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Temperature dependence of deformation mechanism in single crystal Ni-base superalloy①

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Abstract: The tensile behavior of a new single crystal Ni-base superalloy was studied at various temperatures. Specimens were strained to fracture in the temperature range from 20 ℃ to 1 000 ℃. $\sigma_{0.2}$ is essentially unaffected by temperatures between 20 ℃ and 400 ℃. At higher temperatures it increases until it reaches a maximum at about 800 ℃. Beyond 800 ℃ a sharp decrease of strength is observed. There is a slight fluctuation in ductility between 20 ℃ and 800 ℃. The elongation to fracture increases from 10% to 36% as the temperature increases from 800 ℃ to 1 000 ℃. The deformation is dominated by γ' shearing and the high-density dislocations are observed in matrix channels at low temperatures. The dislocation microstructure is inhomogeneous due to the formation of dislocation concentrations with high-density tangling at intermediate temperatures. The networks deposited at the γ/γ' interfaces prevent dislocations from entering the γ' precipitates at high temperatures.

Key words: single crystal; Ni-base superalloy; tensile; microstructure; dislocation
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1 INTRODUCTION

Although the main goal of developing single crystal Ni-base superalloy is to increase their creep strength[1,2], the tensile deformation behavior should be sufficiently addressed. In particular, a comparison between dislocation structures after fracture at different temperatures should be made. Most studies dealt with the microstructure prior to the tensile fracture[3-7]. The results show that there are fluctuations in strength and ductility with respect to temperature, such that three regions can be distinguished[8]. The Ni-base superalloys with rafted structure[9] exhibit marked drop in tensile ductility, depending on the strain rate, in moist environment at room temperature. The embrittlement behavior of these alloys is accompanied by fracture on {100} planes. However, it is necessary to study the microstructure after fracture to get a better understanding of the single crystal Ni-base superalloy. For the purpose of failure analysis and prevention, in this paper the authors presents a TEM characterization of the dislocation microstructure after tensile fracture. The elastic modulus and strain hardening exponent are also examined.

2 EXPERIMENTAL

A Co-Cr-W-Mo-Al-Ti-Ta-Ni system single crystal Ni-base superalloy of [001] orientation was produced by means of crystal-selector method in a directional solidification vacuum furnace under a high thermal gradient. The longitudinal orientations of all specimens were within 10° deviating from [001] orientation.

Tensile specimens, 5 mm in diameter and 25 mm in gauge length, were prepared. Tests were conducted at 20, 200, 400, 600, 700, 800, 900, and 1 000 ℃. The specimens were induction heated, and the temperature gradient no more than ±2 ℃ was maintained over the gauge length. The strain extensometer was attached to the gauge section of each specimen to measure the strain change during tensile test. All the specimens were tested at a constant cross head displacement rate of 0.5 mm/min (before yielding) and 2.5 mm/min (after yielding). The specimen lengths before and after fracture were measured to calculate the elongation to fracture (assuming that all the strain occurred in the gauge section).

The samples investigated on JSM-6301F SEM were ground, polished and etched in a chemical solution composed of 20 g $CuSO_4$, 100 mL hydrochloric acid and 80 mL H_2O. Disks for TEM samples were cut from the gauge length of the specimens deformed under different conditions normal to the loading axis. Thin foils were prepared by twin-jet electropolishing using an electrolyte of 7% per-

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chloric acid and 93% acetic acid at a temperature of – 25 ℃. Dislocation configurations were characterized by using a TECNAIG^2-20 TEM operated at 100 kV.

3 RESULTS AND DISCUSSION

3.1 Microstructure

The as-cast microstructure of specimen consists of a disordered face-centered cubic (fcc) matrix (γ), an ordered precipitate (γ') and γ-γ' eutectics as shown in Figs. 1(a) and (b). The primary γ' particles with average edge length of 2 μm are observed in as-cast samples. The γ-γ' eutectics are distributed at dendrite boundaries, often near micropores, showing that they are formed during the last stage of solidification. Table 1 lists the conditions of solution heat treatment and two-stage annealing heat treatment. The average secondary γ' cube is 0.6 μm in length produced during aging after partial solution heat treatment in Fig. 1(c). No "hyperfine" γ' is found. The initial dislocation density is extremely low in the heat-treated material. The total γ' volume fraction is determined to be more than 70%.

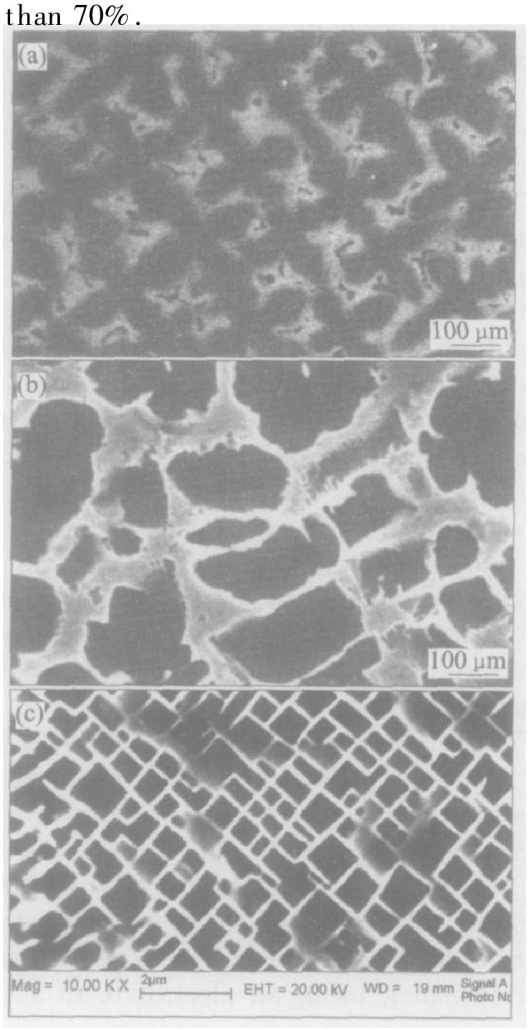

Fig. 1 Microstructures of specimens
(a), (b) —As-cast;
(c) —In dendritic area after full heat treatment

Table 1 Heat treatment schedule of alloy

Solution heat treatment	1 300 ℃ for 8 h/AC+ 1 305 ℃ for 8 h/AC+ 1 310 ℃ for 8 h/AC
Annealing heat treatment	1 080 ℃ for 8 h/AC+ 870 ℃ for 24 h/AC

AC: Air-cooling

3.2 Mechanical behavior

Fig. 2 shows a plot of the 0.2% offset yield strength ($\sigma_{0.2}$) and ductility (represented by elongation to fracture) as a function of temperature. $\sigma_{0.2}$ is essentially unaffected by temperatures between 20 ℃ and 400 ℃. At higher temperatures it increases until it reaches a maximum at about 800 ℃. Beyond 800 ℃ a sharp decrease of strength is observed. There are slight fluctuations in ductility between 20 ℃ and 800 ℃. The elongation to fracture increases from 10% to 36% as the temperature is increased from 800 ℃ to 1 000 ℃. The presence of the facets indicates a cleavage fracture mode below 800 ℃, while the fracture mode is microvoid coalescence mechanism at 1 000 ℃.

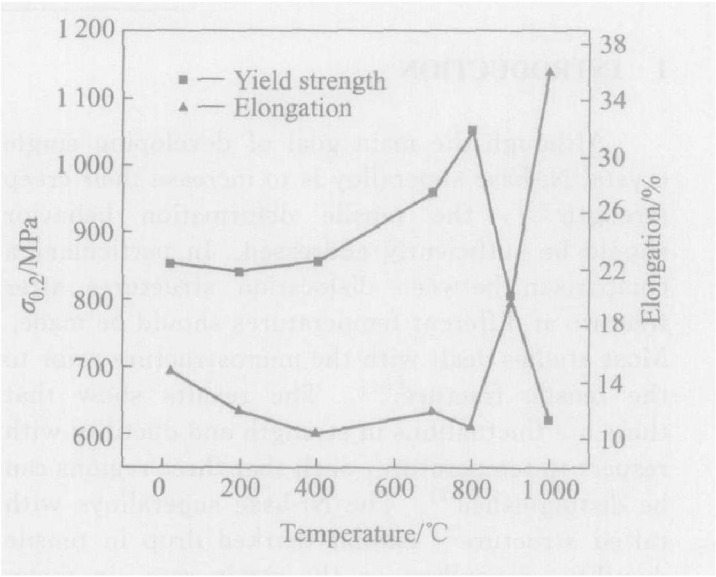

Fig. 2 Influence of temperature on yield strength and elongation

Fig. 3 illustrates the influence of temperature on strain-hardening exponent, n, and elastic modulus, E. It is evident that n value decreases as a whole with an increase in temperature. Nickel is a fcc material and is expected to have a high strain-hardening exponent. However, the present material has a very low strain-hardening exponent, which indicates that the stacking fault energy of this alloy must be high. At high temperature, the diffusion-controlled processes are expected; dislocations are more mobile by climb, so the strain-hardening exponent decreases with increasing temperature.

It is well known that the bonding force among atoms determines the elastic modulus, one of the intrinsic natures of materials. This bonding force

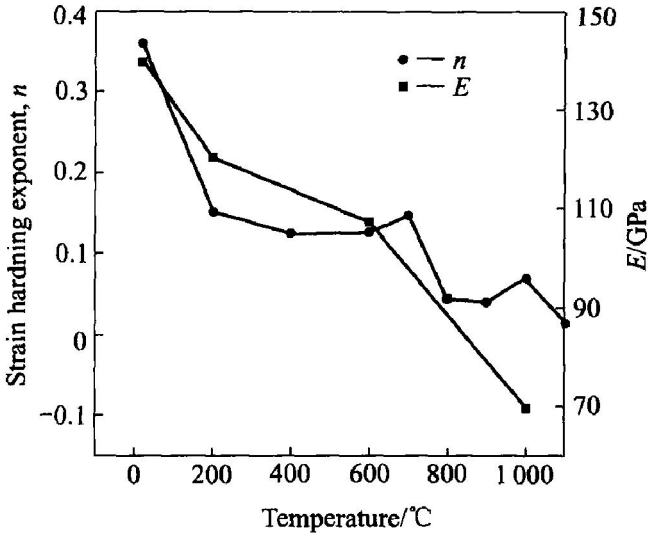

Fig. 3 Influence of temperature on strain hardening exponent and elastic modulus

is related not only to the crystal structure but also to the distances among atoms, and it can be affected by alloying addition, heat treatment and plastic deformation[10]. Since the same heat treatment and chemical composition are carried out in this study, the elastic modulus mainly depends on the plastic deformation temperature. Interestingly, this alloy maintains a high elastic modulus at high temperatures. The elastic modulus of a multiphase alloy is mainly determined by the modulus of its individual phase and by their volume fractions[11].

3.3 Deformation substructure

The superalloy is strengthened by two main strengthening mechanisms: solid solution hardening and precipitation hardening. Elements such as W, Mo, Co and Cr are the most potent solid solution strengtheners. Ni, Ti and Al are γ' formers and together with significant amounts of Mo and W strengthening the alloy through a precipitation hardening mechanism. Some factors such as: coherency strains at γ-γ' interface, elastic modulus difference between γ and γ' matrix, lattice mismatch, long range ordering of γ' and anti-phase boundary (APB), produced during the movement of dislocations through γ' particles, strengthen the Ni-base superalloy via the precipitation hardening mechanism[12].

The matrix is mainly strengthened by γ' precipitates in single crystal superalloy. Dislocation pairs interact strongly with these precipitates. The common attribute of microstructure deformed at room temperature is slip bands shearing both phases on the {111} primary glide plane. Outside these slip bands the microstructure is practically free of dislocations. As shown in Fig. 4(a), the high dislocation density in the matrix arises from parallel, paired dislocation loops that do not depend on the orientation of the matrix channels relative to the tensile axis. It can be proved that the dislocation loops are indeed pairs and not dipoles[13]. Small rugged and elongated stacking fault loops are observed on {111} planes in the γ' phase, which differ considerably from the extended super stacking faults (SSFs) created at the γ'/γ interfaces during high temperature deformation. Many hair-pin shaped dislocations arrays are shown in Fig. 4(b). At 20 ℃, the deformation pattern is inhomogeneous, the dislocation density is high in the matrix. But, instead of long straight dislocation lines, areas of short dislocation segments are observed. These short straight dislocation segments have been identified as screw type and parallel to ⟨110⟩ direction, attached by an edge segment. They may be produced by extension of matrix dislocation loops between the precipitates leaving extended screw segments along the γ-γ' interfaces. $\sigma_{0.2}$ is almost the same between 20 ℃ and 400 ℃ because of the similar dislocation substructure. The shearing of the γ' particles is obtained by two mechanisms[15]. Fig. 4(b) also illustrates the first mechanism, where straight dislocation segments are seen in γ' precipitates. The second mechanism, which is detected through the creation of stacking faults left by a/3⟨112⟩ superpartial γ' dislocations gliding on {111} planes in the precipitates. Almost every γ' particle contains stacking faults, as shown in Fig. 4(c). These dislocations can recombine with other matrix dislocations in different directions. Detailed analysis of the fault nature reveals that a unit matrix dislocation of a/2⟨110⟩ dissociates into a/3⟨112⟩ and a/6⟨112⟩ partials[16]. An extrinsic stacking fault is created in both γ and γ' phases presumably behind the leading partial of a/3⟨112⟩. At the same time, the movement of the trailing partial of a/6⟨112⟩ eliminates the stacking fault in the matrix but leaves a partial dislocation loop around the faulted precipitates[17]. A large amount of stacking faults are distributed in γ', thus leading to the observed high value of strength of about 1 050 MPa. Fig. 4(d) shows the microstructure developed at 1 000 ℃. The coarsening γ' precipitates are not cut by dislocations and the dislocation substructure is confined to the matrix. The interfacial dislocations coalesce to form a homogeneous, regular array. The networks are hexagonal in nature, and consist of primarily two types of dislocations: pure edge dislocations lying on {011} planes with ⟨100⟩ line directions, and mixed dislocations lying on {111} planes with ⟨110⟩ line directions. The networks deposited at the γ/γ' interfaces prevent dislocations from entering the γ' precipitates and this may explain why shearing of precipitates rarely occurs at 1 000 ℃.

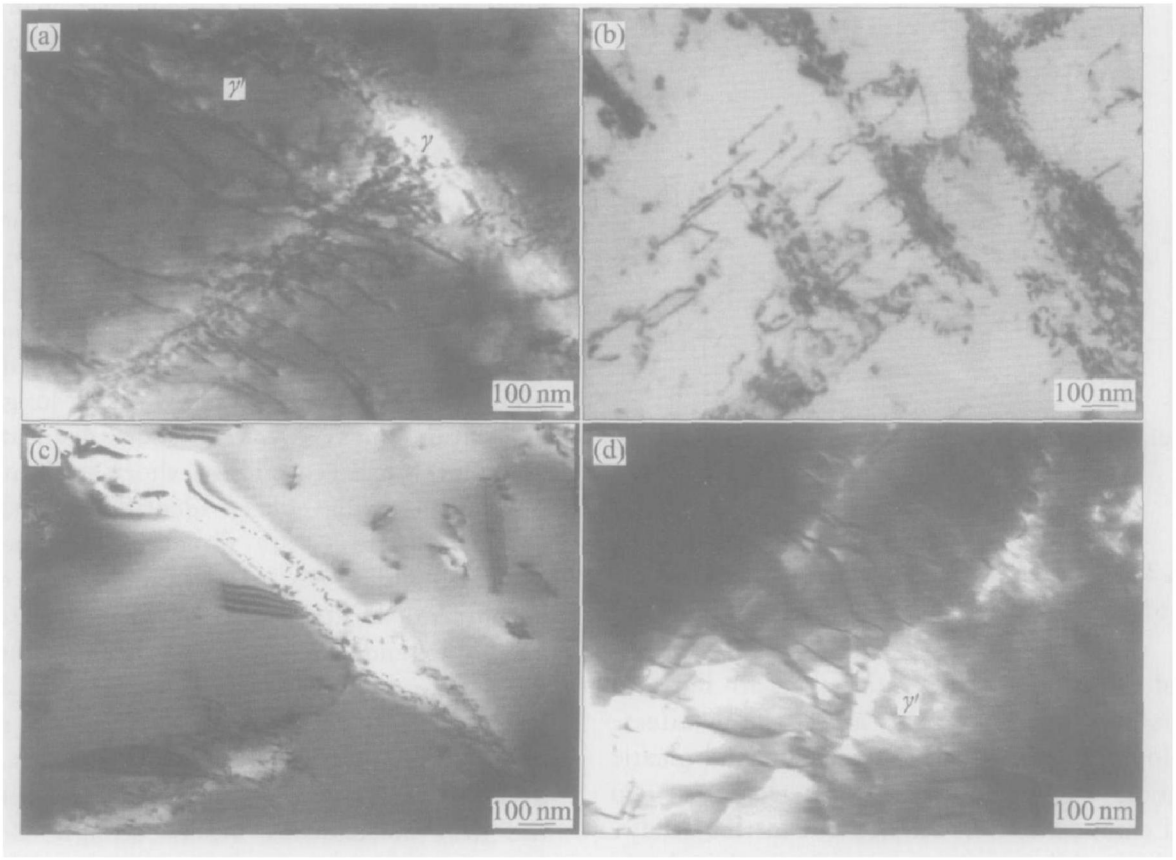

Fig. 4 Comparison of dislocation microstructures after yielding at different temperatures
(a) —20 ℃; (b) —400 ℃; (c) —800 ℃; (d) —1 000 ℃

Furthermore the hyperfine γ′ precipitates remain in the matrix. The coarsening of γ′ precipitate contributes to higher ductility and lower strength.

4 CONCLUSIONS

1) $\sigma_{0.2}$ is essentially unaffected by temperatures between 20 ℃ and 400 ℃. At higher temperatures it increases until it reaches a maximum at about 800 ℃. Beyond 800 ℃ a sharp decrease of strength is observed. There are small fluctuations in ductility between 20 ℃ and 800 ℃. The elongation to fracture increases from 10% to 36% as the temperature is increased from 800 ℃ to 1 000 ℃. The strain-hardening exponent decreases with an increase in temperature. This alloy maintains a high elastic modulus at high temperatures.

2) At low temperatures, the deformation is dominated by γ′ shearing and high dislocation densities are observed in matrix channels. At intermediate temperatures, the dislocation microstructure is inhomogeneous due to the formation of dislocation concentrations with high-density tangling. At high temperatures, the networks deposited at the γ/γ′ interfaces prevent dislocations from entering the γ′ particles. The coarsening of γ′ precipitate contributes to higher ductility and lower strength.

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