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Observation of annealing twin nucleation at triple lines in nickel during grain growth

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ABSTRACT

Three-dimensional near-field high-energy X-ray diffraction microscopy has been used to observe the formation of new twinned grains in high purity Ni during annealing at 800 °C. In the fully recrystallized microstructure annealed at 800 °C, twinned grains form along triple lines. Both the grain boundary character and the grain boundary dihedral angles were measured before and after the twin formed. These measurements make it possible to show that although each new twinned grain increases the total grain boundary area, it reduces the total grain boundary energy.

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1. Introduction

Annealing twins, separated from parent grains by long straight grain boundaries, are one of the most common and easily recognizable features in face centered cubic (FCC) metals with stacking fault energies less than about 0.15 J/m^2. Examples include metals and alloys such as Ni, Cu, Au, brass, and superalloys. Most notable in materials that have undergone recrystallization and grain growth, the twin boundary disorientation is 60° about a common ⟨1 1 1⟩ axis. When the boundary lies in the (1 1 1) plane of both crystals, the unique, low energy structure of these (coherent) twins [1,2] confers improved intergranular properties; this is one of the special boundaries that are exploited in grain boundary engineering [3–5]. Twins in FCC metals have also been implicated in the strengthening of nanostructured Cu [6], the nucleation of fatigue cracks [7], and the stagnation of grain growth [8]. While methods to increase the twin boundary concentration through thermomechanical processes are known [3–5,9,10], we know much less about the mechanism of annealing twin formation.

The proposed mechanisms for twin formation can be classified into three categories. The first is that twins form when crystals that already have a twin relationship impinge during growth [11,12]. The second assumes that a twin forms when growth occurs and a layer of atoms on the (1 1 1) plane is misplaced in the twin relationship; further growth on this misplaced layer leads to a twinned crystal [13–16]. The third involves the replacement of higher energy grain boundaries with a combination of a twin boundary and lower energy grain boundaries [17–20]. The available evidence cannot discriminate between these mechanisms for the case of grain growth because twins form within bulk metals that are opaque to visible light and electron beams. This makes it impossible to use standard probes to observe the three dimensional structure and crystal orientations before and after the twin has formed. For example, serial sectioning cannot be directly applied to this problem because the sample is destroyed during analysis [1,21,22] and transmission electron microscopy can only visualize the structure within very thin, nearly two-dimensional, samples. However, the recent development of near-field high-energy X-ray diffraction microscopy (nf-HEDM) [23–28] and X-ray diffraction contrast tomography [29,30] enables non-destructive measurements of the shapes and orientations of grains within a bulk sample at sequential stages during annealing.

In this paper, we consider the boundary replacement mechanism for twin formation during normal grain growth. Fig. 1

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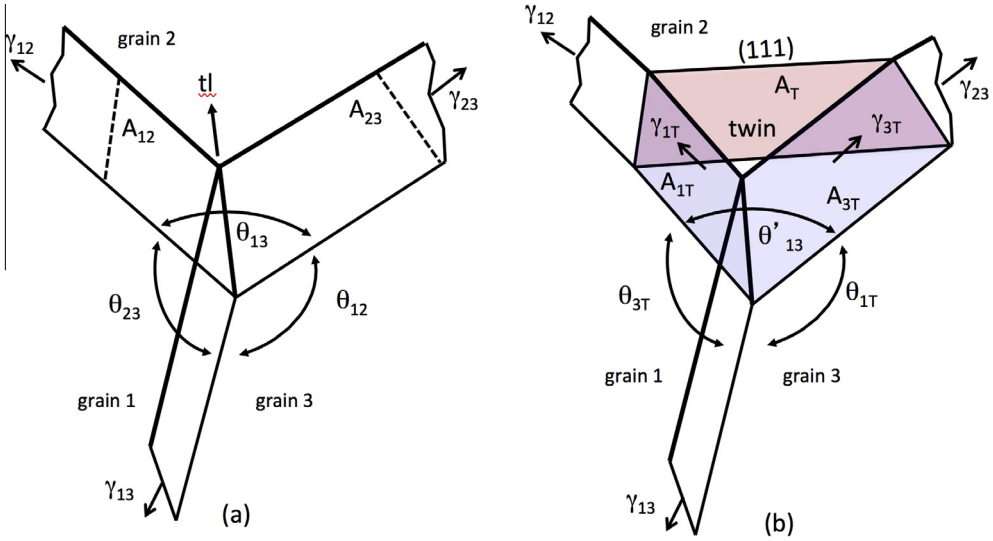

Fig. 1. A schematic illustration of a triple junction before (a) and after (b) a new grain, twinned with respect to grain 2 and labeled 'twin', is inserted. Three grain boundaries separating grains 1, 2, and 3, meet along the triple line labeled *tl*. The schematic defines all of the parameters referred to throughout this paper. Adapted from Murr [20].

illustrates the most important aspects of the theory [20]. The figure depicts three grains (labeled 1, 2, and 3) meeting along a triple line. The grain boundary energies per area (γ_{ij}), dihedral angles (θ_{ij}), and areas (A_{ij}), are labeled with subscripts that denote the relevant crystals. If a part of grain 2 adjacent to the triple line is replaced by a new grain with the twin disorientation, then a twin boundary is introduced and assumed to be on a (1 1 1) plane. The formation of this new grain reduces the energy if the following condition is met [20]:

$$A_{1T}\gamma_{1T} + A_{3T}\gamma_{3T} + A_T\gamma_T < A_{12}\gamma_{12} + A_{23}\gamma_{23} \quad (1)$$

Given the relatively low energy of the twin (γ_T = 0.06 J/m^2 for Ni) and the significant anisotropy of other grain boundaries [2], there should be many combinations of grains where this condition is met. According to Herring's [31] interfacial equilibrium condition, a decrease in the total energy should lead to observable changes in the dihedral angles such that $\theta'_{13} < \theta_{13}$ and $\theta_{23} + \theta_{12} < \theta_{1T} + \theta_{3T}$.

Fullman and Fisher [17], and later Murr [20], provided strong albeit indirect evidence for the mechanism depicted in Fig. 1. With previously available techniques, it was simply not possible to observe the same triple junction before and after the event. The repetitive non-destructive three-dimensional structural characterization afforded by nf-HEDM [23–28] makes it possible to directly observe the boundary replacement mechanism, if it occurs, and to evaluate the grain boundary crystallography and grain boundary dihedral angles. In this paper, results from high purity Ni annealed at 800 °C show that twins form at triple lines, as depicted above, and that the process decreases the total interfacial energy.

2. Methods

The three-dimensional microstructure of a Ni sample was measured in three different grain growth anneal states using nf-HEDM. The data from each anneal state was compiled as a discrete cubic grid of integers (grain identifiers) that are associated with a set of three Euler angles that describe the orientation of the crystal with respect to the external reference frame. The size of each voxel is 2 μm × 2 μm × 4 μm, in *x*, *y* and *z*, respectively. The x-y plane spanned a total of 600 × 600 voxels, while the *z* dimension was greater than or equal to 70 in each anneal state. The experiment was performed at Argonne National Laboratory's Advanced Photon Source, utilizing the 1-ID beam line to collect orientation diffraction patterns using 65 keV x-rays. The initial sample was a 99.999% pure 1 mm diameter nickel wire obtained from Alfa Aesar, which was first homogenized for two hours at 750 °C to obtain a reasonable grain size for the nf-HEDM method of about 25 μm. The sample was then characterized after three successive annealing steps, all at 800 °C and in a 3% H_2/97% N_2 environment to limit oxidation. The three annealing times were 23 min, 30 min, and 25 min. Details of the nf-HEDM experiment and data analysis are given elsewhere [23–28].

The normals to the grain boundary planes, also referred to as the grain boundary orientations, were determined in the following way; a more detailed description can be found in references [1,22,32]. First, the traces of each grain boundary on two-dimensional layers were approximated by line segments using the method described by Wright and Larsen [33]. Triple points were identified as three grain boundary traces that shared an end point. Triple points between three crystals with the same orientations on adjacent layers were assumed to be connected by a triple line; the vector product of the triple line and a grain boundary line segment provides the grain boundary normal. The dihedral angles were calculated from the angles between the grain boundary normals.

3. Results

An example of the three-dimensional Ni microstructure, interpreted from the nf-HEDM data, is illustrated in Fig. 2. The microstructure of the wire shaped specimen was established by a 2 h anneal at 750 °C that recrystallized damage from the wire drawing process. The microstructure contains many twins, which are apparent as long straight boundaries. One particular twinned grain that is visible on two perpendicular sections is indicated by the white arrows. The microstructure was measured in the initial state and after three sequential anneals (each for about 30 min) at 800 °C. Three-dimensional representations of the microstructures after the three anneals are illustrated in Fig. S1 and show that annealing led to modest but obvious changes in the microstructure. For example, the average grain size (equivalent spherical diameter) increased from 68 μm in anneal state 1 to 72 μm in anneal state 2 and was roughly constant after that (see Fig. S2).

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