Modeling the effect of thermal – mechanical processing parameters on the density and length fraction of twin boundaries in Ni-base superalloy RR1000

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Abstract

Enhancement of the physical and mechanical properties of polycrystalline Ni-base superalloys may be achieved through control of the grain boundary structure and is dependent on the optimization of the thermal-mechanical processing parameters. Superalloys containing grain boundary networks that are comprised with a sufficiently high fraction of $\Sigma 3$ /twin boundaries have been reported to exhibit enhanced creep and fatigue properties. In this report, the density and length fraction of twin boundaries in annealed samples of powder processed Ni-base superalloy RR1000 were quantified and expressed as a function of the average grain diameter. The results were found to be consistent with classical models relating density and length fraction to grain size. The effects of varying hot deformation parameters on twin density and length fraction were also quantified and modified models were derived to describe the relationships.

Keywords- Grain boundary engineering, Twin, $\Sigma 3$, Density, RR1000.

1. Introduction

Ni-base superalloys are widely used in advanced gas turbine engines due to their superior fatigue, creep, and corrosion resistance in high temperature environments. Among the critical components that constitute turbine engines, materials used as structures for turbine blade and disk applications require the highest temperature capability as they operate directly within the pathways of

the combustion gases. Single crystal superalloys are state-of-the-art for use as blades in advanced turbine engines where creep deformation tends to be the principal life-limiting mechanism [1,2]. Conversely, polycrystalline Ni-base superalloys are used as disk rotors that operate at relatively lower temperatures and higher stresses where fatigue and fracture properties are of much greater concern [3-6]. However, as the operating temperature of advanced gas turbine engines increase to improve performance and reduce emissions, this requires turbine disk materials to possess a temperature capability of 750°C or above. At these elevated temperatures, creep and resistance to hot corrosion become important mechanisms that will interact with fatigue mechanisms. For this reason, improvements in the physical and mechanical properties of polycrystalline Ni-base superalloys are required for the development of next generation gas turbine engines.

Grain boundary engineering (GBE) has been used to improve the properties of various polycrystalline materials by modifying the character of the grain boundaries in the microstructure of the alloy [7]. Introducing special low- Σ boundaries, such as $\Sigma 3$ twin boundaries, is key to GBE and the improvement of properties such as resistance to corrosion [8], creep deformation [9], and fatigue crack propagation/initiation [10,11]. In polycrystalline materials, each grain boundary can be characterized by its orientation defined by the two adjacent grains. The coherency between the lattices of the two neighbor grains is defined using the coincidence site lattice (CSL) model which classifies the degree of coherency using a Σ value attributed to the grain boundary [12]. Low CSL boundaries, or $\Sigma > 29$, possess large concentrations of crystalline defects and vacancies which promote diffusive mechanisms at elevated temperatures thereby leading to environmental degradation and poor resistance to creep deformation. Conversely, coherent interfaces i.e. high CSL boundaries, or $\Sigma < 29$, contain few crystalline defects and therefore, the mechanisms by which diffusion and mass transport occur along these interfaces are minimized. Employing GBE techniques to introduce a high proportion of $\Sigma 3$ boundaries in the material and improve the properties of the alloy may potentially

extend the temperature capability of existing Ni-base superalloys and enable them to accommodate operation at higher temperatures.

To date, GBE has been used in a limited number of commercialized applications [13] and consists of iterations of cold rolling to 5 to 20% strain at room temperature followed by short time annealing of a few minutes [14]. Due to the high stresses associated with cold work combined with the iterative deformation and anneal cycles, conventional processing routes for GBE are not ideally suited for production of physically large and geometrically complex Ni-base superalloys components. In addition, the thermal inertia involved during heat treatment of physically large and complex shaped components is not compatible with short annealing times. Consequently, GBE approaches that utilize thermo-mechanical processes capable of producing near net shape components at stresses applicable to the forging capabilities of current turbine engine manufacturers need to be considered. In order to understand the fundamental mechanisms governing the formation of the desirable $\Sigma 3$ boundaries and facilitate the development of a robust GBE processing route for Ni-base superalloys, models need to be developed to effectively relate the effects of thermo-mechanical process parameters and the grain boundary character of the material.

2. Experimental procedure

In this investigation, 10 mm diameter discs of Ni-base superalloy RR1000, of composition listed in Table 1, were cut from an as-consolidated billet for a series of heat treatments. The processing route and characteristics of RR1000 have been described previously [15]. The billet was first heat treated at 1110°C for 4h prior to annealing in order to minimize residual strains originating from the hot extrusion process used to consolidate the billet. Heat treatment of the samples was performed at a sub-solvus temperature of 1115°C and two super-solvus temperatures of 1140°C and 1150°C, for times ranging from 5min to 6h. The microstructures and grain boundaries were

characterized using an Oxford Instrument Nordlys-HKL electron back scatter diffractometer (EBSD) attached to a JEOL JSM 5900-LV scanning electron microscope (SEM). A minimum of three regions measuring 417×333 px were mapped at the geometric center of all of the surfaces where strains were uniform and constants. Oxford Instrument Channel 5 software was used to obtain $\Sigma 3$ (misorientation $\Delta\theta = 60^\circ \pm 5^\circ$), and random boundary ($\Delta\theta \geq 15^\circ$) maps necessary for the calculation of the length fraction and density of twin boundaries. The twin density was defined as $p = \frac{n_p \times \Delta}{A}$ where p represents the density of $\Sigma 3$ boundaries in μm^{-1} , n_p is the number of map-pixels that compose the $\Sigma 3$ boundaries in the EBSD map, Δ is the step size in μm , and A is the surface area in μm^2 . In order to minimize errors originating from the grain boundaries thickness in the calculation of the twin density, the step size was kept constant at $0.48 \mu\text{m}$ for most of the samples. The step size was adjusted to account for conditions in which extensive grain growth occurred and the error in the calculation of the twin density was calculated to be less than 10%. In all instances, the grain sizes were calculated from the EBSD maps with a minimum grain diameter of 4.2Δ in order to account for the limit resolution of the EBSD. This corresponds to a grain size of $2 \mu\text{m}$ for scans taken at a step size of $0.48 \mu\text{m}$. For the as-deformed samples containing micron-sized microstructural features, a step size of $0.24 \mu\text{m}$ was used during EBSD characterization and a minimum grain size was defined as $1.5 \mu\text{m}$ in order to exclude dataset biasing from coarse primary γ' precipitates.

In addition to assessment of the grain size and boundary character corresponding to the heat treated billet material, the effect of varying hot deformation parameters was also investigated. Hot deformation of cylindrical samples measuring 10 mm in diameter and 15 mm in height was performed on a Gleeble 3500 system at strain rates of $0.01/\text{s}$ and $0.05/\text{s}$, final strains of 0.2 and 0.5 , and deformation temperatures of 1020°C , 1060°C , and 1100°C in order to simulate thermo-mechanical forging. The samples were rapidly heated to the set temperature in 1 min , allowed to

thermally soak for 3 min under a load of ~ 12 MPa prior to deformation. Graphite was used to minimize friction during deformation and avoid excessive barreling of the sample. Following compression, the samples were sectioned parallel to the deformation axis. The deformed samples were subsequently annealed for various times at either the sub-solvus temperature of 1115°C or super-solvus temperature of 1145°C . In addition to the $\Sigma 3$ boundary maps, local misorientation maps were obtained from Channel 5 using a 5×5 px filter size from scans taken at a finer step size of $0.06 \mu\text{m}$. The same scans were used to observe and quantify the fraction of low angle boundaries.

Table 1. Composition of RR1000 (wt%).

Ni	Cr	Co	Mo	Ti	Al	Ta	Hf	Zr	C	B
Bal.	15	18.5	5	3.6	3	2	0.5	0.06	0.027	0.015

3. Results and discussion

3.1 Twin density and length fraction in heat treated samples of RR1000

The experimental results of twin density for the heat treated discs were plotted in Fig. 1 as a function of the average grain size. As expected, the smallest grain sizes were associated with sub-solvus heat treatment at 1115°C and ranged from 4.9 to $5.9 \mu\text{m}$ after 5 min and 2h at temperature, respectively. The respective twin densities were found to be nominally constant although a slight increase was observed when compared to the original heat treated billet and associated with the early stages of grain growth where migrating boundaries can generate annealing twins. However, the extensive grain growth that occurred during super-solvus heat treatment, made possible by the dissolution of the primary γ' precipitates originally pinning the grain boundaries [16], was found to decrease the twin density from $0.26 \mu\text{m}^{-1}$ to $0.10 \mu\text{m}^{-1}$ after heat treatment at 1140°C for 5 min and 1150°C for 45min respectively. The decrease in twin density with increasing grain size during

dynamic grain growth has been reported to be related to the loss of the twin boundaries located in shrinking grains [17]. Fig. 2 shows the grain boundary maps arranged by increasing grain size and reveals the expansion of the random grain boundary network during which the twin boundaries located inside the grains will grow and sometimes detach from the random grain boundaries to form internal twins in larger grains.

The formation of annealing twins has been described and several models have been developed over the years. The growth accident model is based on the formation of a coherent twin boundary due to a stacking error at the migrating grain boundary during grain growth [18] and has been supported by a number of recent investigations [17,19]. Based on the suggested formation mechanisms, two expressions of the twin density in FCC metals during grain growth regime have been commonly used: Gleiter [20] expressed the probability that a $\{111\}$ plane is a coherent twin boundary leading to the expression of the twin density as a function of the grain size and annealing temperature, and Pande et al. [21] showed that the twin density is proportional to the grain size only. Pande et al.'s model has been validated in a number of studies and the same grain size dependence can be seen in the RR1000 samples, Fig. 1. Regardless of the annealing temperature and time, the twin density is shown to be proportional to the grain size. Therefore, Pande et al.'s expression was chosen for comparison with our experimental data and was formulated in Eq. (1):

$$p = \frac{1}{D} k \ln \left(\frac{D}{D_0} \right) \quad (1)$$

where p is the twin intersection per unit length i.e. twin density, D is the grain size, k is a constant independent of temperature, and D_0 is the critical grain size. D_0 has been defined as the minimum grain size below which no twin boundaries may exist. In certain instances, detailed EBSD characterization of the as-deformed and annealed RR1000 samples using a step size of 0.06 μm

revealed the presence of sub-micron grains within the microstructure that are approximately the same size as the coarse γ' precipitates. Although twin boundaries were occasionally observed within grains possessing an average diameter of $\sim 0.50 \mu\text{m}$, accounting for these sub-micron features does not significantly influence the calculated values of twin density and length fraction. Compared to values used in investigations that used Pande et al.'s model, D_0 was found equal to $1 \mu\text{m}$ [22], $1.5 \mu\text{m}$ [19] and $2.5 \mu\text{m}$ [17] for pure Ni, and $1.5 \mu\text{m}$ for Cu alloys [19]. Although Pande et al.'s equation better describes the independence of the annealing temperature or time as long as the grain size is known, its expression contains a fitting constant that has not been determined to this date whereas Gleiter's equation does not. However, Pande et al. proposed the following relation $k = K(\gamma_{rb}/\gamma_{tb})$, where γ_{rb} is the random grain boundary energy, γ_{tb} is the twin boundary energy, and K is another proportionality constant. By fitting Eq. (1) to the experimental data of RR1000 in Fig. 1 with the critical grain size $D_0 = 0.50 \mu\text{m}$, the resulting constant k was calculated to be $0.65 \text{ m}^2\text{J}^{-1}$. Compared to the value of $0.2 \text{ m}^2\text{J}^{-1}$ for pure Ni [22], this k value is significantly higher and the difference can likely be attributed to the stacking fault energy of the alloy in the expression of k [23]. Moreover, recent atomistic simulations have revealed considerable spreads in the values of random grain boundary energy [24] and more experiments are necessary to determine a more accurate expression of the twin density without the proportionality constant.

Fig. 3 shows the twin length fraction as a function of the average grain diameter. The twin length fraction was found to increase with increasing grain size as expected from the decrease in the overall length of the random grain boundary network that occurs during grain growth. The rate of increase, however, was found to decrease with increasing grain size. This phenomenon can be likely attributed to the loss of twin boundaries located within shrinking grains [17]. As seen in Fig. 4, during grain growth, the length of the random grain boundaries decreases substantially faster than the length of the twin boundaries. Consequently, extensive grain growth should result in a limit in twin length

fraction as observed in other investigations [25] and an appropriate fitting curve would be an exponential decay as formulated in Eq. (2),

$$F_{\Sigma 3} = F_l \left\{ 1 - \exp \left(-\frac{D}{D_l} \right) \right\} \quad (2)$$

where $F_{\Sigma 3}$ is the length fraction of twin boundaries, F_l is the fraction limit i.e. maximum twin length fraction, and D_l is the starting grain size prior to heat treatments. Fitting of the equation using $D_l = \sim 4.8 \mu\text{m}$, calculated from the sample prior to thermal treatments, on the experimental data resulted in the curve shown in Fig. 3 and a fraction limit $F_l = \sim 54.7\%$.

3.2 The effect of hot deformation on the twin density and length fraction

Figs. 1 and 3 correspond to the twin density and length fraction of heat treated RR1000 billet material that was not subjected to any mechanical deformation and therefore, these models for density and length fraction can be used as reference curves to investigate the effect of varying thermal-mechanical processing parameters on the grain boundary character in deformed RR1000 samples. The experimental results of twin density and length fraction from the samples deformed at 1020, 1060, and 1100°C at a strain rate of 0.05/s to final strains of 0.2 and 0.5 after sub and super-solvus annealing for 1h are plotted in Fig. 5a and 5b, respectively, along with the model curves from Figs. 1 and 3. Interestingly, increasing the deformation temperature and decreasing the final strain resulted in lower values of twin density and length fraction. Following sub-solvus annealing (smaller grain sizes), the difference in twin density is significant in comparison to super-solvus annealing (larger grain sizes) where the twin density falls between $\sim 0.05 \mu\text{m}^{-1}$ and $\sim 0.10 \mu\text{m}^{-1}$. This can be attributed to the dynamic reduction of twin boundaries during compression [16]. Following compression, few twin boundaries remain in the microstructure of the alloy and subsequent annealing

will assist in relieving the stored strain energy and induce the formation of twin boundaries. Therefore, higher twin densities are expected in samples that have been deformed under conditions where dislocation-based plasticity mechanisms dominate (1020°C compression temperature) than samples that had been deformed under conditions where super-plastic deformation mechanisms are operative (1100°C compression temperature) [16] and no intragranular misorientations are produced, Fig. 6. As a result, both the twin density Fig. 5a and length fraction Fig. 5b are lower in samples exhibiting little to no intragranular misorientations following deformation.

Samples deformed at 1020°C with a strain rate of 0.01/s to a final strain of 0.2 were annealed for various times to investigate the variation of the twin density and length fraction with grain size. Despite reducing the density and fraction of twin boundaries during deformation, upon annealing, the samples exhibited behaviors that were nominally identical to the non-deformed billet material, Fig. 5a-b. Since similar microstructural changes were observed for the samples deformed at other temperatures and strain rates, Eqs. (1) and (2) were modified to take into account the effect of uniaxial deformation at high temperature. Starting with the twin density, the Pande model was modified to incorporate the effect of strain during cold working [25]. By considering the difference in Gibbs energy between growing and shrinking grains $\Delta G^0 = 4\gamma_{rb}/D$ [20] and substituting this term into Eq. (1), the formulation of the twin density becomes $p = k_a \Delta G^0 \ln(D/D_0)$, where k_a is a constant independent of temperature. Following, the magnitude of cold work was related to the amount of residual strain by Li et al. [25] using $\Delta G^0 = (4\gamma_{rb}/D)(1+k_b s^2)$ where k_b is material constant and s is the amount of plastic strain induced during deformation, prior to annealing. Li et al. justified the use of s^2 from the expression of the residual strain energy in a material $Ee^2/2$, where E is the elastic modulus of the material and e is the relative strain [19,25]. Although the three major parameters that influence the amount of residual plastic strain in the material after hot deformation are deformation temperature, strain, and strain rate, the existing form for the difference in Gibbs energy only accounts for strain

resulting from cold work. However, the stored energy can be calculated from EBSD data [26] and used directly in the formulation of the twin density. Subsequently, the expression of the Gibbs energy can be modified to account for the strain energy stored during deformation: $\Delta G^0 = (4\gamma_{rb}/D)(1+k_c E_s)$, where E_s is the average calculated stored energy and k_c is a dimensionless material constant. Using $E_s = S_{lb} \langle \gamma(P(\Delta\theta)) \rangle$ [26] where S_{lb} is the area per unit volume for the low-angle boundaries and $\langle \gamma(P(\Delta\theta)) \rangle$ is the value of the grain boundary energy γ from the misorientation angle distribution $P(\Delta\theta)$ obtained from the EBSD low-angle grain boundaries maps Fig. 6a-c. The grain boundary energy was then calculated for low-angle boundaries ($2.5^\circ < \Delta\theta < 15^\circ$) using the Read-Shockley equation:

$$\gamma = \begin{cases} \gamma_m \left(\frac{\Delta\theta}{\theta_m} \right) \left[1 - \ln \left(\frac{\Delta\theta}{\theta_m} \right) \right] & \text{for } \theta \leq \theta_m \\ \gamma_m & \text{for } \theta > \theta_m \end{cases}$$

(3)

In Eq. 3, γ_m is the specific energy of high-angle boundaries and θ_m is the misorientation limit for low-angle grain boundaries chosen at 15° . The addition of E_s to Pande's model results in a vertical translation of the density curve towards higher values with increasing amount of misorientation and is consistent with other reported studies on the effect of deformation on twin boundary formation [19,25]. However, it should be noted that although a higher degree of misorientation did result in higher densities of twin boundaries in RR1000, deformation parameters that led to superplastic flow behavior were found to result in a considerable decrease in the maximum twin density during sub-sequent annealing. This phenomenon can likely be attributed to grain boundary sliding, rotation and grain growth mechanisms that occur during deformation that annihilate the existing twin boundaries and do not provide enough strain energy for the creation of new twin boundaries during subsequent annealing. As a result, the value of D_0 in Eq. (1) was

modified so that the slope can be adjusted. Based on the previous observations, D_0 can be a function of the grain size following compression, prior to annealing, and therefore be replaced by $k_d D_{ascp}$, where k_d is a dimensionless material constant and D_{ascp} is the grain size in the as-compressed condition. The resulting twin density equation then becomes Eq. (4):

$$p = k \frac{(1 + k_1 E_s)}{D} \ln \left(\frac{D}{k_2 D_{ascp}} \right) \quad (4)$$

Eq. (4) was plotted in Fig. 5a with the corresponding values of stored energy of 0.021, 0.112, and $0.597 \times 10^5 \text{ Jm}^{-3}$ measured from the EBSD data, Fig. 6, and $D_{ascp} = 6.0, 3.2,$ and $2.8 \text{ }\mu\text{m}$ for samples deformed to 50% strain at 1100, 1060, and 1020°C, respectively, and $k = 0.65 \text{ m}^2\text{J}^{-1}$ as calculated from Eq. (1). Using $k_1 = 0.30$ and $k_2 = 0.27$, the modified model for predicting twin density as a function of hot deformation parameters was found to be in good agreement with the experimental data. However, some discrepancies were observed when comparing the model to the samples deformed at 1100°C where superplastic flow mechanisms are dominant and only a limited degree of intragranular misorientation was present. Following the same reasoning as for the twin density, Eq. (2) was also modified accordingly to account for the effects of hot deformation:

$$F_{\Sigma 3} = F^* (1 + C_1 E_s) \left\{ 1 - \exp \left(- \frac{D}{C_2 D_{ascp}} \right) \right\} \quad (5)$$

where F^* , C_1 and C_2 are dimensionless material constants equal to 49%, 0.17 and 1.49, respectively. Using the same values of E_s and D_{ascp} as for the twin density, a comparison of the model and the experimental results is shown in Fig. 5b. Again, the changes in the twin length fraction resulting from the varying deformation temperature are effectively captured.

4. Conclusion

In conclusion, considerable changes in twin density and length fraction were observed as a function of the grain size in powder processed Ni-base superalloy RR1000 when hot deformation parameters were varied. Deformation conditions promoting superplastic flow resulted in a minimal twin density and length fraction upon annealing, whereas parameters resulting in the storage of strain energy in the form of intragranular misorientations were found to increase both the density and length fraction of twin boundaries for a given grain size. Within the range of deformation parameters studied in this investigation, Pande and Gleiter's model for predicting twin density and length fraction as a function of grain size were effectively modified to account for the effects of hot deformation.

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Figure captions

Fig. 1. Experimental results of twin density in RR1000 for the samples annealed at 1115°C, 1140°C, and 1150°C as a function of the average grain diameter with comparison to Pande et al.'s model for a critical grain size equal to 0.50 μm .

Fig. 2. EBSD grain boundary maps showing the random boundaries in black and twin boundaries in red for RR1000 after various heat treatments ordered according to increasing grain size. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 3. Experimental results of twin length fraction in RR1000 for the samples annealed at 1115°C, 1140°C, and 1150°C as a function of the average grain diameter with fitting curve using Eq. (2).

Fig. 4. Experimental results of length of random and $\Sigma 3$ boundaries from the EBSD grain boundary maps at constant magnification and step size for the heat treated samples of RR1000.

Fig. 5. Twin density (a) and length fraction (b) as a function of the average grain diameter for the samples deformed at 1020°C, 1060°C, and 1100°C to final strains of 0.2 and 0.5 and strain rate of 0.05/s after sub-solvus and super-solvus annealing as well as 1020°C 0.2 0.01/s after sub-solvus and supersolvus annealing at different times with comparison to the curves of Fig. 1 and 3 and the model of Eqs. (4) and (5) for samples having received uniaxial compression at high temperature.

Fig. 6. Local misorientations with relative misorientation maps and grain boundaries maps ($>15^\circ$ black, $>10^\circ$ blue, $>5^\circ$ green, $>2.5^\circ$ red) for the samples deformed to a final strain of 0.5, strain rate of 0.05/s, and temperatures of (a) 1020°C, (b) 1060°C, and (c) 1100°C. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

(a)

(b)

(c)