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Twin related domains in 3D microstructures of conventionally processed and grain boundary engineered materials

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ABSTRACT

The concept of twin-limited microstructures has been explored in the literature as a crystallographically constrained grain boundary network connected via only coincident site lattice (CSL) boundaries. The advent of orientation imaging has made classification of twin-related domains (TRD) or any other orientation cluster experimentally accessible in 2D using EBSD. With the emergence of 3D orientation mapping, a comparison of TRDs in measured 3D microstructures is performed and compared against their 2D counterparts. The TRD analysis is performed on a conventionally processed (CP) and a grain boundary engineered (EM) high purity copper sample that have been subjected to successive anneal procedures to promote grain growth. The EM sample shows extremely large TRDs which begin to approach that of a twin-limited microstructure, while the TRDs in the CP sample remain relatively small and remote.

1. Introduction

Microstructural scalar measures have been directly correlated with material performance for a long time. For example, the well-known Hall-Petch scaling relationship [1,2] quantitatively relates decreasing grain size with increased yield strength. Whereas Hall and Petch did not distinguish between the varieties of grain boundary types, a decrease in grain size necessarily implies more grain boundary area per unit volume. There was also a growing realization that the specific crystallography of individual grain boundaries was of importance for determining materials properties. Grain boundary engineering of face-centered cubic metals and alloys as a concept encompassing an increase in the fraction of boundaries in a microstructure that are special, or associated with a relatively highly ordered crystallographic arrangement [3], was introduced more than 30 years ago [4]. Early research in grain boundary engineering focused on obtaining a similar relationship between the fraction of special boundaries and properties, such as corrosion and cracking. Increasing the fraction of special boundaries, $\Sigma \leq 29$, strongly correlated with decreased corrosion rates and arresting of intergranular crack propagation [5].

A scalar measure, the special fraction, has been used as a proxy to differentiate conventionally processed microstructures (CP) from grain boundary engineered microstructures (EM). However, a natural question arises as to what constitutes a special boundary because it is generally understand that the topological configuration of boundaries affects the properties of materials [6]. So, while it is agreed that the topology of the grain boundary network differs substantially between the two types of microstructures, thus giving rise to the improved properties exhibited in grain boundary engineered materials [7], the attempts to quantifiably connect structure with properties are still very much a work in progress.

The experimental observation of crack blunting at Σ3 boundaries during intergranular stress corrosion cracking [8,9] has added yet another layer to understanding the role boundaries play, forcing us to consider the makeup of boundary junctions [7,10], and how their specific configurations could, for instance, either allow or inhibit crack growth. If we assume all Σ3 boundaries will stop crack propagation and all other boundaries will allow it to proceed, then triple junctions where 2 Σ3 s meet will stop the progression of a crack. Knowing the fraction of these special types of triple junctions is more informative than knowing just the fraction of special boundaries. The incorporation of the triple junction distribution (TJD) in the grain boundary network is an initial step toward looking at higher order correlations beyond just grain boundaries, which are representative two point correlations.

Once a triple junction is characterized as impeding crack

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propagation through the microstructure, then a collection of such objects could form to completely close off any path through the microstructure. The concept of spanning clusters and percolation theory can be invoked to explain transport properties in the microstructure [11–13]. Below a critical threshold, island clusters form which act as barriers but do not completely rule out a path for the crack to proceed from end to end of the microstructure. A crack or diffusion process is forced to go around these obstacles. However, above a critical threshold, there exists no path around the special clusters that act as barriers. Crack promoting paths form islands within a sea of crack-inhibiting clusters. In 2D, a cluster is said to be percolating if it completely spans the microstructure. If crack promoting boundaries percolate the structure, crack propagation is expected to proceed by way of the shortest path possible. Conversely, if crack inhibiting boundaries like Σ3 s percolate the structure, crack propagation can be stunted.

The concept of a twin related domain (TRD) was first introduced by Reed [14] to describe a cluster of grains in which every grain in the cluster is connected to at least one other grain via a Σ3 boundary. The CSL-based group theoretic description implies that every grain in the TRD cluster is related to any other grain in the cluster via a $\Sigma 3^N$ relation. The mathematics of rotations resulting from a twin-dominated structure have been studied in a theoretical context [14–18]. Reed showed a direct relationship between the TRD length scale and material performance [19]. Others have correlated enhancing of mechanical properties [20,21], electronic properties [22–24], and grain growth stagnation [25,26] with grain boundary engineering processing steps. Given that TRD development is a consequence of grain boundary engineering, these results likely indirectly correlate with TRD development as well. Further attempts to quantify the depth of orientation diversity within a TRD are at the forefront of current TRD characterization [27].

Reed's matching of TRD length scale to material properties combined information typically derived in 2D from EBSD scans with fracture roughness measurements, a 3D topographic mapping. Since crack propagation proceeds out of plane, the shape of the TRD as an obstacle is of some importance. For grain boundary engineered materials, relatively convex grain shapes in 2D can look drastically different in 3D, taking on non-convex shapes [28]. Stereological assumptions perform poorly in this case. The morphology of TRDs in 3D, whether convex or not, could have implications for percolating paths of cracks, for instance.

To answer some of these outstanding questions, we non-destructively measure the full 3D microstructure of two complementary copper samples, one that has been grain boundary engineered and the other conventionally processed. Since microstructures are never static the two samples were subjected to the same annealing treatment and re-measured to study the evolution of the TRD populations. These observations are interpreted in the context of common microstructure metrics and also in the context of standard 2D measurements. The implications of these results are then considered in the context of experimental resolution limitations, operational definition of the clusters, possibility of TRD fragmentation, measured special fractions, and expected critical thresholds.

2. Methods

2.1. Experimental measurement

In this study, two physical samples of pure copper were studied through several stages of annealing. Initially 1.1 *mm* diameter cylindrical samples of conventionally processed (CP) and grain boundary engineered (EM) copper were produced with similar grain sizes. The CP sample had an average sphere-equivalent diameter grain size of $\langle d \rangle = 18.2\ \mu m$, and the EM sample had $\langle d \rangle = 24.4\ \mu m$, initially. These samples were then experimentally mapped using the near-field high-energy diffraction microscopy (nf-HEDM) [29–31] technique at Sector 1 of the Advanced Photon Source at Argonne National Laboratory allowing for interrogation of 0.4 mm^3 volume. The measurement volume is limited by the allotted beam time at the facility. The crystallographic orientation data was then reconstructed from the X-ray scattering data using the IceNine forward modeling package [32] resulting in 0.4 mm^3 total volume at a resolution of 2 $\mu m \times 2\ \mu m \times 4\ \mu m$. Each volume represents ~10^7 individual data voxels with unique crystallographic orientation. Taking advantage of the fact that nf-HEDM is a non-destructive technique, we were able to repeatedly map the same physical volume multiple times. We were able to map both samples before and after annealing (at identical conditions for 2 *hrs* at 500 C° with flowing inert gas). Due to the limited beam time allotment at the experimental facility, only the EM sample was

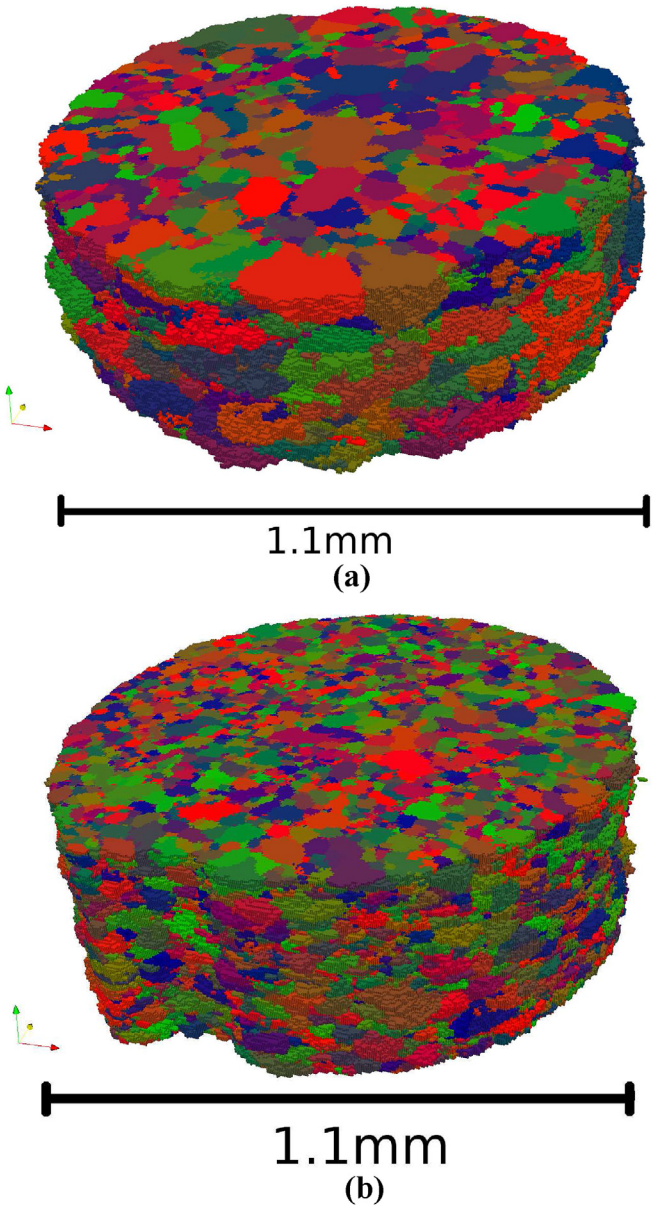

Fig. 1. 3D visual for the (a) EM sample, and (b) CP sample prior to annealing. The grain size differences are visually apparent here.