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Deconvoluting error in measurement of low angle misorientation distribution

Nitin Kumar Sharma, Shashank Shekhar*

Department of Materials Science and Engineering, Indian Institute of Technology Kanpur, Kanpur, 208016, India

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ABSTRACT

Misorientation angle distribution gives information about the type and fraction of grain boundaries present in a material. Since grain boundaries affect various mechanical and functional properties of the material, the distribution of grain boundary misorientation is important in order to evaluate these properties. This becomes particularly important when we want to study the microstructure in finer detail, such as understanding the average misorientation within a grain. One of the techniques increasingly used in past two decades for characterization of grain boundary misorientation is electron back scatter diffraction (EBSD). Reliable detection of very small misorientation angles using conventional EBSD system is quite challenging due to the presence of measurement error. This makes the comprehensive characterization of microstructures difficult and prone to error. In order to prevent such problems, it is important to understand the nature of measurement error and find ways to minimize it. The present work aims to elucidate the effect of measurement error on the observed misorientation angle and its statistical distribution in low misorientation angle regime. A true strain of 0.3 was imposed during cold-rolling of Cu-5%Zn alloy sample. The rolled sample was then subjected to *in-situ* heating from room temperature to 500 °C (~ 0.58 Tm). It was found that the overall measurement error in misorientation distribution consists of random error caused by limited angular precision and systematic error which manifests primarily in the statistical distribution of low angle misorientation. In this work, we show a way to deconvolute this overall error based on the measurement technique. We further show that this systematic error is not limited to any particular measurement technique, rather related to the presence of a lower bound in the measurement.

1. Introduction

Grain boundary character distribution (GBCD) is an important microstructural factor in determining the mechanical and functional properties such as resistance to intergranular corrosion, fatigue and creep etc. (Palumbo et al., 1991; Watanabe, 1984). Characterization of grain boundary type or more fundamentally the measurement of misorientation between two adjacent crystal orientations has been routinely carried out using electron back scatter diffraction (EBSD) (Schwartz et al., 2009). Wilkinson (Wilkinson, 2001) explained that since misorientation is calculated using orientation data, precision of the measurement is mainly dependent on error in the measurement of orientation data itself. For higher misorientation angles, this value of precision is relatively smaller as compared to lower misorientation angles and hence, the measurement of high misorientation angles can be made with very high precision, using EBSD. However, for lower angles, it has been shown that the limited precision can be traced directly to the error in measurement of the orientation (Humphreys, 2001; Humphreys et al., 1999; Prior, 1999; Schwartz et al., 2009; Wilkinson, 2001; Wilson and Spanos, 2001). This is particularly true when one wants to study finer details of a microstructure using parameters like Kernel Average Misorientation (KAM) and Grain Average Misorientation (GAM) in EBSD.

This measurement error leads to scatter in measured crystal orientation data, also called as orientation noise which makes the accurate measurement of misorientation between such crystal orientations difficult and hence prone to error. Thus, precision in measurement of relative crystal orientation becomes a limiting factor in the characterization of very low angle grain boundaries and hence it makes the application of EBSD inadequate for characterization of microstructures having low misorientation values. Error in the measured misorientation angles affects the nature of misorientation angle distribution as it can be easily shown that the measured misorientation distribution is actually the sum of error distribution and the true distribution of misorientation

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Abbreviations: EBSD, electron back-scatter diffraction; GBCD, grain boundary character distribution; KAM, kernel average misorientation; GAM, grain average misorientation; LAGB, low angle grain boundary; TEM, transmission electron microscopy

* Corresponding author.

E-mail address: shashank@iitk.ac.in (S. Shekhar).

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angles (Humphreys, 1999, 2001). Orientation noise is dependent on various microscopic factors including but not limited to pixels in CCD camera, calibration, sample alignment and accuracy of pattern solving algorithms such as hough transform (Humphreys, 1999, 2001; Humphreys et al., 1999).

As discussed by Humphreys (Humphreys, 1999), there are two approaches for a more reliable characterization of misorientation angle lower than ~2°. In the first approach, the focus is on reducing the orientation noise so as to improve the precision with which misorientation angle is measured. Since this orientation noise is caused by limited angular precision or the scatter in measured orientation data, this kind of error is random in nature and one way to minimize it is statistical averaging. Averaging the scattered orientation data is the simplest method for reducing the orientation noise (Humphreys, 2001) and hence making the lower angle data more reliable. Other methods to reduce the noise include changing the operating conditions such as probe current to enhance the pattern quality which has been shown to affect the angular precision (Humphreys and Brough, 1999). But since the change in operating conditions for better resolution and precision leads to decreased pattern acquiring speed, it is not always a good choice for application needing larger area maps for statistical analysis. Some recent methods have shown more ways related to certain advancement in the pattern matching and pattern solving techniques including cross-correlation in order to improve the precision of measured orientation (Bate et al., 2005; Brough et al., 2006; El-Dasher et al., 2003; Wright et al., 2011). Although the use of these methods has led to improved angular precision (~0.1°), these methods are quite resource intensive and require saving all the electron back scatter pattern (EBSP) data and hence take up enormous computer space and, in turn, lead to increased cost for overall EBSD characterization. Moreover, the approaches discussed thus far for reducing the orientation noise and improving the angular precision only reduce the error in individual measurements of orientation and hence the misorientation. However, distribution of misorientation angles is also important when dealing with various microstructural parameters, such as grain boundary character distribution (GBCD), grain orientation spread (GOS), grain average misorientation (GAM) and is also prone to error. Moreover, as we also show in present work, this error in the distribution of misorientation angles is largely caused due to the presence of another type of measurement error, which is systematic in nature and the approaches discussed above have a very minimal effect on that.

The second approach for more reliable EBSD characterization can be carried out using the conventional EBSD system itself (*i.e.* without any modification in the operating conditions or pattern matching and solving techniques) by deconvoluting the error from the observed data. This approach is limited to boundary statistics such as misorientation distribution. Since this approach focuses on the distribution of misorientation angles, it takes care of both forms of measurement error, *i.e.* random error caused by measurement of individual misorientation and also systematic error caused by lower bound of the measurement. This methodology involves first formulating the overall measurement error as a function of misorientation angle. This error can then be subtracted from the experimental distribution to obtain the true nature of misorientation distribution for a particular material and sample condition (Humphreys, 1999). This is akin to the technique applied by Stokes for deconvoluting error in x-ray diffraction data (Stokes, 1948).

The above-mentioned approach is not entirely new and has been discussed earlier for characterization of sub-grain boundaries for deformed state (Humphreys, 1999). However, in the present work, apart from the error deconvolution, the nature of measurement error (*i.e.* whether random or systematic) has also been investigated. This work primarily deals with the error in the distribution of measured misorientation rather than error in the measurement of individual misorientation itself. The current study is not limited to the investigation of measurement error in EBSD technique alone, but it also explores the nature of misorientation error irrespective of the characterization techniques used for measurement.

Table 1
Composition of Cu-Zn alloy

Element	Cu	Zn	Fe	C	Sn	Si	Pb	Ag, Al, S
Wt.%	95.2	4.64	0.048	0.026	0.030	0.022	0.016	< 0.003

2. Materials and methods

A single phase FCC material Cu-Zn alloy was taken as a test material for current study. The complete composition of the alloy is given hereby in Table 1 as determined by optical emission spectroscopy (OES). The sample was initially given a solution heat treatment at 650 °C for 3 h. Heat treated sample was then cold rolled to 26% reduction in thickness (true strain: 0.3). The rolled material was then heated *in-situ* in order to understand the changes in microstructure during annealing. The maximum temperature of heating was 500 °C.

Microstructural results in the form of EBSD band contrast micrographs and misorientation angle distribution curves for all the sample conditions have been described elsewhere (Sharma and Shekhar, 2017a). It has been shown through these results that with the progress in annealing time new strain-free recrystallized grains are formed. These strain-free grains started to form at 500 °C below which only recovery was observed. Recrystallization continued at 500 °C up to 120 min after which no further changes were observed in the microstructure and hence sample condition 120 m was taken as a fully recrystallized state. Electron back scatter diffraction (EBSD) micrographs were taken at various soaking temperatures (in between room temperature and 500 °C) and at the maximum temperature (*i.e.* 500 °C) with the progress of annealing time. Each EBSD scan took about 10 min to complete and hence the temperature of the heating stage was kept constant for that 10 min interval and once the scan was complete, the temperature was increased for further heating. All the information related to grain boundary misorientation was captured using the post-processing of raw data obtained from EBSD scans. These misorientation values were then used for plotting the distribution curves for each of the sample conditions. EBSD acquisition involved the use of Oxford Nordlys detector at the acceleration voltage and working distance of 20 kV and 11 mm respectively. Post-processing of EBSD data was done using HKL Channel-5 software.

3. Results and discussions

3.1. Misorientation angle distribution during annealing

In order to have a closer look at the nature of misorientation angle distribution, curves for recrystallized condition (*i.e.* 120 m) of Cu-Zn alloy along with a few other annealed FCC materials (namely Inconel 600 alloy, 316 L stainless steel and pure copper) are plotted for a small range of misorientation values (in the range 0–5°) and shown in Fig. 1. Details on chemical composition and processing of these materials (Inconel alloy (Vaid et al., 2016), 316 L stainless steel (Sharma and Shekhar, 2017b) and pure copper: annealed at 650 °C for 3 h) can be found in given references. As we see in Fig. 1, the nature of distribution curve remains similar irrespective of the nature of the material. This type of distribution consists of, first, an increase in fraction starting from 0°, then, a peak at some low misorientation angle (typically between 0–1°) and a decrease in fraction thereafter.

It is well established that population of grain boundaries are in general inversely related to their energy (Beladi and Rohrer, 2013; Dillon and Rohrer, 2009; Rohrer et al., 2010) and hence using this consideration, one would expect a monotonically decreasing fraction of low angle grain boundaries (LAGBs) with increasing misorientation. The population of boundaries also depend on the mode of their