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The formation and characterization of large twin related domains

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ABSTRACT

The enhanced properties of grain boundary engineered metals are a result of their unique microstructures, which contain large clusters of twinned grains, called twin related domains. These large twin related domains in grain boundary engineered Ni were found to form through recrystallization. Orientation mapping showed that sparse nucleation and multiple twinning resulted in twin related domains containing hundreds of grains connected together in complex morphologies. A correlation was found between the size of the twin related domains and the overall twin boundary fraction. The same correlation was also observed in Cu and a Ni superalloy, showing that this is a general observation for grain boundary engineered microstructures. This finding can be understood through the topology of the twin related domains and an accompanying scaling relation is provided. The crystal orientations contained within each twin related domain were observed to depend on both the spatial correlation of twinning variants and the degree of branching in the twin boundary network. The results suggest a natural way of quantifying grain boundary engineered microstructures and provide a step toward making a closer connection between processing, microstructure, and performance.

1. Introduction

Material properties can often be improved by controlling both the type and arrangement of grain boundaries, a process called grain boundary (GB) engineering [1]. These improvements have generally been linked to a profusion of twin boundaries, labeled as Σ3 in the coincident site lattice (CSL) framework, making twin boundary fraction an important metric [2]. For example, Lin et al. [3] saw that the intergranular corrosion resistance of Inconel 600 was proportional to twin fraction. On the other hand, it has also been recognized that grain boundary topology, which cannot be assessed from boundary fraction, affects crack propagation [4,5]. Taking a more holistic view, Gertsman and Henager [6] observed that clusters of many grains were mutually connected by twin boundaries in GB engineered Cu-Ni. Within each cluster, every grain was related by a $\Sigma 3^n$ misorientation [6]. This clustering concept was formalized by Reed and Kumar, who called such features twin related domains (TRDs) and offered a mathematical framework for their analysis [7]. Reed et al. [8] then showed how the length scale of TRDs can be used explain the fracture roughness of GB engineered and conventionally processed Ni subjected to intergranular stress corrosion. Likewise, Lind et al. [9] showed that TRD size affects the progress of thermal grain coarsening. As a whole, these studies demonstrate how the concept of TRDs can be a valuable tool for understanding how GB engineering affects material properties.

With mounting evidence that TRDs are important microstructural features, it is desirable to know more about how they form. The statistical increase in $\Sigma 3^n$ GBs brought about by low-strain recrystallization has been known for some time [10]. The mechanism for this increase is Σ3 twinning, with higher order $\Sigma 3^n$ GBs being formed by incidental Σ3 interactions [11]. Indeed, twinning is fundamental to recrystallization, providing the only mechanism for producing orientations significantly different than those present in the deformed material, as reviewed by Berger et al. [12]. Dynamic recrystallization experiments on single crystal copper showed that all of the new orientations form as the result of twin chains [13]. A similar result was found for recrystallized aluminum, despite its high stacking fault energy [14]. Twinning can start in the early stages of nucleation [15], and continue to occur during boundary

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migration [16]. The first of these twins tend to occur along the primary or conjugate slip planes of the deformed material [17], while subsequent twins are more likely to form parallel to the moving growth front [16]. Twinning is suppressed when the growth front becomes a 30–40° ⟨111⟩ type boundary [14,17]. The areal density of these twins is proportional to the prior strain and inversely to the prior grain size [18].

While well understood on an individual basis, there remains more to learn about how these twinning events produce the sorts of large twin related domains that appear to be so important to the properties of GB engineered materials. Qualitatively, the thousand-member TRDs in a GB engineered material bear little resemblance to those in a conventionally processed one. Lind et al. [9] have shown that these materials have topological differences that must be considered in order to understand how large TRDs can be built from relatively few twins. It remains to employ this idea in a predictive relationship between TRD size and twin fraction. There also remains much to learn about the internal structure of GB engineered TRDs, and how it develops. Using metrics proposed by Cayron [19], Lind et al. inferred that TRDs in GB engineered materials contain fewer unique orientations relative to their size than do TRDs in conventionally processed materials. How this difference arises in two materials of identical stacking fault energy is currently unknown.

In this paper, we seek to understand how the size, topology and internal structure of TRDs develop during recrystallization. TRD formation is investigated by a series of interrupted annealing experiments, combined with orientation mapping. TRD boundary topology is considered, along with the probability of TRD coalescence. This leads to a scaling relationship linking TRD size to the twin fraction of the microstructure at large. Finally, we focus on understanding the internal structure of TRDs by applying metrics previously proposed in the literature and by examining their graph structure. These results are then used to examine how large GB engineered TRDs compare to smaller conventional ones. Taken as a whole, this provides a description of how GB engineered microstructures form and how to naturally quantify them.

2. Materials and methods

Varying degrees of cold work were applied to samples of commercial purity Ni (UNS N02201), oxygen-free electronic Cu (UNS C10100) and Inconel 718 (UNS N07718), followed by heat treatment. These materials were chosen because they can be readily grain boundary engineered and have a range of low to medium stacking fault energies. Inconel in particular is also of industrial significance in applications that can potentially benefit from GB engineering. The levels of deformation were selected to cover a range of GB engineering and conventional processes, with details presented in Table 1. The GB engineering treatments involve small deformations and are expected to produce high twin fractions, while the conventional processes have larger deformations and produce microstructures typical of commercially wrought material. Rolling was performed in a 10 inch mill, with no single-pass of less than 5%, and heat treatments were performed in air. Sample thicknesses are also listed in Table 1 because larger samples were observed to require longer times for complete recrystallization, presumably because they reached temperature more slowly. Specimens were water quenched, except for the Inconel samples, which were air cooled. Different material lots are identified in Table 1 because initial grain size and trace impurity content could have an effect.

Standard metallography techniques were used to prepare samples for electron backscatter diffraction (EBSD). Final polishing was performed with electropolishing when the stored plastic strain was of interest, in other cases mechanical polishing with colloidal silica was considered sufficient. EBSD data was collected using a Quanta 200 scanning electron microscope (FEI, Hillsboro, OR) equipped with a Hikari XP2 EBSD camera (EDAX, Mahwah, NJ). The map area and step sizes were selected to be suitable for the grain and TRD sizes of each sample, as listed in Table 1. The grain sizes are given as the circle equivalent diameter of the mean grain area. The orientation data analysis began with a standard dilation cleanup using commercial software (EDAX). Except when considering stored plastic strain, each grain was assigned a single average orientation and a 5° threshold was used for grain reconstruction.

Each TRD was reconstructed with a depth-first search for grains connected by Σ3 boundaries. This algorithm builds the twin network by starting at a random grain and exploring along a branch of twinned grains until no new twins are found, at which point it backtracks to another unexplored branch and the process repeats until every branch has been traversed. This search routine was repeated until every grain in the microstructure had been assigned to its parent TRD. A restrictive ±1° threshold was applied to identify

Table 1
Processing, EBSD, and microstructural parameters. Table lists the rolling reduction, and heat treatment temperature and time for each material. Iterated steps are noted by the number of iterations followed by an 'x' and the details of the repeated step. The supplier lots and thickness after rolling are also included. The EBSD step and map sizes are listed, and can be compared to the grain and TRD sizes. Twin number fraction is also listed for comparison with other literature.

ID	Material	Processing method	Thickness (in)	EBSD step size (μm)	EBSD map area (mm^2)	Grain size (μm)	TRD size (quadratic mean)	Twin number fraction
1	Cu (1)[a]	60%, 500° C/30 min	N/A	1	4	17	4.7	0.25
2	Cu (1)[a]	2×(20%, 500° C/30 min)	N/A	1	4	32	18.4	0.36
3	Cu (2)	60%[b], 280° C/195 min	0.133	0.5	0.25	7	5.3	0.27
4	Cu (2)	60%[b], 500° C/30 min	0.133	0.5	1	8	2.4	0.17
5	Cu (2)	(60%[b], 500° C/30 min) + (20%, 500° C/10 min)	0.107	0.5	1	12	16.7	0.35
6	Cu (2)	(60%[b], 500° C/30 min) + 2×(20%, 500° C/10 min)	0.086	0.5	1	18	24.4	0.39
7	Cu (2)	(60%[b], 500° C/30 min) + 3×(20%, 500° C/10 min)	0.065	0.5	1	18	15.7	0.36
8	Ni (1)[c]	5×(20%, 900° C/15 min) + 900° C/60 min	0.413	2	4	54	3.0	0.22
9	Ni (1)[c]	3×(5%, 900° C/15 min) + 400° C/24 h	0.881	2	4	82	8.8	0.35
10	Ni (2)	25%, 800° C/120 min	0.286	2	4	96	1.9	0.14
11	Ni (2)	5%, 900° C/15 min	0.361	2	4	92	11.8	0.38
12	Ni (3)	25%, 900° C/60 min	0.388	2	4	62	2.0	0.12
13	Ni (3)	5%, 900° C/15 min	0.492	2	4	59	15.5	0.38
14	Inconel (1)	25%, 1020° C/30 min air cooled	0.375	2	3	24	2.8	0.21
15	Inconel (1)	as-received, 1030° C/60 min air cooled	0.500	2	3	49	4.6	0.24

[a] These materials are the same used in Blobaum et al. [38], and were produced with a forging operation.
[b] Rolled immediately after prolonged submersion in liquid nitrogen.
[c] The materials are the same used in Bechtle et al. [33].