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Refinements in phase fraction determination of textured alloys from transmission diffraction data

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The use of high-energy synchrotron X-ray diffraction sources has become increasingly common for high-quality phase fraction measurements and microstructural evolution experiments. While the high flux, large volume illuminated and large number of diffraction vectors should reduce common sources of uncertainty and bias, the distribution of the diffraction vectors may still cause bias in the phase fraction measurement. This hypothesis of bias was investigated with example experimental data and synthetic data. The authors found that there may be bias depending on the sample texture, the distribution of diffraction vectors and the hkl planes used in the phase fraction measurement, even for nearly complete coverage of a pole figure. The authors developed a series of geometry-based correction values that reduced the measurement bias due to sampling scheme and texture in the phase fraction measurement by an order of magnitude. The efficacy of these corrections was demonstrated with application to both experimental and synthetic data.

1. Background

The use of synchrotron light sources has enabled a wide array of techniques for analyzing microstructural evolution in structural materials, often in three dimensions (Poulsen, 2004; Suter *et al.*, 2006; Robinson & Harder, 2009; Johnson *et al.*, 2008; Simons *et al.*, 2015; Hayashi *et al.*, 2019). Key improvements include the wide use of large area detectors enabling a large number of diffraction vectors (or large volume of reciprocal space) to be probed simultaneously, deep penetration of high-energy X-rays enabling transmission experiments with relatively simple diffraction geometries, and high fluxes enabling greatly reduced count times in comparison with laboratory X-ray sources.

These advances have made *in situ* quantification of phase fraction evolution during thermomechanical loading possible on a wide variety of multiphase and metastable materials (Babu *et al.*, 2002; Elmer *et al.*, 2005, 2007; Stone *et al.*, 2008; Zhao *et al.*, 2017; Weißensteiner *et al.*, 2019; Oh *et al.*, 2020). Furthermore, insufficient counting statistics and limited reciprocal-space coverage are often no longer limiting sources of error. Improved accuracy, necessary for manufacturing process qualification and model validation (Allison *et al.*, 2006; Levine *et al.*, 2020), can be achieved.

However, unless explicitly adjusted, phase fraction measurements using integrated intensities (ASTM, 2013) rely on the assumption of a uniform (or random) crystallographic texture (or preferred orientation) distribution. Diffractionbased phase fraction measurements build from the powder diffraction community, where samples are frequently

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processed to create a fine powder to minimize texture effects. This process of powdering a sample is ruled out if the asreceived sample, initial phase fractions and/or initial crystallographic texture from prior thermomechanical processing are of interest; if the processing conditions, resulting phase fractions and/or crystallographic texture evolution as a function of new thermomechanical processing are of interest; and/or if the material under investigation is composed of metastable materials.

One approach that has been employed to account for the effect of texture is to measure over a large number of diffraction vectors. Classically this has been done via a tiltand-rotate technique developed by Miller (1968) and recommended in the widely used Society of Automotive Engineers (SAE, now SAE International) publication on retained austenite measurements (Jatczak *et al.*, 1980). These and similar approaches have the tacit assumption that, if there are a large number of diffraction vectors, the texture effect is negligible.

These assumptions were tested in prior work by Creuziger *et al.* (2018*a*). As shown by Creuziger *et al.* (2018*a*,*b*), the series of diffraction vectors (or sampling scheme) used to measure phase fractions can cause oversampling of specific areas of the pole figure depending on the distribution of diffraction vectors. The oversampling occurs even for a large number of diffraction vectors such as used in the tilt-and-rotate sampling method. The oversampled regions in turn can cause bias errors in phase fraction calculations if the material under study has crystallographic texture. The magnitude and sign of the bias error were found to be a function of the sampling scheme, peak combination used in the phase fraction calculation and texture of each phase. The number of peaks used seemed to be a less significant factor than the number of diffraction vectors used and the evenness of diffraction vector distribution.

One of the sampling schemes considered by Creuziger *et al.* (2018*a*) was a diffraction ring recorded at high (infinite) energies. However, at many high-energy sources the samples may be rotated while in the beam, which results in the recording of a series of diffraction rings. An example of this approach and subsequent phase fraction calculation is shown by Phan *et al.* (2019) for data measured at the Cornell High Energy Synchrotron Source (CHESS) facility.

Phan *et al.* (2019) made a specimen from additively manufactured 17-4 stainless steel which was deformed in tension during *in situ* X-ray diffraction. Nominally, the wrought form of this alloy is in the martensitic phase with a low-to-moderate amount of crystallographic texture, whereas the additively manufactured material contains a significant volume fraction of austenite and is heavily textured. X-ray diffraction data were collected as a function of applied strain to measure evolution in the crystallographic texture and phase fraction simultaneously. The results showed the austenite phase fraction started at approximately 0.54 (zero strain) and decreased to 0.12 at a strain level of 0.03. The austenitic phase had significantly more crystallographic texture in comparison with the martensitic phase, but there was minimal evolution of the texture in this strain range.

The phase fraction calculations of Phan *et al.* (2019) were performed by summing all diffraction data to create a single intensity 2θ plot at each deformation step. While it is clear that the method of rotating a sample and collecting a complete diffraction ring results in a large number of diffraction vectors and produces nearly complete pole figures, it is not clear if the phase fractions calculated by summing all diffraction data are free of bias errors. This paper assesses the hypothesis that this sampling scheme may result in bias errors.

2. Methods

Parameters described by Phan *et al.* (2019) and data provided in the companion data publication (Pagan, 2019) were used as an example data set. Only the initial load point was considered as the texture evolution in Phan *et al.*'s study was minimal. Based on the experimental parameters, geometrical corrections to the intensity data due to oversampling were developed. Paralleling the prior work of Creuziger *et al.* (2018*a*), synthetic textures were used to investigate the robustness of the rotated ring sampling scheme to particular texture components and applied to a reduced series of rotation angles.

2.1. Example data parameters

The following list of key parameters from the Phan *et al.* (2019) paper were implemented in this work:

(i) Energy of 61.332 keV (wavelength 0.0202 nm).

(ii) The axis and angle conventions used in this paper and shown in Fig. 1 are the same as those used by Phan *et al.* (2019).

(iii) Debye rings are recorded at a given rotation (ω) and each Debye ring is divided into 72 azimuthal (η) bins 5° wide. Midpoints of the bins are used for the diffraction vectors and start at $\eta = 0^{\circ}$.

(iv) A series of Debye rings were collected in 36 increments from $\omega -90^{\circ}$ to 90° (5° step) as the specimen was continuously rotated about the Y axis.





Schematic of a diffraction experiment at CHESS with axis conventions for X, Y, Z, η , ω and 2θ .

Adam Creuziger et al. • Refinements in phase fraction determination 1481 electronic reprint (v) Peak profiles at each η , ω and 2θ were used to calculate the intensity and diffraction vector. The resulting intensities were summed together for all diffraction vectors for each *hkl*.

(vi) Three austenite (γ phase) *hkls* (111), (200) and (220) and three martensite/ferrite (α'/α phase) *hkls* (110), (200) and (211) were measured, but only the austenite (111) and martensite/ferrite (110) were used to calculate the reported phase fractions.

(vii) The resulting phase fractions were 0.54 austenite and 0.46 martensite/ferrite, prior to any applied load.

2.2. Example intensity data

Intensity data for the plots shown by Phan *et al.* (2019), available from the data publication (Pagan, 2019), were used in this work, expressed in multiples of a uniform (or random) distribution (MUD or MRD). Additional notes and code to import these data have been included as release 2.1.1 by Creuziger *et al.* (2018b). Fig. 2 shows the pole figures calculated from the work of Pagan (2019) plotted using the software package *MTEX* (Hielscher & Schaeben, 2008). By depicting the data at each diffraction vector instead of as a filled contour, the distribution of the diffraction vectors over the entire pole figure is shown. Visible in this plot is how the density of points increases when traversing from the *XZ* plane to near the rotation axis (*Y*) and the presence of a small unmeasured region parallel to the rotation axis (*Y*). Instead of

projecting the intensity data onto a sphere, equal-area stereographic projection was used and only the top of the sphere is shown.

2.3. Bias error quantification

The code developed by Creuziger *et al.* (2018*b*) was used to assess the bias errors from the sampling scheme used by Phan *et al.* (2019). Using the guidance of Phan *et al.* (2019) and with the assistance of those authors, the sampling scheme used was added to the code base of Creuziger *et al.* (2018*b*) and termed 'rotated ring'. A depiction of this sampling scheme with an ω range of $\pm 90^{\circ}$ is shown in Fig. 3(*a*), using the parameters described above. For each *hkl*, the sampling scheme was adjusted to account for the slight change in 2θ for each reflection.

Two additional sampling schemes were tested for bias errors. As some of the ancillary equipment used at highenergy sources does not permit complete rotation of the sample, the results from these schemes were of additional interest. These schemes, also depicted in Fig. 3, represent more limited ranges in ω of $\pm 60^{\circ}$ and $\pm 30^{\circ}$.

2.4. Phase fraction calculations

A common method for calculating phase fractions via diffraction is the reference intensity ratio method (Dinnebier & Billinge, 2009; Pecharsky & Zavalij, 2009). This is frequently



Figure 2

Equal-area stereographic projection pole figures replotted using the *MTEX* software package (Hielscher & Schaeben, 2008) reproduced from Phan *et al.* (2019) via the data set (Pagan, 2019). Pole figures of (top row) the austenite phase and (bottom row) the martensite phase with the *hkl* annotated above each figure. Pole figure normalized intensities are displayed at individual diffraction vectors. Intensity color scale is the multiples of a uniform (or random) distribution (MUD or MRD).

employed for phase fractions in steels (ASTM, 2013). The intensity normalized by theoretical intensity value \bar{I}^{hkl} for each *hkl* reflection is calculated as

$$\bar{I}^{hkl} = \frac{I^{hkl}}{R^{hkl}},\tag{1}$$

where the integrated intensity I^{hkl} is measured from the diffraction data and theoretical intensity R^{hkl} is calculated from the diffraction setup and specimen parameters (Dinnebier & Billinge, 2009; Pecharsky & Zavalij, 2009). Assuming there are only two phases (austenite γ and ferrite/martensite α'), the phase fraction of austenite V_{γ} is then calculated from the rule of mixtures as

$$V_{\gamma} = \frac{\bar{I}_{\gamma}}{\bar{I}_{\gamma} + \bar{I}_{\alpha'}},\tag{2}$$

where the intensity normalized by a theoretical value for each phase (j) is the average over the (m) *hkl* planes measured:

$$\bar{I}_j = \frac{1}{m} \sum_{1}^{m} \bar{I}^m.$$
(3)

These equations contain the tacit assumption that there is no crystallographic texture, *i.e.* there is a 'random' distribution of diffracting particles or uniform crystallographic texture. For materials with crystallographic texture, the integrated intensity $I^{hkl}(\alpha, \beta)$ is also a function of the sample texture and the location of the diffraction vector (α, β) on the pole figure. To represent crystallographic texture data, the intensity data normalized for pole figures $\hat{I}^{hkl}(\alpha, \beta)$ are calculated using equation (4) (Kocks *et al.*, 1998):

$$\hat{I}^{hkl}(\alpha,\beta) = I^{hkl}(\alpha,\beta) \frac{\sum_{i=1}^{i} \sin \alpha_{i}}{\sum_{i=1}^{i} I^{hkl}(\alpha,\beta) \sin \alpha_{i}}.$$
(4)

The term \hat{I}^{hkl} will be used to indicate the pole figure normalized intensity over all measured (α, β) for a single *hkl* and \hat{I}_j to indicate the average pole figure normalized intensity for all *hkl* measured for phase *j* as in equation (3).

The pole figure normalized intensity \hat{I}^{hkl} from an untextured material or idealized sampling scheme that completely accounts for texture would result in a value equal to 1 (since

uniform texture has MUD = 1). To include these data in the phase fraction calculation, an imposed austenite phase fraction ξ is required to convert between the pole figure normalization and the theoretical intensity normalization:

$$\bar{I}^{hkl} = \xi \hat{I}^{hkl}.$$
(5)

Rewriting equation (2) to use the pole figure normalized intensity for the phase fraction calculation results in

$$V_{\gamma} = \frac{\xi \hat{I}_{\gamma}}{\xi \hat{I}_{\gamma} + (1 - \xi) \hat{I}_{\alpha'}}.$$
 (6)

In this work the calculations for bias errors due to texture and sampling scheme use the normalized intensities from the pole figure data \hat{I}^{hkl} . To assess bias errors, an imposed phase fraction of ξ is set and the calculated austenite volume fraction V_{γ} is determined as a function of texture, sampling scheme and *hkl*.

Using this approach sidesteps the majority of factors used to calculate the theoretical intensity values but preserves the preferred orientation (or crystallographic texture) factor [equation 8.41 of Pecharsky & Zavalij (2009)] as a function of position on the pole figure in determining V_{γ} . In addition, any deviation from the value of ξ in the calculated phase fraction V_{γ} is a bias error caused by sampling and texture. Conversely equation (6) can be reversed and the bias errors in the calculated phase fraction and normalized intensities can be used to determine the 'true' phase fraction ξ^* for a specific sampling scheme and peak combination.

2.5. Correction factors

While it is hypothesized that the $\omega \pm 90^{\circ}$ sampling scheme may oversample portions of the pole figure near the Y axis as shown in Fig. 2 and cause bias errors in the phase fraction, the nearly complete pole figure coverage and regularity of the ω $\pm 90^{\circ}$ sampling scheme permit correction factors to be developed for this particular scheme. Correction factors are only possible for the $\omega \pm 90^{\circ}$ sampling scheme as this scheme has nearly complete pole figures but uneven coverage of the pole figures. Additionally, the nearly complete pole figure coverage can be used to accurately estimate an orientation distribution



Figure 3

Diffraction vectors for a range of $\omega \pm 90^{\circ}(a)$, $\omega \pm 60^{\circ}(b)$ and $\omega \pm 30^{\circ}(c)$ rotated ring sampling schemes shown on an equal-area stereographic projection. Points assume 5° increments in ω rotation about the Y axis, a 5° binning in the azimuthal direction η of the detector and $\theta = 2.5^{\circ}$. Equal-area stereographic projection.

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function (ODF) (Bunge, 2015). For the $\omega \pm 60^{\circ}$ and $\omega \pm 30^{\circ}$ schemes, there are significant gaps in pole figure coverage, resulting in partial pole figures. While partial pole figures can be used for ODF estimation, the authors are not aware of any investigation using partial pole figures such as those shown in Fig. 3 to determine the accuracy of the estimated ODF. As the accuracy of the ODF is not established, corrections for these sampling schemes were beyond the scope of this work.

The first correction factor takes into account the variation in distribution density of diffraction vectors. There is a higher density of points near the Y axis, visible in Figs. 2 and 3. While the stereographic representation distorts this to some extent, the higher density is due to the fact that the areas covered by $\eta = 0^{\circ}$ (XZ plane) and $\eta = 90^{\circ}$ (near the Y axis) are quite different, while the number of points in each area remains the same (ω rotation increments). However, the intensity data for these two areas are typically in practice summed without considering this difference in area, as was the case in the work of Phan *et al.* (2019).

To account for the difference in area, it is possible to multiply the intensity data by a weighting factor. This weighting factor would be a function of the area covered. To calculate the area covered, the pole figure sphere is discretized into spherical zones (or spherical segments). A cross section of a spherical zone is shown in Fig. 4(a). The cross section does not have the distortion inherent in the spherical projections used in Figs. 2 and 3. Examples of the change in area as one approaches the Y axis are shown in Fig. 4(b). Using the boundaries between azimuthal (η) bins as the bases $(r_1 \text{ and } r_2)$ of a zone of the sphere [shaded region in Fig. 4(a)], it is possible to calculate the area covered by each azimuthal (η) bin. Note that when rotating by ω about the Y axis, the Y component of the diffraction vector does not change for each η bin. The area for each azimuthal bin is the area of the zone $(A_z = 2\pi rh)$, where h is the height of the segment. Normalizing this area by the area of the sphere $(A_s = 4\pi r^2)$ and a unit



Figure 4

(a) Cross section sketch of a spherical zone (blue). Upper (r_1) and lower (r_2) boundaries of the zone (bases of a zone) are depicted. (b) Depiction of the difference in area on a sphere. Bases of a spherical zone with $\theta = 2.5^{\circ}$ and azimuthal bins of $\eta = 0^{\circ}$ (blue) and $\eta = 90^{\circ}$ (red) are shown. Baselines are set by the midpoint between adjacent azimuthal bin boundaries except where noted in the text.

sphere (r = 1), the weighting factor reduces to h/2 for each zone.

Two locations require additional adjustment. The midpoint between azimuthal bin boundaries is used for the bases with the exception of points nearest the rotation axis ($\eta = 90^{\circ}$, $\eta = 270^{\circ}$). For these points there is no additional point closer to the Y axis to determine the midpoint. In this case one base is set to the azimuthal bin boundary and the other is placed at the midpoint of this arc (90°) and the weighting is calculated on the basis of these points. The second adjustment occurs at an ω rotation of $\pm 90^{\circ}$ as these points appear on both the top and bottom of the pole figure. An additional factor of $\frac{1}{2}$ is applied to points at $\omega = \pm 90^{\circ}$. All of these weight factors are summed and normalized such that the sum of points equals 1.

The second correction factor accounts for the small unmeasured region along the Y axis (rotation axis). The weighting factors alone cannot correct for values not measured. However, in the $\omega \pm 90^{\circ}$ sampling scheme there is sufficient pole figure data to calculate an ODF. From the ODF, recalculated pole figures can be calculated and the intensity data from these recalculated pole figures used to fill in the missing region. In this second correction factor, the intensity parallel to the Y axis $\bar{I}_{\parallel Y}^{hkl}$ in the recalculated pole figures and the unmeasured area calculated as a spherical cap from the azimuthal bin boundary at $\eta = 90^{\circ}$ or $\eta = 270^{\circ}$ were used. When normalized by the area of a unit sphere the area of the spherical caps at $\pm Y$ is equal to $A_c = 1 - \cos \theta$. The intensity data from the spherical cap are then combined with the weighted normalized intensity via equation (7):

$$\bar{I}^{hkl} = A_{c} \bar{I}^{hkl}_{\parallel Y} + \bar{I}^{hkl}_{weighted} (1 - A_{c}).$$
(7)

2.6. Synthetic textures

To check the robustness of the phase fraction measurement, in addition to the example texture data from Phan *et al.* (2019)

shown in Fig. 2, the phase fractions from synthetic texture components were tested using synthetic ODFs. This paper uses the same series of seven austenitic textures (uniform, cube, Goss, beta fiber, brass, S and copper) and 13 martensitic (ferritic) textures [uniform, shear, Goss, rotated Goss (RGoss), alpha fiber, alpha1, alpha2, alpha3, alpha4, gamma fiber, gamma1, gamma2 and 554 (O554)] as used by Creuziger *et al.* (2018*a*).

3. Results

Fig. 5 shows the diffraction vectors for $\theta = 2.5^{\circ}$ and $\theta = 5.0^{\circ}$, similar to the data from Phan *et al.* (2019). Echoing the data in Fig. 2 there is a higher density of points near the rotation axis and a small region that is unmeasured near the

rotation axis. This unmeasured region is correspondingly larger at larger values of 2θ . While it is clear that the density of points varies in this sampling scheme, it may be hard to interpret from the displayed scattering vectors alone. However, a method to depict the density of points is available in the work of Kington (2015) as an orientation density contour map. The pole figure is discretized into a series of grid points and the normalized number of orientations within a patch 1% of the total area is returned for each grid point. While generally correct, the discretization inherent in this method can lead to visible artifacts from the grid size and smoothing. Figs. 5(b) and 5(e) show that the points near the $\pm Y$ axis are oversampled with oversampling multiples (orientation densities) exceeding 4, supporting our hypothesis. In addition, a line with a higher density of orientations from +Y to -Y is visible where X = 0. This is due to the duplication of points with diffraction vectors symmetrically equivalent at a rotation of $\pm 90^{\circ}$. Figs. 5(c) and 5(f) depict the sampling scheme when corrected by the weighting factor described in the Correction factors section above. As shown, applying the weighting factor produces a nearly uniform distribution of oversampling multiples with a value of 1.

3.1. Example data

From the pole figures shown in Fig. 2, ODFs were calculated for each phase. These ODFs were used to recalculate the pole figures used in the phase fraction calculations. Fig. 6 shows the recalculated austenite pole figures and the recalculated martensite pole figures. There are slight differences between the original pole figures and the recalculated data, particularly visible in the martensite (211) data.

Table 1

Pole figure normalized intensity values \hat{I}^{hkl} and relative error for the unweighted $\omega \pm 90^{\circ}$, $\omega \pm 60^{\circ}$ and $\omega \pm 30^{\circ}$ range sampling schemes and example data from Phan *et al.* (2019).

A value of 1 represents an ideal sampling scheme that corrects for texture and/ or a uniform texture distribution in the sample. Values are quoted beyond the significant figures for replicability testing.

	θ	$\omega \pm 90^{\circ}$	Bias error	$\omega \pm 60^{\circ}$	Bias error	$\omega \pm 30^{\circ}$	Bias error
A111	2.8462°	1.0386	3.86%	1.0515	5.15%	1.1422	14.22%
A200	3.2870°	1.0599	5.99%	1.1190	11.90%	1.2100	21.00%
A220	4.6511°	0.9717	-2.83%	0.9566	-4.34%	0.8712	-12.88%
Average		1.0234	2.34%	1.0424	4.24%	1.0745	7.45%
M110	2.7904°	1.0483	4.83%	1.0721	7.21%	1.1668	16.68%
M200	3.9478°	0.9575	-4.25%	0.9626	-3.74%	0.8659	-13.41%
M211	4.8370°	0.9954	-0.46%	0.9892	-1.08%	0.9888	-1.12%
Average		1.0004	0.04%	1.0080	0.80%	1.0072	0.72%

The pole figure normalized intensity for each reflection \hat{I}^{hkl} is calculated from the pole figures shown in Fig. 6 for the sampling schemes shown in Fig. 3. Table 1 lists the normalized intensity for each hkl and sampling scheme in the unweighted condition. The θ angle was adjusted to match the position of the hkl peak. Any deviation from a value of 1 indicates a bias error in the normalized intensity. Bias errors are also shown as a relative error value.

Considering the data from the $\omega \pm 90^{\circ}$ case there is a clear trend: if the intensity parallel to the Y axis is >1 MUD (visible in Fig. 6), the unweighted data are also >1 (Table 1). This is consistent with oversampling occurring along the Y axis as shown in Fig. 5. However, despite the oversampling near the Y direction exceeding $4\times$ as shown in Fig. 5, there is not a



Figure 5

Locations of the diffraction vectors projected onto an equal-area pole figure for $\theta = 2.5^{\circ}$ as (a) a series of rotated diffraction rings, (b) the density of points as a contour plot unweighted and (c) the density of points weighted. Subfigures (d), (e) and (f) repeat these for $\theta = 5^{\circ}$.

commensurate bias error in this sampling scheme. The largest relative errors are approximately 5%. If the ω range is restricted, the bias error increases significantly, exceeding 10% in the $\omega \pm 60^{\circ}$ case and exceeding 20% in the $\omega \pm 30^{\circ}$ case. For the textures observed by Phan *et al.* (2019), the largest errors are in the austenite (200) peak and the martensite (110) peak.

Phan *et al.* (2019) used the A111 and M110 peaks and $\omega \pm 90^{\circ}$ for the phase fraction calculation in that work, reporting phase fractions of 0.54 austenite and 0.46 martensite prior to deformation. As shown in Table 1, both of these peaks have bias errors of the same sign and nearly the same value. Using these values, the true phase fraction ξ^* would be 0.542, which is more precise than the uncertainty implied from the significant figures in their work. If, instead of using a single peak, the intensity values for all three austenite peaks and all three martensite peaks are averaged, the calculated phase fraction V_{γ} would change to be slightly outside the significant figures reported [$\xi = 0.54$ in the work of Phan *et al.* (2019), $V_{\gamma} = 0.546$ in this work], owing to the non-offsetting bias errors in the austenite phase.

Examining the $\omega \pm 60^{\circ}$ and $\omega \pm 30^{\circ}$ cases indicates that a similarly small error in phase fraction would occur for this example texture and use of only the single peak pair, with V_{γ} values of 0.535 and 0.534, respectively, for $\xi^* = 0.54$. Including three austenite and three martensite peaks results in larger values of V_{γ} of 0.548 and 0.556, respectively, the larger bias errors in phase fraction again owing to the non-offsetting bias errors in the normalized intensity in the austenite phase.

The results above demonstrate that bias errors are observed due to the sampling scheme when the correction factors

Table 2

Normalized intensity values for the unweighted, weighted correction, and weighted and with unmeasured region correction cases for the range of $\omega \pm 90^{\circ}$.

Relative error values for each peak and the average of all three peaks are included. Values are quoted beyond the significant figures for replicability testing.

	Unweighted	% error	Weighted	% error	Unmeasured region	% error
A111	1.0386	3.86%	0.9987	-0.13%	0.9991	-0.09%
A200	1.0599	5.99%	0.9975	-0.25%	0.9989	-0.11%
A220	0.9717	-2.83%	1.0039	0.39%	1.0027	0.27%
Average	1.0234	2.34%	1.0000	0.00%	1.0002	0.02%
M110	1.0483	4.83%	0.9985	-0.15%	0.9991	-0.09%
M200	0.9575	-4.25%	1.0012	0.12%	1.0006	0.06%
M211	0.9954	-0.46%	1.0005	0.05%	1.0002	0.02%
Average	1.0004	0.04%	1.0001	0.01%	0.9999	-0.01%

described in the *Methods* section are not applied. The effect of applying each correction factor is shown in Table 2. Note that this technique is only applied in the $\omega \pm 90^{\circ}$ case due to the large gaps in pole figure coverage in the $\omega \pm 60^{\circ}$ and $\pm 30^{\circ}$ sampling schemes. The relative error for each peak is reduced by an order of magnitude or more when the weighting factor is included. The largest relative error is 0.39% for the A220 case, with the relative error for the other *hkl* peaks of a similar magnitude. While the weighting factor alone does decrease error, it does not eliminate error owing to the missing measurement points along the rotation axis. In the weighted case, the sign of the error now has an inverse relationship to the intensity parallel to the Y axis and the unweighted case.



Figure 6

(Top) Recalculated austenite pole figures. (Bottom) Recalculated martensite pole figures.

For this example data set, the bias errors for the average normalized intensity values are near zero due to offsetting bias errors in the three *hkl* average.

Normalized intensity values after applying the second correction for the unmeasured region are also shown in Table 2. Most of the relative errors decrease by approximately one-half when compared with the weighted scheme. However, the error in the austenite (220) does not decrease substantially.

3.2. Synthetic data

The results above demonstrate that, while there were bias errors in the normalized intensity, the resulting bias errors in phase fraction were minimal due to the texture of their sample and the peak pairing choice of Phan *et al.* (2019). However, this is only one example texture. To check the robustness of the phase fraction measurement to different peak pairings, ω range and weighted sampling scheme, the volume fractions for a series of texture components were calculated. In these plots the imposed phase fraction ξ is set to 0.54 for comparison with the experimental data above. The angle θ had minimal influence on the volume fractions, so a value of $\theta = 2.79^{\circ}$ (M110) was used for all calculations. A half-width of 20° was set for each of the texture components. The phase fractions for each combination of textures are expressed as a heatmap matrix.



Figure 7

Calculated phase fractions for a series of austenite and martensite texture components. Using the A111/M110 peak pairing, range of $\omega \pm 90^{\circ}$, unweighted.



Figure 8

Calculated phase fractions for a series of austenite and martensite texture components. Using the A111/M110 peak pairing, range of $\omega \pm 90^{\circ}$, weighted.

The A111/M110 peak pairing and $\omega \pm 90^{\circ}$ unweighted sampling scheme is shown in Fig. 7. The largest bias error occurs for the austenite brass and martensite rotated Goss component combination, with a calculated phase fraction $V_{\gamma} =$ 0.612, overestimating the imposed phase fraction $\xi = 0.54$ by 14% of value. The most significant underestimation occurs for the austenite cube and martensite shear component combination, with a calculated phase fraction $V_{\gamma} = 0.473$, underestimating by 13% of value. The austenite textures tend to have a slightly more significant effect, shown by the common row colors. Martensite textures (column colors) seem to have less of an effect.

The A111/M110 peak pairing and $\omega \pm 90^{\circ}$ weighted sampling scheme is shown in Fig. 8. The bias errors are substantially reduced for all texture components. With the weighting correction, the range of calculated phase fractions V_{γ} is 0.542–0.538, under- or overestimating by 0.3% of value.

If the $\omega \pm 90^{\circ}$ unweighted sampling scheme uses all three measured austenite *hkls* (111), (200) and (220) and three martensite *hkls* (110), (200) and (211) to calculate the average normalized intensity, the calculated volume fractions are as shown in Fig. 9. The range of calculated phase fractions V_{γ} is 0.557–0.524. The bias errors are substantially reduced from the single peak pair used in Fig. 7, but are larger than if the weighting scheme is used as in Fig. 8. Again, the austenite textures tend to have a slightly more significant effect, with the exception of the martensite rotated Goss texture components.

The A111/M110 peak pairing and $\omega \pm 60^{\circ}$ unweighted sampling scheme is shown in Fig. 10. The overall pattern of bias errors is similar to that seen in Fig. 7 for the $\omega \pm 90^{\circ}$ data. The largest bias error occurs again for the austenite brass and martensite rotated Goss component combination, with a calculated phase fraction $V_{\gamma} = 0.613$, slightly greater than the $\omega \pm 90^{\circ}$ case. The most significant underestimation occurs for the austenite copper and martensite Goss combination, with a calculated phase fraction $V_{\gamma} = 0.486$. With this sampling scheme the austenite textures and martensite textures seem to have approximately even effects, with the exception of the austenite Goss and brass textures. As stated above, weighted sampling schemes for $\omega \pm 60^{\circ}$ and $\omega \pm 30^{\circ}$ are outside the scope of this paper due to insufficient pole figure coverage.



Figure 9

Calculated phase fractions for a series of austenite and martensite texture components. Using the average intensity from the three austenite and three martensite peaks, range of $\omega \pm 90^{\circ}$, unweighted.

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Figure 10

Calculated phase fractions for a series of austenite and martensite texture components. Using the A111/M110 peak pairing, range of ω $\pm60^\circ,$ unweighted.



Figure 11

Calculated phase fractions for a series of austenite and martensite texture components. Using the A111/M110 peak pairing, range of $\omega \pm 30^{\circ}$, unweighted.

The A111/M110 peak pairing and $\omega \pm 30^{\circ}$ unweighted sampling scheme is shown in Fig. 11. The overall pattern of bias errors is also similar to that seen in Fig. 7 for the $\omega \pm 90^{\circ}$ data and Fig. 10 for the $\omega \pm 60^{\circ}$ data. The largest bias error occurs in this case for the austenite Goss and martensite rotated Goss component combination, with a calculated phase fraction $V_{\gamma} = 0.628$. The most significant underestimation now occurs for the austenite cube and four different martensite textures (gamma1, gamma2, gamma fiber, O554), with calculated phase fractions $V_{\gamma} = 0.408$ or 0.409. Like the $\omega \pm 90^{\circ}$ case, the austenite textures seem to dominate the phase fraction values. The austenite cube texture leads to significant underestimation of the phase fraction, and the martensite rotated Goss leads to overestimation in all cases except when paired with the austenite cube texture.

4. Discussion

The hypothesis that the rotated ring sampling scheme employed by Phan *et al.* (2019) may result in bias errors is supported. These bias errors have relative errors on the order of 5% for the unweighted $\omega \pm 90^{\circ}$ sampling scheme. The large number of points and wide coverage of the pole figure in the ω $\pm 90^{\circ}$ sampling scheme seem to help minimize bias errors, even with uneven distribution.

The phase fractions reported by Phan *et al.* (2019) have no significant bias error due to offsetting bias errors in the two peaks used in the phase fraction calculation. While phase fraction calculations are possible with only two peaks, this method lacks many diagnostics that could address other sources of error. If the three peaks for each phase were used in the phase fraction calculation, the normalized intensities for each *hkl* would have smaller bias errors, but counterintuitively this would result in a larger error in the phase fraction than the two peaks used by Phan *et al.* (2019) due to non-offsetting errors.

Application of the weighting factor developed in this work largely corrects for bias errors, reducing the relative error of the normalized intensities to the order of 0.3%. A second-order correction for the region not measured reduces the relative error further to approximately 0.15%. In these cases, including all three peaks in the average for the example data reduces the relative error to approximately 0.01%.

There are some slight differences between original and recalculated pole figures, particularly in the martensite (211) peak. This should not greatly affect the calculated phase fractions, as the texture intensity is near a uniform or untextured state (MUD = 1). However, this does demonstrate a gap in the accuracy of ODF calculations that will be explored in future work. A complementary topic for future work would be the demonstration of accurate ODF reconstruction for reduced pole figure coverage in the $\omega \pm 60^{\circ}$ and $\omega \pm 30^{\circ}$ cases. Accurate ODF reconstruction would permit corrections similar to the one performed for the $\omega \pm 90^{\circ}$ sampling scheme.

In the synthetic data, the single peak pair results in relative errors in the phase fraction in the range of $\pm 14\%$. Using the weighted values and the single peak pair, the relative error range of the phase fraction is reduced to $\pm 0.3\%$. Using all three peaks for each phase decreases the relative errors in the phase fraction to the range of $\pm 2\%$ for the unweighted scheme compared with the single pair. The weighted values for all three peaks result in values comparable to the single pair weighted scheme (not shown). Reducing the coverage in ω to $\pm 60^{\circ}$ and $\pm 30^{\circ}$ leads to slightly larger ranges of errors than the unweighted single peak pair. As there may be additional sampling schemes of interest, the tools developed are available to investigate additional textures and sampling schemes (see Creuziger *et al.*, 2018*b*).

In summary, a large number of diffraction vectors alone are not sufficient to ensure accuracy in phase fraction measurements. Increased accuracy can be achieved by even coverage of the pole figure, appropriate weighting of diffraction data and incorporation of texture data from prior knowledge, or with fitting methods that include texture models. Uneven coverage can be problematic as many subsequent analysis steps tacitly assume that all data should be equally weighted, even if there is significant oversampling of some areas. For experiments conducted without corrections, the synthetic data presented in this work provide a guide as to which textures may be particularly problematic for phase fraction determination.

5. Conclusions

The authors find that bias errors did occur due to texture, hkl peaks chosen and sampling scheme. These bias errors did not significantly affect the results of Phan *et al.* (2019) due to offsetting bias errors in each hkl used. Synthetic data show particular texture and peak choice combinations that may affect phase fraction calculations if these textures are present. The correction factors developed in this work decreased bias errors by an order of magnitude when applied to the experimental data, and reduced them to comparable levels in the synthetic data.

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