



Effect of strain rate on microstructure evolution of a nickel-based superalloy during hot deformation



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ABSTRACT

The hot deformation behavior of a nickel-based superalloy was investigated by means of isothermal compression tests in the strain rate range of 0.001–10 s⁻¹ at 1110 °C. Transmission electron microscope (TEM) and electron backscatter diffraction (EBSD) technique were used to study the effect of strain rate on the microstructure evolution of the alloy during hot deformation. The results revealed that the dynamic recrystallization (DRX) process was stimulated at high strain rates ($\dot{\epsilon} \geq 5$ s⁻¹) due to the high dislocation density and adiabatic temperature rise. Meanwhile, high nucleation of DRX and low grain growth led to the fine DRX grains. In the strain rate range of 0.001–1 s⁻¹, the volume fraction of DRX grains increased with the decreasing strain rate, and the grain growth gradually governed the DRX process. Moreover, the strain rate has an important effect on DDRX and CDRX during hot deformation. On the other hand, particular attention was also paid to the evolution of twin boundaries during hot deformation. It was found that there was a lower fraction of $\Sigma 3$ boundaries at the intermediate strain rate of 1 s⁻¹, while the fractions of $\Sigma 3$ boundaries were much higher at both the lower strain rates ($\dot{\epsilon} \leq 0.1$ s⁻¹) and higher strain rates ($\dot{\epsilon} \geq 5$ s⁻¹).

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1. Introduction

Nickel-based superalloys have been widely used in the aeronautical, chemical, nuclear, petrochemical, and marine applications due to their excellent mechanical, physical and anticorrosion properties [1–5]. However, the flow behaviors of these alloys are very complex during hot deformation, and control of microstructure is of great importance for optimizing the final properties of products [6–9]. In these alloys, work hardening, dynamic recovery (DRV) and dynamic recrystallization (DRX) often occur during hot deformation, resulting in the complex microstructure evolution [2,10]. Among them, DRX can reduce deformation resistance and lead to significant grain refinement, and it is beneficial for the mechanical properties and formability of the materials [11,12]. Moreover, there are two main nucleation mechanisms of DRX in the nickel-based superalloys, i.e. discontinuous DRX (DDRX) and continuous DRX (CDRX) [13,14], which can occur simultaneously [15]. The occurrence of DDRX is closely related to the stored energy within materials [16], which is mainly in form of the dislocations generated from plastic deformation [17].

Meanwhile, the occurrence of CDRX is also closely related to the dislocation density in materials [18,19].

In addition, strain rate seems to have a complex effect on the DRX process. For superalloy 718, it was reported that the DRX process was accelerated in the low strain rate domain ($\dot{\epsilon} < 1$ s⁻¹) [20]. The acceleration of DRX process was also observed in the high strain rate domain ($\dot{\epsilon} = 10$ s⁻¹) for superalloy 625 [15]. Recently, Mandal et al. [16] observed that the DRX process was accelerated in both the high and low strain rate domains but became sluggish at the intermediated strain rate domain in a nitrogen-enhanced 316L(N) austenitic stainless steel. Therefore, systematical investigations on the evolution of DRX microstructure in the superalloys under different strain rates are quite necessary.

The properties of nickel-based superalloys are largely controlled by their grain boundaries (GBs), which can be characterized by coincidence site lattice (CSL) model [21]. The beneficial boundaries, so-called special boundaries, are usually based on $\Sigma 3$ twin boundaries and other related low- Σ CSL boundaries [22]. As a kind of special boundaries, the $\Sigma 3$ twin boundaries can enhance the high temperature mechanical properties of nickel-based alloys [23–25], and they can also play an important role in obtaining desired properties through grain boundary engineering [26]. In addition, it has been reported that the primary twins ($\Sigma 3$) and

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higher order twins ($\Sigma 9$ and $\Sigma 27$) can accelerate the DRX process for materials with low stacking fault energy [27,28]. However, few researches have been carried out on the variation of $\Sigma 3^n$ ($n = 1, 2, 3$) boundaries with strain rate during hot deformation for the nickel-based superalloys. Therefore, further researches are necessary.

This study was aimed to investigate the effect of strain rate on the microstructure evolution and nucleation mechanisms of DRX for a nickel-based superalloy during hot deformation. Analyses of grain boundary misorientations were systematically carried out to investigate the nucleation as well as the progress of DRX under various strain rates. Moreover, particular attention was paid to the evolution of twin boundaries during hot deformation.

2. Experimental procedures

The chemical composition (wt.%) of the alloy used in this investigation is listed in Table 1. The nickel-based superalloy is a kind of γ' -hardened superalloy with low stacking fault energy, which has been widely used in gas turbines, combustion systems and other high-temperature applications due to its excellent high-temperature strength, creep resistance and corrosion resistance [2]. Particularly, the highest service temperature of the alloy can reach about 1000 °C. In the alloy, the elements of Ti and Al can make a great contribution to form γ' phase (Ni_3AlTi), which is the main strengthening phase. The content of γ' precipitates (L1₂ structure) in the alloy is about 20 wt.%. Meanwhile, the elements of W, Mo and Co are used as solution strengthening elements, and the elements of B, Ce and Mg are used as grain boundary strengthening elements. On the other hand, the density of the alloy is 8.47 g/cm³. The dissolution temperature of the alloy is in the temperature range of 1345–1390 °C [29].

Cylindrical specimens of 8 mm in diameter and 12 mm in height were machined from a cylindrical forging bar of the alloy. Before compression tests, the specimens were solution treated at 1100 °C for 30 min, and then air-cooled to room temperature. The microstructure of the alloy after solution treatment is shown in Fig. 1. Fig. 1(a) shows the inverse pole figure, which is composed of equiaxed grains and a few annealing twins. The orientation imaging microscopy (OIM) map is shown in Fig. 1(b), in which $\Sigma 3$, $\Sigma 9$ and $\Sigma 27$ boundaries are represented by red lines, green lines and blue lines, respectively. It should be noted that the fraction of $\Sigma 3$ boundaries is about 51% in the specimen after solution treatment. The misorientation angle distribution is described in Fig. 1(c). It can be found that most of the misorientation angle is larger than 15°, indicating that majority of grain boundaries are high angle grain boundaries (HAGBs). In other words, the fraction of low angle grain boundaries (LAGBs) with the misorientation angle lower than 10° is relatively low. Furthermore, the grain size distribution is shown in Fig. 1(d). The average grain size of the alloy after solution treatment is about 96.5 μm.

Compression tests were carried out on a Gleeble-1500D testing system in the strain rate range of 0.001–10 s⁻¹ at 1110 °C. All the tests were performed with a heating rate of 10 °C/s. Before deformation, the specimens were preserved for 3 min at the testing temperature, which was controlled to ±2 °C. During compression tests, the load-stroke curves were automatically converted into true stress–true strain curves using standard equations. After compression tests, the specimens were quenched to room temperature

immediately by water with minimum delay. The deformed specimens were cut along the compression axis for microstructure observation. The foils for TEM examination were prepared by hand grinding to a thickness of 70 μm. After that, these foils were thinned by the method of twin-jet electropolishing with a solution of 10% perchloric acid in ethanol. TEM examination was conducted with Tecnai G2 F30 microscope operated at 300 kV. The EBSD measurements and analyses were performed by using TSL system (OIM™ 5 software) attached to a FEI Quanta 200FEG scanning electron microscope. The samples for EBSD investigation were electropolished with a solution of 10% perchloric acid in ethanol at 20–30 V for 30 s at room temperature. EBSD maps were obtained with a step size of 0.25–1 μm depending on the grain size.

3. Results and discussion

3.1. Flow behavior

Fig. 2 shows the true stress–true strain curves of the alloy deformed a 0.7 true strain at 1110 °C with different strain rates. Obviously, there is an obvious work-hardening stage at the strain less than 0.2, which is mainly caused by the increase of dislocation density and the formation of poorly developed subgrain boundaries [30]. With the increasing strain, the effect of work hardening can be partially neutralized by the occurrence of dynamic softening mechanisms, leading to the decrease of flow stress. In general, the hot deformation is a competing process between the work hardening and dynamic softening. As $\dot{\varepsilon} \leq 1 \text{ s}^{-1}$, a steady-state flow stress can be reached when the work hardening and dynamic softening mechanisms reached a dynamic equilibrium. At the strain rates of 5 s⁻¹ and 10 s⁻¹, the curves exhibit continuous flow softening. Similar results were also reported in Alloy Haynes 230 [31] and alloy 800H [32], which were caused by the adiabatic heating at high strain rate. Generally, such flow behaviors are consistent with the previous results for nickel-based superalloys with low stacking fault energy, indicating the occurrence of DRX during hot deformation [33,34].

In addition, the amount of adiabatic temperature rise could be estimated by using the following equation [35]:

$$\Delta T = \frac{0.95\eta}{\rho C_p} \int_0^\varepsilon \sigma d\varepsilon \quad (1)$$

where ρ is the density (8.47 g/cm³), C_p is the specific heat (above 1000 °C, C_p is 0.812 J/(g K) for the alloy), σ is the flow stress, ε is the true strain, and η is the thermal efficiency, which can be calculated by using the following equations [36]:

$$\eta = \begin{cases} 0 & \dot{\varepsilon} \leq 10^{-3} \text{ s}^{-1} \\ 0.316 \log \dot{\varepsilon} + 0.95 & 10^{-3} \text{ s}^{-1} < \dot{\varepsilon} < 1 \text{ s}^{-1} \\ 0.95 & \dot{\varepsilon} \geq 1 \text{ s}^{-1} \end{cases} \quad (2)$$

Fig. 3 shows the adiabatic temperature rise (ΔT) in the alloy deformed a 0.7 true strain at 1110 °C with various strain rates. The values of ΔT can reach above 20 K at the strain rates higher than 1 s⁻¹, while the values of ΔT are below 10 K at the strain rates lower than 0.1 s⁻¹. Therefore, the adiabatic temperature rise was much more significant in the alloy deformed at high strain rate.

It is well known that the temperature rise during hot deformation can not only simulate the process of DRX, but also lead to a rapid reduction in the rate of work hardening [29,37]. At the strain rates of 5 s⁻¹ and 10 s⁻¹, a dynamic balance between the work hardening and dynamic softening was not reached during hot deformation, and DRX became dominant due to the effect of adiabatic heating. In other words, the significant adiabatic temperature rise led to the intensive DRX behavior of the alloy at high strain rates, which can be confirmed by the further analysis of

Table 1
Chemical composition of the alloy (wt.%).

Element	C	Cr	Fe	W	Mo	Al	Co	Ti
wt.%	0.045	18.30	0.24	5.90	4.02	2.19	6.40	1.16
Element	Si	B	Mg	Ce	Mn	P	S	Ni
wt.%	0.05	0.003	≤0.01	≤0.02	0.02	0.007	0.002	Bal.

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