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Flow and fracture behaviour of Ni_3(Al · Ti) single crystals tested in tension

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The deformation and the fracture behaviour of the [0 0 1] orientated Ni_3(Al.Ti) single crystals were investigated to determine the relation between the positive temperature dependence of the flow stress in the γ'-phase and the malleability of nickel-base superalloys. The positive temperature dependence of the flow stress is observed in the [0 0 1] orientation below about 1000 K (T_p) and the failure occurs in a catastrophic and brittle manner after considerable plastic deformation. The room temperature fracture stress increases with increase in the angle θ between the [0 0 1] orientation and the tensile axis at 290 K, and it is well expressed by a crack propagation criterion only by considering the effect of the normal stress on the {1 0 0} cleavage plane. The cleavage fracture stress for the [0 0 1] orientation is nearly independent of temperature below T_p, while the elongation decreases with temperature in contrast to the yield stress. The cleavage fracture of Ni_3(Al.Ti) single crystals is explained by the rapid decrease of the mobile dislocation density due to a dislocation pinning mechanism based on the cube cross-slipping of the screw superdislocations which causes the positive temperature dependence of the flow stress. The insufficiency of the malleability of nickel-base superalloys seems to be attributed to that of the γ'-Ni_3(Al.Ti) phase, and the hot working of nickel-base superalloys near T_p in the γ'-phase should be avoided.

1. Introduction

Nickel-base superalloys are widely used in applications requiring high strength at elevated temperatures. Most of these alloys are precipitation hardened by a Ni_3(Al.Ti) γ'-phase which has an ordered f c c structure ($L1_2$ intermetallic compound) and precipitates coherently in a nickel-rich f c c (γ) solid solution. The excellent high-temperature mechanical properties of commercial nickel-base superalloys are primarily attributed to unusual high-temperature mechanical properties of the γ'-phase; namely, the positive temperature dependence of the flow stress. The strength of a given superalloy depends on such factors as volume fraction, particle size, coarsening rate and composition (antiphase boundary energy) of the γ' precipitate. For example, the volume fraction of the γ'-phase varies widely from about 0.2 in Nimonic 80A to about 0.6 in Mar-M200. Since the alloys with large volume fractions of γ' cannot be fabricated by the normal hot-forming procedure, they are usually used in the cast condition. However, it is at present not clear whether the insufficient malleability of nickel-base superalloys at elevated temperatures originates from the characteristic of the γ'-phase or not, because most concern has been concentrated on the positive temperature dependence of the flow stress, and the malleability of the γ'-phase (fracture behaviour and ductility) has been little investigated to date.

We have recently found that the $L1_2$ intermetallic compound Ni_3Ge single crystals break by cleavage on the {1 0 0} plane at 290 K [1], and concluded that the cleavage fracture of Ni_3Ge is attributed to the rapid decrease of mobile dislocation density on {1 1 1}⟨1 1 0⟩ slip systems due

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to a dislocation pinning mechanism based on the cube cross-slipping of screw superdislocations, which is characteristic of the $L1_2$ intermetallic compound and causes the positive temperature dependence of the flow stress. In other words, the cleavage fracture of the $L1_2$ compounds is closely related to the positive temperature dependence of the flow stress. Since $Ni_3(Al.Ti)$ has the same crystal structure ($L1_2$ type) as Ni_3Ge, and similarly shows positive temperature dependence, it may well be that $Ni_3(Al.Ti)$ single crystals also break by cleavage. If $Ni_3(Al.Ti)$ single crystals break by cleavage on the {1 0 0} plane, then the [0 0 1] orientated specimen breaks most easily, namely, at the lowest fracture stress, because the resolved normal stress to (0 0 1) is largest for this orientation.

On the other hand, it is well-known that the rate of increase of the yield stress with temperature is lowest [2–9], and the temperature of the peak yield stress is highest for the [0 0 1] orientation. Thus, the [0 0 1] orientation in the $L1_2$ intermetallic compounds is thought to behave uniquely in both plastic deformation and fracture behaviour.

In the present investigation, we will examine the deformation and fracture behaviour of the [0 0 1] orientated $Ni_3(Al.Ti)$ single crystals at elevated temperatures to determine the relation between the positive temperature dependence of the flow stress in the γ'-phase and the malleability of nickel-base superalloys.

2. Experimental procedure

A single crystal of $Ni_3(Al.Ti)$, (75 at.% Ni, 15 at.% Al and 10 at.% Ti) was grown by the Bridgman method under purified argon atmosphere using a high-purity alumina crucible. The single crystal was homogenized for 50 h at 1323 K in a vacuum of 1.33 mPa. Tensile specimens were cut from the one single crystal excluding both ends of it using a spark cutting machine. The specimens had rectangular cross-sections and a gauge size of approximately 1 mm x 2 mm x 12 mm. Five kinds of orientations were chosen on the [0 0 1]–[$\bar{1}$ 1 1] symmetry line so that double or multiple slips were operative from the initial stage of deformation. The orientation of specimens was determined by the Laue back-reflection method, and their orientations were shown to be within ± 2° of the expected ones. After spark cutting, the damaged layer of the specimens was removed by electrolytic polishing (10% H_2SO_4, 90% CH_3COOH) and then the specimens were annealed again for 24 h at 1323 K. Finally, all specimens were electrolytically polished. Tensile tests were carried out at 290 K for five kinds of specimens using an Instron-type testing machine. Furthermore, the tests were performed between 290 and 1273 K for [0 0 1] orientated specimens in a vacuum of 1.33 mPa. The specimens were pulled at an initial strain rate of $1.3 \times 10^{-3}\ sec^{-1}$.

3. Results

Fig. 1 shows the tensile stress–strain curves of $Ni_3(Al.Ti)$ single crystals tested at 290K, where the [A] orientation is located on the [0 0 1] – [1 1 1] symmetry line and 11 degrees from the [1 1 1] orientation. The orientation dependence of the tensile stress–strain curves of $Ni_3(Al.Ti)$ single crystals is similar to that of Ni_3Ge [1, 9] namely, both the yield stress (σ) and the maximum work-hardening rate $(d\sigma/d\epsilon)_M$ increase when nearing the [1 1 1] orientation. This orientation dependence may be attributed to the cube cross-slipping of screw superdislocations which causes the positive temperature dependence of the flow stress as proposed previously [8, 9]. The elongation and the yield stress of $Ni_3(Al.Ti)$ single crystals are larger and lower than those of Ni_3Ge [1], respectively, although the fracture stresses of both compounds are almost the same. No serrated flow which generally resulted from the dynamic strain-ageing or deformation twinning was detected on a load–elongation curves. Failure occurs in a catastrophic and brittle manner after considerable

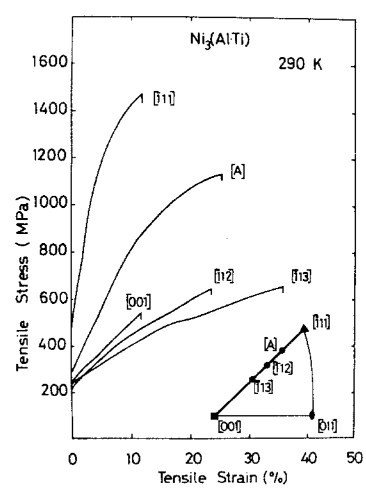

Figure 1 The tensile stress–strain curves of $Ni_3(Al.Ti)$ single crystals at 290K.

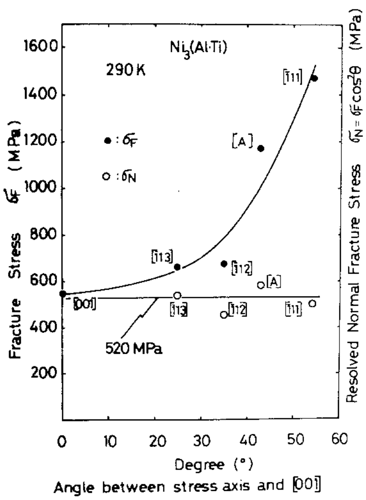

Figure 2 The fracture stress, σ_F, and the resolved normal fracture stress to the (0 0 1) plane, σ_N, of $Ni_3(Al.Ti)$ single crystals as a function of the angle θ between [0 0 1] and the tensile axis.

plastic deformation, and necking of the specimen is not observed below T_p (peak temperature of the flow stress).

Fig. 2 shows the fracture stress, σ_F, obtained from Fig. 1 and the resolved normal fracture stress to the (0 0 1) plane, $\sigma_N = \sigma_F \cos^2\theta$, as a function of the angle θ between the [0 0 1] orientation and the tensile axes. σ_F increases with increasing θ, namely, when nearing the [$\bar{1}$ 1 1] orientation in the same manner as Ni_3Ge [1]. On the other hand, σ_N is almost constant independent of the tensile axes at 290K. This relation suggests that the fracture occurs by the cleavage on the (0 0 1) plane when resolved normal flow stress attains to the cleavage strength of the (0 0 1) plane.

Fig. 3 shows the temperature dependence of

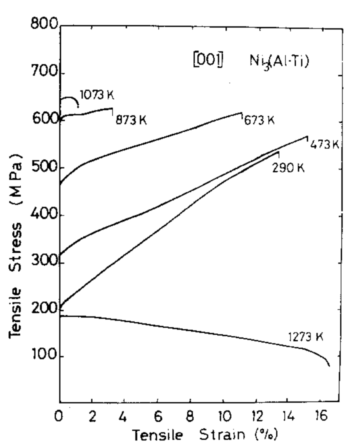

Figure 3 The temperature dependence of the tensile stress–strain curves of [0 0 1] orientated $Ni_3(Al.Ti)$ single crystals.

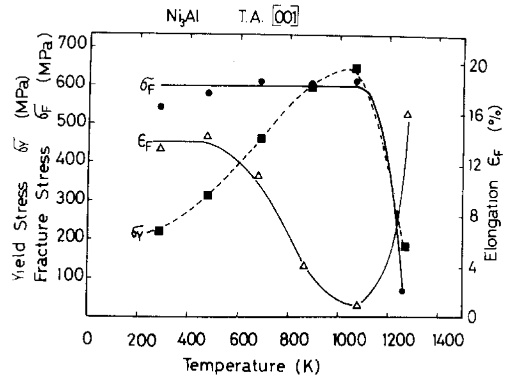

Figure 4 The temperature dependence of the yield stress, σ_Y, the fracture stress, σ_F, and the elongation, ϵ_F, of the [0 0 1] orientated specimens.

the tensile stress–strain curves for the [0 0 1] orientated $Ni_3(Al.Ti)$ single crystals. The stress–strain curves show nearly linear work-hardening below 900K, while they show negative work-hardening, i.e. work-softening above 1100K. No serrated flow was observed at elevated temperatures. Failure occurs also in a catastrophic and brittle manner except at 1273K, where the fracture mode is a ductile one.

Fig. 4 shows the temperature dependence of the yield stress (0.2% proof stress), fracture stress and elongation (fracture strain) for [0 0 1] specimens. The yield stress shows a positive temperature dependence below about 1100 K, and a negative one above that temperature. This tendency is in agreement with the previous one [4, 5], for [0 0 1] specimens. Although the yield stress shows a positive temperature dependence, the fracture stress remains almost constant over the same temperature range, and then decreases with increasing temperature above about T_p. The elongation decreases with increasing temperature, and attains a minimum value near T_p. Therefore, we can say that the elongation exhibits a temperature dependence in the opposite sense to the yield stress. This tendency is in accord with the previous case of binary Ni_3Al single crystals [10].

Fig. 5a and b show scanning electron micrographs of the fracture surface of the [0 0 1] specimens fractured at 290 and 1037 K, respectively. The fracture surface is relatively smooth and normal to its tensile axis, i.e. the fracture surface is nearly (0 0 1). River lines, which are the characteristic of cleavage fracture, are also observed at high magnification, as seen in Fig. 5c.

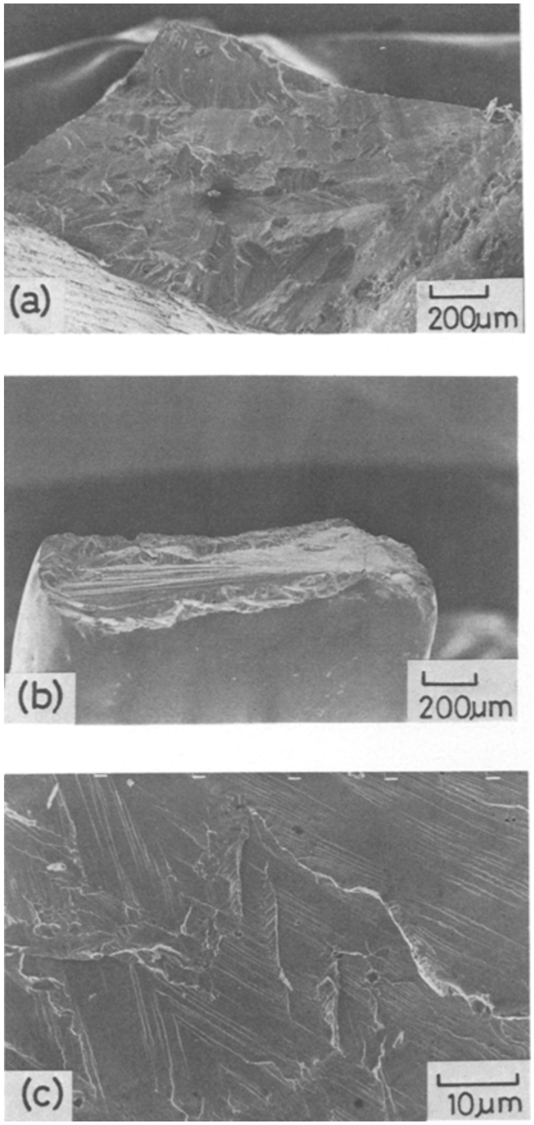

Figure 5 The fracture surfaces of [0 0 1] specimens fractured at 290K and 1073K. (a) 290K, (b) 1073K, (c) 290K.

4. Discussion
4.1. The positive temperature dependence of yield stress
It has been confirmed that the yield stress of the [0 0 1] orientated $Ni_3(Al.Ti)$ single crystals also shows a positive temperature dependence. We have recently investigated the mechanism of positive temperature dependence of the flow stress in Ni_3 (Al.Ti) and Ni_3Ge single crystals [8, 9] and have concluded that it may be attributed to the cube cross-slipping of the screw superdislocations, i.e. the screw superdislocations on the {1 1 1} slip planes tend to cross-slip onto the {1 0 0} plane to reduce the APB energy. The resistance to dislocation motion is so large on the {1 0 0} plane that the cross-slipped segments prevent the subsequent motion of the remaining parts of the dislocations on the {1 1 1}⟨1 0 0⟩ slip systems. The yield stress increases with increase in the number of cube cross-slipped parts. Since the probability of cube cross-slip increases with temperature, the yield stress shows a positive temperature dependence. On the other hand, at higher temperatures the {1 0 0}⟨1 1 0⟩ slip, which shows a normal temperature dependence, takes the place of {1 1 1}⟨1 1 0⟩ slip, because the CRSS of the latter becomes much higher than that of the former. Thus, the temperature dependence of the yield stress in Fig. 4 will also be reasonably explained by the above mechanisms.

4.2. The reasons for the occurrence of cleavage fracture in Ni_3(Al.Ti) single crystals
It has generally been accepted that cleavage fracture is unlikely to occur in normal f c c metals and alloys except for the environment embrittlement as the liquid metal embrittlement, although many f c c compounds (e.g. TiC and MgO), cleave on the {1 0 0} plane.

The most dominant quantity which controls the cleavage resistance of materials is generally considered to be the energy dissipated by various processes near the tip of a crack. The most important dissipative process is plastic deformation. Since cleavage is a dynamic process, the rate of energy absorption by plastic flow is a primary quantity, which depends on the plastic strain rate. The plastic strain rate ($\dot{\epsilon}$) is expressed as follows.

$$\dot{\epsilon} = b\rho\overline{V} \tag{1}$$

Where $\boldsymbol{b}$ is the Burgers vector, ρ is the mobile dislocation density and $\overline{V}$ is the average dislocation velocity. There is little variation in $\boldsymbol{b}$ from one crystal to another, but ρ and $\overline{V}$ may vary considerably. If either ρ or $\overline{V}$ is very low, cleavage fracture may be brought about because little energy is absorbed due to low $\dot{\epsilon}$. The reasons for f c c metals and alloys not breaking by cleavage are considered to be as follows:

(1) Dislocation pinning is so weak (or non-existent) that the mobile dislocation density is always very high;

(2) Dislocation velocity is independent of temperature and always very high except in the high-strength alloys.

Both the yield stress and the temperature dependence of the yield stress in normal f c c metals is very low, which makes it difficult for cleavage fracture to occur. On the contrary, the yield stress of $Ni_3(Al.Ti)$ at room temperature is higher than that of normal f c c metals, and increases further with increasing temperature. Thus, there is a large difference in the deformation behaviour between $Ni_3(Al.Ti)$, i.e. f c c-type ordered phase, and f c c metals. This difference may result mainly from the motion of the superdislocation, which is bound by antiphase boundaries (APB). The dislocation motion in the $L1_2$ intermetallic compounds is very unique due to the cube cross-slipping of screw superdislocations [8, 9], i.e. the screw superdislocations on the $\{1\,1\,1\}$ slip plane tend to cross-slip onto the cube $\{1\,0\,0\}$ plane by a thermally activated process to reduce the APB energy, which causes the positive temperature dependence of the flow stress as mentioned in Section 4.1. Once cross-slipping occurs, the motion of the dislocation is retarded. The slowing-down of the velocity brings about an increase in the number of thermally activated portions. Thus, the motion is rapidly decelerated and the dislocation soon becomes perfectly sessile after a short-range motion. This proposition is confirmed by means of *in situ* observation of dislocation motion on the $\{1\,1\,1\}\langle 1\,1\,0\rangle$ slip systems in $Ni_3(Al.Ti)$ single crystals by Nemoto *et al.* [11]. According to them, the $\{1\,1\,1\}$ slip occurs quickly and suddenly, but once the movement of the $\{1\,1\,1\}\langle 1\,1\,0\rangle$ slip dislocation is stopped, which is frequently observed, these previously mobile dislocations become perfectly immobile. On the other hand, $(1\,0\,0)$ slip systems are not operative below T_p, because the frictional stress of the dislocation on the $\{1\,0\,0\}$ slip systems is generally very high at low temperatures. Therefore, the exhaustion of mobile dislocations in $Ni_3(Al.Ti)$ may be brought about by this dislocation pinning mechanism after considerable plastic deformation. Thus, the cleavage fracture of $Ni_3(Al.Ti)$ is rationalized by the exhaustion of mobile dislocation density.

Since the cube cross-slipping (dislocation pinning) increases with temperature, the tendency for cleavage fracture may also increase with temperature. However, the cleavage fracture stress of $Ni_3(Al.Ti)$ single crystals is, in fact, nearly independent of temperature below T_p, as seen in Fig. 4. Consequently, it is considered that two conditions must be fulfilled for the occurrence of cleavage fracture in $Ni_3(Al.Ti)$. The first prerequisite is the very low mobile dislocation density, and the second is that the resolved normal flow stress attains to its cleavage strength of the $\{1\,0\,0\}$ plane. These conditions for $Ni_3(Al.Ti)$ are fulfilled by work-hardening after considerable plastic deformation. For example, the strain-to-fracture (the elongation, ϵ_f is considerably large (Fig. 4) at 290K, because the yield stress is low. On the contrary, elongation is very low at 900K because the yield stress is very high and nearly equals the cleavage strength. Thus, the temperature dependence of the elongation in $Ni_3(Al.Ti)$ single crystals is closely related to the positive temperature dependence of the yield stress. In other words, the positive temperature dependence of the yield stress leads inevitably to the embrittlement of the $Ni_3(Al.Ti)$ single crystals, because the cleavage strength is nearly constant.

As mentioned in Section 1, the nickel-base superalloys with large volume fractions of γ' cannot be fabricated by the usual hot-forming procedures. The insufficiency of the malleability of these alloys at elevated temperatures seems to be attributed to that of the γ'-phase, because the ductility of $Ni_3(Al.Ti)$ single crystals decreases with temperature, and becomes very low near T_p. Therefore, the hot-working of nickel-base superalloys near T_p of the γ'-phase should be avoided.

4.3. Fracture-controlling process

Since it has been found from the present investigation that $Ni_3(Al.Ti)$ fractures by cleavage, we will discuss what process controls cleavage fracture.

Various criteria of crack nucleation and propagation have been put forward to explain cleavage fracture of materials. Among the propagation criteria, which completely neglect the crack nucleation process, the constant normal stress law, based on the Griffith condition for the fracture of brittle solids, is the most simple and fundamental. The critical resolved normal stress law is expressed as follows:

$$\sigma_N = \sigma_F \cos^2\theta = \left(\frac{2E\gamma_p}{\pi(1-\nu^2)c}\right)^{1/2} \qquad (2)$$

Where σ_F is the tensile fracture stress, σ_N is the normal stress on a cleavage crack of length $2c$ lying in the most favourably oriented cleavage plane at an angle of θ° to the tensile axis in a material having an effective surface energy γ_p, Young's modulus E and Poission's ratio ν. Since $\sigma_F \cos^2\theta$ is nearly constant, independent of the angle θ between the [0 0 1] and the tensile axis as seen in Fig. 2, it can be said that the constant normal stress law is satisfied in $Ni_3(Al.Ti)$ single crystals. Therefore, if the resolved normal stress to {1 0 0} planes attains its cleavage strength, about 520 MPa as seen in Fig. 2, the cleavage crack propagates. The reason for σ_F inceasing with increase in the angle θ, is that σ_f is expressed as $\sigma_N/\cos^2\theta$ (where σ_N is constant in the present case) from Equation 2.

On the other hand, it is generally said that we should consider the effect of shear as well as normal stress on the crack propagation process, because a crack is also able to grow under the influence of shear stress. If the effect of shear stress is considered, the propagation criterion for an inclined crack in a tensile specimen is expressed as follows [12]:

$$\sigma_F \cos\theta = \left(\frac{2E\gamma_p}{\pi(1-\nu^2)c}\right)^{1/2} \qquad (3)$$

Fig. 6 shows $\sigma_F \cos\theta$ as a function of θ. It can be seen from this figure that this fracture criterion is not applicable in the present case. Furthermore, the propagation criterion taking account the anisotropy of the Young's modulus is also not applicable, because there is no clear relation between

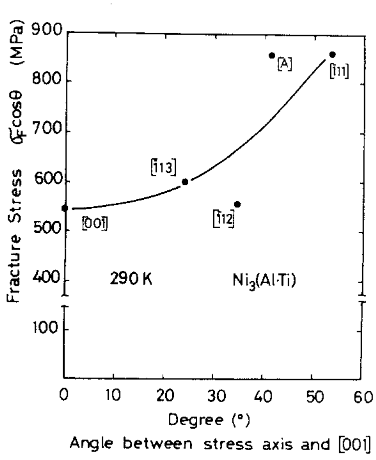

Figure 6 The fracture stress which takes into account the shear as well as the normal stress as a function of the angle θ between [0 0 1] and the tensile axis.

the angle θ and $\sigma_F \cos\theta \{1 + [(\mu_n - \mu_s)/\mu_s] \cdot \sin^2\theta\}^{1/2}$, although the cleavage fracture of Ni_3Ge single crystals has most reasonably been explained by this criterion [1]. On the other hand, there is no crack nucleation criterion which satisfies the experimental data. These experimental facts are in accord with the absence of deformation twinning and considerable plastic deformation. Consequently, it can be concluded from the present investigation that cleavage fracture in $Ni_3(Al.Ti)$ single crystals is governed not by crack nucleation but by propagation, i.e. the crack nucleation process is completely neglected, and the crack propagation process is reasonably explained by only considering the effect of the normal stress on the cleavage plane.

5. Conclusions

The deformation and fracture behaviour of the [0 0 1] orientated $Ni_3(Al.Ti)$ single crystals were investigated to determine the relation between the positive temperature dependence of the flow stress in the γ'-phase and the malleability of nickel-base superalloys. The main results obtained in the present investigation are summarized as follows.

(1) The positive temperature dependence of the flow stress is observed even in the [0 0 1] orientation below about 1000K (T_p), and failure occurs in a catastrophic and brittle manner after considerable plastic deformation.

(2) The fracture stress increases with increase in the angle θ between the [0 0 1] and the tensile axis at 290K, and it is well expressed by a crack propagation criterion only by considering the effect of the normal stress for the {1 0 0} cleavage plane.

(3) The cleavage fracture stress for the [0 0 1] orientation is nearly independent of temperature below T_p, while the elongation decreases with temperature in contrast to the yield stress.

(4) The cleavage fracture of $Ni_3(Al.Ti)$ single crystals is reasonably explained by the rapid decrease of the mobile dislocation density due to a dislocation pinning mechanism based on the cube cross-slipping of the screw superdislocations which causes the positive temperature dependence of the flow stress.

(5) The insufficiency of the malleability of nickel-base superalloys seems to be attributed to that of the γ'-$Ni_3(Al.Ti)$ phase, and the hot-working of nickel-base superalloys near T_p in the γ'-phase should be avoided.

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