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Evolution of Microstructure in Pure Nickel during Processing for Grain Boundary Engineering

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Abstract. Grain boundary engineered (GBE) materials have improved properties that are associated with the high fraction special $\Sigma 3^n$ boundaries in the microstructure, where n = 1, 2, or 3. Previous experimental studies with high purity nickel before and after GBE thermomechanical processing have shown that the fraction of Σ3 boundaries increased by at least factor of two [1]. Electron backscatter diffraction (EBSD) is used to characterize the evolution of these special boundaries throughout the recrystallization process of a 25% cold rolled sample annealed at 490°C. The fractions of the Σ3 boundaries and coherent twins have been measured over time revealing a steadily increasing population throughout the entire microstructure. However, when only the recrystallized regions are analyzed, the population of Σ3 boundaries initially increases rapidly and then stagnates over time. The evolution of the population of triple junctions containing special boundaries was also measured.

Introduction

Grain boundary engineered (GBE) materials feature improved material properties including reduced crack growth rate and improved corrosion resistance properties. The most notable property of grain boundary engineered materials is the increased number of special boundaries in the material [2]. Grain boundary misorientations are classified by the coincident site lattice (CSL) theory. The CSL theory designates the misorientation based on the inverse of the number of overlapping lattice sites [3]. While most high angle boundaries have high energy, the coherent Σ3 boundary, a symmetric twist 60 degree misorientation on the (111) plane, or an annealing twin, has been found to provide unique properties to the microstructure because of its low energy. The presence of these boundaries in Ni-based superalloys have been shown to impede intergranular stress corrosion cracking [4]. Other special boundaries of interest are the Σ9 and Σ27. The population of these boundaries has been observed to increase during GBE processing and they are assumed to play a role in the GBE properties [5].

It has also been concluded from previous studies that the proportion of special boundaries alone does not account for the improved material properties [6]. Most researchers agree that the grain boundary network and how the special boundaries are connected to one another in the microstructures play a significantly larger role. To characterize the connectivity, triple junctions are classified based on the number of special versus the number of random boundaries at the junction. Scientists have also applied percolation theory for triple junction characterization [7]. Upon reaching some threshold of special boundaries in the microstructure as well as a high degree of connectivity, the material will exhibit GBE properties. Traditional microstructures feature high percentages of completely random high angle triple junctions, while a GBE treated material is expected to behave oppositely as the degree of connectivity of the special boundaries in the grain boundary network develops.

GBE studies have focused on how the CSL content of the initial non-GBE microstructure compares to the final GBE microstructure as a result of varying the thermo-mechanical processing variables [8]. While different strains and annealing temperatures can alter the special boundary content, a more interesting point is that these steps can be iterated to increase the fraction of special boundaries typically up to 60-70%. However, after a certain number of cycles or too large a strain, the special boundary fraction actually begins to decrease [9]. This motivates the need to observe how the microstructure evolves prior to reaching the limiting concentration of special boundaries. The purpose of this paper is to characterize how the fractions of special boundaries change over time in one annealing cycle in both the overall microstructure and specifically in the recrystallized regions. Triple junction classification reveals that the microstructure exhibits additional changes not just associated with increasing population of special boundaries, but also decreasing fractions of random triple junctions.

Experimental Procedure

A high purity nickel rod (diameter of 5mm) of 99.999% purity was obtained from Alfa Aesar, and sample specimens were sectioned from the rod of approximately 2 mm thickness. Samples were homogenized by annealing at 410°C for 24 hours and then cold rolled to 25% reduction. Samples were mechanically polished starting with SiC 600 grit and diamond abrasives and finishing with a final polish of colloidal silica of 0.05 μm.

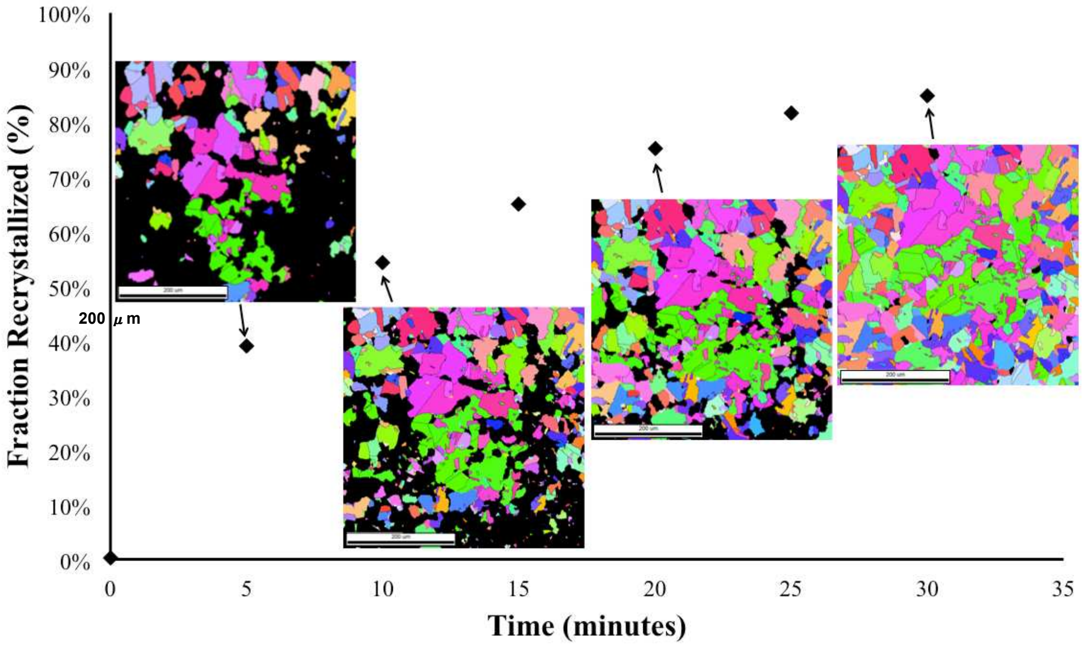

Figure 1. Time evolution of the volume fraction of grains with a GOS below 1.0°

Crystal orientations of the planar surface in the rolling and traverse planes after deformation were observed by EBSD using an EDAX/TSL acquisition software with a Hikari EBSD detector on a Quanta 200 FEG SEM. Orientation maps with an area of 450 μm x 450 μm, were acquired with a 1.5 μm step size. The samples were repeatedly annealed for 5 minutes at a time at 490°C in a dry Ar/H_2 tube furnace until fully recrystallized at 30 minutes. Orientation maps of the same areas were captured after each anneal. EBSD data was processed using OIM Data Analysis software from for finding recrystallization fraction and determining the CSL fractions based on Brandon criterion [3]. Further data processing on triple junction analysis was achieved using the exported reconstructed boundary segments.

Results and Discussion

The grain orientation spread (GOS) was used to partition the recrystallized and deformed regions of the microstructure. Recrystallization progress was measured with both hardness testing and GOS. The progress of recrystallization against time is shown in Fig.1, along with some of the corresponding EBSD maps partitioned with a GOS < 1.0°. It should be noted that partitioning by GOS only includes the boundaries between grains satisfying the GOS condition implemented (between fully recrystallized grains), and none of the boundaries between the recrystallized and deformed grains.

Partitioning by GOS to only include the recrystallizing grains reveals a very interesting behavior in the length fraction of special boundaries present in the new grains as shown in Figure 2. Note that the Σ3 fraction of boundaries approaches a maximum at 10 minutes, Fig. 2(a) (which corresponds to approximately 50% recrystallization), and then decreases over time. By contrast, the Σ9 fraction continues to increase throughout the annealing process. Similarly the behavior of the Σ27a and Σ27b fractions is also apparent in Fig. 2(b), where the Σ27a shows a small increase while the Σ27b fluctuates slightly.

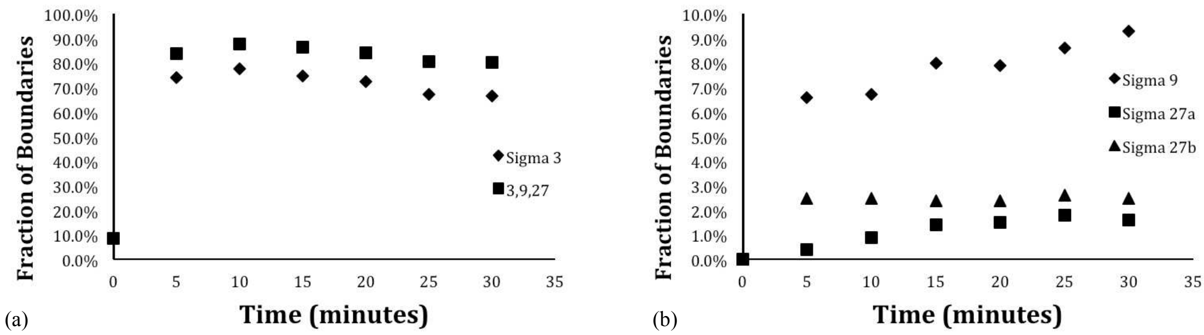

Figure 2. The fraction of boundaries only from the recrystallized portion of the microstructure a) Σ3 boundaries and all special boundaries; b) Σ9 and Σ27 boundaries only.

Analysis of the data shows that the high fraction of Σ3 boundaries initiates in the recrystallizing fraction of the GBE material, but then decreases rather quickly after reaching 50% recrystallized. It should also be noted though that 70% Σ3 boundaries in the final anneal is close to the saturation limits found in GBE Ni materials [1]. This behavior suggests that there are fewer Σ3 boundaries generated during the later stages of recrystallization and starting regime of grain growth. The continued increase of Σ9 boundaries most likely arises from the geometric requirements of special boundaries interaction at triple points as the recrystallizing grains grow. The intersection of two Σ3 boundaries in the same grain forms a Σ9 boundary. Similarly the increase of Σ27a and Σ27b will arise from interactions of Σ3 and Σ9 boundaries. As recrystallization progresses, the length fractions of Σ9 and Σ27 boundaries increase from these interactions, but not enough to compensate the decrease of Σ3 boundaries and hence the overall decrease of total fraction of special boundaries in Fig. 2(a).

Figure 3 illustrates the change in triple junction nature of the entire microstructure throughout the recrystallization process. The decrease in the RRR triple junctions is expected along with a pronounced increase of ΣΣΣ types of triple junctions. This is associated with both the increase of special boundary fractions as noted from Fig. 2(a) and the increasing connectivity as recrystallizing regions impinge on each other as seen from Figure 1. Specifically focusing on 3,3,9 combination reveals that this configuration contributes to the largest increase of ΣΣΣ triple junctions according to Table 1. This is expected as two Σ3 boundaries that meet must form Σ9 at the triple junction as suggested earlier [5]. Again, this behavior correlates with the increasing fraction of Σ9 boundaries observed in Fig. 2(b).

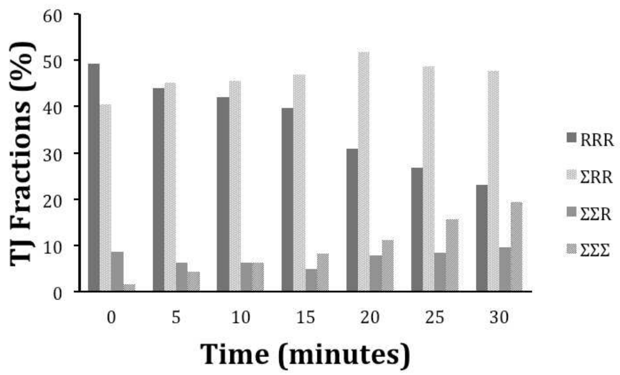

Figure 3. Fraction of triple junctions based on random boundaries vs. special boundaries.

Table 1. Fraction of 3/3/9 type triple junctions compared to total ΣΣΣ triple junctions.

Boundary Type	**Time (minutes)**						
	0	**5**	**10**	**15**	**20**	**25**	**30**
ΣΣΣ	1.6%	4.3%	6.2%	8.2%	11.1%	15.7%	19.3%
→ 3/3/9	1.5%	3.9%	5.3%	6.7%	8.9%	11.5%	14.7%

Summary

The evolution of special boundaries in the microstructures can be observed throughout the recrystallization process. In the recrystallized regions of the maps, the fraction of Σ3 increases rapidly at first and then somewhat decreases, consistent with what was reported for stainless steel [10]. Consideration of the recrystallization region also provides a clearer observation on the behavior of Σ9 and Σ27 boundaries. The grain boundary characteristics and how the triple junction character changes over time were quantified, all of which strengthens our knowledge of how GBE is accomplished.

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