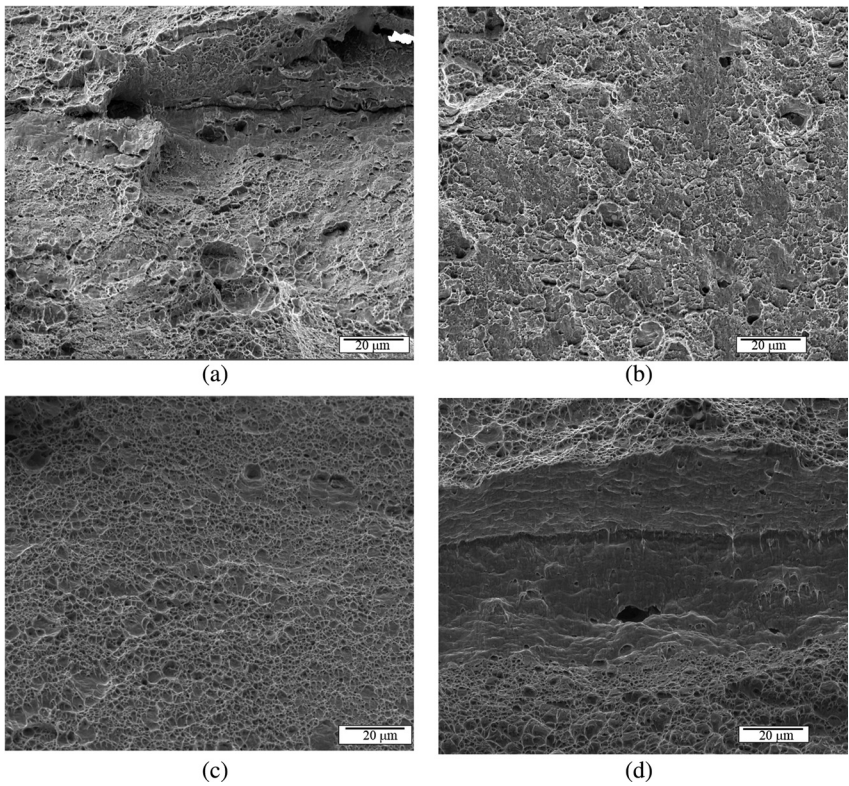


plasticity, and strain softening associated with an adiabatic increase in temperature arising due to the dynamic nature of the high strain rate tests [20,38,39]. Mason, Rosakis, and Ravichandran [40] have shown that approximately 80 % of the energy is converted to heat under dynamic rates of loading. The increase in the tensile strength at the highest strain rate with enhanced ductility, and increased post uniform elongation, all indicate that the steel strengthens and toughens at higher strain rates. Further study of the fracture surfaces was performed to shed more light on the failure mechanisms operating at various strain rates.

QUANTITATIVE FRACTOGRAPHY

Fig. 7 shows representative fracture surface images used for systematic quantification of the features defined in Fig. 4 for the lowest (Fig. 7a and b) and the highest (Fig. 7c and d) strain rates. All fracture surfaces showed large area fractions of dimpled ductile regions often attributed to the nucleation, growth, and coalescence of

FIG. 7 Representative SEM images of fractured tensile samples tested at (a), (b) 10^{-4} /s and (c), (d) 2,500/s showing the qualitative difference in the fracture morphology and revealing increasing ductile response at the highest strain rate.



voids. At the higher strain rates, qualitatively, the fracture surface portrays more dimpled ductile features, which has been observed by other authors for DP steels [11,20]. In comparing various studies, differences in chemistry and morphological features of the two constituent phases should be kept in mind. More importantly, this study focuses on quantitative characterization of fracture features as a function of strain rates.

Fig. 8 shows the variation of the area fraction of facets with secondary cracks (Fig. 8a), number density of pullouts (Fig. 8b), and average dimple size as a function of strain rate (Fig. 8c). The dimple sizes and the pullouts do not show a significant trend as a function of strain rate. The area fraction of secondary cracks shows an increase but occupies a small area of the fracture surfaces on average and hence is not discussed in detail here.

Fig. 9 shows the variation of the area fraction of ductile dimpled (Fig. 9a) and faceted (Fig. 9b) regions plotted as a function of the strain rate. Although the area fraction measurements indicate a large scatter, the mean values (based on a statistically unbiased method) appear to linearly increase with the logarithm of the strain rate. Similar observations have been reported in a press hardened, fully martensitic steel as well [41]. The area fraction of dimpled regions doubles at the highest strain rate, and the area fraction of faceted regions decreases by more than a factor of two when compared to the lowest strain rate.

These results illustrate that although some fracture features are not changing as a function of strain rate, the area fractions participating in dimpled fracture

FIG. 8 The variation of (a) facets with secondary cracks, (b) number density of pullouts, and (c) dimple size as a function of strain rate.

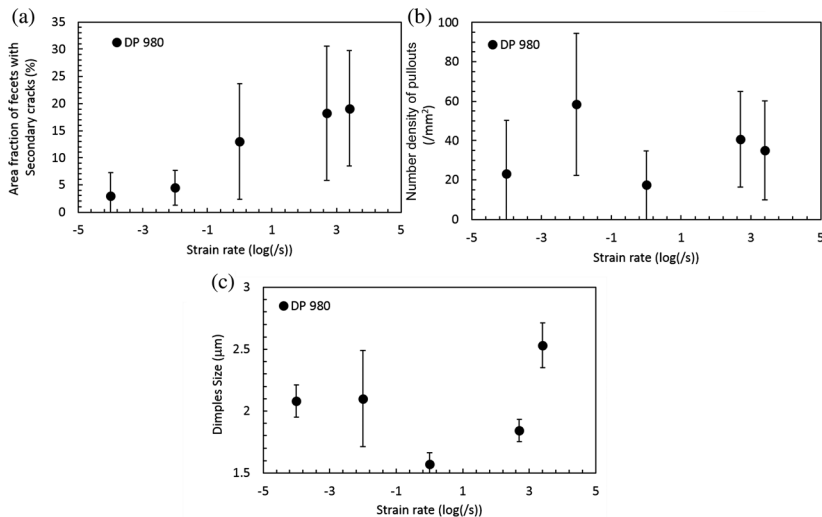
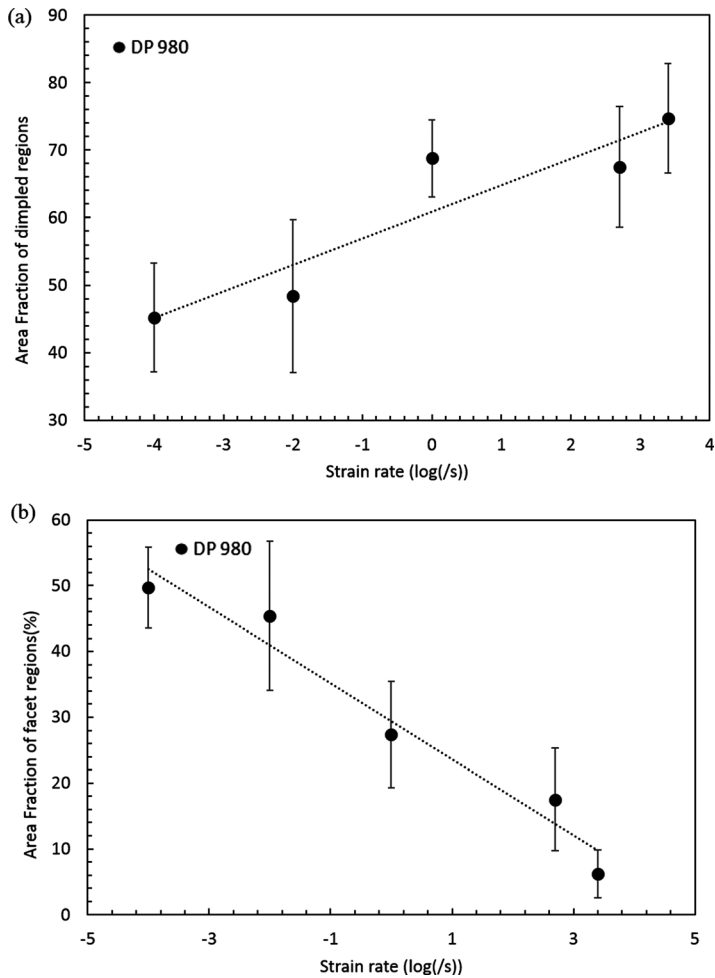


FIG. 9 Quantitative fractography results showing the variation of area fractions of (a) dimpled regions and (b) facets as a function of strain rate.



versus faceted fracture are strain rate sensitive, and the material exhibits deformation and failure via ductile fracture at high strain rates. These results correlate well with the corresponding increase in ductility measured at the higher strain rates (Fig. 6b).

Previous studies [6,11] under quasistatic strain rates have associated the observed facets (indicative of brittle failure) with the shear fracture of martensite. The decreasing area fraction of the facets with increase in strain rate observed in the current work suggests an increased resistance to the faceted mechanism of martensite fracture at higher strain rates, which in turn can affect ductility.

The current observations revealed that at higher strain rates, DP 980 shows an improved tensile strength and higher ductility, suggesting a toughening mechanism that is supported by an increase in the area fraction of ductile fracture regions at the higher strain rates. More detailed studies to discern the roles of individual constituent phases (ferrite and martensite) in promoting or inhibiting specific fracture micromechanisms at play at various strain rates currently are underway.

Conclusion

Uniaxial tensile tests conducted using samples of a constant geometry illustrate that the tensile strength, ductility, uniform strain, and postuniform strain increase at higher strain rates. Specimens tested at the higher strain rates (500/s to 2,500/s) showed considerable local deformation and plasticity that can be partly attributed to the adiabatic heating experienced during dynamic testing. The area fraction of the flat featureless facets associated with martensite fracture is reduced at high strain rates. The area fraction of dimpled regions increases as a function of strain rate, suggesting that ductile fracture mechanisms are activated and preferred under high strain rates.

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Microstructure Analysis for the Detection of Intermetallic Phases Predictive of Toughness and Corrosion Resistance in Duplex and Lean Duplex Steels

Citation

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ABSTRACT

Metallographic techniques for microstructure analysis are used for detection of the presence of intermetallic phases in duplex stainless steels to the extent that toughness or corrosion resistance is affected significantly. Duplex (austenitic-ferritic) stainless steels are susceptible to the formation of intermetallic compounds during exposures in the temperature range from approximately 600°F to 1,750°F (320°C to 955°C). ASTM A923, *Standard Test Methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic/Ferritic Stainless Steels*, Test Method A (Sodium Hydroxide Etch Test for Classification of Etch Structures of Duplex Stainless Steels), is used as a rapid screening method to detect the presence or absence of intermetallic phases to the extent that they are detrimental to the toughness and corrosion resistance of the material. The use of these etch structures depends on the test method for which the specimens are being screened. Recent revisions have incorporated additional alloys that may be evaluated in accordance with this specification. For lean

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duplex alloys (composed of 30 % to 70 % ferrite content with a typical alloy composition having chromium [Cr] greater than 17 % and molybdenum [Mo] less than 1 % and with additions of nickel, manganese, nitrogen, and controlled low carbon content as well as other alloying elements), ASTM [A1084-15a](#), *Standard Test Method for Detecting Detrimental Phases in Lean Duplex Austenitic/Ferritic Stainless Steels*, provides a metallographic procedure to show the appearance and approximate amount of chromium-containing detrimental phases in lean duplex stainless steel, despite the small size of the detrimental phases and the difficulty in achieving a fully reproducible etch structure. Microstructure results are correlated with toughness test and corrosion test results on a pass/fail basis. This newer process covers lean duplex stainless steels that were not well served by ASTM [A923](#).

Keywords

duplex stainless steels, lean duplex stainless steels, δ -phase in duplex stainless steels, microstructure of duplex stainless steels, ASTM [A923](#), ASTM [A1084](#)

Duplex stainless steels have an austenitic ferritic structure that provides improved resistance to corrosion, especially in chloride environments, in combination with significantly better mechanical properties than the typical austenitic materials.

All duplex stainless steels are susceptible to the formation of intermetallic compounds during exposures in the temperature range from approximately 600°F to 1,750°F (320°C to 955°C).

The speed of these precipitation reactions is a function of composition and thermomechanical history of each individual piece. The presence of these phases is detrimental to toughness and corrosion resistance. Correct heat treatment of duplex stainless steels can eliminate these detrimental phases in the mill product. Rapid cooling of the mill product provides the maximum resistance to formation of detrimental phases by subsequent thermal exposures [1].

These intermetallic phases can seriously undermine the corrosion resistance and toughness of the material. As an alternative to traditional time consuming and costly corrosion tests and Charpy impact fracture toughness tests, metallographic techniques for microstructure analysis can be used for rapid detection of the presence of these intermetallic phases in duplex stainless steels.

ASTM [A923](#) 14, *Standard Test Methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic/Ferritic Stainless Steels* [2], in its original publication, only provided applicability and acceptance standards for the early duplex grades UNS S31803 and UNS S32205.

Three test methods were provided: Test Method A, Sodium Hydroxide Etch Test for Classification of Etch Structures; Test Method B, Charpy Impact Test for Classification of Structures of Duplex Stainless Steels; and Test Method C, Ferric Chloride Corrosion Test for Classification of Structures of Duplex Stainless Steels. It should be noted that the Charpy impact tests in this standard and in

ASTM [A1084](#), *Standard Test Method for Detecting Detrimental Phases in Lean Duplex Austenitic/Ferritic Stainless Steels* [3], as well as not traditional Charpy V notch tests for general toughness but are specifically designed to be used in consideration of the detrimental effects of intermetallic phases.

The sodium hydroxide etch is used as a rapid screening method to detect the presence or absence of intermetallic phases to the extent that they are detrimental to the toughness and corrosion resistance of the material. The solution for etching is prepared by adding 40 g of reagent grade sodium hydroxide (NaOH) to 100 g of distilled water, and the polished specimen should be etched at approximately 1 V dc to 3 V dc for 5 s to 60 s.

The use of these etch structures depends on the test method for which the specimens are being screened. The sodium hydroxide etch test may be used as a stand alone test for the acceptance of material but not for rejection. This test method may also be used with other evaluation tests to provide a rapid method for identifying those specimens that are free of detrimental intermetallic phases as measured in these other tests. The sodium hydroxide etch test may be used to screen photomicrographs. ASTM [A923](#) provides photomicrographs to show classifications of etch structures of a particular stainless steel type that are equivalent to acceptable or to possibly nonacceptable performance for each practice.

In the unaffected structure ([Fig. 1](#) and [2](#)), the ferrite has been etched without revelation of intermetallic phase. The interphase boundaries are smooth.

FIG. 1 Unaffected structure in S31803: Longitudinal section (500× magnification before reproduction).

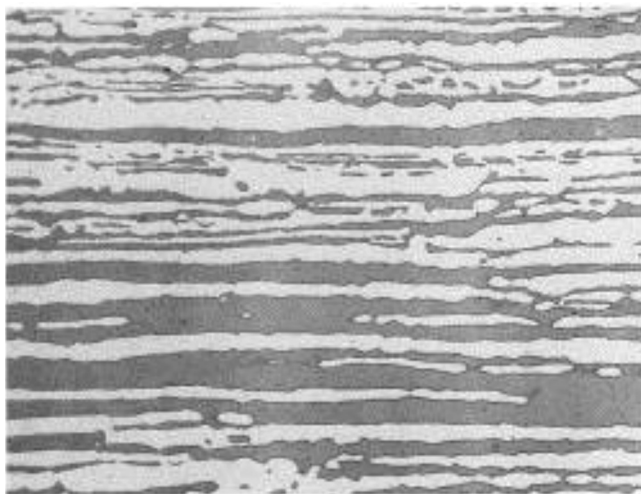
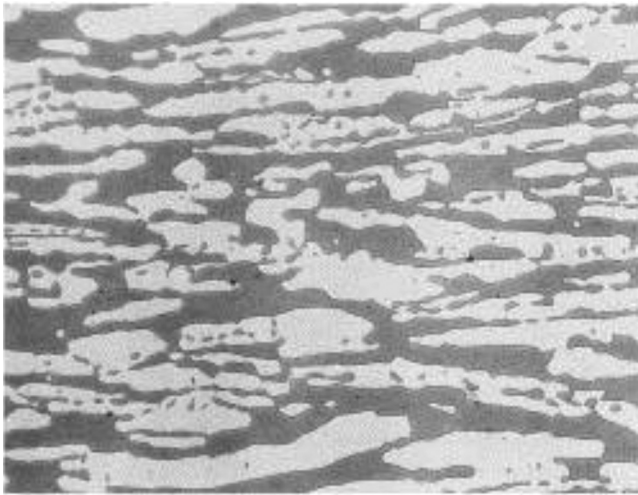


FIG. 2 Unaffected structure in S31803: Transverse section (500× magnification before reproduction).



In the possibly affected structure (Fig. 3 and 4), the ferrite has been etched with isolated indications of possible intermetallic phase. The interphase boundaries may show a fine waviness structure.

FIG. 3 Possibly affected structure in S31803: Longitudinal section (500× magnification before reproduction).

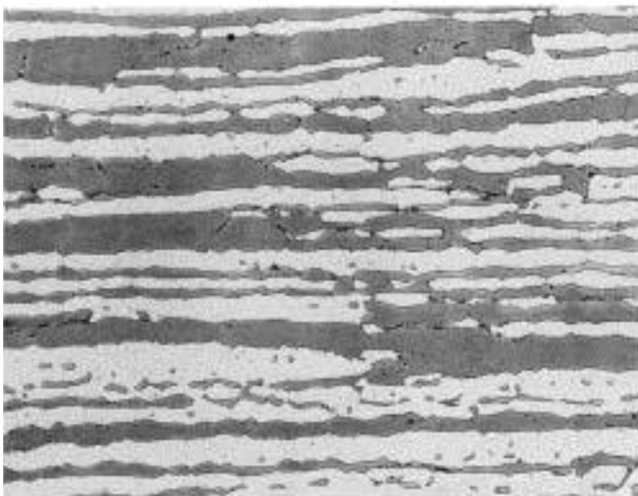
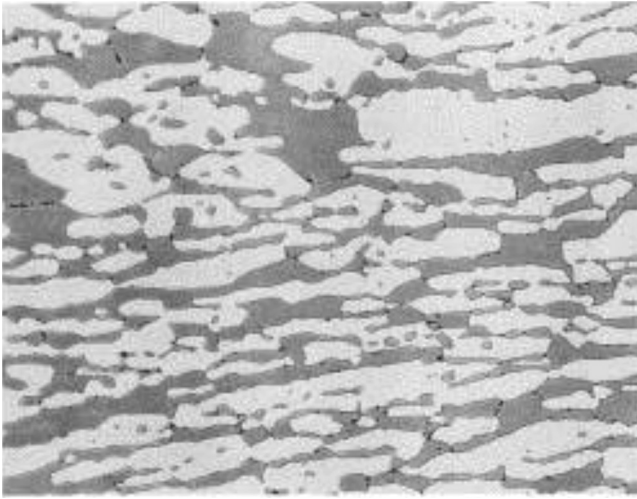


FIG. 4 Possibly affected structure in S31803: Transverse section (500× magnification before reproduction).



In the affected structure (Fig. 5 and 6), the indications of an intermetallic phase are readily revealed before or simultaneously with the staining of the ferrite during etching.

FIG. 5 Affected structure in S31803: Longitudinal section (500× magnification before reproduction).

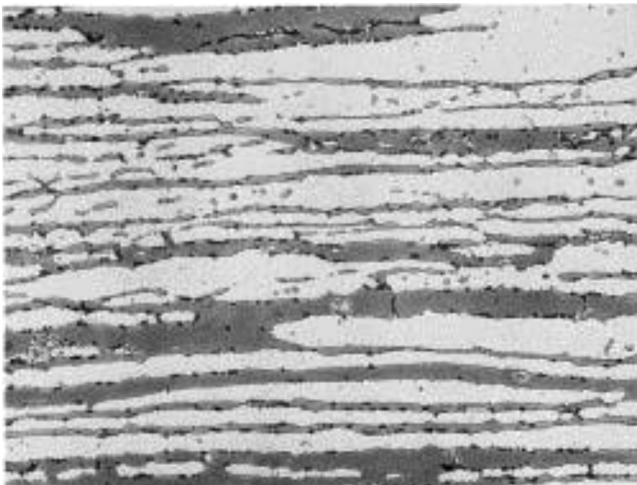
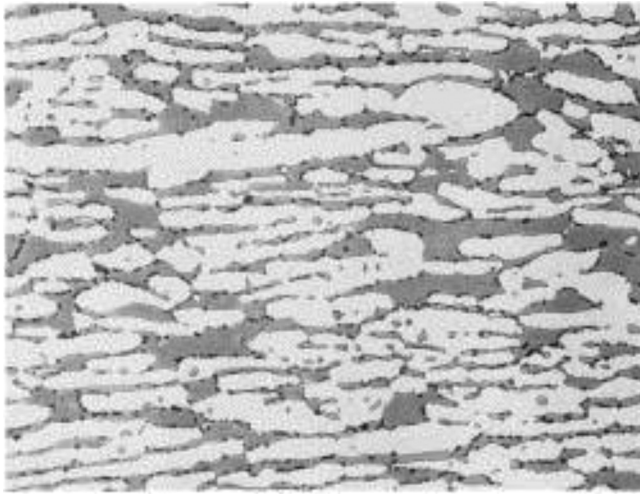


FIG. 6 Affected structure in S31803: Transverse section (500× magnification before reproduction).



When Test Method A is used as a screening test for Test Method B or Test Method C, specimens having acceptable etch structures need not be subjected to Test Method B or Test Method C. When Test Method A results in a possibly affected or affected structure, further testing in accordance with Test Method B or C must be performed to confirm acceptance or rejection of the material.

Recent revisions have incorporated additional alloys that may be evaluated in accordance with this specification. UNS S32750 was added in ASTM [A923 98](#).

With the addition of the first cast duplex grade UNS SJ92205, utilizing the Practice A and Practice B Charpy V notch testing only, ASTM [A923 14](#) deleted wrought from the title becoming *Standard Test Methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic/Ferritic Stainless Steels*. Photomicrographs of the cast structures ([Figs. 7 11](#)) were added as appropriate.

As new grades developed in the market, requests were made and, where compatible, they were incorporated into the standard. Another cast grade, UNS J93404 was added to Practices A and B in ASTM [A923 06](#); wrought Grades UNS S32550 and UNS S32520 were added to Practices A, B, and C in the ASTM [A923 08](#) revision and the current revision ASTM [A923 14](#) saw Grade UNS S32760 added to Practices A, B, and C as well.

These standard test methods have and continue to serve the original and traditional types of duplex stainless steels wells.

Around the year 2000, a group of products collectively known as lean duplex stainless steels (composed of 30 % to 70 % ferrite content with a typical alloy composition having chromium [Cr] greater than 17 % and molybdenum [Mo] less than

FIG. 7 J92205: Unaffected structure statically cast (400× magnification before reproduction).

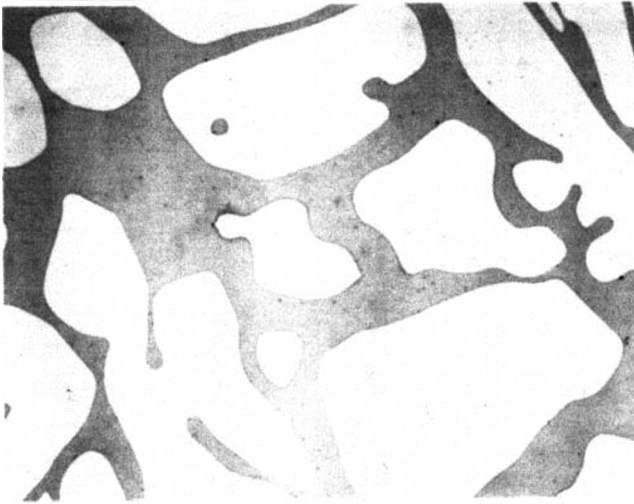
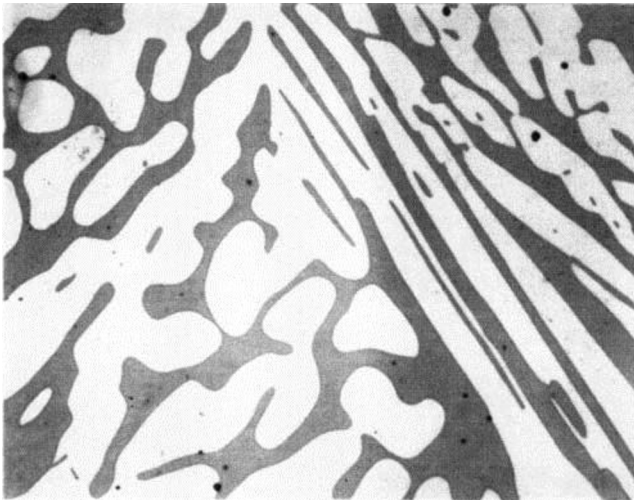


FIG. 8 J92205: Unaffected structure centrifugally cast (400× magnification before reproduction).



1 % and with additions of nickel, manganese, nitrogen, and controlled low carbon content as well as other alloying elements) appeared as economical alternatives to traditional duplex stainless steels and austenitic alloys [4].

FIG. 9 J92205: Possibly affected structure statically cast (400× magnification before reproduction).

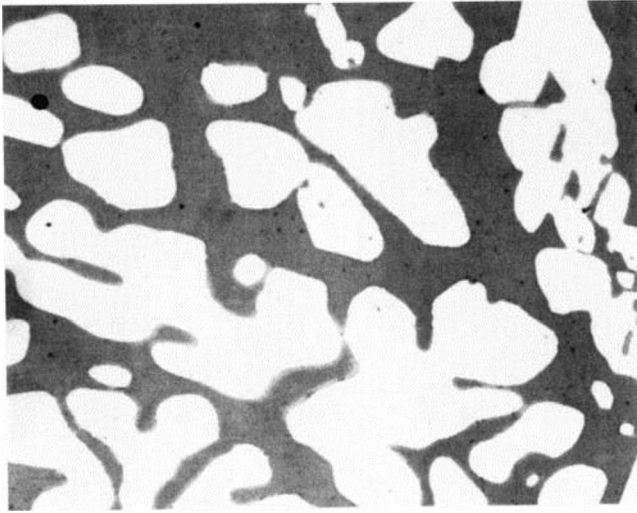


FIG. 10 J92205: Possibly affected structure centrifugally cast (400× magnification before reproduction).

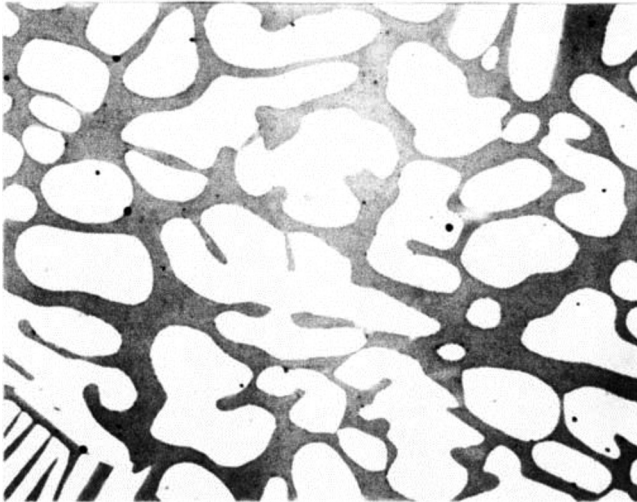
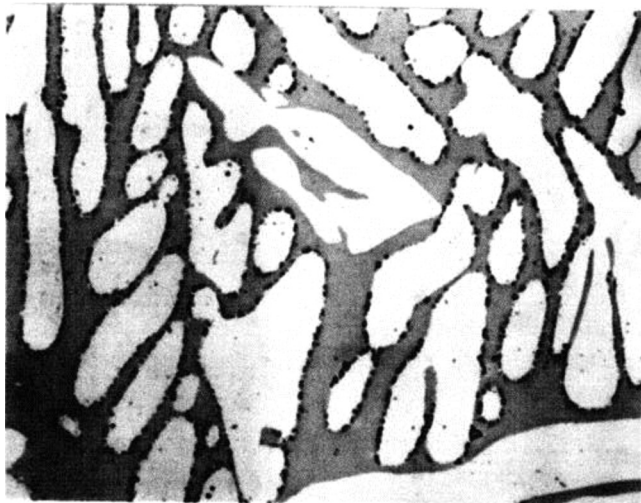


FIG. 11 J92205: Affected structure (400× magnification before reproduction).



The formation of secondary phases in duplex stainless steels as a result of isothermal treatments and continuous cooling has been extensively documented. All duplex stainless steels are prone to nitrides precipitation because they contain significant percentages of nitrogen. Nitrides formation is strictly related to the variation of temperature of the ferrite solubility against nitrogen, which increases at high temperature, bringing ferrite in supersaturated solid solution conditions. This metastable condition may cause nitrides precipitation during either water quenching or isothermal aging [1].

Thermodynamic simulations have shown that σ phase (the main intermetallic compound) is not a structural anomaly resulting from manufacturing errors but belongs to the equilibrium microstructure of all duplex stainless steels. However, studies have shown that the precipitation of intermetallic compounds mainly concerns the high alloyed grades, due to their high content of alloying elements, mainly chromium and molybdenum [1].

Molybdenum, together with chromium, is one of the main constituents of the intermetallic compound, making the material more sensitive to σ precipitation. It appears the molybdenum content may be considered as the crucial factor for the σ phase appearance because it contributes most to the increase in the formation of the intermetallic compound. The harmful effects of this intermetallic phase are well known, and its precipitation generally affects the high alloyed grades, such as UNS S32205 and UNS S32750 [1].

However, due to the low molybdenum content of less than 1 %, it was a metallographic challenge to use the testing parameters of ASTM A923 to reveal the presence of intermetallic phases in lean duplex stainless steels.

ASTM A1084 15a provides a metallographic procedure to show the appearance and approximate amount of chromium containing detrimental phases in lean duplex stainless steel, despite the small size of the detrimental phases and the difficulty in achieving a fully reproducible etch structure [3].

Microstructure results are correlated with toughness test and corrosion test results on a pass/fail basis. This newer process covers lean duplex stainless steels that were not well served by ASTM A923.

ASTM A1084 13 covered Grades UNS S32101 and UNS S32304. In revision, ASTM A1084 15, Grade UNS S32202 was added, and most recently, Grade UNS S82011 was added in revision ASTM A1084 15a [3].

At this time, the etch test in this standard is used for exploratory purposes. The reason for this is the small size of the detrimental phases typically occurring in lean duplex stainless steels and the difficulty in achieving a fully reproducible etch structure, which depends on factors such as specimen size and geometry, etching current and potential, composition of the lean duplex, as well as the amount and type of detrimental phases present. Nonetheless, the test method contained in this standard is still the best known metallographic procedure to show the appearance and approximate amount of detrimental phases in a lean duplex stainless steel.

The solution for etching is prepared by adding 10 g of reagent grade oxalic acid ($C_2O_4H_2$) to 90 ml of distilled water. The polished specimen is etched at approximately 6 V dc to 7 V dc, for 3 s to 20 s.

The detrimental phases in lean duplex stainless steels are typically small in size, and their presence can therefore be difficult to identify. However, the etchant enlarges the appearance of these precipitates. The appearance of the etched microstructure depends strongly on the size and shape of the specimen, as well as on the exact current and potential applied. Anomalous effects around the edges of the specimen can be caused by current density variations during etching; therefore, the light optical microscopy examination should be done predominantly in the central regions of examined specimens.

As there is no formal test result from the metallographic etch method, the actual test method is attached to ASTM A1084 15a as Appendix X1.

Figs. 12 14 (Fig. X2.1, X2.2, and X2.5 in ASTM A1084 15a Appendix X1) show near perfect solution annealed structures. No precipitates or only very small traces of precipitates can be seen in the microstructure. Figs. 15 17 (Fig. X2.3, X2.4, and X2.6 in ASTM A1084 15a Appendix X1) show a structure as can be typically found after proper solution annealing. In the microstructure, a small amount of detrimental phases can be seen. Both samples passed with good margin when tested according to Test Method B or C, or both.

Figs. 18 20 (Fig. X2.7 X2.9 in ASTM A1084 15a Appendix X1) show microstructures that appear to contain higher amounts of detrimental phases that still passed the acceptance criteria in Test Methods B or C, or both. Figs. 21 23 (Fig. X2.10 X2.12 in ASTM A1084 15a Appendix X1) show microstructures that are of samples that have failed the acceptance criteria in Test Methods B or C, or both.

FIG. 12 UNS S32101, thickness 10 mm, (500× magnification), solution annealed 1050°C + WQ, Method B result: Passed 187 J at room temperature, Method C result: Passed 1.01 mdd @ 45°C, 5012 mdd at 50°C [3].

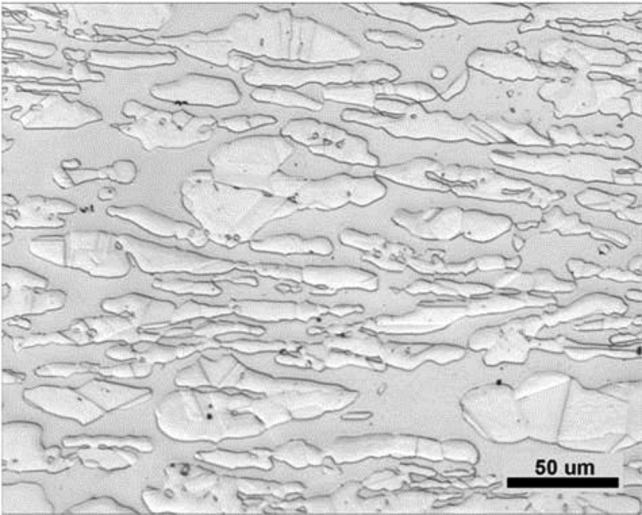


FIG. 13 UNS S32101, thickness 1.5 mm, (1000× magnification), solution annealed 1050°C WQ, Method B result: not tested, Method C result: Passed 0.0 mdd at 50°C, 4968 mdd at 55°C [3].

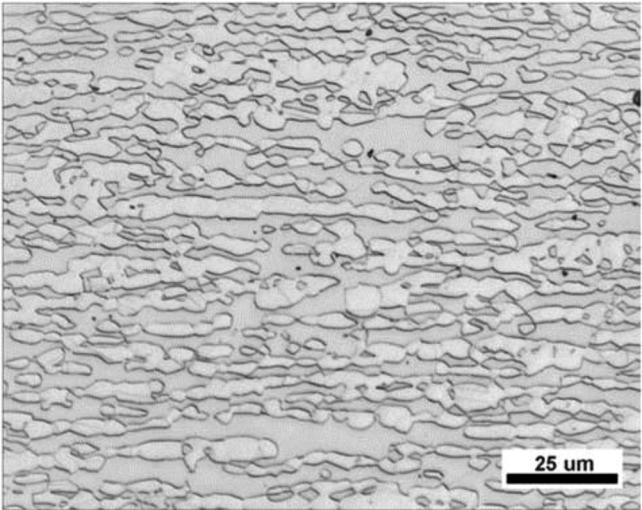


FIG. 14 UNS S32304, thickness 30 mm, (500× magnification), solution annealed 1050°C + WQ, 595 Method B result: Passed 288 J at 10°C, Method C result: Passed 1.57 mdd at 45°C, 4760 mdd 50°C [3].

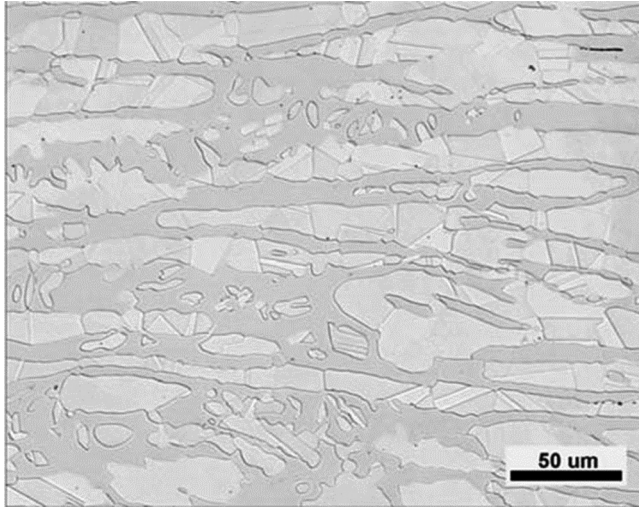


FIG. 15 UNS S32101 thickness 1.5 mm, (1000× magnification), solution annealed 1050°C + 1 min 700°C + WQ, Method B result: not tested, Method C result: Passed 0.19 mdd at 35°C, 1971 mdd at 40° [3].

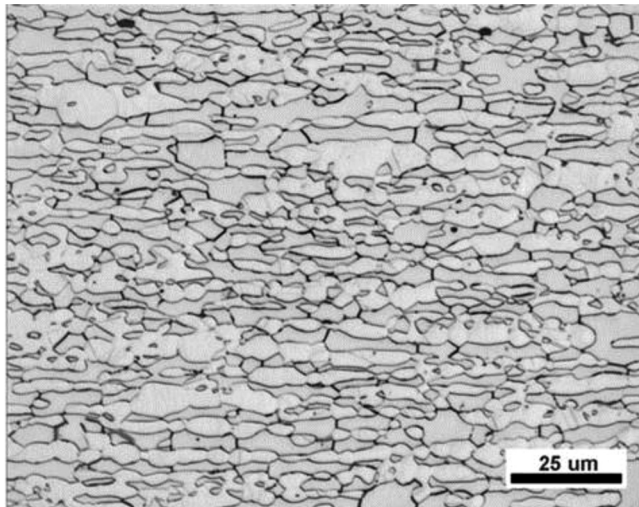


FIG. 16 UNS S32101 thickness 30 mm, (500× magnification), solution annealed 1050°C + 2 min 700°C + WQ. Method B result: Passed 105 J at room temperature, Method C result: Passed 0.29 mdd at 40°C, 3981 mdd at 45°C [3].

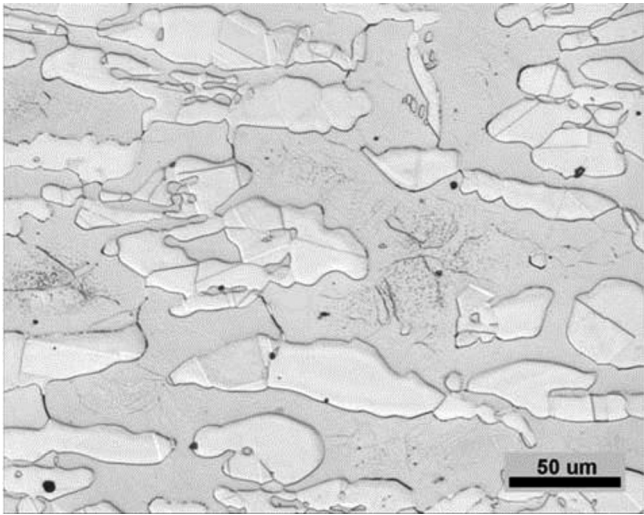


FIG. 17 UNS S2304, thickness 30 mm, (500× magnification), solution annealed 1050°C + 7 min 700°C + WQ, Method B result: Passed 266 J at 10°C, Method C result: Passed 1.85 mdd @ 30°C, 2521 mdd at 35°C [3].

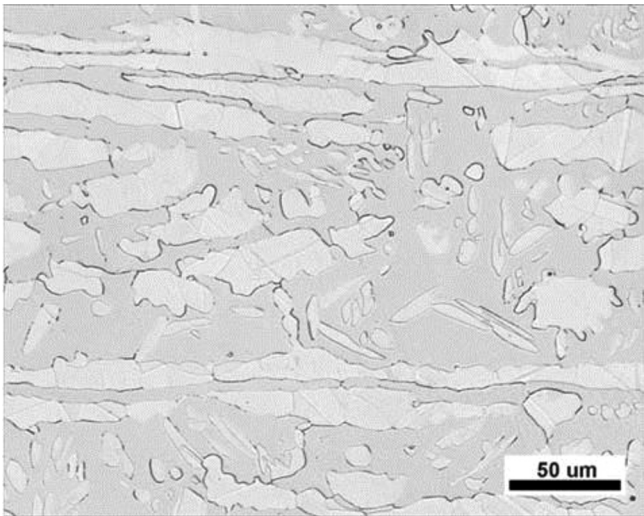


FIG. 18 UNS S32101 thickness 10 mm, (500× magnification), solution annealed 1050°C + 7 min 700°C + WQ. Method B result: Passed 87 J at room temperature, Method C result: Passed 0.47 mdd at 25°C, 803 mdd at 30°C [3].

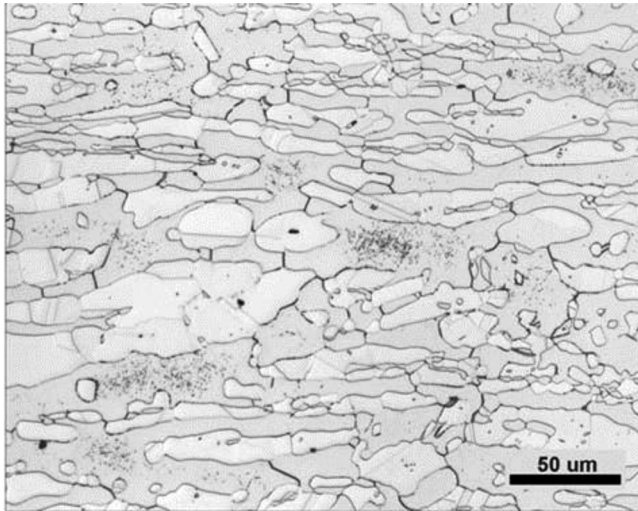


FIG. 19 UNS S32101 thickness 1.5 mm, (1000× magnification), solution annealed 1050°C + 4 min 700°C + WQ, Method B result: not tested, Method C result: Passed 1.11 mdd at 30°C, 3909 mdd at 35°C [3].

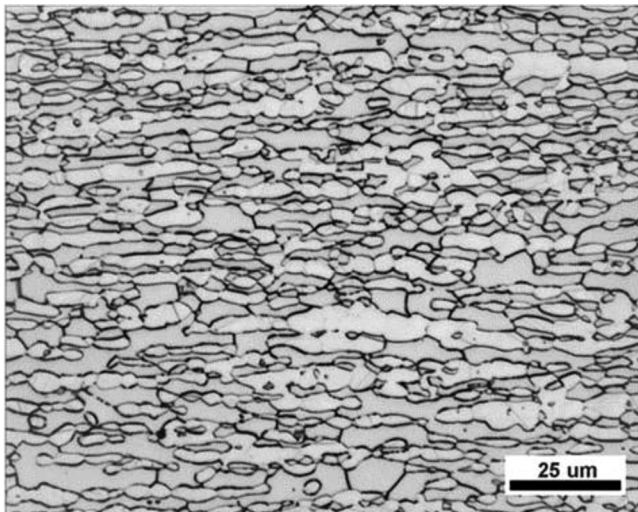


FIG. 20 UNS S2304, thickness 30 mm, (500× magnification), solution annealed 1050°C + 1 h 700°C + WQ, Method B result: Passed 118 J at 10°C, Method C result: Passed 0.15 mdd at 25°C, 1717 mdd at 30°C [3].

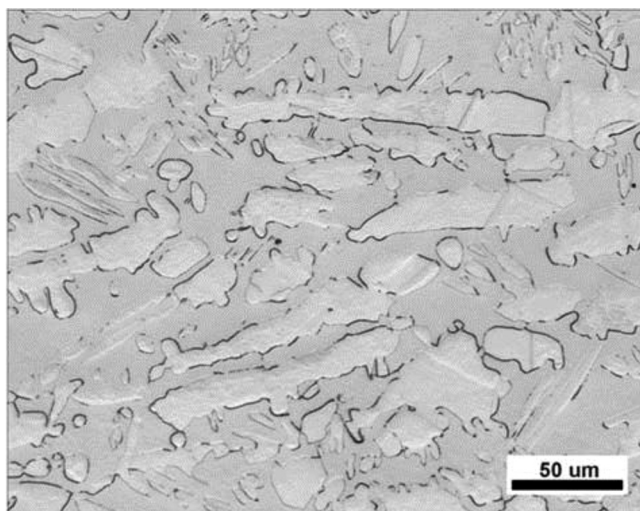


FIG. 21 UNS S32101 thickness 10 mm, (500× magnification), solution annealed 1050°C + 7 min 700°C + WQ, Method B result: Failed 60 J at room temperature, Method C result: Failed 0.80 mdd at 10°C, 275 mdd at 15°C [3].

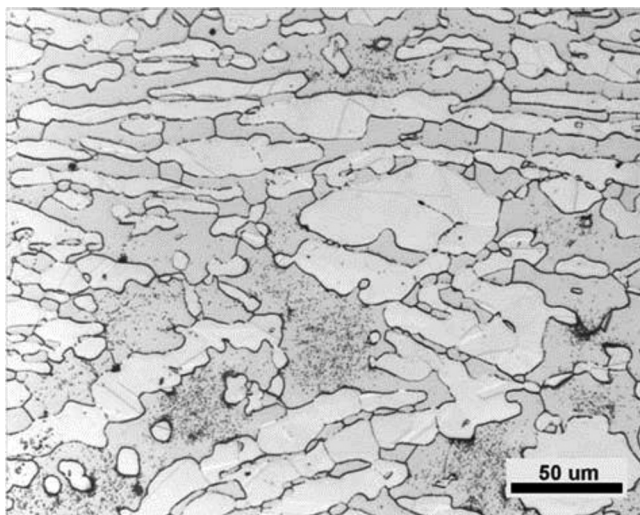


FIG. 22 UNS S32101 thickness 1.5 mm, (1000× magnification), solution annealed 1050°C + 9min 700°C + WQ, Method B result: Not tested, Method C result: Failed 0.74 mdd at 15°C, 1430 mdd at 20°C [3].

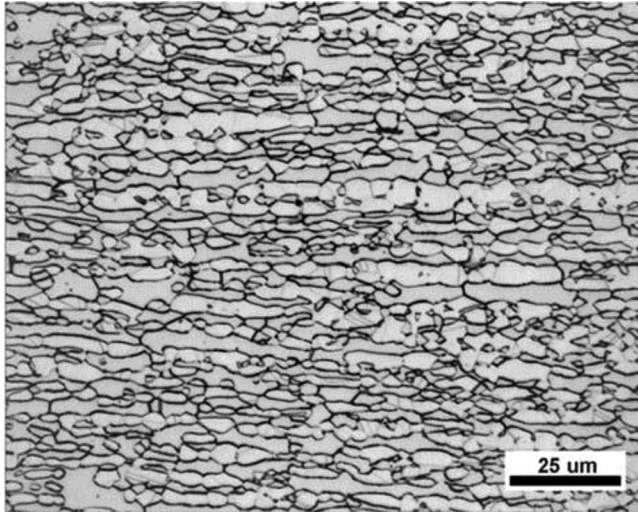
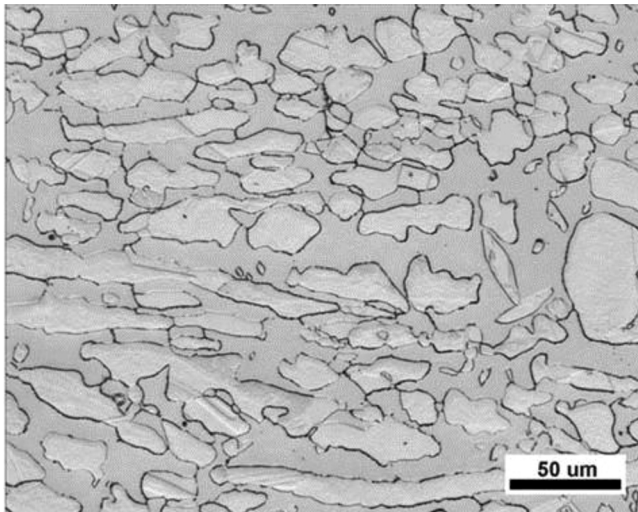


FIG. 23 UNS S32304, thickness 30 mm, (500× magnification), solution annealed 1050°C + 10h 700°C + WQ, Method B result: Not tested, Method C result: Failed 0.0 mdd 15°C, 331 mdd at 20°C [3].



In November 2012, ASTM Subcommittee A01.14 initiated an interlaboratory study activity (ILS #0909) that, among other issues, intended to further evaluate whether sufficiently precise etching specifications can lead to a microstructure characterization that is reliable for screening purposes such as ASTM A923 presently uses for higher alloyed duplex stainless steels. A test matrix was developed, materials producers were contacted for samples, and laboratories were selected, but discussions over the plans, diversions into other issues, and other events seem to have stalled the activity.

It is time to renew the ILS project with a focus on the original primary objective of developing a sufficiently precise etching specification leading to a microstructure characterization that is reliable for screening purposes of lean duplex stainless steels such as ASTM A923 presently uses for higher alloyed duplex stainless steels.

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