Title

Reliability of Twin-Dependent Triple Junction Distributions Measured from a Section Plane

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Abstract

Numerous studies indicate polycrystalline triple junctions are independent microstructural features with distinct properties from their constituent grain boundaries. Despite the influence of triple junctions on material properties, it is impractical to characterize triple junctions on a large scale using current three-dimensional methods. This work demonstrates the ability to characterize twin-dependent triple junction distributions from a section plane by adopting a grain boundary plane stereology. The technique is validated through simulated distributions and simulated electron back-scatter diffraction (EBSD) data. Measures of validation and convergence are adopted to demonstrate the quantitative reliability of the technique as well as the convergence behavior of twin-dependent triple junction distributions. This technique expands the characterization power of EBSD and prepares the way for characterizing general triple junction distributions from a section plane.

Keywords: Triple junction; Triple line; Characterization; Electron backscatter diffraction (EBSD); Stereology; Twin Boundary

1 Introduction

1.1 The Need for Triple Junction Characterization

A polycrystalline triple junction is formed by the common intersection of three grains or crystals within a polycrystal. A fully characterized triple junction consists of three crystals, three grain boundaries, and a common triple line. These features may be macroscopically defined by three crystal orientations g_A , g_B , and g_C , three grain boundary plane orientations by the boundary normals n_1 , n_2 , and n_3 , and a triple line orientation by the vector t . Clearly these 17 parameters are not all independent and steps may be taken to reduce the number of parameters to a minimal number of independent parameters. By referencing all parameters from an internal coordinate system, or a coordinate system attached to one of the crystal lattices, the number of parameters may be reduced to 11 independent parameters of misorientations and plane parameters. To distinguish the application of an internal coordinate system, those parameters referenced from this system are appended with a subscript corresponding to the reference grain, which in Fig. 1 is grain A or g_A [1].

A microstructural distribution provides the normalized probability density as a function of character for an identified microstructural feature. As the dimensionality of a distribution feature increases, the quantity of measured data which must be collected to form the distribution increases as well. For instance, an orientation distribution contains only 3 parameters which consist of three Euler angles. A minute number of measurements would be required to form an orientation distribution as compared to a triple junction distribution containing 11 parameters. Bunge approximated that the quantity of measurements for minimal statistical reliability of a distribution is approximately 10^N , where N is the dimensionality of the desired feature [2].

Clearly, 10^{11} measurements are beyond the capabilities of any current experimental technique. This work brings the dimensionality of a triple junction to more measurable levels by focusing solely upon triple junctions containing a coherent twin boundary.

By exclusively characterizing twin-dependent triple junctions, the dimensionality of the distribution is significantly reduced. Given the mirror orientation symmetry across the grain boundary plane, the misorientation relation:

$$\Delta g_{AB} \bullet \Delta g_{BC} \bullet \Delta g_{CA} = \mathbf{I}$$

allows the two independent misorientations to be described by a single misorientation. As the incorporated twin boundary is a coherent twin, in the considered materials or FCC materials, it always points in a $\langle 111 \rangle$ direction. By defining the reference frame such that the twin boundary or $\mathbf{n}_{2A}$ points in the $[111]$ direction, this plane orientation becomes known. Given this convention, the parameters characterizing a twin-dependent triple junction are reduced to six independent parameters identified in this work as: a boundary plane normal $\mathbf{n}_{1A}$ given by spherical coordinates θ_{1A} and φ_{1A} , a dihedral angle α between $\mathbf{n}_{2A}$ and $\mathbf{n}_{3A}$, and a misorientation Δg_{AB} given by the Euler angles ϕ_1 , Φ , and ϕ_2 in the Bunge convention [3]. These parameters constitute the continuous twin-dependent triple junction distribution given by $\Gamma(\theta_{1A}, \varphi_{1A}, \alpha, \Delta g_{AB})$. The applied distribution parameters are depicted on an idealized triple junction in Fig. 1.

While triple junctions are composed of three constituent grain boundaries, they are distinct microstructural features. This is evidenced by their independent properties and microstructural effects, which are detailed in numerous studies [4–11]. Some of these properties are noted in a previous publication which lays the foundation for this work [12]. Despite

understanding the independent nature of triple junctions, junction properties are not well linked to character. This is in part the result of the lack of an experimental method for characterizing triple junctions on a large scale. This deficiency in an experimental method is the result of the inherent difficulty of measuring the junction's many parameters within three-dimensions.

By developing a method for experimentally characterizing triple junction distributions, junction character could be better linked to microscopic as well as macroscopic material properties. In addition, triple junctions provide grain boundary network information which is unavailable from grain boundary distributions alone [13–16]. A method of characterizing triple junction distributions would also allow grain boundary energy distributions to be easily approximated on a large scale. Grain boundary energy distributions have been extracted over the full range of grain boundary parameters in a few significant studies, but always with great experimental effort [17–22]. By enabling triple junction distributions to be generated from measurements on a section plane, the grain boundary character and energy distributions could be extracted from these data [23,24]. In short, in order to perform integrated materials engineering, triple junctions must first be characterized on a large scale.

1.2 Shortcomings of Current Three-Dimensional Characterization Techniques for Characterizing Triple Junction Distributions

Three-dimensional characterization techniques have seen great advances over the past decade and will continue to increase in efficiency and efficacy [25,26]. The techniques mentioned here are those capable of characterizing polycrystalline microstructures on the grain and grain boundary scale. These techniques include methods of high-energy X-ray tomography and diffraction and serial sectioning methods such as three-dimensional electron back-scatter diffraction (EBSD) by mechanical sectioning, focused ion beam milling, or laser ablation [27–

39]. These techniques have advantages over traditional two-dimensional techniques in that they can reveal information which cannot be extracted from two-dimensional planes [40,41]. Despite these advantages and advances in automated techniques [42,43], the three-dimensional techniques are typically expensive and tend to require substantial time and effort to carry out.

Given the time and effort required to collect data, current three dimensional approaches are incapable of providing the quantity of data necessary for practically forming statistically reliable triple junction distributions. For grain boundary distributions, Morawiec recently showed that despite the great advances in the ability to collect three dimensional datasets, these datasets are still relatively small compared to the quantity of measurements required for reasonable resolution and statistical reliability [44]. He estimated that for the largest dataset measured at the time, the relative error of the grain boundary distribution at 1 MRD was approximately 53% at best. The relative error for the distribution at its peak was approximately 34%. These error estimates were made for a 5-dimensional grain boundary distribution which included both the boundary misorientation and plane orientation. Smaller datasets would likely result in higher estimates of relative error over the full boundary distributions. While the generated grain boundary distributions will require more measurements to reduce the approximate relative errors, triple junction distributions would require substantially more data. Despite the reduction in parameters of a twin-dependent triple junction, the data required for generation of a triple junction distribution is still approximately an order of magnitude greater than that for a grain boundary distribution. It may be concluded that if it is challenging for the three dimensional techniques to generate grain boundary plane distributions with an acceptable relative error, then they would be ineffective or impractical when the quantity of data must be increased an order of magnitude for twin-dependent triple junction distributions.

1.3 Advantages of a Stereological Approach

Despite the inability of current three-dimensional techniques to generate reliable triple junction distributions, the technology exists to generate these distributions through stereology. As defined by Russ, “stereology is the science of the geometrical relationships between a structure that exists in three dimensions and the images of that structure that are fundamentally two-dimensional,” [45]. In this instance, the two-dimensional images would be EBSD maps from a section plane through the triple junctions.

There are several advantages to characterizing a triple junction distribution by a stereological technique. Primarily, a stereological technique could supply the quantity of data necessary for statistical reliability. The nature of a three dimensional technique requires characterizing each microstructural feature from many progressive planes in order to measure the feature with a reasonable resolution. A stereological technique will typically require less time since each individual feature requires a single measurement. From a single measurement applied to many similar features, the three dimensional data is extracted. As previously explained, Bunge gave the rough estimate that approximately 10^6 triple junctions would be required for minimal statistical reliability of a twin-dependent triple junction distribution [2]. Note that this is an approximate value. It will be shown later that this value is a little low but of the correct order of magnitude. Currently, EBSD cameras are being sold with the capability of indexing over 1000 points per second and speeds have been continually increasing [46]. While it might still take several weeks to measure the required quantity of data, it is an automated process and not out of the question.

Additionally, a stereological method does not require reconstruction of a three-dimensional polycrystalline structure. For many of the three-dimensional techniques it is

estimated that researchers often spend more time in reconstruction than in data collection [26,47]. Reconstruction generally may consist of data handling activities such as image processing or alignment. Changes in the reconstruction process have been shown to have a significant effect on results and great care must be taken to ensure the data is reconstructed properly [22]. Given the tedious task of reconstructing polycrystalline structures, care must be taken to ensure errors are not inadvertently introduced into the datasets through the image and data processing steps. For a stereological technique there is no polycrystalline reconstruction process and much of the data handling can be automated. For good statistical reliability the data collection time is still significant, but does not require attention to alignment. In addition, the measured features can be acquired from EBSD scans over many representative regions of a material rather than from a single volume. These reductions in the required effort for image and section processing remove the possibilities of potential errors from these activities. This is not to say that a stereological method is more accurate than a three dimensional approach. As will be emphasized later, this approach provides good results but it is an approximate technique that relies on several assumptions. The emphasis here is that the labor involved in forming distributions from a stereological technique appears significantly reduced from what is required from common three dimensional approaches.

As EBSD systems are commonplace in materials characterization labs, a stereological technique would also put twin-dependent triple junction characterization into the hands of students and researchers everywhere. Many of the advanced three-dimensional characterization techniques, such as the variations of automated serial sectioning or high-energy X-ray diffraction, require expensive highly specialized equipment which is available to only elite research programs. The experimental setup and requisite software for an EBSD-based

stereological method are widespread and relatively inexpensive. This accessibility allows the technique to be used by many researchers and also allows the technique to be easily incorporated into larger studies.

This work describes the framework of a characterization method capable of characterizing twin-dependent triple junction distributions from a single section plane. The previously developed foundational material as well as the adopted stereology will be briefly described. The efficacy of the technique will be demonstrated by some quantitative measures as well as by plots of results generated from simulated distributions. As briefly explained, this work focuses on twin-dependent triple junctions in order to reduce the required measurements to practical levels. Some might dismiss the technique as being ineffective given it doesn't apply to general triple junctions. While the materials available for analysis are seriously reduced, there are a number of materials with low to moderate stacking fault energy which could still be analyzed given they contain an abundance of coherent twins. A number of these materials, such as the grain boundary engineered alloys, have already been the source of intense grain boundary studies given their impressive properties. When analyzing materials with this technique, researchers should be able to put their results in the proper context of their limitations as well as their implications. This technique provides a first look at triple junctions on a large scale which is otherwise unobtainable. The intention of this technique is to also institute the foundation and experience necessary for the development of a generalized triple junction stereological technique.

2 Theory

2.1 Summary of the Developed Foundation

The foundational material necessary for the measurement of triple junctions from a single section plane has been developed and documented in a prior publication [12]. A brief summary of these results is provided here in an attempt to minimally prepare the reader for the development of the experimental method. Further details of this fundamental material, including descriptions of the distribution space and plots, may be found within the previous publication.

While a minimal number of distribution parameters have been established for the twin-dependent triple junction distribution, crystallographic and geometric symmetries result in many symmetrically equivalent triple junctions across the parameter space. These symmetrically equivalent triple junctions correspond to various parameter values. In order to distinguish symmetrically equivalent triple junctions and form the distribution, conventions may be utilized to remove the symmetrically redundant triple junctions. Initially the plane boundary inversion symmetry of the space is eliminated by the following conventions. The misorientation, Δg_{AB} , is identified as the junction misorientation with minimum misorientation angle. Grain *A* is identified as the grain between the coherent twin boundary and the minimum misorientation boundary corresponding to Δg_{AB} . Grain *B* is defined opposite the twin boundary and the remaining grain is identified as grain *C*. The coherent twin plane normal, $\mathbf{n}_{2A}$, always points into grain *A* and the other boundary normals point in the same direction about the triple line as $\mathbf{n}_{2A}$. These assignments and directional conventions ensure the distribution plane inversion symmetry is constrained. These conventions are in use in Fig. 1 which assists in visualizing the conventions. If two twin boundaries are present within a single triple junction, the twin

boundary with character closest to an ideal twin boundary is selected as the known twin with normal $\mathbf{n}_{2A}$. The remaining twin boundary is then managed with the same conventions as any other boundary.

The parameter space of the triple junction distribution is formed by the domains of the parameters. In the method utilized by Saylor et al., the parameter space will be discretized such that $\Gamma(\theta_{1A}, \varphi_{1A}, \alpha, \Delta g_{AB})$ is approximated over the distribution space by discrete representations [18]. The discretization makes possible the practical experimental generation of the distribution and this method has been carried out numerous times in previous studies [21,48–50]. Typically, the parameter space is discretized into equal volume cells which prevents biasing the distribution for any parameters associated with a polar angle over the unit sphere [51]. This is accomplished by implementing the cosine of any polar angles within the parameter space. Morawiec recently showed that this method of producing equal volume cells results in artifacts within the distribution given the variation in angular dimensions across distribution cells [52]. As the twin-dependent triple junction distribution already requires weighting to correct for inherent sampling biases, this work will present results using equal and unequal volume cells over the distribution space [12]. This will allow a secondary distribution for analysis or to assist in identifying artifacts.

The domains of the equal volume cell space boundary parameters are: $0 \leq \varphi_{1A} < 2\pi$, $-1 \leq \cos(\theta_{1A}) < 1$, and $-1 \leq \cos(\alpha) < 1$. The domains of the unequal volume cell space boundary parameters are: $0 \leq \varphi_{1A} < 2\pi$, $0 \leq \theta_{1A} < \pi$, and $0 \leq \alpha < \pi$. The domains of the $\Delta g_{AB}(\phi_1, \Phi, \phi_2)$ Euler angles are: $0 \leq \phi_1 < 2\pi$, $0 \leq \cos(\Phi) < 1$, and $0 \leq \phi_2 < \pi/2$. The $\Delta g_{AB}(\phi_1, \Phi, \phi_2)$ Euler angle domains remain identical for the equal and unequal volume

discretization as this parameterization remains the standard in the field of texture analysis. These parameters are discretized according to the desired resolution. The resolution of the experimental technique increases as the size of the cells or bins decreases. A decrease in cell size also increases the amount of measurements required to reliably form the distribution. As noted by Rohrer et al., while boundary properties begin to vary significantly at resolutions of just 10 degrees, it is uncommon to measure boundaries with a resolution less than 5 degrees. An appropriate resolution may therefore be designated from 5 to 10 degrees [51,53]. This reasoning will be followed for triple junction distributions and the resolution will be set to approximately 10 degrees for the equal and unequal volume space. A resolution of 10 degrees is achieved by equally dividing ϕ_1 and ϕ_{1A} into 36 cells, $\cos(\Phi)$ and ϕ_2 into 9 cells, and $\cos(\theta_{1A})$, θ_{1A} , $\cos(\alpha)$, and α into 18 cells.

It is thermodynamically unfavorable for a triple junction to contain dihedral angles greater than 180 degrees. Given this constraint, there is a significant region of the parameter space which pertains to geometrically impossible triple junctions. The distribution space is therefore a subset of the parameter space and 50% of the boundary space pertains to this geometrically infeasible region. This region is treated as empty space and accounts for the empty regions within the distribution plots.

As mentioned, crystallographic symmetries account for numerous symmetrically equivalent triple junctions across the parameter space. A fundamental zone is a region of the parameter space where each unique triple junction is represented once. In order to uniquely define each triple junction by a single set of parameters, conventions were also implemented to select a single fundamental zone from those present within the parameter space [12]. The selection of a fundamental zone completes the designation of the distribution space within the

parameter space. Distribution values within the space are normalized to multiples of a uniform random distribution (MRD).

As previously noted, given inherent sampling biases the twin-dependent triple junction distribution must be weighted in order to display true distribution values of MRD. Occasionally this weighting forms singular points within the distribution. To prevent these points from obscuring actual distribution values, all values undergo a filtering to remove singular points. The rate of filtering which results in minimal filtering for maximum distribution convergence was identified using a factorial design. Typically less than 1% of the distribution space is filtered from the distribution, though rates slowly increase as the amount of data measured decreases. This filtering accounts for occasional missing cells from the triple junction distributions as might be noticed in presented figures.

2.2 Adoption of Grain Boundary Stereological Approach

2.2.1 Implementation of Grain Boundary Stereology to Characterize Triple Junctions

The majority of the parameters required to fully characterize a triple junction containing a coherent twin boundary are measurable from a two-dimensional section plane. As depicted in Fig. 2a, from a section plane the grain orientations and plane traces are apparent. While the traces don't indicate the orientation of the junction planes, the traces indicate a range of possible orientations. This information may be used to identify the actual orientation of the coherent twin boundary and approximate the orientations of the remaining planes. The method of accomplishing this will be described progressively with Fig. 2.

Commercial EBSD software already contains algorithms for identifying coherent twins according to the proximity of a boundary misorientation angle to a twin misorientation and the boundary trace to the twinning plane trace [54,55]. As depicted in Fig. 2a, once an EBSD map is

generated, the triple junctions containing a coherent twin may be identified as a subset of all the triple junctions. After each twin is identified within every junction, misorientation Δg_{AB} is identified as the minimum misorientation. The normals to each trace and Grain A , B , and C can then be assigned according to the established conventions as depicted in Fig. 2b. As illustrated in Fig. 2c, the normal vector to the twin plane, or $\mathbf{n}_2$ as identified in the sample or laboratory reference frame, must be normal to the trace of the twin in the section plane. Therefore, a range of potential normal vectors may be identified over a hemisphere about the trace. Note that when $\mathbf{n}_2$ is represented by the spherical angles θ_2 and φ_2 , angle φ_2 is known while the range of θ_2 values provides potential normals. As this method is applied to FCC materials, it is known the twin boundary normal points in a $\langle 111 \rangle$ direction within grain A . The potential normals may be transformed to the crystal coordinate system according to the transformation:

$$\mathbf{n}_{2A} = g_A \bullet \mathbf{n}_2$$

The normal closest to a $\langle 111 \rangle$ direction is selected as the true normal and the grain A reference frame, or g_A , is rotated such that $\mathbf{n}_{2A}$ points in the $[111]$ direction according to the relation:

$$g_A = g_A^{n_{2A}[111]} = C_{rotat[111]} \bullet g_A^{initial}$$

Where $C_{rotat[111]}$ are elements of $SO(3)$ belonging to the octahedral (or cubic) symmetry group.

The selected $C_{rotat[111]}$ symmetry operator is applied to grain B , or g_B , so the Δg_{AB} misorientation remains consistent. Once $\mathbf{n}_{2A}$ points in the $[111]$ orientation it becomes known and doesn't require representation in the distribution space. The only unknown parameters remaining within the triple junction are θ_1 and θ_3 or the polar angles of $\mathbf{n}_1$ and $\mathbf{n}_3$. In order to complete characterization of the triple junction, only $\mathbf{n}_1$ or rather θ_1 must be identified. Once

this plane is known, the constraints of the junction geometry allows $\mathbf{n}_3$ to be constructed solely from the known value φ_3 .

To characterize $\mathbf{n}_1$ we can conveniently adopt the grain boundary stereology as already developed by Saylor et al. A brief summary of this stereology will be provided here, but further details and proofs may be found in the original publication of the technique [50]. This stereology functions in a similar manner to what has already been described and demonstrated for identification of $\mathbf{n}_{2A}$ in Fig. 2c. For a given grain boundary trace, potential plane orientations are obtained. For every potential boundary orientation and associated misorientation, the boundary length of the corresponding trace is added to a cell within a discretized five parameter grain boundary space. The discrete space acts as an accumulator array since potential boundaries are progressively stored within the array. Because every set of potential boundaries contains the true boundary orientation, as more and more sets of potential boundaries are added to the array, eventually the peaks of the true distribution will become apparent. Clearly while the true grain boundary plane orientations are added to the accumulator array, many false orientations are also included. To compensate for this error within the distribution, an approximate background subtraction is performed to subtract the erroneous grain boundaries from the distribution.

As only $\mathbf{n}_1$ must be characterized to finish forming the triple junction distribution, the grain boundary stereology may be directly applied. Potential $\mathbf{n}_1$ normals are formed about the plane 1 trace and for every potential $\mathbf{n}_1$ normal a $\mathbf{n}_3$ may be found according to the potential junction geometry. Both of these are transformed to the crystal coordinate frame and a unit value corresponding to the potential triple junction geometry may then be added to the twin-dependent triple junction space. After this occurs for every twin-dependent triple junction trace

in the section plane, the background subtraction is applied. The values within the distribution space are then normalized to values of MRD.

2.2.2 Advantages and Disadvantages of the Grain Boundary Plane Stereology

There are many advantages as well as a few notable disadvantages of the grain boundary stereology. The primary advantage of the technique is its ease of application. The technique is exceptionally simple compared to three dimensional techniques since it relies exclusively upon a two-dimensional EBSD orientation map. This accounts for the many studies which have employed this technique [20,22,56–59]. As already noted, this ease of application along with the high data collection capacity of automated EBSD systems enables collecting the quantity of data necessary to form twin-dependent triple junction distributions.

The primary disadvantage of the technique is its inherent errors given its employed approximations. One of the primary approximations of most concern is that of a random distribution. In estimating an appropriate background subtraction, the applied distribution was assumed to be random. While this enables the subtraction to be easily performed, the end result is that the accuracy of the technique decreases as the sharpness of a measured texture increases. This can clearly become problematic when attempting to analyze microstructures with a high degree of anisotropy. As noted by Morawiec in a recent review of the technique, this assumption makes it difficult to separate genuine features of distributions from artifacts of the method. He also correctly observes that at times this technique has been applied with no accounting for its potential inaccuracies [44]. These are valuable observations which should be accounted for when analyzing results and drawing conclusions through this technique.

Despite its potential inaccuracies, this technique is still very valuable as a characterization method. In previous studies good quantitative as well as qualitative results have

been obtained and validated. In these instances, distributions generated from the stereology were compared to distributions obtained through a three-dimensional characterization technique and good agreement was obtained [20–22]. This method has shown reliability for common textures and while results will become less accurate as anisotropy increases, this method can still provide general features of a distribution. As this technique in some applications provides quantitative results and consistently provides qualitative results, it may be effectively used by recognizing its limitations.

The value of this technique is also appreciated when considering the reliability of three-dimensional results. As mentioned in the Introduction, the current three-dimensional grain boundary plane characterization techniques have difficulty in collecting the quantity of data necessary for reliable distributions. Morawiec suggested focusing on the most reliable points in the distribution which are the peaks. This could be accomplished by measuring the grain boundary misorientations and plane orientations separately. Once the peaks are identified, the characterization techniques could focus upon the boundary planes at the peaks of the misorientation distribution and vice versa. This would enable a sufficient amount of data for statistical reliability to be collected in these regions of the distributions [44]. While this is a valuable insight, identifying regions of interest is still a challenging task. A stereological technique is perfectly suited to this task. As it is a relatively simple and inexpensive technique, it could relatively easily identify regions of interest for boundary or triple junction distributions. In addition to identifying distribution areas of interest, the twin-dependent triple junction stereological technique could be applied in parallel with three dimensional approaches. This was successfully demonstrated by Rohrer who used grain boundary plane stereology to refine his three-dimensional reconstruction of serial sectioned EBSD data [22]. This was carried out by

comparing initial results to stereological results. When the results differed significantly, refinements were made to the reconstruction process which brought the results into closer agreement.

3 Results

3.1 Validating the Method

The proposed technique of generating twin-dependent triple junctions from a section plane was validated by applying the method to a known twin-dependent triple junction distribution. The results between the known and stereologically generated distribution were compared to assess the accuracy of the procedure. The method of validation employed in this work is very similar to the approach utilized by Saylor et al. for the grain boundary stereology [50]. A simulated twin-dependent triple junction distribution was generated and then two-dimensional EBSD data, as would be collected in a section plane, were generated from this known distribution. The simulated distribution was produced in the method outlined by Morawiec for generating non-uniform distributions [60]. An arbitrary peak was selected within the distribution space at $\mathbf{n}_{1A} = [1\bar{2}\bar{1}]$, $\mathbf{n}_{2A} = [111]$, and $\mathbf{n}_{3A} = [\bar{2}10]$. The frequency function, which is Gaussian in nature and forms the peak in the distribution, was adopted from that used by Saylor et al. [50]. The function is given by:

$$F = A \exp \left(- \left(\frac{\omega}{B} \right)^2 \right) + C$$

Where factors A , B , and C were selected as 10, 15, and 3.5. These factors differ from those used by Saylor as they were adapted for the change in space. ω is the average angle between $\mathbf{n}_{1A}$ and $\mathbf{n}_{3A}$ of the selected peak triple junction and the $\mathbf{n}_{1A}$ and $\mathbf{n}_{3A}$ planes of a randomly generated

junction. Clearly, values with greater proximity to the peak value result in a larger frequency value F . A distribution was formed by conditionally accepting randomly generated junctions. Random twin-dependent triple junctions were formed and a frequency value F generated for every junction. This F value was then compared to a randomly generated number within the range $[0, F_{peak}]$, where F_{peak} is the maximum possible frequency value or where ω equals 0. The triple junction was accepted and added to the distribution space if its corresponding F value was greater than or equal to the randomly generated number. When a triple junction was added to the space, a unit value was added to the cell within the distribution space corresponding to the triple junction character.

The experimental data were generated from the simulated distribution by adapting the method implemented by Saylor [50]. A random orientation g_A was formed and the neighboring orientation g_B was generated according to a preselected misorientation relationship Δg_{AB} . Each accepted junction and associated crystal frame was randomly rotated such that the twin pointed to a $\langle 111 \rangle$ direction. Each junction was then rotated by g_A^{-1} , the inverse of the crystal reference frame, so as to rotate the values to the sample reference frame. Trace vectors were then generated from the triple junction boundary planes. At this point the trace vectors and Δg_{AB} appeared as if extracted from section plane EBSD data. These data generated from the simulated distribution were then characterized by the stereological method. If the experimentally generated distribution resembled the simulated distribution, the technique could be considered successful.

3.2 Visual Validation of the Technique

The results of the validation method are produced visually by plotting the simulated and stereologically generated distributions in Figs. 3 through 8. The validation was performed for a

fixed misorientation in order to simplify the computational process. The conclusions drawn from inferring these results to the full space will be discussed within the Conclusions section. The fixed misorientation was selected as a 20 degree rotation about $[952]$. Originally this rotation was selected given it is at the center of the stereographic triangle and not subject to any special bi-crystal symmetries [18,51,61,62]. Now that the method of weighting the distribution to remove sampling biases is implemented, bi-crystal symmetries should have no effect and the misorientation angle/axis selection is arbitrary.

Fig. 3 depicts a simulated distribution generated in the equal volume cell distribution space, while Fig. 5 depicts the same distribution in the unequal volume cell distribution space. This distribution was generated with an arbitrary peak at $\mathbf{n}_{1A} = [1\bar{2}\bar{1}]$, $\mathbf{n}_{2A} = [111]$, and $\mathbf{n}_{3A} = [\bar{2}10]$ as previously mentioned. Figs. 3a and 3b consist of the southern and northern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of $\cos(\alpha)$ at center cell values. Figs. 5a and 5b consist of the southern and northern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of α at center cell values. Fig. 3 or 5 represent the full distribution boundary space at a fixed misorientation. The white areas of the plots represent the regions of the parameter space pertaining to thermodynamically or rather geometrically infeasible triple junctions. The distribution space within the parameter space pertains to the colored areas of the plots. As apparent in the plots, the distribution discrete cells are plotted directly with no interpolation between cells. This accounts for the pixel-like colored squares of the projections. While these raw distribution plots are not as visually appealing as a plot that has been artificially smoothed, the results are true to the actual distribution. Further details for interpreting these plots may be found in the aforementioned previous publication [12].

The EBSD or stereographic data used to generate the equal volume distribution in Fig. 4 were created from the triple junctions within the simulated distribution in Fig. 3. Likewise, the stereographic data used to generate the unequal volume distribution in Fig. 6 were created from the triple junctions within the simulated distribution in Fig. 5. The experimental distributions in Figs. 4 and 6 were generated from 500,000 triple junction measurements at a fixed misorientation. The simulated distributions in Figs. 3 and 4 were generated from approximately 500,000 triple junctions at a fixed misorientation. The slight difference in triple junctions implemented in the experimental and simulated distributions is a result of the filtering process.

Visual comparison of the distributions provides a simple analysis of the stereological technique. As apparent when comparing Figs. 3 and 4 and Figs. 5 and 6, at these high data levels the technique shows excellent visual agreement. The stereologically produced distributions in Figs. 4 and 6 demonstrate some minor peak broadening as evident in the northern and southern hemisphere of the distributions. This is a slight artifact of the technique and the broadening may be anticipated when generating results. The stereologically reconstructed distributions demonstrate good agreement in peak magnitude. While the peak magnitudes happened to be very close in these instances, there is still some variance between the simulated and reconstructed magnitudes at this data level. For 12 distributions in both the equal and unequal distribution spaces, the average difference between the simulated and reconstructed distribution peak magnitudes was 0.30 and 0.32. The standard deviation of these peak difference values was 0.1787 and 0.1626.

To demonstrate the performance of the technique over a range of data levels, simulated and stereologically reconstructed experimental distributions have been generated within the equal volume cell distribution space for decreasing numbers of triple junctions. For conciseness

and greater visual detail, only the projection containing the distribution peak will be plotted at each data level. Figs. 7a – 7f contain the simulated distribution peak projections for distributions composed of approximately 500,000, 250,000, 100,000, 50,000, 25,000, and 10,000 twin-dependent junctions at a fixed misorientation. Figs. 8a – 8f contain the corresponding stereologically generated distribution peak projections at the same data levels as those found in Figs. 7a – 7f. For example, Fig. 7a is the peak projection for a simulated distribution composed of approximately 500,000 triple junctions. Fig. 8a is the stereologically generated distribution formed from 500,000 triple junction measurements produced from the distribution in Fig. 7a.

This comparison at decreasing data levels visually demonstrates the performance of the stereological technique as well as the distribution convergence behavior. It is apparent that as the distribution data input decreases, the scatter increases within the distributions. This appears to occur equally for both the simulated and stereological distributions. The stereologically generated distributions also tend to perform poorer as the distributions diverge. Thus, the performance of the stereological technique must be considered in the context of the quantity of data inputs. From examining the distributions, it is evident that the distribution peak was identified by the stereological technique all the way down to 25,000 triple junctions. This demonstrates a degree of robustness within the technique since the peak was identified despite significant scatter within the distributions. As apparent in the simulated distribution at 10,000 triple junctions, this number of junctions was not sufficient to form a peak in the distribution. Therefore, there was nothing for the stereological technique to reconstruct. While the peaks were identified down to approximately 25,000 triple junctions, the peak magnitudes were closely approximated only until 100,000–50,000 triple junctions. Below this point the magnitudes between the simulated and stereological distributions began to vary significantly. This behavior

indicates that the method can at least provide qualitative identification of distribution peaks at low levels of data, but care must be taken to ensure sufficient data were measured to reliably form the distribution character. If quantitative results are expected from the stereological technique, significant data must be collected in order to form a reliable distribution. Clearly, significant data are required to form a reliable distribution even if all parameters were known, as indicated by the simulated distributions. Further insight into the reliability of the technique as well as the distribution convergence behavior can be gained by examining several quantitative performance factors.

3.3 Quantitative Validation of the Technique

A measure of relative distribution convergence may be created by considering the number of data inputs per cell within the distribution space. This has been employed previously when analyzing results from grain boundary distributions [22]. As the number of triple junctions within each cell increases, the distribution converges and the results become more reliable. A summary of the triple junctions per cell has been created in Table 1. This work was performed at 10 degrees resolution and will focus on the results within Table 1a. The first two columns of Table 1a indicate the number of triple junctions implemented in the distribution at a fixed misorientation and the number of cells in the grain boundary space at a fixed misorientation. The change in cells across the rows in the second column is the result of the distribution filtering. The third column is the number of triple junctions per cell if the included triple junctions from the first column were randomly dispersed throughout the distribution space. In this application the distribution was not random; therefore, the distribution peak cells will have values greater than this number while the valley cells will be lower. The difference in the triple junctions per cell at the distribution peaks and valleys depends on the degree of distribution anisotropy. While

the number of random triple junctions per cell does not represent the actual distribution of triple junctions within the space, this value may still provide a simple method of determining relative convergence. For example, for the distributions formed at a fixed misorientation with 10,000 triple junctions, a random distribution would have provided less than two triple junctions per cell. In the anisotropic case, the minimum values would have been even lower than this. Clearly, this wasn't nearly enough to form a distribution. Note that the distributions began converging and the stereological procedure began reconstructing the peaks accurately from 50,000 – 100,000 triple junctions at a fixed misorientation. This corresponds to 8.63 – 17.25 random triple junctions per cell.

A better comparison of distribution similarity, to be used for the stereological validation, may be provided by comparing the distributions over the full distribution space. Morawiec has provided a method for comparing smooth orientation functions/distributions according to the relation:

$$\frac{1}{2} \int |f_1(O) - f_2(O)| dO$$

Where f_1 and f_2 are the distributions of interest and O is a given orientation. Given the normalization of f_1 and f_2 , the result of this method provides a number between 0 and 1 with 0 corresponding to identical distributions [60]. The technique may be adapted to the discrete twin-dependent triple junction distribution in an attempt to provide a quantitative measure of similarity between functions. While this technique should be applied to smooth distributions, the distributions in this application will be assumed smooth enough. To adapt this method to the discretized twin-dependent triple junction distribution the following relation may be used:

$$\frac{1}{2N} \sum_{i=1}^N |\Gamma_{sim}(i) - \Gamma_{ster}(i)|$$

Where $\Gamma_{sim}(i)$ and $\Gamma_{ster}(i)$ are the discrete simulated and stereologically generated triple junction distributions at cell i and N is the number of cells within the distribution space. Identical to the relation given by Morawiec, this relation provides difference values in the range of 0 to 1. The adapted relation has been applied to the distributions whose peaks are listed in Figs. 7 and 8. A graph of these results is shown in Fig. 9. As apparent in the figure, the difference values seem to accurately describe the similarities of the figures with a quantitative value. Most notable is the rate of change of the curve's negative slope. This value begins increasing dramatically below 100,000 triple junctions where the magnitudes of the simulated and stereologically generated distributions begin diverging. For this simple application, this technique seems to hold good promise in providing insights into distribution results.

An additional quantitative measure of the simulated and stereological distributions may be implemented by computing the distribution statistical entropy. As mentioned in Section 3.2, the reconstructed distributions display a minor quantity of peak broadening. As explained by Morawiec, the statistical entropy (unitless) provides a measure of distribution sharpness according to the relation:

$$S = -\int [f(O) - \ln(f(O))] dO$$

where $f(O)$ is the distribution of interest. The relation provides a value of zero for a random distribution and negative infinity for a distribution gathered about a single peak [60]. The statistical entropy may be computed for either the simulated and stereologically reconstructed discrete distributions according to the relation:

$$S = -\sum_{i=1}^N \Gamma_{sim}(i) \ln(\Gamma_{sim}(i))$$

where in this instance $\Gamma_{sim}(i)$ is the discrete simulated distribution. As this distribution is discrete rather than continuous the output is in the range of zero for a uniform distribution and approximately negative 56,000 for a distribution at a fixed misorientation. The statistical entropy for the simulated and stereological distribution is displayed in Fig. 10. In the figure it is clear that the stereologically reconstructed distributions maintain a smaller statistical entropy given the broadened peak. As for the difference values, the slope of the comparison curves begins changing rapidly below 100,000 triple junctions where the distributions begin diverging and the peaks become sporadic.

4 Conclusions

The fourth column of Table 1a corresponds to the approximate quantity of triple junctions necessary for expanding the results at a fixed misorientation to the full distribution space. It is somewhat alarming to note that the reasonable convergence obtained at 50,000 – 100,000 triple junctions at a fixed misorientation corresponds to 16 – 32 million triple junctions in the full space. In this same column the lowest levels of convergence and stereological validation, which provided only rough qualitative results, would require measuring anywhere from 3-8 million triple junctions. This would result in significant experimental time despite using two-dimensional EBSD measurements on a single section plane. Clearly, the 10^6 triple junction approximation provided by Bunge is of the correct order of magnitude but on the low end of data requirements [2]. As apparent in the fourth column of Table 1a, quantitative results with better reliability would require data at another order of magnitude which becomes impractical.

The amount of data required to generate the full distribution with reasonable reliability may be reduced to practical levels by slightly reducing the distribution resolution. Table 1a indicates that a full distribution with a resolution of 10 degrees requires approximately 3.24 million triple junctions for 1.72 random triple junctions per cell. This data quantity was not sufficient to form the distribution peak. By reducing the resolution to approximately 12.9 degrees, or 7 cells per 90 degrees, 3.81 million triple junctions provides approximately 9.23 random triple junctions per cell. By comparing the levels of random triple junctions per cell, this is better relative convergence than could be achieved with 16.2 million triple junctions at 10 degrees resolution. This dramatic improvement in convergence with a slight decrease in resolution is the nature of a discretized high-dimensional space. Small changes in the numbers of cells on each level compound across all levels for a large reduction in total numbers of cells. Table 1c is provided as a reference for the data reduction and improved convergence for a resolution of 15 degrees. While data suggests boundary properties vary above 10 degrees, reducing the resolution slightly would enable characterizations with much greater reliability [53]. This is a reasonable approach for the current limitations in the speed of experimental measurements. Despite a loss of resolution, the distributions could be formed reliably and some conclusions drawn. As automated data collection methods improve, smaller resolutions could be attempted.

Further improvements in the distribution convergence rates could be achieved without reducing the resolution. This could be accomplished by implementing more sophisticated array cell assignment functions. In this study triple junctions were assigned to cells with a unit value. The location of a value within the cell made no difference in assignment. If unit values were divided between cells according to their proximity to cell borders, the distribution would be

naturally smoothed. For instance, if a value lands practically at a cell border the unit value should be split between the neighboring cells. If a value lands at the center of a cell the entire unit value should be assigned to that cell. Anything in between these extremes is divided between cells according to proximity to the border. This would provide a more converged distribution for the same quantity of data without interpolation or approximate methods of smoothing.

The results of this study indicate that this is an effective method for twin-dependent triple junction characterization on a large scale. Care must be taken to ensure sufficient data is measured to provide reliable results, though this is the case with all distributions of microstructural features. The ease and speed of experimental measurements through automated EBSD on a single section plane enables this technique to measure the high quantities of data necessary for generating twin-dependent triple junction distributions. The removal of the need to reconstruct a three-dimensional polycrystalline microstructure gives this technique a further advantage in ease and speed over common three-dimensional approaches. The accessibility of the experimental equipment necessary for this approach also puts this method into the hands of researchers everywhere and empowers them with an additional tool. While this method has limited application to materials with a high fraction of coherent twin boundaries, it will provide the first experimental look at triple junctions on a large scale. The experience and framework gained through this work will also prepare the foundation for a generalized approach in the future.

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Figure Captions

Fig. 1

The conventions and six independent parameters within the twin-dependent triple junction distribution.

Fig. 2

The progression of steps to characterize a twin-dependent triple junction from a section plane. (a) Identify the coherent twin trace in the section plane. (b) Apply developed conventions to the twin-dependent triple junction grains and trace normals. (c) Identify the twin boundary plane orientation by selecting the potential trace normal nearest a $\langle 111 \rangle$ direction. Transform the normal to the $[111]$ direction.

Fig. 3

Simulated distribution generated in the equal volume cell distribution space. The peak of the distribution is located at $\mathbf{n}_{1A} = [1\bar{2}\bar{1}]$, $\mathbf{n}_{2A} = [111]$, and $\mathbf{n}_{3A} = [\bar{2}10]$ or $\cos(\alpha) = -.2582$. (a) Southern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of $\cos(\alpha)$ at center cell values. (b) Northern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of $\cos(\alpha)$ at center cell values.

Fig. 4

Stereologically generated distribution from simulated experimental data created from the junctions in Fig. 3. Comparison of Fig. 3 and Fig. 4 provide a qualitative visual validation of the implemented stereological method. (a) Southern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of $\cos(\alpha)$ at center cell values. (b) Northern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of $\cos(\alpha)$ at center cell values.

Fig. 5

Simulated distribution generated in the unequal volume cell distribution space. The peak of the distribution is located at $\mathbf{n}_{1A} = [1\bar{2}\bar{1}]$, $\mathbf{n}_{2A} = [111]$, and $\mathbf{n}_{3A} = [\bar{2}10]$ or $\alpha = 104.96^\circ$. (a) Southern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of α at center cell values. (b) Northern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of α at center cell values.

Fig. 6

Stereologically generated distribution from simulated experimental data created from the junctions in Fig. 5. Comparison of Fig. 5 and Fig. 6 provide a qualitative visual validation of the implemented stereological method. (a) Southern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of α at center cell values. (b) Northern hemispheres of the stereographic projections of $\mathbf{n}_{1A}$ for fixed values of α at center cell values.

Fig. 7

Simulated distribution peak projections composed of approximately (a) 500,000 (b) 250,000 (c) 100,000 (d) 50,000 (e) 25,000 (f) 10,000 twin-dependent junctions at a fixed misorientation.

Fig. 8

Distribution peak projections stereologically generated from (a) 500,000 (b) 250,000 (c) 100,000 (d) 50,000 (e) 25,000 (f) 10,000 simulated measurements of twin-dependent junctions at a fixed misorientation.

Fig. 9

Difference values of similarity between the simulated and stereologically reconstructed boundaries in Fig. 7 and Fig. 8.

Fig. 10

Distribution statistical entropy values for the simulated and stereologically reconstructed boundaries in Fig. 7 and Fig. 8.

Table 1

Summary of cells at a fixed misorientation, triple junctions per cell, and triple junctions for the full space for varying levels of fixed misorientation data. (a) Actual data taken from the simulated distributions in Fig. 7. (b) Values calculated for a 12.9 degree resolution at the 10 degree resolution filtering levels. (c) Values calculated for a 15 degree resolution at the 10 degree resolution filtering levels.

Fig 1

Fig 2a

Fig 2b

Fig 2c

Fig 3a

Fig 3b

Fig 4a

Fig 4b

Fig 5a

Fig 5b

Fig 6a

Fig 6b

Fig 7a

$$\cos(\alpha) = -0.2582$$

Fig 7b

$$\cos(\alpha) = -0.2582$$

Fig 7c

$$\cos(\alpha) = -0.2582$$

Fig 7d

$$\cos(\alpha) = -0.2582$$

Fig 7e

$$\cos(\alpha) = -0.2582$$

Fig 7f

$$\cos(\alpha) = -0.2582$$

Fig 8a

$$\cos(\alpha) = -0.2582$$

Fig 8b

$$\cos(\alpha) = -0.2582$$

Fig 8c

$$\cos(\alpha) = -0.2582$$

Fig 8d

$$\cos(\alpha) = -0.2582$$

Fig 8e

$$\cos(\alpha) = -0.2582$$

Fig 8f

$$\cos(\alpha) = -0.2582$$

Fig 9

Fig 10

Triple Junctions at Fixed Misorientation

10° Resolution			
Triple Junctions Fixed Misorientation	Simulated Cells Fixed Misorientation	Random Junctions Per Cell	Approximate Junctions Full Space
5.00E+05	5790.52	86.05	1.62E+08
2.50E+05	5778.78	43.07	8.10E+07
1.00E+05	5763.70	17.25	3.24E+07
5.00E+04	5758.74	8.63	1.62E+07
2.50E+04	5755.69	4.32	8.10E+06
1.00E+04	5754.61	1.72	3.24E+06

12.9° Resolution			
Triple Junctions Fixed Misorientation	Approximate Cells Fixed Misorientation	Random Junctions Per Cell	Approximate Junctions Full Space
5.00E+05	2724.54	183.52	7.62E+07
2.50E+05	2719.02	91.94	3.81E+07
1.00E+05	2711.92	36.87	1.52E+07
5.00E+04	2709.59	18.45	7.62E+06
2.50E+04	2708.15	9.23	3.81E+06
1.00E+04	2707.65	3.69	1.52E+06

15° Resolution			
Triple Junctions Fixed Misorientation	Approximate Cells Fixed Misorientation	Random Junctions Per Cell	Approximate Junctions Full Space
5.00E+05	1715.75	291.42	4.80E+07
2.50E+05	1712.27	146.01	2.40E+07
1.00E+05	1707.80	58.55	9.60E+06
5.00E+04	1706.33	29.30	4.80E+06
2.50E+04	1705.43	14.66	2.40E+06
1.00E+04	1705.11	5.86	9.60E+05

Table 1

Summary of cells at a fixed misorientation, triple junctions per cell, and triple junctions for the full space for varying levels of fixed misorientation data. (a) Actual data taken from the simulated distributions in Figure 7. (b) Values calculated for a 12.9 degree resolution at the10

degree resolution filtering levels. (c) Values calculated for a 15 degree resolution at the 10 degree resolution filtering levels.

Title

Reliability of Twin-Dependent Triple Junction Distributions Measured from a Section Plane

Authors and Affiliations

Graden B. Hardy^{a,1} and David P. Field^{a,2}

Graphical Abstract

The distribution of planes forming a triple junction with a given twin boundary is shown partially in the stereographic projections below from a given projection. The plot on the left shows the ideal/measured distribution and the plot on the right shows the distribution obtained from the stereological method presented here.