RELATIONSHIP BETWEEN RAFTED γ' MORPHOLOGY AND CREEP

RESISTANCE OF A NICKEL-BASED SUPERALLOY

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Abstract

The effect of the rafted γ' precipitates on the creep resistance of the conventionally cast superalloy IN-100 was investigated at the creep conditions of 900 °C/276 MPa. The creep rate was observed to be minimum when the rafted γ' size aspect ratio a/b was close to ~ 1 or ~ 2 , where a and b are the rafted γ' particle sizes perpendicular and parallel to the applied tensile stress axis, respectively, whereas the creep rupture life showed the highest value when the initial-to-rafted γ' volume fraction ratio and the initial to rafted γ' size ratio were minimum. In addition, it was also observed that the ratio of the initial-to-rafted γ' particle surface area per unit volume controls the creep rate in the secondary and tertiary stages. It was predicted that the creep deformation mechanism changes from the Orowan bowing in the secondary stage to the particle shearing in the final stage.

Superalloys 1992

Edited by S.D. Antolovich, R.W. Stusrud, R.A. MacKay,
D.L. Anton, T. Khan, R.D. Kissinger, D.L. Klarstrom
The Minerals, Metals & Materials Society, 1992

Introduction

The initial γ' precipitate morphology in cast IN-100 is usually in the form of cubes or spheres, depending on the misfit parameter between γ' and the γ -matrix. The strengthening contribution of γ' is partly dependent on the size, spacing, shape and volume fraction of the particles (1). However, during the creep exposure, γ' coarsening can take place and the particles may remain equiaxed (2), become irregularly shaped (3) or coarsen into rod or rafted morphologies (4).

As mentioned above, under an applied stress at elevated temperatures, the discrete type γ' particles link up to form rafted γ' morphology. The γ/γ' lamellae are believed to strengthen the material during creep, because this rafted morphology of the γ' phase essentially eliminated γ' particle by passing (5), which is the creep mechanism normally operative in conventional Ni-based superalloy at elevated temperatures and low stress.

Experimental Procedure

The effects of rafted γ' morphology after creep rupture on the creep properties of conventionally cast IN-100 were investigated. The bulk composition is (in wt.%): 10.0 Cr, 15.0 Co, 4.7 Ti, 5.5 Al, 3.0 Mo, 0.18 C, 0.06 Zr, 0.014 B, 1.0 V, balance Ni. The variations of the rafted γ' dispersion was achieved by using the following conditions before the creep test: (i) as-cast structure at different cooling rates; (ii) heat treatment I: 1107 °C/4 h, furnace cooling and 871 °C/12 h, furnace cooling; (iii) heat treatment II: 1220 °C/1 h, furnace cooling and 1100 °C/1 h, furnace cooling. These conditions have assured that the initial γ' dispersions were varied. As a result, the different rafted γ' morphology after creep was also obtained.

The creep tests were carried out up to failure at 900 °C by uniaxial constant-load testing with an initial stress of 276 MPa. Creep specimens with 4 mm gauge diameter were used. The temperature variation over the testing time was kept within ± 0.5 °C. The creep elongation was recorded continuously using differential transformers.

The metallographic examinations of the rafted γ' morphology from the gauge sections of the specimens were made using the point counting method (Cambridge Instruments Q520 system) on scanning electron microscopy (SEM) photographs.

Experimental Results

Figure 1 illustrates the effect of the rafted γ' particles expressed by the rafted γ' size aspect ratio a/b on the minimum creep rate $\dot{\epsilon}_m$. a and b are the average sizes of the rafted γ' particles perpendicular and parallel to the applied tensile stress axis, respectively. The a/b ratios were measured for all creep ruptured specimens to establish quantitatively the morphological changes of γ' under the present creep condition. $\dot{\epsilon}_m$ is maximum at a critical ratio of $a/b \sim 1.5$. There is a rather good correlation between the minimum creep rate and the aspect ratio a/b , which indicates the degree of the shape changes or the degree in flatness of rafted γ' particles during creep process.

To investigate the different morphologies and distributions of the rafted γ' particles Fig. 1 was divided into four regions (A, B, C, D). Figure 2 shows the typical micrographs from these regions. As can be seen in Fig. 2a, in region A the microstructure does not show the properly developed rafted γ' morphology which consists of the complex, equiaxed or rounded

and randomly distributed γ' particles after creep fracture. In this region the aspect ratio a/b is close to unity. However, in region B, which corresponds to the peak in Fig. 1, the γ' particles have become rafted, whereas in region C the rafted structure has developed further. Finally, in region D where $a/b \sim 2$ the rafted particles are more finely and regularly distributed, and also the particle spacing is finer than those in regions C or B.

Figure 3 illustrates the effect of the rafted γ' size aspect ratio a/b on the specific perimeter ratio PE_I/PE_R , where PE_I and PE_R are the mean specific perimeters for the initial and the rafted γ' particles. Note that the specific perimeter is the γ'/γ -matrix interface length per unit area of γ' particle. As illustrated in Figs 1 and 3, the specific perimeter ratio decreases up to $a/b \sim 1.5$ whereas the minimum creep rate increases. Above this a/b -value the behaviour reverses. There was not any relationship between PE_I or PE_R and the aspect ratio.

Figure 4 shows the effect of the volume fraction ratio V_I/V_R on the creep rupture life t_R , where V_I and V_R are the volume fractions for the initial and rafted γ' particles, respectively. Decreasing the volume fraction ratio continuously elongates the creep rupture life. The correlation between the specific perimeter ratio PE_I/PE_R and the volume fraction ratio V_I/V_R in Fig. 5 indicates a similar behaviour.

The plotting of the creep rupture life as a function of the initial-to-rafted γ' size ratio d_I/d_R is shown in Fig. 6. As this figure indicates decreasing the initial-to-rafted γ' size ratio continuously increases the creep rupture life. There is a rather inverse linear relationship between the specific perimeter ratio PE_I/PE_R and the initial to rafted γ' size ratio d_I/d_R (Fig. 7), similar to that in Fig. 6. The dependence of the creep rupture life on the specific perimeter ratio is also plotted to show the effect of PE_I/PE_R ratio on t_R clearly (Fig. 8). The creep rupture life increases with increasing PE_I/PE_R .

Discussion

Figure 1 indicates the effect of the aspect ratio a/b on the minimum creep rate $\dot{\epsilon}_m$. As Figs 1 and 2 illustrate minimum creep rate, i.e. a higher creep resistance, it achieved for the two types of rafted γ' morphologies: (i) the complex, equiaxed and rounded type γ' particles in the fractured specimen, which has an aspect ratio close to 1; and (ii) the rafted γ' particles are finely and regularly distributed throughout the microstructure with an aspect ratio close to 2. In addition, as can be seen in Fig. 9, the γ' volume fraction variations in the crept specimens are minimum at these two conditions. As a result, for the minimum creep rate to be kept at the lowest value the γ' volume fraction ratio V_I/V_R should also be as low as possible.

As seen in Figs 1 and 3 there is a reverse relationship between the minimum creep rate $\dot{\epsilon}_m$ and the specific perimeter ratio PE_I/PE_R as a function of the rafted γ' size aspect ratio a/b . The variation of the specific perimeter ratio in Fig. 3 indicates the relative variation of the interface energy between the γ and γ' phase. From Figs 1 and 3 it is concluded that the minimum creep rate depends not only on the interface energies between the rafted γ' particles and the γ phase but on the relative variation of these energies for the initial structure (ΔE_I) and the rafted γ' particles (ΔE_R), i.e. $\Delta E_I/\Delta E_R$. Therefore, it is the surface or interface energy ratio ($\Delta E_I/\Delta E_R$) controlling the creep resistance of the alloy.

As seen in Fig. 4 the creep rupture life shows a rather good correlation with V_I/V_R . Again, the correlation of PE_I/PE_R as a function of V_I/V_R (Fig. 5) indicates that the creep rupture life depends on the relative variation of the interface energies $\Delta E_I/\Delta E_R$ for the initial and the

Figure 1 - The correlation between the minimum creep rate $\dot{\epsilon}_m$ and the aspect ratio a/b of the rafted γ' particles.

b) Region B;

Figure 2 - Different morphological features of the rafted γ' particles after creep fracture in the regions A, B, C and D of Fig. 1
a) Region A;

c) Region C;

d) Region D.

Figure 4 - Effect of the volume fraction ratio V_I/V_R on the creep rupture life t_R , where V_I and V_R are the volume fractions for the initial and rafted γ' particles.

Figure 3 - Relationship between the specific perimeter ratio PE_I/PE_R and the rafted γ' aspect ratio a/b , where PE_I and PE_R are the specific perimeters for the initial and rafted γ' particles.

Figure 5 - The relationship between the specific perimeter ratio PE_I/PE_R and the volume fraction ratio V_I/V_R .

rafted γ' particles. The creep rupture life also shows good correlation with d_i/d_R (Fig. 6). Figure 7 indicates linear inverse relationship between PE_i/PE_R and d_i/d_R which confirms the previous observation (Fig. 3) that the relative variation of the energies between the γ/γ' interfaces for the initial and the rafted γ' particles directly controls the creep rupture life (Fig. 8).

The plotting of the a/b ratio, which is the controlling microstructural parameter for the minimum creep rate of the alloy, against the initial γ' size d_i did not show an unambiguous relationship. However, the plotting a/b against d_i/d_R leads to a rather good correlation (Fig. 10). The curve indicates that a/b is controlled by d_i/d_R ; increasing d_i/d_R from ~ 0.5 to ~ 2 decreases a/b from ~ 2 to ~ 1 . In conclusion, in the present results the initial γ' microstructural parameters do not affect the rafted γ' parameters directly. Rather these affects take place in terms of ratios. For instance, in the past it was observed on the single crystals (6,7) that the creep properties improved as the raft thickness decreased and the number of γ/γ' interfaces increased, whereas in the present work, the γ/γ' interfaces alone in the rafted structure do not control the creep deformation, and it is the PE_i/PE_R ratio, where PE_i and PE_R indicate the level of interface energies in the γ/γ' interface for the initial and rafted structure, which controls the creep resistance and rupture life.

An attempt has been made to predict the effect of rafted γ' particles on the creep resistance in terms of the existing deformation mechanisms, namely, shearing or cutting the precipitated particles (8), looping between particles by Orowan bowing mechanism (9). The following expressions for particle shearing ($\Delta\tau_{ps}$) and Orowan bowing ($\Delta\tau_{OB}$) threshold stresses (or the critical resolved shear stress, CRSS) as possible creep deformation mechanisms have been used in this investigation to predict the resistance made by the γ' precipitates using the observed rafted γ' dispersion parameters:

$$\Delta\tau_{ps} = \frac{\gamma}{2b} \left[\left(\frac{4\gamma R_s f}{\pi\Gamma} \right)^{1/2} - f \right] \quad (1)$$

$$\Delta\tau_{OB} = \frac{0.4\mu b}{\pi\lambda_s} \cdot \frac{\ln \left(\frac{2R_s}{b} \right)}{(1-\nu)^{1/2}} \quad (2)$$

where γ is the anti-phase boundary (APB) energy, b the Burgers vector, R_s the average radius, f the particle volume fraction, μ the shear modulus, $\Gamma = (1/2) \mu b^2$ dislocation line tension, ν the Poisson's ratio and λ_s can be determined by (8): $\lambda_s = [(\pi/f)^{1/2} - 2]R_s$.

Equations (1) and (2) show that there are two microstructural variables, viz. volume fraction and particle radius (or particle spacing λ_s). Using the following values the variations of the threshold stresses for the particle shearing (Eqn. (1)) and Orowan bowing (Eqn. (2)) have been calculated using the observed microstructural variables as a function of the observed γ' size aspect ratio, the volume fraction ratio and the initial-to-rafted γ' size ratio:

$\mu = 4 \times 10^6$ MPa, $b = 2.5 \times 10^{-10}$ m, $\nu = 0.33$, $\gamma = 0.17$ Jm⁻² (10).

Figure 6 - Effect of the initial-to-rafted γ' particle size ratio d_I/d_R on the creep rupture life t_R .

Figure 8 - Effect of the specific perimeter ratio PE_I/PE_R on the creep rupture life t_R .

Figure 7 - Relationship between the specific perimeter ratio PE_I/PE_R and the initial-to-rafted γ' ratio d_I/d_R .

Figure 9 - Relationship between the volume fraction ratio V_I/V_R and the γ' aspect ratio a/b .

The results are given in Figs 11 - 13. Figure 13 illustrates the variation of the threshold stress for Orowan bowing, $\Delta\tau_{OB}$, as a function of a/b . The resistance against deformation decreases as a/b increases up to ~ 1.5 . Further increasing of a/b increases the resistance against deformation. Therefore, it is possible that during the secondary creep stage the Orowan bowing is the operative creep deformation mechanism (Fig. 1). However, the plotting of the threshold stress for Orowan bowing as a function of the d_I/d_R or V_I/V_R ratios did not show any relationship with the creep rupture life or strain. Figures 12 and 13 show the dependence of the threshold stress for particle shearing, $\Delta\tau_{ps}$, as a function of the initial-to-rafted γ' volume fraction V_I/V_R and the initial-to-rafted γ' size d_I/d_R ratios. These two figures indicate that the increase of both, V_I/V_R and d_I/d_R , reduce the resistance against deformation. Therefore, from the similarities of the curves in Figs 12, 13, 4 and 6 it is concluded that in the final stage of creep or during the tertiary stage the particle shearing is the creep deformation mechanism at the present creep conditions (900 °C/276 MPa).

The question now arises why the creep deformation mechanism changes from Orowan bowing in the secondary stage to the more difficult particle shearing in the final or tertiary stage. It is possible that the shape change of the γ' particles during creep is responsible for the change in the creep deformation mechanism. In fact, evidence of shearing has been observed in rafts during creep in CMSX-2 (11) and in NASAIR 100 (12). It has been further postulated (5,6,12) that this shearing mechanism is impeded by the densely spaced networks of misfit dislocations which develop at the γ/γ' interfaces.

Conclusions

The minimum creep rate has its lowest value when the γ' size aspect ratio a/b is close to 1 or 2. At these values the γ' volume fraction ratio V_I/V_R is minimum. The creep rupture life is maximum when the volume fraction ratio V_I/V_R or the initial-to-rafted γ' size ratio d_I/d_R is minimum. At lowest γ' volume fraction ratios the rafted γ' particles are either equiaxed and rounded type with an a/b close to 1 or finely and regularly distributed close to 2. The creep in the secondary and tertiary stages is controlled not by the surface energy between the γ and γ' phases alone but by the initial-to-rafted γ' specific perimeter ratio PE_I/PE_R or the interface energy ratio $\Delta E_I/\Delta E_R$. The prediction of the creep deformation mechanism at the present creep conditions (900 °C/276 MPa) has shown that it changes from Orowan bowing in the secondary stage to particle shearing in the final stage.

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Figure 10 - Correlation between the rafted γ' aspect ratio a/b and the initial-to-rafted γ' size ratio d_I/d_R .

Figure 12 - The threshold stress for particle shearing, $\Delta\tau_{ps}$, versus the initial-to-rafted γ' volume fraction ratio V_I/V_R .

Figure 11 - The threshold stress for Orowan bowing, $\Delta\tau_{ob}$, versus the rafted γ' aspect ratio a/b , showing a minimum at $a/b \sim 1.5$.

Figure 13 - Relationship between the threshold stress for particle shearing, $\Delta\tau_{ps}$, and the initial-to-rafted γ' size ratio d_I/d_R .

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