Uncertainty in retained austenite measurements applied to individual crystallographic orientations

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Abstract. A technique to measure the phase volume fraction of an individual orientation and the uncertainty in the measurement is demonstrated in this paper. The technique of complete pole figure averaging using neutron diffraction was used to assess the phase fraction of retained austenite in transformation induced plasticity (TRIP) steels and quantify the uncertainty in the phase fraction. In parallel, an ensemble of orientation distribution functions was calculated to assess crystallographic volume fractions of particular orientations and the uncertainty of these volume fractions using Monte Carlo techniques. These methods were combined to measure the retained austenite phase volume fraction of an individual orientation.

1. Introduction

The accurate measurement of the phase fraction of retained austenite in manufactured steels remains a challenge. Protocols for measurement of retained austenite using x-ray diffraction [1], [2] are often employed, despite the caveat that these methods only apply to uniform (random) texture distributions. Due to the strong crystallographic texture caused by deformation during processing, these assumptions are typically not valid for rolled sheet steel. Current interests in transformation induced plasticity (TRIP) steels have brought a new application to retained austenite measurements that are outside the scope of uniformly distributed textures.

The overall phase fraction of austenite in TRIP steels is of interest as the rate of transformation is thought to strongly affect mechanical strength and ductility [3]. There is data to indicate that the transformation will not be distributed evenly as a function of orientation, but particular stress states will cause some orientations to transform at a higher rate than other orientations [4]. Therefore, the distribution of retained austenite as a function of crystallographic orientation and the rate of transformation of a particular orientation is also of interest.

The accurate measurement of retained austenite is difficult enough [5], but to compare between retained austenite values, not only are the values themselves required, but a metric on the uncertainty as well. Recently, a technique using complete pole figure averaging using neutron diffraction was developed to measure the retained austenite in textured TRIP steels as well as provide an estimate on the uncertainty in the phase fraction [6]. These techniques provide a method to assess the overall phase fraction of retained austenite.

To assess individual orientations, volume fractions from orientation distribution functions (ODFs) are required. Pole figures recorded as part of the phase fraction measurement were used to calculate these ODFs. As with the phase fraction of retained austenite, not only is a metric on the volume fraction required, but also the uncertainty in the volume fraction.

A technique has been demonstrated to measure the uncertainty at each point in the ODF using Monte Carlo methods on the diffracted neutron counts at each location in the pole figures [7]. By combining the values and uncertainties of phase fraction and crystallographic orientation volume fraction measurements, the retained austenite volume fraction at a particular orientation can be calculated.

2. Experimental methods

These methods were applied to an as-received TRIP 700 sample. The sample was measured using the BT8 beamline at the NIST Center for Neutron Research [8]. Four complete austenite pole figures and three complete ferrite pole figures were measured on the same sample with three different pole figure resolutions on a hexagonal mesh: 4° (highest resolution), 5° and 6° (lowest resolution). The sample was not removed or remounted between scans. The acquisition time was constant per point, so the total time for a complete pole figure is approximately 1.4 times more for the 5° mesh compared to the 6° mesh, and 2.2 times longer for the 4° mesh compared to the 6° mesh.

3. Results and discussion

Pole figure data were analyzed using the program PF [9] to measure the phase fraction of retained austenite. Each point in the pole figure was summed together to create a single peak for each reflection. The channel width for peak fitting (using Gaussian functions) was set to 4 standard deviations on either side of the peak to ensure an accurate background value, with the exception of the 111 austenite peak which was done manually due to interference from the 110 ferrite peak. These peak and background values were then used to calculate the phase fraction as described in [6]. From these data, phase fractions and uncertainties were calculated, shown in Table 1.

Table 1: Phase fraction of retained austenite, calculated by the procedure described in [6].

Mesh	Retained Austenite Phase Fraction	Absolute Uncertainty	Relative Uncertainty
6° (331 pts)	0.147	0.0142	9.66%
5° (469 pts)	0.146	0.0134	9.18%
4° (721 pts)	0.145	0.0131	9.03%

Table 1 shows that the phase fraction is the same within the uncertainty of the measurement. The relative uncertainty values are nearly the same as those observed in [6]. The relative uncertainty decreases slowly as the number of points increase, decreasing only by 0.5% when twice as many points were measured.

ODF calculations were performed using the texture analysis package mTex [10]. The rest of this paper will focus on the austenite ODF. Triclinic sample symmetry and cubic crystal symmetry were applied in the analysis. In order to estimate the uncertainty, a Monte Carlo approach was implemented in mTex. Individual points on the pole figure were perturbed using Poisson noise proportional to the peak and background intensities. These perturbed pole figures were then used to calculate an ODF. Crystallographic volume fractions (VFs) of selected crystallographic orientations were extracted over a series of misorientation angle thresholds.

For the examples shown here 5000 perturbed ODFs were calculated. From this ensemble of ODFs a mean and standard deviation were calculated for seven different orientations commonly investigated in face centered cubic materials: Cube, Goss, RGoss, RCube (Shear), Copper, Brass and S. Three different misorientation angle thresholds were investigated for

each of the orientations: 30°, 10°, and 1°. The term error in this calculation is defined as (unperturbed – mean) / unperturbed. This paper does not address the absolute error quantified from a known ODF, nor is the difference between the input pole figures and the recalculated pole figures considered. Table 2 shows the ODF volume fractions for selected orientations (Cube and Brass) as a function of mesh density and misorienation angle.

Table 2: Volume fractions of Brass and Cube orientations as a function of misorientation for mesh resolutions of 6°, 5° and 4°. The terms 'Mis Angle' refers to the misorientation angle threshold, 'g' for the particular crystallographic orientation. 'Uniform VF' is the volume fraction of a uniform texture distribution for the orientation and misorientation angle indicated. 'ODF VF' refers to volume fraction extracted from the ODF calculated from the unperturbed pole figures. 'Mean VF' refers to mean volume fraction extracted from the ensemble of ODFs calculated from perturbed pole figures. 'St Dev VF' refers to standard deviation of volume fractions extracted from the ensemble of ODFs calculated from perturbed pole figures. 'Rel Uncert' refers to the standard deviation divided by the mean volume fraction. ×10

Mesh	Mis Angle	g	Uniform VF	ODF VF	Mean VF	Relative Error	St Dev VF	Rel Uncert
6°	30°	Cube	1.80E-01	1.55E-01	1.56E-01	0.81%	2.43E-03	1.56%
6°	10°	Cube	6.76E-03	6.00E-03	6.18E-03	3.01%	4.24E-04	6.86%
6°	1°	Cube	6.77E-06	6.13E-06	6.40E-06	4.26%	7.09E-07	11.08%
6°	30°	Brass	1.80E-01	2.46E-01	2.49E-01	1.11%	2.58E-03	1.04%
6°	10°	Brass	6.76E-03	2.02E-02	2.04E-02	1.10%	7.54E-04	3.70%
6°	1°	Brass	6.77E-06	2.34E-05	2.36E-05	0.75%	1.35E-06	5.72%
5°	30°	Cube	1.80E-01	1.54E-01	1.54E-01	0.18%	1.50E-03	0.97%
5°	10°	Cube	6.76E-03	6.18E-03	6.19E-03	0.18%	3.14E-04	5.07%
5°	1°	Cube	6.77E-06	6.43E-06	6.46E-06	0.55%	6.33E-07	9.79%
5°	30°	Brass	1.80E-01	2.49E-01	2.50E-01	0.34%	1.83E-03	0.73%
5°	10°	Brass	6.76E-03	2.05E-02	2.08E-02	1.41%	5.73E-04	2.75%
5°	1°	Brass	6.77E-06	2.51E-05	2.56E-05	1.73%	1.38E-06	5.39%
4°	30°	Cube	1.80E-01	1.56E-01	1.56E-01	0.08%	1.46E-03	0.93%
4°	10°	Cube	6.76E-03	7.05E-03	6.96E-03	1.26%	3.43E-04	4.93%
4°	1°	Cube	6.77E-06	7.92E-06	7.75E-06	2.16%	8.31E-07	10.72%
4°	30°	Brass	1.80E-01	2.47E-01	2.48E-01	0.34%	1.63E-03	0.66%
4°	10°	Brass	6.76E-03	2.15E-02	2.18E-02	1.37%	5.33E-04	2.45%
4°	1°	Brass	6.77E-06	2.52E-05	2.56E-05	1.54%	1.46E-06	5.72%

In all cases, uncertainty increases as the misorientation angle decreases. This behavior is expected as a small misorientation angle means the ODF is only being evaluated at a few points, which are varying. When summing many points together for a volume fraction, the effect of variation at each of those points often attenuates the uncertainty. Similarly, the error generally decreases as the mesh resolution is reduced, but increases as the misorientation angle is decreased. Error is less than uncertainty in most cases, but often of a similar magnitude.

The root sum of squares method of uncertainty propagation was used to assess the combined effects of phase fraction uncertainty and ODF volume fraction uncertainty, shown in Table 3. These values shown in Table 3 are then a measure on the phase volume fraction of an individual orientation and the uncertainty of that phase volume fraction.

Table 3: Phase volume fraction of an individual orientation and the uncertainty for 10° misorientation angle and 3 different mesh resolutions. 'V' represents the phase volume fraction, and 'sigma' the propagated standard deviations.

Mesh	Cube		Brass	
	V	Sigma	V	Sigma
6°	9.08E-04	1.08E-04	2.99E-03	3.10E-04
5°	9.04E-04	9.47E-05	3.04E-03	2.91E-04
4°	1.01E-03	1.04E-04	3.16E-03	2.96E-04

4. Summary

The combination of phase fraction and crystallographic volume fractions has been demonstrated in this paper. The uncertainties in the phase fraction measurement and crystallographic volume fractions have been quantified and used to provide a metric on the uncertainty in the phase volume fraction. By measuring the phase volume fraction as a function of applied strain, the effect of orientation on the overall deformation can be measured and compared to predictions for transformation rate [4].

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