STRESS COARSENING OF γ' AND ITS INFLUENCE ON CREEP
PROPERTIES OF A SINGLE CRYSTAL SUPERALLOY

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The influence of stress annealing on the γ' morphology in single crystals of a Ni-13Al-9Mo-2Ta (at. pct.) alloy has been examined. In agreement with previous work, it has been found that the γ' morphology depends on the direction and sense of the applied stress. A crystal subjected to $\langle 100 \rangle$ tensile creep develops platelets, or rafts of γ' in orientations perpendicular to the applied stress. Due to the large negative γ/γ' misfit (~ 0.7 pct.) in this alloy, the γ/γ' interfaces are decorated with a high density of misfit dislocations. These microstructural changes exert a profound influence on creep properties, at least in the $\langle 100 \rangle$ orientation. Under a stress of 207 MPa at 1038°C the rafted γ' structure exhibits a rupture life of over 400 hrs., which compares with ~ 100 hours for unrafted material. The observed creep properties in this new alloy compare favorably with those reported for D.S. MAR-M200. About a 75°C advantage in metal temperature capability is found in the high temperature range. The overall creep response of this alloy, first of a new class of 'super single crystal' (SSC) alloys, is comparable with the best available alloys.

INTRODUCTION

For many years changes in the morphology of γ' have been observed after prolonged creep testing at elevated temperatures (1-4). These observations have been viewed with some concern in polycrystals and have led to studies where the stress-induced morphological changes were essentially eliminated by reducing the γ/γ' lattice misfit. Furthermore, the influence of external stress on the morphology of stress coarsened γ' has been determined for several alloys (5-7). Tensile and compressive stress annealing produced γ' platelets or rods, depending on the direction and sense of the applied stress. Since the yield behavior of these composite morphologies were shown to drop abruptly at about the same temperature, 760°C (8), the advantage of prolonged high temperature creep strength enhancement was not anticipated. In what follows the effect of stress coarsening of semi-coherent γ' precipitates during the creep of a Ni-Mo-Al-Ta alloy, MMT 143 (9), is discussed.

PROCEDURE

Single crystals in predetermined orientations were produced by directional solidification, using the seeded Bridgman technique. The rate of solidification was 40 cm/hr for $\langle 100 \rangle$ oriented crystals and 20 cm/hr for $\langle 110 \rangle$ and $\langle 111 \rangle$ crystals. Under these conditions, the primary dendrite spacing was ~.02 cm for the $\langle 100 \rangle$ orientation and somewhat coarser for the other two orientations. All crystals were given a solution treatment above the γ' solvus at 1032°C for 16 hours to completely homogenize the cast structure. The time estimated to reduce the amplitude of microsegregation to 1% of the original value was obtained from the formula (10):

$$t = \frac{\lambda^2 \ln \delta}{4\pi^2 D} = \frac{0.12 \lambda^2}{D} \quad \text{for 1\% residual microsegregation } (\delta = 0.01)$$

for typical values

$$\lambda = .02 \text{ cm (primary dendrite spacing)}$$

$$D = .85 \exp \frac{-64400}{RT} \text{ cm}^2/\text{sec} \quad \left(\begin{array}{l} \text{diffusivity of} \\ \text{Mo in Ni} \end{array} \right)$$

$t \sim 10 \text{ hours}$

Photomicrographs of the alloy in the as-cast and solution treated condition are shown in Fig. 1. A microprobe trace of the homogenized alloy showed no residual microsegregation.

Creep tests were conducted in air using paired LVDT's attached to the specimen with clamps and alumina extension rods. Creep curves were obtained at 1038°C 207 MPa and at 899°C 414 MPa. The specimens were tested in two conditions. The first was in the air cooled condition after homogenization. In this state, the alloy consisted of a fine uniform distribution of γ' precipitates ($\sim 1\mu\text{m}$) produced during cooling (Fig. 2a). The second condition was one in which the alloy was given a post homogenization heat treatment of 4 hours at 1080°C followed by 16 hours at 870°C which simulates a typical coating cycle for turbine blades. This produced a coarser γ' distribution which also contained a high degree of misfit dislocations at the γ' particle interfaces (Fig. 2b).

DISCUSSION OF RESULTS

Typical creep curves for [100] [110] and [111] oriented specimens tested at 899°C and 1038°C are shown in Figs. 3 and 4. As can be seen, the alloy exhibits a substantial orientation dependence for creep response. At the high temperature, the [111] orientation exhibits the best creep life. In addition, a dramatic improvement of the creep life for the [100] orientation was observed at 1038°C when tested after being solution treated only. In this condition, the rupture life was increased by four times and the minimum creep rate was reduced to $\sim 10^{-5}\text{hr}^{-1}$, comparable with the [111] orientation. No improvement was observed in the [110] or [111] orientations when similarly tested in the solution treated condition. Some differences between the two heat treatments

Figure 1 Effect of Solution Treatment (1320°C , 16 hrs) on Chemical Homogeneity (a) As-cast (b) Solutionized

Figure 2 Effect of Heat Treatment on Size and Shape of γ' Precipitates (a) Solutioned/Air Cool (b) Aged 4 hrs. 1080°C /16 hr 870°C

were observed at 899°C for the [100] crystals but not nearly so dramatic as at 1038°C. The [111] orientation was again superior but not as much as at 1038°C.

The difference in the behavior of the [100] crystals was attributed to a finely spaced, lamellar array or 'raft' of γ' which apparently develops very early in the creep testing of these materials at 1038°C. Similar rafting occurs in the aged [100] specimens but it is not nearly so extensive laterally and much more coarsely spaced. The rafts were very stable as can be seen in Fig. 5 which was taken from a specimen which has been creep tested at 1038°C 207 MPa for 445 hours. Subsequent testing of these materials has shown two important features. Once the [100] crystals have been pre-'rafted' for 15 hours at 1038°C 207 MPa they can be heat treated isothermally at 1080°C for 4 hours and maintain their creep strength at 1038°C. Also, when pre-'rafted' at 1038°C they show improved creep resistance when tested at 899°C (Fig. 4).

As previously mentioned, the fine rafts of γ' which develop in this alloy are unusual both with regard to the fine spacing and to the lateral extent. This feature was attributed to the large lattice misfit ($a_0^{\gamma} \sim 3.61\text{\AA}$, $a_0^{\gamma'} \sim 3.585\text{\AA}$) which generates sufficient interfacial strain to produce misfit dislocations at elevated temperature (Fig. 2b). When the alloy was allowed to age or coarsen under an applied stress along the [100] axis, the combination of internal and external strain apparently leads to very rapid generation of fine rafts of γ' lamellae. This would provide an ideal structure for creep resistance in the [100] orientation because circumvention of the γ' phase by climb would be eliminated. Significant creep can occur only by dislocation penetration of the γ' phase and it is postulated that the misfit dislocation nets at the interface retards this process. Therefore, superior properties are obtained when the rafts are made as fine as possible. The performance of the [111] orientation is explained, in contrast, due to the low CRSS for {111} $\langle 110 \rangle$ slip in this orientation. Also, the interfacial dislocations might be expected to provide an additional drag stress on matrix dislocations migrating around the γ' particles.

Figure 3 1038°C, 207 MPa Creep Anisotropy of Aged (4 hrs 1080°C/16 hrs 870°C) Crystals Compared with Solutioned only [100].

Figure 4 899°C, 414 MPa Creep Anisotropy of Aged (4 hrs 1080°C/16 hrs 870°C) Crystal Compared with Solutioned only [100].

FUTURE PERSPECTIVE

The present work indicates that chemical homogeneity is desirable for achieving optimum creep properties in the single crystal alloy. Other work indicates that phase instability in alloy single crystals need not be detrimental with respect to creep properties, as it tends to be in polycrystalline materials due to grain boundary embrittlement. This suggests a new direction in alloy design specifically for homogeneous single crystals, in which phase instability in the solid state is deliberately introduced and exploited to obtain controlled polyphase structures with improved high temperature properties. An example of such a structure occurs in an alloy containing more Ta than MMT 143, which can be induced to precipitate Ni_3Ta (δ -phase) by annealing at high temperatures. The resulting structure consists of $\gamma+\gamma'$, boxed in by a three dimensional interpenetrating network of Ni_3Ta lamellae. Uniform microstructures of this type are possible only in perfectly homogeneous single crystal alloys. Even so, the presence of dislocation sub-boundaries does modify the precipitate morphology. It remains to be shown in future work to what extent the presence of dislocation sub-boundaries influences the properties of polyphase single crystal alloys.

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Figure 5 Rafted γ' Structure Observed in [100] Creep Specimen (1038°C 207 MPa and 450 hrs).

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