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The role of deformation temperature and strain on grain boundary engineering of Inconel 600

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ABSTRACT

The present study investigates the effect of deformation temperature and strain on the formation of twin boundaries in Inconel 600. With a constant strain rate of 0.003/s, deformation temperatures were varied between 25 °C and 982 °C while strains varied between 11% and 80%. The resulting microstructures were characterized using electron back scatter diffraction both immediately following deformation and after an annealing cycle at 1025 °C. Comparison of the grain boundary characters associated with the samples enabled correlation of the processing parameters to the formation of desirable coherent twin boundaries and the identification of a deformation mechanism map. Processing parameters associated with strain annealing and dynamic recovery were found to promote the formation and retention of twin boundaries, while statically and dynamically recrystallized microstructures tend to contain large proportions of random grain boundaries that mitigate the effects of grain boundary engineering.

1. Introduction

Inconel 600 is a commercially available Ni–Cr–Fe alloy that exhibits a good combination of strength, formability and environmental resistance at elevated temperatures. This combination of properties makes these materials ideally suited for use as structural components in chemical processing equipment, nuclear reactors and aerospace applications [1–4]. To improve the overall efficiency and performance of many of these applications, advanced design concepts require many of these structures to operate under higher loads and temperatures. These continuous changes serve as the primary motivation for the development of innovative new materials, alloys and manufacturing processes. Pertaining to some of the recent advances in processing, grain boundary engineering (GBE) has been used to enhance the mechanical and environmental resistance of polycrystalline engineering alloys [5–11]. Property improvements associated with GBE have been attributed to the increase in the overall proportion of "special boundaries" that possess a large number of coincident lattice sites along grain boundaries. Grain boundaries in polycrystalline alloys can be classified on the basis of these coincident site lattices (CSL) and the reciprocal density of the coinciding sites can be designated as Σ. From this description, "special boundaries" are typically defined as those with $\Sigma < 29$, with the Σ3 twin boundaries being the most desirable ones to have distributed within the random boundary network. Polycrystalline alloys that contain a sufficiently high fraction of these special CSL boundaries that break up the interconnectivity of the pre-existing random grain boundary network [12–14] exhibit improved strength [15], creep performance [16] resistance to intergranular fracture [9–11], stress corrosion cracking [11–17] and fatigue crack propagation [18–19].

Recent advances in the development of electron back-scatter diffraction (EBSD) characterization tools have enabled materials scientists and engineers to develop a significantly better understanding of the role of crystallographic texture and grain boundary character on the properties of virtually all classes of materials. Despite some successes in achieving significantly improved properties via GBE, these advances have not yet been fully realized in the production of physically large bulk structures. This can largely be attributed to the existing practices for GBE that utilizes multiple iterations of cold rolling to 5–20% strain at room temperature followed by short annealing cycles of a few minutes after each deformation step. Since each iteration of deformation and annealing imparts only a modest increase in the fraction of twin and special grain boundaries, multiple iterations are required to achieve a sufficiently high fraction (~50–60%) of "special grain boundaries" that result in the improved properties. From a commercial manufacturing perspective, room temperature deformation of high strength Ni-base superalloys of interest is impractical as these materials can withstand only limited deformation without cracking, forging die materials cannot withstand the forging stresses required, and forging equipment would limit the size and complexity of potential component configurations. Moreover, the short annealing times employed to limit recrystallization and grain

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growth are not compatible with the large thermal inertia inherent in large complex structures. Moreover, the multiple deformation and thermal processing cycles would add manufacturing lead time and cost even if the aforementioned limitations could be overcome. As such, the current approaches used for GBE are not ideally suited for the fabrication of physically large, complex components for propulsion and power generation applications.

In polycrystalline materials, the character of the grain boundaries is extremely important and greatly influences both the physical and mechanical properties of the material, particularly at elevated temperatures [20–22]. In recent years, ultra-fine grain processing techniques have been developed to produce bulk metallic materials with sub-micron grain sizes [23–27]. At relatively low temperatures, the high densities of grain boundaries serve as obstacles to restrict dislocation activity and resist fatigue crack propagation. In these instances, the Hall–Petch relationship is valid for numerous material systems and relates changes in the yield strength to grain boundary density or grain size. However, for structural materials intended for use at elevated temperatures, fine grain sizes and high densities of grain boundary area are not necessarily beneficial as grain boundary sliding and the accumulation of cavitation damage contribute to the degradation of creep properties. The random grain orientations that exist in isotropic polycrystalline materials lead to the formation of a random grain boundary network consisting of a statistical distribution of grain boundary orientations. Depending on the relative orientations, the coherency of the individual grains with their neighbors or CSL at the boundary may vary significantly. CSL boundaries, with $\Sigma > 29$, tend to contain large concentrations of crystalline defects and vacancies that serve to both weaken the interface and promote diffusive mechanisms at elevated temperatures. Under these conditions, grain boundary diffusion and sliding are accelerated along these boundaries. This leads to environmental degradation and poor resistance to creep deformation. On the other hand, adjacent or neighboring grains that exhibit CSL boundaries, with $\Sigma < 29$, have relatively coherent interfaces that contain few crystalline defects that would serve to weaken the boundaries. In this regard, coherent twin boundaries with $\Sigma = 3$ are highly desirable features in grain boundary engineered materials since they possess some of the lowest grain boundary energies [5–16,28,29]. Moreover, since there are comparatively fewer vacancies and defects along low Σ interfaces, the mechanisms by which diffusion and mass transport occurs along the interfaces are also more sluggish. Thus, polycrystalline materials containing high proportions of special CSL boundaries are desirable for use at elevated temperatures. In order to induce the formation of sufficiently large fractions of special grain boundaries, strain energy in the form of dislocations first needs to be stored in the material. Upon subsequent annealing, the supplied thermal energy enables the system to effectively utilize the stored strain energy to form twins and other special CSL boundaries. It is still not entirely clear whether the underlying mechanisms responsible can be attributed to dislocation recovery or grain boundary migration induced by the differences in strain energy. In this paper, the effect of various hot deformation processing parameters on the formation of $\Sigma 3$ boundaries in IN600 was investigated.

2. Procedure

Fully recrystallized hot rolled bars of IN600 with a composition listed in Table 1, were machined into cylindrical compression specimens with a diameter of 10 mm and a height of 15 mm. Using an MTS servo-hydraulic testing system equipped with a resistance furnace, a total of 16 samples were compressed at a strain rate of 0.003/s to true strains of 11%, 28%, 51% and 80% at temperatures of 25 °C, 538 °C, 760 °C and 982 °C. Strains were measured using a MTS high temperature extensometer with alumina extension arms while temperatures were measured by averaging the values obtained from three thermocouples attached directly to the surface of the sample. For tests conducted at elevated temperatures, the samples were immediately quenched in water following deformation in order to preserve representative microstructures. The compressed samples were sectioned longitudinally along their centers using a low speed diamond saw. For each deformation temperature and strain level, one half of the sample was subjected to an annealing treatment that consisted of heating the sample from room temperature to 1025 °C at a rate of 23.8 °C/min and an 18 min isothermal hold at 1025 °C. To investigate the effect of annealing time on the formation of $\Sigma 3$ boundaries, multiple samples were deformed to 25% strain at 25 °C and sectioned. These samples were placed directly into a furnace set at 1025 °C and annealed for 5, 18, 38 and 60 min followed by a water quench.

Table 1
Nominal composition of Inconel 600 (wt%).

Ni	Cr	Fe	C	Mn	Si	Cu
Bal.	15.5	8.0	0.15	1.0	0.50	0.50

Following deformation and/or heat treatment, all of the samples were prepared using standard metallographic procedures that incorporated a final polish using 0.05 μm colloidal silica. A JEOL 5900 equipped with an Oxford Instruments Nordlys Nano EBSD system was used to measure the character of the grain boundaries and quantify the average intragranular misorientations. Multiple EBSD scans were taken from each sample to produce statistically significant results and provide quantitative information at different length scales. High magnification scans were taken over an area spanning $40 \times 40\ \mu m^2$ using a 0.1 μm step size. These EBSD scans were used to produce inverse pole figures showing the individual grain orientations and the magnitude of the intragranular misorientations. Lower magnification scans over an area of $350 \times 350\ \mu m^2$ using 0.7 μm step size were used to generate datasets that contained statistically significant numbers of grains for determination of the grain boundary characters. Data from samples in the as-deformed condition were compared to those following annealing.

3. Results

In the as-received or hot rolled condition, the IN600 sample contained equiaxed grains with an average grain size of approximately 7.7 μm, Fig. 1a. Twin or $\Sigma 3$ boundaries were observed in this initial condition and EBSD analyses revealed that their length fraction (total $\Sigma 3$ length/total length of all boundaries) was 36%. The grain orientation map for hot rolled IN600, shown in Fig. 1b, also reveals that there were no measurable changes in the intragranular misorientations that correspond to plastic deformation or the presence of pre-existing dislocation networks and is consistent with a fully recrystallized microstructure.

Cylindrical compression samples were deformed to various strain levels at different temperatures. Depending on the deformation temperature, the samples exhibited characteristic flow behaviors, Fig. 2. The peak flow stresses were observed to decrease dramatically as a function of temperature and ranged from 143 MPa at 25 °C to 16 MPa at 982 °C. Additionally, the degree or magnitude of strain hardening was also observed to decrease as deformation temperatures increased. Only a modest amount of strain hardening was evident at 760 °C and no strain hardening was observed during deformation at 982 °C. As samples were deformed to various levels of true strain at these temperatures, the corresponding changes in the microstructure were characterized. In the as-deformed condition, the magnitude of the intragranular