Quantitative assessment of strain and heat treatment on twin formation in commercially pure nickel

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Abstract

Thermomechanical treatment comprising cold working to 6% strain followed by annealing at temperatures in the range 700 ^{o}C to 1000 ^{o}C greatly increases the fraction of special boundaries, primarily $\Sigma 3$ type. The accompanying generation of annealing twins is analysed using the accident growth model due to Gleiter and the phenomenological model of Pande et al. It is shown that Gleiter's model, which contains no scaling factors, when used correctly, predicts the twin density in Cu alloys and pure nickel. The model due to Pande et al also predicts the twin density in Ni but this model incorporates two scaling factors that detract from its generality.
Keywords: special grain boundaries, annealing twins, copper alloys, nickel.

1   Introduction

It has been long accepted that thermomechanical treatments can alter the size, shape and crystallographic orientation of grains. In 1984, Watanabe [1] proposed that the type of grain boundaries present could also be controlled sand thus the concept of "grain boundary engineering" was initiated. Grain boundary engineering has been used to increase the fraction of "special grain boundaries", usually defined in terms of coincidence lattice site (CSL) boundaries. A high fraction of low Σ CSL boundaries $\{\Sigma \ (\leq 29)\}$ has been shown to improve resistance to intergranular crack propagation in Inconel 600 [2, 3] and to substantially improve the elevated temperature crack growth resistance of a nickel-based superalloy [4]. For type 304 stainless steel and alloy 600, high

Σ CSL boundary fractions resulted in reduced crack growth rates. High special grain boundary fractions also resulted in a significant reduction of intergranular stress corrosion cracking in irradiated type 304 stainless steel [5]. It has been clearly demonstrated that high fractions for Σ CSL special boundaries can substantially improve the resistance to creep, fatigue, and environmental degradation of engineering alloys.

The purpose of the present investigation was to increase the fraction of ΣCSL special boundaries in commercially pure nickel by employing an appropriate thermomechanical treatment. Following treatment, the fraction of special boundaries was determined using orientation imaging microscopy and the annealing twin density was also determined. The experimental values for annealing twin density are compared to the theoretical models of Gleiter [6] and Pande et al [7].

2   Experimental

The material used for this investigation was commercial purity nickel sheet, 3mm thick, mill annealed at 750°C. Work completed by Guyot et al [8] indicated that cold rolling 6% reduction in area would be a good starting point for the investigation. Therefore, strips of the as-received sheet were cold rolled 6% reduction in area and annealed for various times at temperatures from 500°C to 1000°C to obtain a range of grain sizes. Following the anneals, the fraction of special grain boundaries was determined using orientation imaging software EDAX/TSL, version 3.5, on a JEOL 5900 SEM equipped with an ultra thin, Oxford EDS window. Grain sizes and annealing twin densities were determined using the linear intercept method on a Zeiss microscope equipped with a Clemex Vision Image Analysis System. The results from at least 600 intercepts were averaged to obtain the values.

3   Results and discussion

The fraction of special boundaries after ten minutes at the annealing temperature is shown in fig 1. Over 80% of the special boundaries were of the Σ3 type with small fractions of Σ9, Σ27 and other types. The effect of annealing time at 700°C on the fraction of special boundaries is given in fig 2 which shows that the optimum annealing time at 700°C is 2.2 X 10^5 s or about 61 hours (220,000 s). The maximum 75% of special boundaries obtained by annealing at 700°C for 61 hours is the same as the maximum percentage of 75% obtained after annealing at 900°C for 10 minutes (600s). If the formation of special boundaries is diffusion controlled, then the product of Dt, where D is the diffusion coefficient and t is the annealing time, should be similar for the optimum times at the various annealing temperatures. However, the formation of special boundaries could be controlled by either grain boundary diffusion or lattice diffusion so both cases must be considered. For the grain boundary diffusion coefficients, we use the results of Čermák [9] who obtained $D_{gb} = 5.1$ X 10^{-15} exp $(-120,000/RT)$ m^2/s where R = 8.3145 J·K^{-1}· mole^{-1} and T is the absolute

temperature. (Actually, Čermák's results were primarily for the grain boundary diffusion of Co in Ni, but he obtained a few results for the grain boundary diffusion of Ni in Ni that were essentially identical to the values for the diffusion of Co in Ni. Since Co and Ni atoms are very similar, Čermák concluded that the grain boundary diffusion of Ni in Ni was similar to that for the grain boundary diffusion of Co in Ni. Further, Čermák's results agree well with the earlier results of Wazzan [10].)
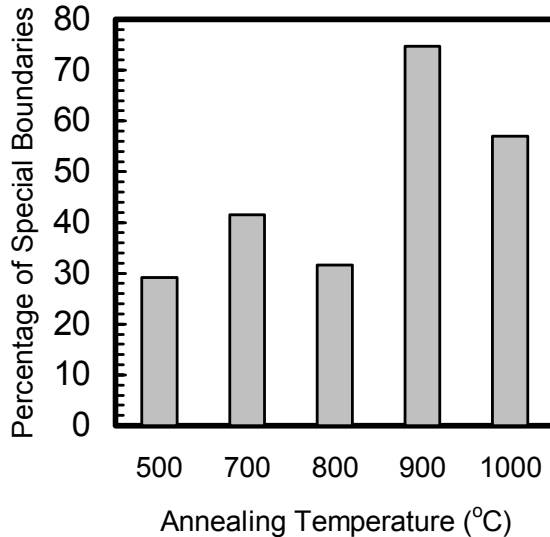
Figure 1:   Percentage of special boundaries after cold rolling 6% reduction in area and annealing for 10 minutes.
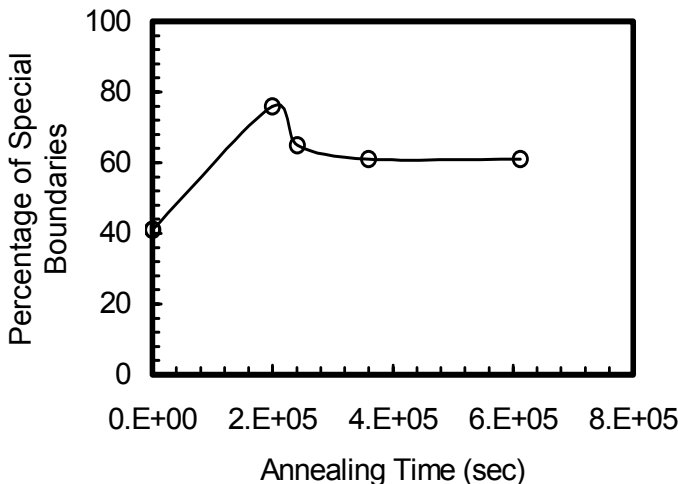
Figure 2:   Percentage of special boundaries after cold rolling 6% reduction in area and annealing at 700°C.

For 700°C, the grain boundary diffusion coefficient is 1.84×10^{-21} m²/s and for an annealing time of 220,000 s, the product $Dt = 4.1 \times 10^{-16}$ m². For 900°C, the grain boundary diffusion coefficient is 2.31×10^{-20} m²/s and for an annealing time of 600s, the product $Dt = 1.4 \times 10^{-17}$ m². For grain boundary diffusion, the Dt values for the two temperatures differ by a factor of about 40.

The lattice diffusion coefficient for the diffusion of Ni in Ni is given by $D_{latt} = 1.82 \times 10^{-4} \exp(-285,000/RT)$ m²/s [11]. For 700°C, the lattice diffusion coefficient is 9.13×10^{-20} m²/s and for an annealing time of 220,000 s, the product $Dt = 2.0 \times 10^{-14}$ m². For 900°C, the lattice diffusion coefficient is 3.71×10^{-17} m²/s and for an annealing time of 600s, the product $Dt = 2.2 \times 10^{-14}$ m². For lattice diffusion, the Dt values for the two temperatures are almost identical. Therefore, it appears that the formation of special grain boundaries in commercially pure Ni is controlled by lattice diffusion.

The annealing twin densities for annealing temperatures of 700°C, 800°C, and 1000°C are plotted versus grain size in figs 3-5 respectively. Two models were used to calculate the theoretical values for the twin density. The first is due to Gleiter [6] who proposed an atomistic model for the formation of annealing twins. Gleiter proposed that the probability, p, of a {111} plane being a coherent twin plane is given by:

$$p = \exp\left\{\sigma_z \left(Q - kT\ln\frac{\Delta G^0}{kT}\right) \middle/ \left[kT\sigma_z - \frac{\pi kT\varepsilon^2 h^2}{\left(Q - kT\ln\frac{\Delta G^0}{kT}\right)}\right]\right\}. \tag{1}$$

In eqn (1), σ_z is the surface energy of a coherent twin boundary, Q is the activation energy for grain boundary migration, ΔG^0 is the difference in the Gibbs' free energy between the growing and the shrinking grain, h is the step height formed by the twin nucleus (taken as the distance between {111} planes), ε is the surface energy of a step of height h, and k and T have their usual meanings. The twin density is simply calculated from the product of the number of {111} planes per unit length and the probability that any {111} is a coherent twin plane. Gleiter, noting that kT << Q, deleted the entire term $kT\ln(\Delta G^0/kT)$ from the denominator of eqn (1) (but not the numerator) and obtained a simplified equation. The use of Gleiter's simplified equation usually results in a temperature dependence as well as a grain size dependence for the twin density. Several investigators [7, 12–15] have obtained experimental results (or have proposed) that the twin density is essentially independent of temperature and have therefore considered Gleiter's theory as incorrect. However, Li et al [15] showed that the entire term $\{kT\ln(\Delta G^0/kT)\}$ is in fact not negligible and should not be removed from the denominator of eqn (1). Li et al also showed that when Gleiter's complete equation is used, the twinning probability in Cu-3 wt% Al alloy with a grain size of 300 μm is essentially independent of temperature as shown in fig 6. This is in agreement with the observations of Meyers and McCowan [13] as well as Pande et al [14] who suggested that the twinning probability for 300 μm grain size Cu-3 wt% Al alloy is independent of the annealing temperature with a constant value of $p \approx 2.75 \times 10^{-5}$ (fig 6).
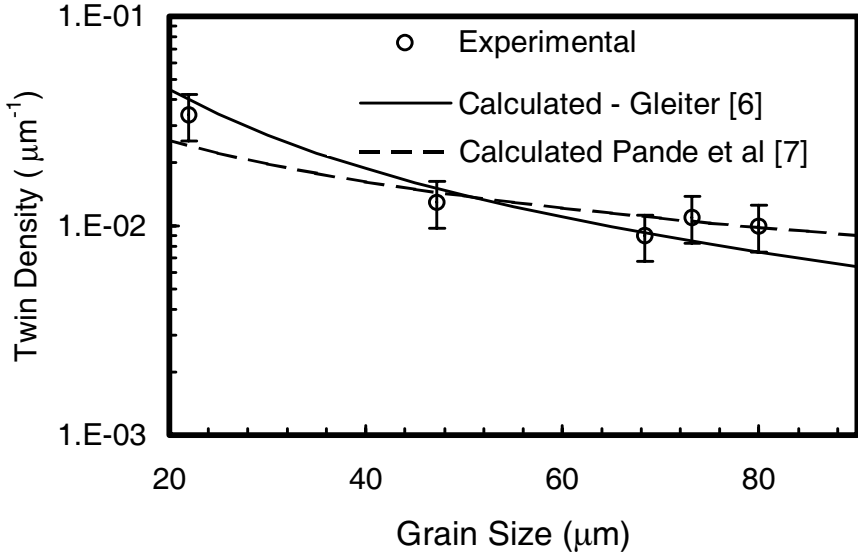
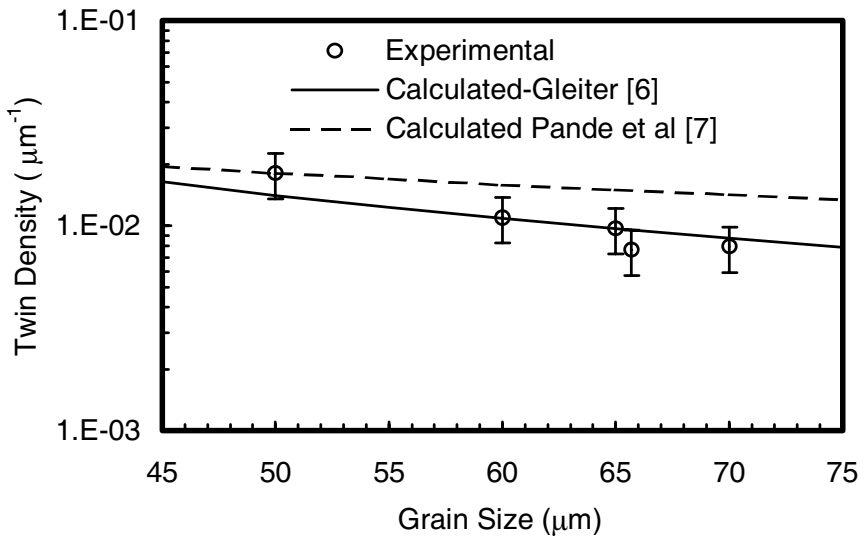Figure 3: Twinning density for nickel annealed at 700°C. (δ = 1.04 J/m^2, σ_z = 0.0367 J/m^2).

Figure 4: Twinning density for nickel annealed at 800°C. (δ = 0.994 J/m^2, σ_z = 0.0333 J/m^2).
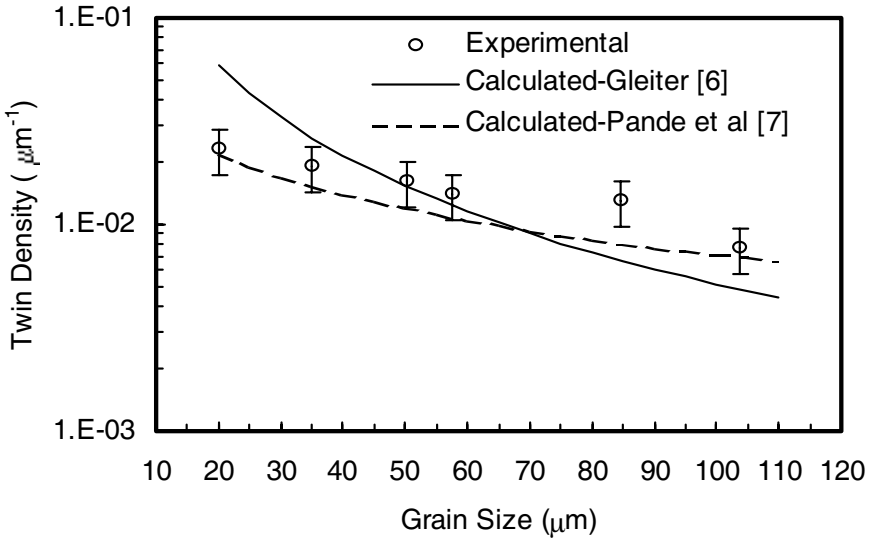
Figure 5:   Twinning density for nickel annealed at 1000°C. ($\delta = 0.899$ J/m^2, $\sigma_z = 0.026$ J/m^2).
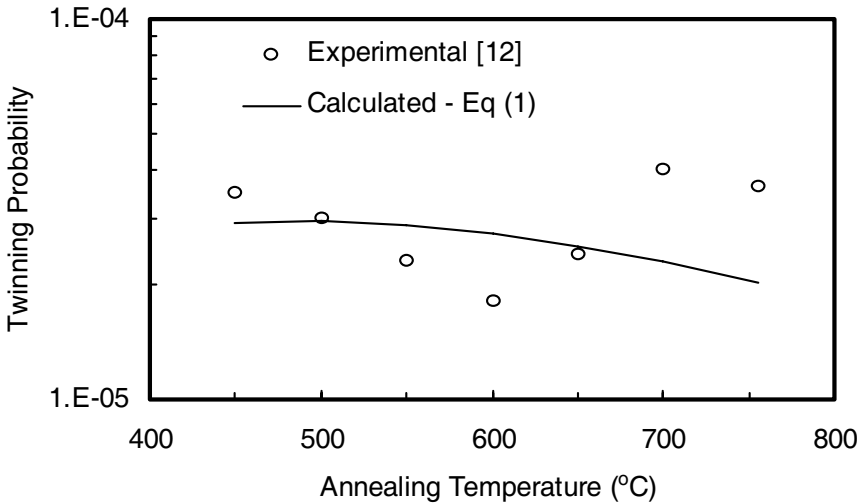

Figure 6:   Comparison of experimental values of twinning probability with values calculated from Gleiter's theory, eqn (1), for a grain size d = 300 μm. (σ_z=0.01037 J/m^2, Q=1.9 X 10^{-19} J/atom, δ=0.6 J/m^2, h=2.097 X 10^{-10} m, $\varepsilon = \delta/2 = 0.3$ J/m^2, $\Delta G^0 = 4\delta/d = 8$ X 10^3 J/m^3 = 9.58 X 10^{-26} J/atom).

Assuming that the annealing twin density is independent of annealing temperature, Pande et al [7, 14] developed a phenomenological equation giving the probability of a {111} plane being a twinning plane as:

$$p = \frac{k\delta}{d} \ell n\left(\frac{d}{d_0}\right) \tag{2}$$

where δ is the grain boundary energy, d is the grain diameter, d_0 is a material constant representing the smallest grain size for which twinning can occur, and k is a scaling factor also dependent upon the material.
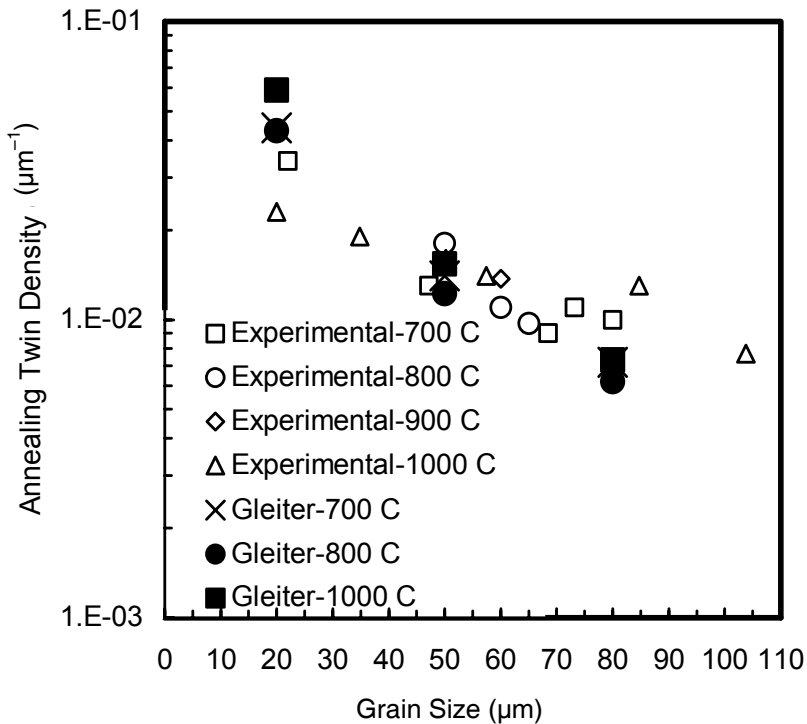

Figure 7: Experimental results for the twin density compared to values calculated from eqn (1) for different grain sizes and annealing temperatures.

The experimental values for the annealing twin densities in commercially pure Ni are compared with the values calculated from both eqn (1) and eqn (2) in figs 3 to 5. The values used for the variables in eqn (1) were Q = 120,000 J/mole = 2.0 X 10^{-19} J/atom and h = 2.0344 X 10^{-10} m. The value of the step energy, ε, is taken as $\varepsilon = \delta/2$ [6] where δ is the grain boundary energy which varies with temperature and is taken from data given by Murr [16]. The value of ΔG^0 is

taken as $\Delta G^0 = 4\delta/d$ [6] and converted to the appropriate units using the atomic weight and density of pure Ni. Following Gleiter [6], the surface energy of a coherent twin boundary, σ_z, was used as an adjustable parameter to obtain a good fit with the experimental data. The values of twin density calculated from the Gleiter model, eqn (1), are given by the solid lines in figs 3-5.

Pande et al [14] suggested that the constant d_0 in eqn (2) should be about 1 μm for Ni. Choosing values of $d_0 = 1.5$ μm and $k = 1.9$ X 10^{-11} m^3/J results in reasonable agreement with the experimental values as shown by the dashed lines in figs 3-5. However, the formulation due to Gleiter is more general in that it uses only one adjustable parameter, σ_z, and this is approximately 0.03 J/m for the temperature range 700°C - 1000°C. The value of $\sigma_z = 0.03$ seems reasonable in comparison to the values of $\sigma_z = 0.01$ for Cu - 3 wt% Al and $\sigma_z = 0.02$ J/m^2 for pure Cu [15]. Further, use of the Gleiter formulation, eqn (1), results in calculated values for the twin density that are essentially independent of temperature even though the formulation explicitly contains the temperature. This is illustrated in fig 7 where the calculated twin density is essentially independent of temperature and agrees well with experimental values.

4 Conclusions

1. The percentage of special grain boundaries in commercially pure Ni can be increased from about 30% in the as received material to about 75% in material cold rolled 6% reduction in area and annealed at temperatures from 700°C to 1000°C.

2. The special boundaries are primarily Σ3 type.

3. The formation of special grain boundaries in commercially pure Ni appears to be controlled by lattice diffusion.

4, The annealing twin density in commercially pure Ni is essentially independent of temperature for annealing temperatures from 700°C to 1000°C.

5. Values for the annealing twin density calculated from the Gleiter model and including all terms in the equation, agree well with experimental values.

6. The phenomenological equation due to Pande et al [7, 14], eqn (2), results in calculated values for annealing twin densities that agree well with experimental values using values of $d_0 = 1.5$ μm and $k = 1.9$ X 10^{-11} m^3/J.

Acknowledgements

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