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Assessment of bias errors caused by texture and sampling methods in diffraction-based steel phase measurements

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Abstract

Many advanced high-strength steels rely on a metastable austenite phase for improvements in strength and formability. To date, no method has demonstrated the ability to provide accurate austenite phase fraction measurements in textured steels. Several techniques have been proposed, such as averaging the intensity of several peaks and/or summation of intensity from several sample orientations. The series of numerical experiments performed in this work sought to quantify the effects of texture on the measurement of the austenite phase fraction, with an emphasis on techniques suitable for laboratory X-ray diffraction. Simulated diffraction profiles were created with the following variables: texture components for the ferrite and austenite phases, the sharpness of each of the texture components, the number of peaks used for averaging in the phase fraction calculation, and the sampling scheme used for sample orientation summation in the phase fraction calculation. The resulting phase fraction calculations showed that texture, the number of peak pairs and the sampling method have a drastic effect on phase fraction measurements, causing significant bias errors. Hexagonal grids produced minimal bias errors and demonstrated a robust method of measuring phase fractions in textured materials.

Keywords

austenite; phase fraction; simulation; diffraction

1. Introduction

Many advanced high-strength steels (AHSS) developed in recent years, such as dual phase, complex phase, transformation induced plasticity (TRIP), and quenching and partitioning (Q&P), rely on multiple microstructural constituents for their strength and formability (Keeler & Kimchi, 2015). The face centered cubic (f.c.c.) metastable austenite phase is considered to be a key constituent in new third generation AHSS (3GAHSS). The US Department of Energy (DoE) has set target properties for 3GAHSS of 1200 MPa

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tensile strength with 30% elongation or 1500 MPa tensile strength with 25% elongation (Grants.gov, 2012). De Moor et al. (2010) and Matlock et al. (2012) predicted that varying the stability of austenite and introducing new microstructure concepts will result in steel with strength and ductility combinations between first and second generation AHSS, potentially meeting these DoE target values. New processing techniques for Q&P (Speer et al., 2011; Paravicini Bagliani et al., 2013) and medium Mn TRIP (Gibbs et al., 2011) steels have demonstrated material processing methods that result in properties within the range of 3GAHSS. These methods are currently under active development for production (Thomas, 2015). A key measurement challenge is the accurate and precise measurement of the phase fraction of austenite, both in the as-received state and as a function of deformation.

Although phase fraction measurements are a mature topic, recent round robin results show that typical practices for austenite phase fraction measurements result in significant variation (Jacques et al., 2009). Eight samples were tested using six different measurement techniques, one of which was X-ray diffraction (XRD). Examples of the range of austenite phase fraction values measured with XRD are 0.099–0.213 in one sample and 0.109–0.296 in a separate sample. Examination of the XRD methods used in the round robin shows differences in sample preparation, X-ray radiation, number of peaks measured, measurement conditions and analysis methods, indicating a lack of accepted techniques (Jacques et al., 2009). The remainder of this paper focuses solely on diffraction techniques, with an emphasis on techniques that may be suitable for laboratory X-ray systems.

1.1. Diffraction-based austenite phase fraction measurements

Phase fraction measurements on steel using XRD that did not require external calibration were first pioneered by Averbach (1947). Integrated intensities of the austenite and martensite¹ phases were measured and averaged for two austenite ($F^{A,hkl}$) and three martensite ($F^{F,hkl}$) diffraction peaks (Averbach & Cohen, 1948; Averbach et al., 1950). Theoretical intensities ($R^{A,hkl}$ and $R^{F,hkl}$) were calculated for each peak (i), which include assumptions of the elemental distribution in each unit cell but neglect details of the diffractometer such as beam intensity and detector efficiency. The intensities of the three normalized martensite peaks were averaged together, and assuming the absence of other phases, the sum of averaged martensite (number of peaks n_F) and austenite (number of peaks n_A) phases was set equal to 1. The austenite phase fraction (V_A) can then be determined *via* equation (1):

$$V_A = \frac{(1/n_A) \sum_{i=1}^{n_A} (I_i^{A,hkl} / R_i^{A,hkl})}{(1/n_F) \sum_{i=1}^{n_F} (I_i^{F,hkl} / R_i^{F,hkl}) + (1/n_A) \sum_{i=1}^{n_A} (I_i^{A,hkl} / R_i^{A,hkl})}. \quad (1)$$

¹The resolution limits of the equipment used by Averbach (1947) prevented distinguishing between the body centered tetragonal martensite (M or α') phase and the body centered cubic ferrite (F or α) phase. Distinguishing these phases remains a challenge for laboratory XRD systems. Diffraction peaks from ferrite and martensite are often considered a single peak when integrated. Discussion of the martensite phase is limited to this paragraph to accurately describe the experiment performed by Averbach. However, the rest of this paper focuses on the ferrite phase: hence the use of the notation F^{hkl} for consistency.

While the focus of this paper is on steel phases, this method has been extended to other phases including titanium (Averbach et al., 1959).

1.2. Crystallographic texture in phase fraction measurements

Absent in equation (1) is the dependence of integrated intensities on sample orientation. This dependence, referred to as crystallographic texture or preferred orientation, occurs when some crystal orientations are aligned with a particular sample orientation more frequently than if they were uniformly (or randomly) distributed. This dependence is often visualized in a pole figure, where the integrated intensity for a selected peak is plotted onto a projection of sample orientations (Klug & Alexander, 1974). Variation of intensity on a pole figure is therefore an indication of crystallographic texture, and is often expressed in multiples of a uniform (or random) distribution.

Using the example of a single sample orientation, if the integrated intensity in that particular sample orientation is larger (or smaller) than the intensity that would occur in a uniform distribution, then the phase fractions calculated using equation (1) may contain bias errors caused by the corresponding increase (or decrease) in the integrated intensity. These bias errors may be difficult to detect, as the true value for a material is generally unknown and the repeatability inherent in the bias errors may give a false sense of accuracy. As the majority of steel products undergo some type of deformation during production (rolling, forging *etc.*) or during use of the material, it is almost assured that a steel product will have some degree of crystallographic texture caused by the deformation. However, the magnitude of bias error due to texture has not been quantified.

The diffraction community often makes use of powdered samples to avoid these issues of crystallographic texture. This process typically involves mechanically crushing the sample to create a fine powder with a resulting minimal crystallographic texture. However, as the austenite phase in steel may be susceptible to deformation-induced transformation, this method is inapplicable. The challenge is to develop an accurate phase fraction measurement technique that can be applied to bulk samples that have an unknown crystallographic texture.

An additional technique used in the powder diffraction community is Rietveld refinement. Rietveld refinement was not analyzed in this paper as the focus was on the integrated intensity method. While many Rietveld refinement packages include some ability to include texture, the parameter space needed to rigorously assess the accuracy of phase fraction measurements on textured materials is quite large (specific program, crystallographic texture fitting method, resolution of texture fitting method, number of peaks and sample orientations, *etc.*) and this approach is therefore outside of the scope of this paper.

As summarized by Cullity (1978), there are two general methods to account for crystallographic texture in phase fraction measurements: averaging the intensity of several peaks and the summation of the intensity along a set of independent sample orientations. With regards to averaging the normalized intensity of several peaks, the number of peaks used for the averaging for austenite phase fractions varies widely in the reported literature. Averbach (1947) used three ferrite and two austenite peaks; Miller (1964) advocated the use of two austenite and one ferrite peaks, but focused on the use of high multiplicity planes;

Arnell et al. (1968) and Beu (1952) recommended three ferrite and three austenite peaks; while Gullbert & Lagneborg (1966) used four ferrite and four austenite peaks. A similar range of variation is found in prior round robins, from one to four peak pairs (austenite and ferrite) reported by Hinton (1987) and two to five peak pairs in the results reported by Jacques et al. (2009). There seems to be a general belief in the literature that averaging over more peaks results in a more accurate measurement, but there has been no rigorous investigation to support this hypothesis.

The second method to account for crystallographic texture involves summation of intensity over a set of sample orientations. The sample axes common in rolled products – rolling direction (RD), transverse direction (TD) and normal direction (ND) – are used in this paper. The simplest and most widely used set of sample orientations are single orientations, typically with the diffraction vector [referred to as the diffracting plane (Klug & Alexander, 1974) or the reflecting plane normal (Cullity, 1978)] aligned with ND, TD or RD. However, Lopata & Kula (1965) criticized the use of ND, TD or RD single orientations, as these are often greatly affected by crystallographic texture and additional sample orientations should be used. An alternative single orientation was proposed by Morris (1967). Morris analyzed several pole figures to find a sample orientation where there was minimal crystallographic texture (*i.e.* the normalized pole figure intensity value was near one times uniform or one times random) with a low gradient in intensity. These studies indicated that a single orientation 30° from TD in the ND–TD plane would meet these criteria. In transmission mode, complete Debye-Scherrer rings can be recorded with a corresponding cone of diffraction vectors (*e.g.* Hu et al., 2017). Measurements of additional sample orientations have been achieved by rotation (Durnin & Ridal, 1968), tilt or simultaneous ‘tilt & rotation’ of the sample while recording a single peak (Lopata & Kula, 1965; Miller, 1968; Jatzak et al., 1980). These techniques continuously expose the sample to the X-ray beam during sample motion and thus the peak intensity is a summation of several sample orientations, but the peak intensity at a specific sample orientation cannot be recovered. An alternative method is to rotate the sample to a discrete set of points projected on the pole figure and sum the resulting diffraction patterns. The rotation of the sample to fixed points is not common for phase fraction measurements, but it is widely used for pole figure measurements and texture analysis. In addition to an equal angle grid, hexagonal grids (Matthies & Wenk, 1992; Rizzie, 2008) and geometric grids (Helming et al., 1998) have been developed. However, moving the sample to a series of sample orientations will require additional time per phase fraction measurement compared with a single sample orientation.

An alternative to accounting for texture is to avoid calculating phase fractions when the sample exceeds a ‘significant’ amount of texture, with the definition of significance variable in the literature. This has been implemented by comparing the integrated or normalized intensities of selected peaks and prohibiting the calculation of phase fractions outside of specified bounds. The Society of Automotive Engineers (SAE) special publication SP-453 (Jatzak et al., 1980), widely used as a practical guide for austenite phase fraction measurements, gives bounds that the ratio of integrated intensities from the 220 and 200 austenite peaks should be in the range of 1.2–1.8 when using Cr radiation, approximating 20% of value bounds of the theoretical intensity ratio. Ratios outside these bounds indicate that the phase fractions may not be accurate. In the round robin reported by Hinton (1987),

the range is shifted to 1.1–1.7. In both papers, there is no explicit range expressed for the ferrite phase. The only standard for XRD-based phase fraction measurements of austenite (ASTM E975-13, 2013) further restricts the applicability of the measurement, requiring that the texture be ‘near random’, defined as peak ratios for austenite 220 and 200 reflections within the range of 1.1–1.7 (Cr radiation) or 0.7–0.5 (Mo radiation) and ferrite 211 and 200 peak ratios within the range of 8–11 (Cr) or 1.5–2.2 (Mo).

To summarize, there is no integrated-intensity-based method, *via* either averaging of several peaks or summation over a set of sample orientations, that has been demonstrated to provide accurate austenite phase fraction values in textured steels. It is recognized that crystallographic texture may cause bias errors in phase fraction measurements. Bias errors due to texture would be difficult to assess experimentally, as the nature of bias errors means that they result in repeatable measurements, providing repeatability without accuracy. The goal of this paper is to test the following hypotheses:

1. Crystallographic texture will cause some averaging or summation methods to be systematically biased.
2. Averaging over more peaks will result in improved accuracy of the phase fraction measurement compared with fewer peaks.
3. Using simultaneous tilt & rotation to sum intensity over several sample orientations will result in reduced bias errors compared with single orientations.
4. Moving the sample to points on a hexagonal grid will result in a lower bias errors than continuous tilt & rotation.

The results of this investigation will provide guidance on the range and significance of bias errors caused by the crystallographic texture and methods used to measure austenite phase fractions.

2. Methods

To test these hypotheses, simulated diffraction profiles and pole figures were generated. This allowed repeatable and explicit control of the following variables in the phase fraction calculation: the texture components for the ferrite and austenite phases in the simulated sample; the sharpness of each of the texture components; the number of peaks used for averaging in the phase fraction calculation; and the sampling scheme used for summation over a set of sample orientations.

Although there are other significant sources of error and uncertainty in phase fraction measurements, such as the interference of carbide peaks and grain size, the items above were the only variables considered in this work. These variables can be separated into those that the researcher has explicit control of (number of peaks and sampling scheme) and variables where the researcher has no direct control (texture components and sharpness). The data and analysis methods used in this paper are available as a published data set to facilitate data transfer and reproducibility (Creuziger et al., 2018).

Only two phases were considered, austenite (space group 225, $a = 3.6467 \text{ \AA}$) and ferrite (space group 229, $a = 2.8665 \text{ \AA}$) (Brandes & Brook, 1992). Additional phases such as carbides or martensites were not included in this analysis. To avoid the effects of alloying elements, only Fe atoms were used in structure factor calculations necessary for theoretical intensities for both phases. Similarly, variable absorption effects from the two phases and/or variable grain sizes were not included in this analysis. The interaction volume of the simulated sample was assumed to be large, such that spatial variations and grain-size effects were negligible.

The phase fractions for this investigation were fixed at 0.25 austenite and 0.75 ferrite. The calculated austenite phase fractions were determined using equation (1) for different combinations of texture, sampling and peak quantity variables. In the reported results, any deviation from the nominal austenite phase fraction of 0.25 represents a bias error in the calculated austenite phase fraction.

Diffraction profiles were created using the software package *MAUD* (Lutterotti, 1997; Ferrari & Lutterotti, 1994; Lutterotti et al., 1997; Matthies et al., 1997). For simplicity, a single X-ray wavelength and a zero background X-ray intensity was used. The theoretical copper $K\alpha_1$ ($K-L_3$) wavelength of $1.540538 \text{ \AA}$ (Deslattes et al., 2005) was used. An example intensity *versus* 2θ plot with uniform (random) austenite and ferrite textures is shown in Fig. 1.

2.1. Crystallographic texture components and sharpness

The first set of variables considered were the crystallographic textures. Kocks et al. (1998) provide a detailed discussion of crystallographic texture representations, so only pertinent details will be summarized here. In the present paper, orientation distribution functions (ODFs) will be used to display crystallographic texture information. Although there are a variety of possible functional forms and conventions used to describe the ODF, the content is largely the same. The Bunge angle convention $[\varphi_1, \Phi, \varphi_2]$ was used in this paper.

For a given type of deformation and crystal symmetry, specific orientations become more intense. These specific orientations, or texture components, are expressed as a series of Euler angles or $(hkl) [uvw]$ pairs and often have a shorthand name associated with them. Texture components known to occur in steels are described by Bleck et al. (1991), Kestens & Jonas (2005) and Jonas et al. (2006). These include texture components generated during hot rolling and recrystallization of the fully austenitic microstructure, transformation of the austenite phase to the ferrite phase upon cooling, and cold rolling of the ferrite phase. A selection of these texture components are listed in Table 1. Many of the texture components listed in Table 1 appear on a single ODF cross section, the $\varphi_2 = 45^\circ$ section. Fig. 2 is a key for the $\varphi_2 = 45^\circ$ cross section with cubic crystal symmetry and orthotropic sample symmetry, showing the location in Euler space of the texture components.

Fig. 3 shows example ferrite and austenite $\varphi_2 = 45^\circ$ ODF cross sections for two AHSS, a TRIP 700 and a TRIP 780 steel. For the austenite phase texture, components of brass, copper and occasionally cube occur more frequently in the bulk material than in a uniform (or random) distribution, and gamma and alpha fiber orientations occur more frequently than

in a uniform distribution in the ferrite phase. The ODFs shown in Fig. 3 were calculated *via* pole figure inversion using the software package *MTEX* (Hielscher & Schaeben, 2008; Bachmann et al., 2010) with complete pole figures measured at the NIST Center for Neutron Research, beamline BT-8 (Brand et al., 1997). These TRIP materials have been analyzed previously (Gnäupel-Herold & Creuziger, 2011) and are similar to other AHSS for which ODFs can be found in the literature (De Knijf et al., 2014; de Meyer et al., 2001; Barrales-Mora et al., 2011). To characterize the sharpness of the ODF, texture index (Morawiec, 2004) values were used. For the ODFs shown in Fig. 3, the TRIP 700 ferrite phase has a texture index of 1.5 and the austenite phase has a texture index of 1.4. The TRIP 780 ODFs have sharper textures, particularly the ferrite phase with a texture index of 2.9, and the texture index for the austenite phase is 1.6.

As discussed with the example ODFs, examination of individual texture components is a typical technique in ODF analysis. In a complementary way, these texture components can be used as building blocks to approximate an ODF. The texture components listed in Table 1 were used to create a more general set of textures than those shown in Fig. 3 to test the robustness of the phase fraction measurement for different sampling schemes and numbers of peaks used in averaging. As samples with all crystals exactly aligned with a particular texture component are rarely observed experimentally, a distribution about each texture component was set. To create textures comparable to those shown in Fig. 3, a unimodal ODF was created at each texture component orientation with a ‘deLaValeePoussin’ kernel function and a 20° half-width. For the fiber textures, a series of unimodal ODFs were added together to approximate a fiber texture. This provided consistency in intensity values with the series of single texture components. These ODFs are depicted in Fig. 4. The texture indices of each ODF with a half-width equal to 20° are included in Table 1. Additional textures with half-widths between 10 and 50° [not shown here, but included in the data set of Creuziger et al. (2018)] were also calculated. This range of half-widths results in a corresponding texture sharpness range of between 1 (uniform distribution) and 23 (heavily textured), measured *via* the texture index value.

2.2. Peak averaging

The second variable was the number of peaks that were used to average the intensity of each phase. Three groups of peaks were selected: two pairs, the austenite peaks 200, 220 and the ferrite peaks 200, 211; four pairs, the austenite peaks 111, 200, 220, 311 and the ferrite peaks 110, 200, 211, 220; max unique, the austenite peaks 111, 200, 220, 311, 331, 420, 422, 511 and the ferrite peaks 110, 200, 211, 310, 222, 321.

The two and four pair groups are comparable to methods listed in the literature. The max unique group includes all possible diffraction peaks when using Cu radiation and skips those peaks that are higher-order reflections of peaks already included in the set. For example, the 222 austenite peak was not included in this set, as it already contains the 111 austenite peak.

2.3. Sample orientation summation

To investigate the effects of sample orientation summation, 13 different sampling schemes were explored. These schemes are depicted in Fig. 5. Each diffraction vector in the set was

assumed to be aligned with the corresponding point shown in the sampling scheme. These schemes fall into four categories: (1) single orientations, (2) ‘rings’, (3) rotation and tilt, and (4) hexagonal grids. First were single orientations, with the diffraction vector aligned with either RD, TD or ND of a rolled sheet. In addition, the sample orientation 30° toward ND from TD described by Morris (1967) was included. The ‘rings’ were discretized as 5° increments in the planes perpendicular to RD, TD or ND. Such a method is consistent with complete Debye-Scherrer rings measured in transmission mode. Diffraction vectors were approximated to be in a plane perpendicular to the incoming beam, *i.e.* a diffraction cone of infinitesimal height. This approximates a high-energy source and two-dimensional detector geometry. The third category comprised rotation, tilt and simultaneous tilt & rotation schemes. The rotation axis, the diffraction vector and ND were coincident, and the tilt axis was aligned with TD (tilting along the ND-RD plane). Tilt was applied as a triangle wave at 56 cycles per minute and rotation was applied at 30 rotations per minute in accordance with the literature (Jatczak et al., 1980). The final category consisted of hexagonal grids. These were based on a fixed angular difference between points. A 60° cutoff in the tilt, simultaneous tilt & rotation and partial hexagonal grid was chosen to represent laboratory XRD systems, where higher tilt angles lead to significant absorption and defocusing. Note that the density of points around ND is much higher in the tilt & rotation scheme than in the hexagonal scheme, indicating that ND is measured more frequently, or oversampled.

2.4. Phase fraction calculation

To assess the bias errors in the phase fraction calculation, the following procedure was used. The ODFs shown in Figs. 3 and 4 were exported from *MTEX* to *MAUD*. *MAUD* was used to generate diffraction profiles from the example ODFs shown in Fig. 3 and to calculate pole figures from all ODFs. Using the sampling scheme, the number of peaks and the phase fraction defined above, intensity values were taken from pole figures and used to calculate the phase fractions according to equation (1). As the intensity values were taken from the pole figure, the effective theoretical intensities ($R^{A,hkl}$ and $R^{F,hkl}$) were removed from equation (1) as the pole figures are already normalized for each peak. The pole figure normalization effectively results in a perfect theoretical correction for each peak, and the intensity was only affected by texture, as the phase fraction was fixed. Linear interpolation of the pole figure data was required to avoid jumps in intensity at the rotation boundary of 0°/360°.

While zero bias error is a noble goal, in practice bias errors may be tolerated, provided that they are known and/or less than other sources of error. For this work, bias errors within the bounds of ±5% of the value were considered ‘tolerable errors’. Calculated austenite phase fractions between 0.2375 and 0.2625 fall within this range.

3. Results

The combination of variables explored resulted in a large array of data. The data presented here show the calculated austenite phase fraction for each variable. The nominal value at 0.25 is marked for reference. Any deviation from this nominal value is a bias error caused

by the combination of texture and sampling methods. All of the calculations and plots are included in the published data set (Creuziger et al., 2018).

As a demonstration of how bias errors are caused by texture, Fig. 6 shows simulated diffraction profiles for a uniform texture and the two example TRIP textures. Intensity data from two peak pairs and a single sample orientation (ND) are shown. The changes in intensity for each peak were caused solely by texture. In the TRIP 700 case, the austenite 200 peak decreases while the other three peaks increase. Using these peak intensities, the calculated phase fraction is 0.239, with a bias error of -4.3% . The peaks for the TRIP 780 texture behave similarly, albeit with the austenite 200 peak decreasing more than the same peak in TRIP 700 and the two ferrite peaks increasing more. These changes result in a larger bias error for the TRIP 780 example, with a calculated phase fraction of 0.181 and a bias error of -27% .

Fig. 7 shows the calculated austenite phase fractions for TRIP 700 and TRIP 780 textures as a function of peak averaging and sample orientation summation scheme. These results are grouped by category: 'single', 'ring', 'tilt & rotate' and 'hex'. For each of the sampling schemes the three peak groups are listed, with the distance of the bar graph from the nominal 0.25 austenite showing the magnitude of the bias error. The two peak single ND phase fractions calculated above are included in this plot.

For both experimental textures, the single orientations generally have the largest bias errors of all the categories. The single orientations also show there is no convergence of the bias errors as the number of peaks is increased. The Morris orientation does not have significantly smaller bias errors than any of the other single sample orientations, but the range is generally smaller. For the TRIP 700 texture, some single orientations such as the ND and Morris orientation when four peak pairs were used result in tolerable bias errors. Summation of intensity along a ring leads to reduced bias errors compared with single orientations and tilt and/or rotation methods, and also shows no convergence with increasing number of peaks. Rotation alone resulted in bias errors identical to those of the single ND orientation. Tilt and tilt & rotate methods reduced some of the bias error, but the bias errors are generally greater than those for the ring schemes. Additionally, the tilt & rotate method showed bias errors that appear as scaled copies of the single ND orientation, indicative of the oversampling of ND inherent in this method. The hexagonal grids have minimal bias errors, but the 22.5° full grid has larger bias errors than either of the finer grids.

Comparing the TRIP 700 and TRIP 780 plots, there is general agreement of the sign for the bias errors, but the magnitudes are markedly different. The TRIP 780 bias errors were larger than those in the TRIP 700 texture; there is a correlation between larger texture index values and larger bias errors. In comparison to all of the other categories, the hexagonal grids have tolerable bias errors, even when only two peak pairs are considered.

In Fig. 7, there is no clear convergence of the calculated austenite phase fraction when the number of peaks is increased. The case of the ND sample orientation and max unique peak group is used as an example to explain this observation. The bias errors in the max unique group are among the largest, despite the high number of peaks included in the average.

Examination of the intensity for each of the individual peaks reveals the bias error is largely caused by the ferrite 222 peak. Examination of the ferrite texture shown in Fig. 3 reveals that the texture of this phase is dominated by the gamma fiber, where the (111) crystal plane is parallel to ND. Therefore, it is not surprising that the texture of this sample will have significant bias errors for any peak averaging method that includes the 222 peak and sample orientation summation limited to a single orientation (ND). However, if the texture is not known *a priori*, these bias errors may not be detected.

The data shown in Fig. 7 depict bias errors for a single austenite and ferrite texture combination (the TRIP 700 and TRIP 780 example textures). To more rigorously determine the potential magnitude of bias errors, all possible combinations of the austenite and ferrite single component textures shown in Fig. 4 (91 combinations in total) were calculated. Each combination is depicted as an individual point in Fig. 8. Note that the austenite phase fraction scale was increased from Fig. 7 because of the larger bias errors that were observed. In Fig. 8 there is an apparent convergence of bias errors as more peaks were included in the average. However, the linkage between bias errors for the same combination of austenite and ferrite textures was not shown, so non-converging bias errors similar to those in Fig. 7 may have occurred.

Fig. 8 provides further support for the observations seen in Fig. 7. The single sample orientation category has the largest bias errors. The Morris sample orientation shows improvement over other single orientations. Summation over a ring decreases the bias errors relative to single orientations and tilt, rotation and tilt & rotation. Tilt and/or rotation does not improve bias errors dramatically compared with single orientations. The hexagonal grids perform the best, but some bias errors larger than the tolerable bias error range are seen if only two peak pairs are used in conjunction with the partial grid.

To quantify these trends, the number of texture combinations shown in Fig. 8 that were within the tolerable bias error range are listed in Table 2. As the number of peaks increases, the number of combinations with tolerable bias errors also increases, with the exception of the TD single orientation which decreases at four peaks. The RD, TD and Morris single orientations do not exceed 40% of the combinations falling within the 5% bias error range until the max unique peak averaging is used. For the ring sampling schemes and four peaks, more than 60% of the texture combinations are within 5% bias errors. This total reaches 85 and 90% in RD and ND, respectively, when the max unique averaging is used for the ring schemes. However, with the exception of the two peak partial hexagonal grid, all other hexagonal grid schemes were successful at reducing bias errors to less than 5% of the value in all texture combinations.

Turning attention to texture sharpness, this was varied by changing the half-width of the texture distribution from 10 to 50° in 5° steps. For the 91 austenite and ferrite single component texture combinations, this resulted in 945 combinations of textures. The calculated austenite phase fraction for a single sample orientation, single ND, is plotted as a function of the average texture index of ferrite and austenite in Fig. 9. The average texture indices of the TRIP 700 and TRIP 780 ODFs are shown by vertical lines to mark example texture indices of AHSS. Fig. 10(a), a subset of Fig. 9 with texture index plotted up to

1.0020, shows that there is no bias error at a texture index of 1, which is a uniform (random) texture. However, bias errors increased as texture index increased, particularly for the two peak pair data set. There was some skew observed to bias errors: the two and four peak data sets tend to have positive bias errors (calculated phase fractions greater than 0.25), while the max unique data set tended to have more negative bias errors. For some particularly sharp textures, with average texture indices above 6, calculated austenite phase fractions equal to 0.0 or greater than 0.8 were observed. The additional line, showing the applicability limit for ASTM E975, is discussed below.

As stated in the introduction, ASTM E975 uses austenite and ferrite peak ratios as limits on the applicability of the standard. Using the range of different textures shown in Fig. 9, and fixed sample orientation single ND, texture combinations that fell within the peak ratios given by ASTM E975 are plotted in Fig. 10(b). No points with a texture index greater than 1.06 met the peak ratio criteria of ASTM E975. A texture index of 1.06 was added as a line in Fig. 9. The small average texture index range shows that this standard only applies to a very limited range of texture indices. However, even with this extremely small degree of texture, there are still bias errors in the phase fraction measurement that can exceed the 5% of value tolerable bias error range for two peak pairs.

4. Discussion

In the introduction, four hypotheses were presented. The first hypothesis, that the texture and sampling method would have a drastic effect on the phase fraction measurements, is supported by the data, namely by the wide bias errors shown in Figs. 7, 8 and 9. In Fig. 7, the greatest bias errors occur for TRIP 780, TD single, two peak and four peak conditions. The two peak condition had a calculated austenite phase fraction of 0.350 and a +40% bias error, and the four peak condition had a phase fraction of 0.122 and a -51% bias error. Fig. 8 shows that the bias errors can be even larger for other texture combinations; for example, several sampling conditions for two peak pairs resulted in bias errors exceeding +100 and -60%. All averaging and summation methods are free from bias errors if there is uniform (random) texture (*i.e.* a texture index equal to 1). However, bias errors quickly increase as texture increases, as shown in Fig. 9. For highly textured materials, bias errors can exceed +200% or reach -100% (meaning no austenite is detected). As shown in Fig. 10(a), divergence from the austenite phase fraction value of 0.25 begins at average texture indices less than 1.001. Furthermore, as texture tends to increase with deformation (Kocks et al., 1998), bias errors in phase fraction measurements may similarly increase with deformation. Single orientations seemed most susceptible to bias errors. Rings resulted in tolerable bias errors for more than 60% of the texture cases explored when four or max unique peaks were used, but did not eliminate bias errors in all cases. Hexagonal grids were highly successful at reducing bias errors, even when two peaks were used.

The second hypothesis, that averaging over more peaks will result in improved accuracy, is generally supported by the data. In Fig. 8, the scatter in the two pair averages is always largest within a sampling method compared with the four pair and max unique averages. However, certain texture combinations contradict this hypothesis, such as the example textures used in Fig. 7. Single sample orientations and tilt and rotation combinations

are particularly susceptible to bias errors, even when considering many peaks. The large difference in the range of bias errors for the ring sampling schemes between two peak and four peak averaging, as shown in Fig. 8, is a dramatic improvement and is relevant for transmission diffraction experiments. Additionally, the already low bias errors for the full hexagonal grid conditions were not significantly improved by increasing the number of peak pairs.

The third hypothesis, that tilt and rotation would reduce bias errors compared with single orientations, is also generally supported by the data, but the bias errors may still be significant and larger than those for ring schemes. Continuous tilt & rotation methods, such as those described by Miller (1968) and Jatczak et al. (1980), oversample some of the sample directions, as shown by the higher density of points near the ND sample axis in Fig. 5. While a large proportion of the pole figure is covered with this scheme, the oversampling of ND is also demonstrated by the similar magnitude and sign of bias errors to the single ND orientation for the TRIP steel textures in Fig. 7. Other equal angle pole figure methods, where the rotational increment is fixed for all tilt angles, may also suffer from this type of bias error attributed to oversampling. Rotation alone results in bias errors identical to the ND (rotation axis) sample orientation. This is caused by the coincidence of the diffraction vector and the rotation axis. With this alignment, no additional sample orientations are measured as there is no change in the orientation of the diffraction vector as a function of rotation angle. Rotation of the sample may improve results, but only as an effect of increasing the interaction volume of the sample (*i.e.* if there are too few grains in the interaction volume for sufficient sampling). Translation of the sample perpendicular to the diffraction plane may provide similar results in increasing the interaction volume.

Hexagonal grids, as proposed in hypothesis (4), stand out as the best method studied thus far to avoid bias errors. These grids minimize oversampling of any single sample orientation and therefore effectively minimize texture effects in the phase fraction calculation. Even without the further improvements discussed below, four peaks and a partial hexagonal grid produced bias errors less than $\pm 5\%$ of the value for all textures shown in Figs. 3 and 4.

There are two obvious objections to the widespread use of hexagonal grids that should be addressed: the effects of high tilt angles in reflection mode and the additional time required. With respect to the limitations on tilt angle in reflection mode, a solution has been provided in the literature, but not applied to phase fraction measurements. Lopata & Kula (1962) first presented the idea of mounting a sample such that the [111] sample axis is the surface normal. This idea was repeated in the classic texts of Klug & Alexander (1974) and Cullity (1978). However, in all cases this was only introduced as a method to perform pole figure measurements, not as a method to reduce bias errors due to texture in phase fraction measurements. With this sample shape, the ND, TD and RD sample axes are separated by 55° from the sample surface, well within the range of most reflection mode XRD systems, and reasonably free of significant defocusing effects. Additionally, any crystal symmetry should be measurable, as long as there is orthotropic sample symmetry. Including orthotropic sample symmetry would also allow for measuring only on a quarter of the pole figure, thus limiting the oversampling of RD and TD, which are measured twice compared with once for ND.

The other downside to the hexagonal grid methods is the time required. As the number of peaks or discrete sample orientations is increased in these methods, there will be a corresponding increase in the time required for motor motion and each phase fraction measurement. The 5° full hex grid implemented here requires nearly 1000 individual orientations. The coarse and partial grids explored here dramatically reduce the counting time compared with the 5° full hex grid, and only the partial grid with two peak pairs results in bias errors greater than $\pm 5\%$ for the textures explored. However, even the coarse grid would require more time than the conventional tilt and rotation methods for the same number of peak pairs. It may be possible to decrease the time required for a hexagonal grid further, as the methods presented here have not been optimized for minimum time and bias errors as a function of density of points and/or pole figure coverage required. For example, if orthotropic sample symmetry is assumed, the measurement time could be reduced by 25%. However, optimization of the density of points would require some prior knowledge of the crystallographic texture under investigation and its sharpness.

The results shown in Fig. 10(b) demonstrate the success of ASTM E975 in ensuring the texture is near random, albeit while confining the standard to a narrow range of applicability not often seen in real materials. None of the textures shown in Figs. 3 or 4 (other than uniform) would meet the applicability criteria, providing no standard method of measuring phase fractions in materials with a similar degree of texture sharpness. The introduction of constraints on the ferrite peak ratios in ASTM E975, which were not included in prior work (Jaczak et al., 1980; Hinton, 1987), is critical to avoid bias errors introduced by the ferrite phase, as demonstrated in Fig. 6.

As stated previously, the current study did not consider Rietveld or full-pattern fitting methods, since the aim was to quantify the effects of texture on the methods of phase fraction measurements that utilize integrated intensity ratios. In full-pattern techniques, diffraction spectra from separate sample orientations can be related and simultaneously used to fit the phase fraction, texture and many other parameters. These methods generally fit other factors which are known to affect the diffraction spectra, and phase fraction is only one. A complete study of the diffraction information necessary for simultaneous determination of the phase fraction and texture with minimal bias errors has yet to be reported in the literature. Similarly, techniques that required prior knowledge of the texture or used the diffraction data to calculate and correct for the texture, such as those proposed by Bonarski et al. (1991), hold promise, but were not included in the work presented here.

The focus of this investigation has been to quantify the magnitude of bias errors caused by crystallographic texture and provide a framework for the assessment of these bias errors. There are likely to be many possible alternative sampling schemes that could minimize time and have tolerable bias errors, and readers are encouraged to test and publish innovative schemes. The provided data set (Creuziger et al., 2018) should assist readers in these efforts. The bias errors shown in the present work are a lower bound on the experimental error associated with quantifying phases in multiphase materials, since this analysis only considered effects of crystallographic texture. Many other factors affect the errors in a diffraction-based experiment, such as signal to noise, atom positions, absorption effects, choice of peak fitting models and deviations from the ideal diffraction geometry.

5. Conclusions

A series of numerical experiments, which sought to quantify the effects of texture on the measurement of austenite phase fractions, have been performed in this work and the following conclusions have been drawn:

1. The results demonstrate that texture, the number of peak pairs and the sampling method have a drastic effect on phase fraction measurements.
2. Averaging over a greater number of peaks will generally result in improved accuracy, but the specific texture of a sample may lead to larger bias errors as the number of peaks is increased. Measuring single sample orientations, even when multiple peaks are considered, will result in a large range of possible bias errors due to the texture of the sample.
3. Oversampling during continuous tilt and rotation measurements leads to greater bias errors than ring or hexagonal sampling schemes for most textures.
4. Hexagonal grids produced minimal bias errors and demonstrate a robust way to measure phase fractions in textured materials.

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Figure 1.

Plot of simulated intensity *versus* 2θ for a 0.25 austenite phase fraction and a 0.75 ferrite phase fraction. Crystallographic textures for both phases were set to a uniform (random) distribution. Annotations above the plot show the phase (A – austenite, F – ferrite) and hkl plane of each diffraction peak.

Figure 2. ODF key for cubic crystal symmetry and orthotropic sample symmetry. Selected orientations listed in Table 1 are highlighted in this figure. Only the $\phi_2 = 45^\circ$ cross section is shown.

Figure 3. Example ODFs for austenite (first column) and ferrite (second column) for TRIP 700 (first row) and TRIP 780 (second row) materials. Only the $\varphi_2 = 45^\circ$ cross section is shown.

Figure 4. Common texture components observed in steel. Only the $\varphi_2 = 45^\circ$ cross section is shown. The distribution about each individual orientation was chosen such that the intensity range was comparable to the experimental textures of those in Fig. 3. The φ_1 and Φ axis conventions are the same as shown in Fig. 3.

Figure 5.

Sampling schemes used to explore sample orientation summation. Plot (a) shows the RD, TD and ND axes of an equal area stereographic pole figure. Plot (b) shows the sample orientations for four single sample orientations. Plot (c) depicts sample orientations for ring sampling schemes, labeled by the sample axis aligned to be coincident with the incoming beam. Plots (d), (e) and (f) show the sample orientations possible with rotation, tilt and simultaneous tilt & rotation, respectively. Note that rotations were performed along the sample ND axis, and tilts were performed about the sample TD axis. Plots (g), (h) and (i)

show sample orientations aligned with three types of hexagonal grids, 5° full pole figure coverage, 22.5° full pole figure coverage and 5° partial pole figure coverage, respectively.

Figure 6. Intensity versus 2θ plots for (a) uniform crystallographic texture, (b) texture matching the TRIP 700 example and (c) texture matching the TRIP 780 example. Only the 2θ range for the two peak pairs and the ND single sampling scheme are shown.

Figure 7.

Calculated austenite phase fractions for the two experimental TRIP steel textures shown in Fig. 3 as a function of sampling scheme (shown in Fig. 5) and number of peaks. The end of the bar indicates the calculated value, with the height of the bar a visualization of the bias error from the nominal value of 0.25 austenite. Dashed horizontal lines depict $\pm 5\%$ of value bias errors. The x axis labels apply to both plots.

Figure 8.

Calculated austenite phase fractions as a function of sampling method and number of peaks for 91 texture combinations (fixed HW = 20°). Each circle indicates the calculated value from one texture combination (austenite and ferrite, listed in Fig. 4). The horizontal black line at 0.25 shows the nominal value of austenite and the dashed horizontal lines depict $\pm 5\%$ bias errors. Note the larger austenite phase fraction range compared with Fig. 7.

Figure 9.

Calculated austenite phase fractions as a function of average texture index for 945 texture combinations with varying texture sharpness (half-width). Only the ND single sample orientation is shown. Each circle indicates a value calculated from one combination of texture components selected for the ferrite and austenite phases. The horizontal black line at 0.25 shows the nominal value of austenite, and the dashed horizontal lines depict $\pm 5\%$ bias errors. Vertical lines showing the texture indices of TRIP 700 and TRIP 780 and the ASTM E975 applicability limit are included as a guide. Note the larger phase fraction range compared with Figs. 7 and 8.

Figure 10.

(a) Details of the phase fractions near uniform ($= 1.0000$). Small increases in the texture index can still result in bias errors in the phase fraction. (b) Subset of data shown in Fig. 9 that meet ASTM E975 criteria for 'near random'. No additional points above a texture index of 1.06 met the ASTM E975 criteria. Only the ND single sample orientation is shown. Dashed horizontal lines depict $\pm 5\%$ bias errors. Note the change in scales from Fig. 9.

Table 1

Orientations of typical texture components found in ferrite (F) and austenite (A) phases in steel, adapted from Bleck et al. (1991), Jonas et al. (2006) and Kestens & Jonas (2005), and the texture index for a half-width (HW) of 20°.

Fiber orientations.

Name	Phase	<i>hkl</i>	Sample direction	Texture index at HW = 20°
Alpha fiber	F	(110)	RD	1.4298
Gamma fiber	F	(111)	ND	1.5678
Beta fiber	A	$\hat{\tau}$		1.3827

Single orientations.

Name	Phase	<i>hkl</i>	<i>uvw</i>	Texture index at HW = 20°
Cube	A	(001)	[100]	3.0381
Shear (rotated cube)	F	(001)	[1 $\bar{1}$ 0]	3.0381
Goss	A, F	(011)	[100]	3.0381
Rotated Goss	F	(110)	[1 $\bar{1}$ 0]	3.0381
Brass	A	(110)	[1 $\bar{1}$ 2]	1.7010
S	A	(123)	[$\bar{3}$ 64]	1.3412
Copper	A	(112)	[$\bar{1}$ 11]	1.7010
alpha1	F	(115)	[1 $\bar{1}$ 0]	2.1901
alpha2	F	(113)	[1 $\bar{1}$ 0]	1.7793
alpha3	F	(112)	[1 $\bar{1}$ 0]	1.7010
alpha4	F	(223)	[1 $\bar{1}$ 0]	1.7070
gamma1	F	(111)	[1 $\bar{1}$ 0]	1.7010
gamma2	F	(111)	[1 $\bar{2}$ 1]	1.7010
554	F	(554)	[$\bar{2}$ 25]	1.7191

$\hat{\tau}$ The Beta fiber for f.c.c. crystals is not defined as crystal and sample direction.

Table 2

Number of texture combinations with bias errors within the $\pm 5\%$ of value range, with the percentage (%) of texture combinations out of the 91 possible cases included in parentheses.

Scheme	Two peaks	Four peaks	Max unique
Single ND	13 (14)	15 (16)	32 (35)
Single RD	7 (8)	13 (14)	47 (52)
Single TD	17 (19)	7 (8)	54 (59)
Single Morris	12 (13)	34 (37)	44 (48)
Ring ND	11 (12)	66 (73)	82 (90)
Ring RD	22 (24)	58 (64)	77 (85)
Ring TD	17 (19)	59 (65)	61 (67)
Rotation	16 (18)	30 (33)	46 (51)
60° tilt	13 (14)	15 (16)	32 (35)
Tilt & rotation	18 (20)	24 (26)	49 (53)
Hex 5° full	91 (100)	91 (100)	91 (100)
Hex 22.5° full	91 (100)	91 (100)	91 (100)
Hex 5° partial	71 (78)	91 (100)	91 (100)