



Oriental dependence of microstructure evolution and the related deformation mechanism of a single crystal Ni-base superalloy

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ABSTRACT

The orientational dependence of microstructure evolution and the relevant deformation mechanism of a single crystal superalloy were investigated. It showed that the annealing microstructures of below the γ' solvus were strongly associated with the deformation distribution between the γ' phase and the matrix. Several influence factors concerning the anisotropic deformation distribution were discussed in $\langle 001 \rangle$ and $\langle 111 \rangle$ orientations in this paper. It was concluded that orientations which were favorable to dislocation development in the matrix channels and suppressed the occurrence of particle shearing would enhance the driving force for recrystallization or rafting at high temperatures.

1. Introduction

Single crystal nickel based superalloys are the most reliable materials in use for gas turbine industry. Grain boundaries are eliminated during the evolution from equiaxed polycrystal to single crystal. Elements required for grain boundary strengthening, such as C, Zr and B, are also removed. It increases the melting point as well as the temperature of solution treatment of the alloys, however, recrystallization will become extremely detrimental to their mechanical performance [1,2]. During the solidification, manufacturing and service, plastic deformation will be inevitably introduced, which provides driving force for microstructure evolution. The influence of original microstructures and heat treatment parameters has been well studied [3–5]. However, orientational dependence of microstructure evolution has been rarely reported because most studied focused on the $\langle 001 \rangle$ orientation. Okazaki et al. observed the nucleation anisotropy of recrystallized grains on the indented surfaces with different crystallographic orientations. They argued that the active slip systems played an intrinsic role for this anisotropy phenomenon [6]. Xie et al. investigated the detailed surface topography and distribution of slip bands on the indented (001) and (011) surfaces showing that the size and shape of the recrystallization region is in good accordance with the deformation zone [7]. Further investigation of the relationship between dislocation morphology and recrystallization behavior was not involved in their studies.

Although microstructure evolution of single crystal superalloys is influenced by their significant anisotropy in plastic deformation.

annealing temperature is even more critical. It has been proved previously that single crystal superalloys usually shows a strong tendency of recrystallization and forms normal recrystallized grains above the γ' solvus. Under the circumstances, driving force will generally be sufficient and annealing behaviors tend to be similar for different orientations. In comparison, situation becomes much more complex when annealing temperature is below the γ' solvus. This is commonly attributed to the pinning effect of γ' phase [8–10]. On this occasion, the driving force introduced by predeformation becomes crucial. As a result, deformation orientations will have a strong influence on the annealing microstructure.

This work focused on the anisotropic microstructure evolution below the γ' solvus and related deformation mechanism of a single crystal superalloy oriented in $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$ orientations. Several influence factors concerning the anisotropic deformation distribution were discussed in detail.

2. Experimental

Composition of the studied alloy is 4.8Cr, 9.5Co, 9.0W, 2.5Mo, 6.2Al, 1.2Nb, 8.5Ta, 2.4Re with minor B and Hf and balance Ni. Solution and aging treatment was carried out on the alloy as follows: 1290 °C/1 h+1300 °C/2 h+1315 °C/4 h+air cooling+1120 °C/4 h+air cooling+870 °C/32 h+air cooling. The γ' solvus of this alloy is about 1305 °C.

Tensile samples oriented in $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$ orientations had a gauge length of 18 mm and a cross section of

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5 mm–0.4 mm. They were tensioned to a plastic strain of about 4% at room temperature, and tubed in silica glass under argon atmosphere to avoid oxidation. The tensile strain was determined according to a previous study by Cox et al. [9], showing that the critical strain value at room temperature for occurrence of recrystallization below γ' solvus was around 3–4%. The tubed samples were annealed at 1100 °C for 4 h which was far below the γ' solvus. Subsequently, they were mechanically polished and etched in a 100 ml H₂O+100 ml HCl+25 g H₂SO₄ solution. The following microstructure observation was carried out under an Apollo-300 field-emission scanning electron microscope. Outer surfaces instead of cross sections were made for observation due to the limited thickness of the tensile samples. Another set of samples were deformed under the same parameters. They were used for the observation of dislocation structures under a JEM 2100f field-emission transmission electron microscope.

3. Results and discussion

3.1. Annealing microstructures

Annealing microstructure of the $\langle 001 \rangle$ oriented sample was shown in Fig. 1a and b. A typical γ/γ' raft structure had been developed during the heat treatment. The raft lamellas were basically arranged perpendicular to the tensile axis as shown in Fig. 1a. No trace of recrystallization was observed even in slip bands as shown in Fig. 1b.

It was a little surprising that microstructure of the $\langle 011 \rangle$ oriented sample kept almost unchanged after anneal as shown in Fig. 1c. The only exception was the edge of the tensile sample where γ' particles showed a slight tendency of coarsening and coalescence (Fig. 1d). In comparison, outer surface of the $\langle 111 \rangle$ oriented sample was covered with large recrystallization area as shown in Fig. 1e. Highly magnified observation of the area revealed that recrystallization was in fact concentrated near the surface and did not penetrate the sample, because original microstructure beneath the recrystallized layer was exposed as shown in Fig. 1f. Thickness of the recrystallized layer was estimated to be near 100 μm in consideration of the grinding and etching process.

3.2. Dislocation morphology after predeformation

The orientation dependence of annealing microstructures shown above should be mainly attributed to the diverse dislocation morphology introduced by predeformation. Free energy of a crystal will be raised by deformation with the introduction of dislocations, which makes the material thermodynamically unstable. When the deformed material is heated to elevated temperatures, thermally activated restoration process will occur. This process is strongly associated with the stored energy and annealing temperature. Normally, the recovery process involves just partially restoration and reaches a metastable state by annihilation and rearrangement of dislocations [11]. Further restoration proceeds by recrystallization or some other microstructure evolution.

However, most alloys consist of more than one phase. The situation then becomes much more complicated. Because the restoration process is not only influenced by the total stored energy introduced, but also the distribution of it [12,13]. For single crystal superalloys, the microstructure is normally comprised of γ matrix and large volume fraction γ' phase. The presence of the second phase has a remarkable influence on both the deformation structure and the microstructure evolution during anneal. The main aspects involved are concluded as follows: (1) The deformation distribution and the inhomogeneity of deformation caused by the second phase; (2) Deformation structure in the vicinity of the second phase; (3) The pinning effect of the second phase. The first two aspects are strongly orientational dependent for single crystal superalloys.

Dislocation morphology of the $\langle 001 \rangle$ deformed sample was

shown in Fig. 2a. It was observed that deformation was not homogeneously distributed. Dislocation density in the matrix channels parallel to the loading axis was obviously higher. It was associated with the distribution of stress components in matrix channels of different orientations. This issue had been investigated in detail by Arrell and Vallés [14]. They also declared that this inhomogeneity would gradually disappear with increasing strain. Besides deformation in the matrix, continuous shearing of the γ' particles also occurred. For the $\langle 011 \rangle$ orientation, dislocation density in the matrix channels decreased significantly in comparison with the $\langle 001 \rangle$ one. Plastic deformation was chiefly developed in the γ' phase as shown in Fig. 2b. However, the situation was completely opposite for the $\langle 111 \rangle$ one. Dislocations in the second phase were rarely seen. Although it was difficult to identify the accurate γ/γ' boundaries due to the significant overlap of the two phases and the interference of dislocations, it could be deduced that the dense dislocation networks observed in Fig. 2c and d were located inside the matrix channels according to previous observation of similar microstructure [16]. In addition, the intrinsic features of the γ' phase would not allow formation of such dislocation structure.

3.3. Related deformation mechanism and influence factors

3.3.1. The possibility of cross slip

It was important to illuminate origin of the anisotropic deformation structures shown above. As far as FCC structural single crystals were concerned, the anisotropic deformation was commonly attributed to the change of resolved shear stress in different slip systems. This would strongly affect the possibility of cross slip and the propagation of dislocations. In single crystal superalloys, the cubic shape of γ' phase divided the matrix into channels along the three $\{001\}$ interfaces. Plastic deformation was commonly initiated from the matrix and transferred into the γ' phase with further strain. A higher possibility of cross slip would facilitate dislocation propagation in the matrix. Fig. 3 was a sketch map illustrating the interact of matrix dislocation with the interface. This was associated with the potential active slip systems. According to the Schmid law, $\langle 001 \rangle$ orientation equally facilitated 8 octahedral slip systems in comparison with 6 ones for $\langle 111 \rangle$ and only 4 for $\langle 011 \rangle$. Early studies had proved that the probability of cross slip in $\langle 011 \rangle$ oriented FCC single crystals was very low and deformation always started with only one slip system, which would strongly hinder the propagation of dislocations in the matrix channels. This led to shearing of the γ' phase when strain in the matrix was still small [15]. Therefore, the stored energy in the matrix and the driving force for recrystallization was very low. The dense zig-zag dislocation structure in the $\langle 111 \rangle$ oriented sample then could be attributed to its higher possibility of cross slip. D. Bettge attributed it to the multiple cross slip between $(\bar{1}\bar{1}1)$ and $(1\bar{1}\bar{1})$ slip systems along the matrix channels. This mechanism was used to explain the macroscopic slip lines corresponding to cubic slip system in $\langle 111 \rangle$ oriented superalloys [16]. Their experiment was carried out at intermediate temperatures so that the zig-zag dislocations had a very high mobility and could move continuously along the matrix channels. In our study conducted at room temperature, it seemed its mobility was significantly decreased and its distribution usually became localized as shown in Fig. 2d. In fact, if the magnification became lower, we would find that the dislocation networks in Fig. 2c was also a localized area.

3.3.2. Relative orientation between the external stress and the matrix channels

Discrepancy emerged when comparing the $\langle 001 \rangle$ and $\langle 111 \rangle$ oriented samples. At the same level of plastic strain, particle shearing occurred only in the former one. The absence of recrystallization also indicated that stored energy in the matrix was lower for it. As mentioned before, $\langle 001 \rangle$ was of the highest possibility of multiple cross slip among the three orientations. Therefore, some other factors

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