THE EFFECT OF GRAIN SHAPE ON STRESS RUPTURE OF THE OXIDE DISPERSION STRENGTHENED SUPERALLOY INCONEL MA 6000

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Summary

Creep specimens of Inconel MA 6000 with different grain aspect ratios (GAR) ranging from about 4 to 60 were tested at 950° C at a stress of 230 MPa. The resulting rupture times show a pronounced dependence on the GAR below a value of about 20, where fracture is mainly intergranular. At higher GAR values, the rupture time becomes less sensitive to grain shape, and fracture is predominantly transgranular. These results are interpreted in the light of the hypothesis that sliding of the elongated grains along longitudinal grain boundaries, which is a necessary accommodation mechanism, controls the rate of creep damage accumulation on boundaries perpendicular to the applied stress. A quantitative model for the coupling between the two processes is presented. If it is assumed that the oxide dispersoid introduces a threshold stress for sliding, then the transition in the fracture mode with increasing GAR can be explained quantitatively: The reason is that, above a critical GAR of about 20, the damage-induced shear stress in the longitudinal boundaries lies below the threshold, and sliding and damage accumulation on transverse boundaries are suppressed. This approach results in a quantitative description of the beneficial interaction between elongated grain shape, boundary waviness and oxide dispersion.

Introduction

Elongated grains are an effective additional strengthening element in oxide-dispersion strengthened (ODS) superalloys (1-3). Wilcox and Clauer (4) were the first to show that grain shape is more decisive for the high-temperature behaviour of ODS materials than grain size: creep strength scaled with the grain aspect ratio (GAR), which is defined as GAR = L/ℓ (where L and ℓ denote the long and the short grain diameter, respectively); by contrast, no correlation was found with the grain size. Whittenberger (5) discovered threshold stresses below which the creep rates of several ODS alloys were negligible; these thresholds varied, in a linear manner, with the GAR.

It has been suggested (4, 5) that grain boundary sliding must be responsible for this influence of grain shape on the deformation behaviour, but the explanation still appears incomplete: how is sliding along boundaries which are oriented in directions of negligible resolved shear stress brought about? And, can its rate determine the rate of overall deformation?

The aim of the present investigation was to deliberately vary the grain shape in the ODS superalloy Inconel MA 6000 and to study its effect on stress rupture. The results are compared with predictions from a model which assumes that sliding of grains along longitudinal boundaries controls the rate of damage accumulation on the boundaries perpendicular to the applied stress. In this way a quantitative picture of the role played by grain shape in creep and stress rupture is developed.

Experimental

As-extruded bars of MA 6000 were recrystallized by zone annealing at various temperatures and bar travel rates. These treatments produced different grain geometries, with GAR values ranging from about 4 to 60. Creep specimens with a diameter of 4 mm and a gauge length of 24 mm were machined from the recrystallized bars and given the standard heat treatment. Stress rupture tests were performed at 950° C and a stress of 230 MPa, acting in the longitudinal grain direction. The fractured specimens were analyzed by light microscopy and in the SEM.

Results

Fig. 1 shows the rupture time as a function of the GAR. It is seen that the effect of grain shape is quite dramatic at low GAR: increasing the GAR from 4 to 40 results in a prolongation of creep life from about 1 h to more than 100 h. At higher GAR values, the rupture life becomes insensitive to grain shape.

The fracture mode, too, depends on the GAR: it is predominantly intergranular at low GAR, with fracture occurring by pull-out of individual grains (fig. 2a), whereas a high GAR results in mainly transgranular fracture (fig. 2b). Metallographic sectioning reveals copious damage on transverse boundaries in specimens with low GAR (fig. 3). Above a GAR of about 20, no signs of cavitation were detected on grain boundaries.

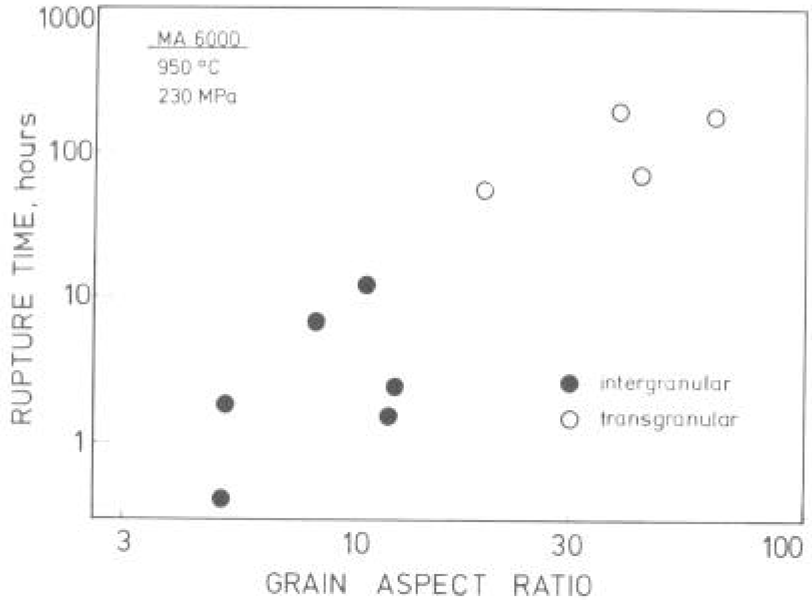

Figure 1 - Rupture time vs. grain aspect ratio (GAR) for MA 6000 at 950° C and 230 MPa. Note the strong grain shape sensitivity, and the transition from intergranular (full circles) to transgranular (open circles) fracture

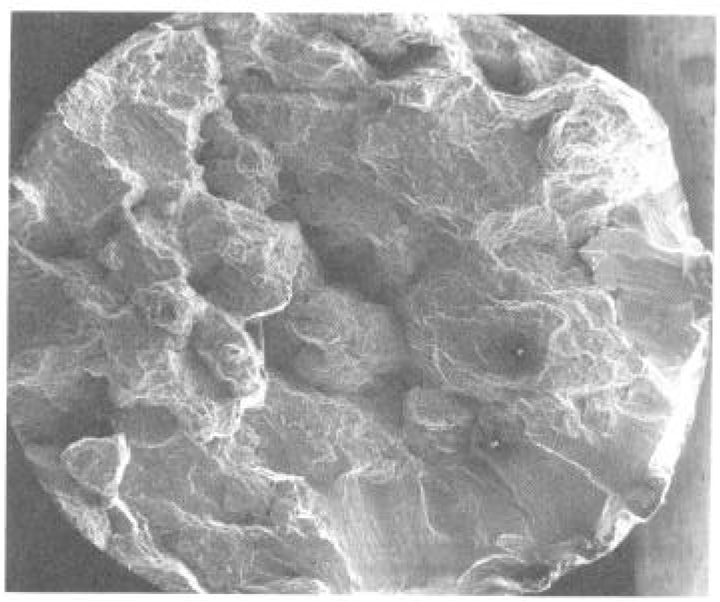

Figure 2a - SEM micrograph showing the fracture surface of a specimen with a GAR of 5: fracture is predominantly intergranular ("pull-out" fracture)
(specimen diameter 4 mm)

Figure 2b - SEM micrograph showing the fracture surface of a specimen with a GAR of 60: fracture is predominantly transgranular (specimen diameter 4 mm)

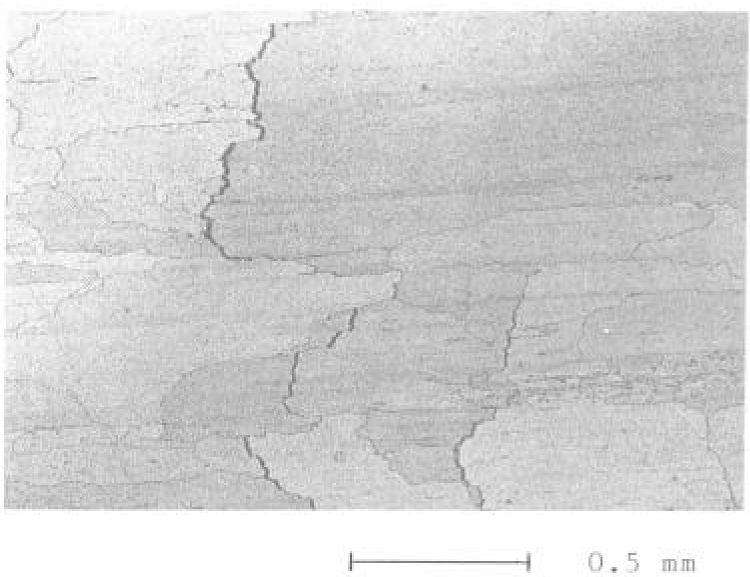

Figure 3 - Creep damage on grain boundaries perpendicular to the applied stress (acting horizontally), typical of specimens with low GAR.

Discussion of Possible Mechanisms

Because the GAR and the absolute grain lengths produced during recrystallization were correlated, we cannot decide unambiguously from our data whether an increase in grain length or in GAR is more beneficial to the stress rupture behaviour. In principle, grain size and grain shape can improve the creep properties of polycrystals in the following ways (which will be discussed in turn):

- by retardation of diffusional creep
- by retardation of grain boundary sliding and switching of grain neighbours
- by retardation of creep damage accumulation

Diffusional creep is slow in large grains, because the distances between vacancy sinks and sources are long. An elongated grain shape slows down diffusional creep even more, as the internal accommodation of the grain strains by grain boundary sliding becomes more difficult (6) and the altered geometric boundary conditions modify the diffusion fields (7, 8). A rough estimate of the strain rate provided by diffusional creep in our material is 10^{-12}/sec (with data from (9)); this represents even an upper bound, because we ignore that grain boundaries in particle-strengthened materials are inefficient sinks and sources for vacancies (10). In conclusion, we can neglect the contribution of diffusional creep to the total deformation in our tests.

Large and elongated grains expose less grain boundary area to the maximum resolved shear stress. Thus grain boundary sliding and grain neighbour switching as primary deformation mechanisms are slowed down. Such an explanation has originally been put forward by Wilcox and Clauer (4) and is discussed by Sellars and Petkovic-Luton (11). It is difficult, however, to envisage such a deformation mode in grain structures with a GAR of two or three; yet our results show that the stress rupture behaviour still improves beyond a GAR of 10. Furthermore this effect should be equally important in transverse loading, yet it is well known that the creep properties of MA 6000 are markedly inferior in that direction.

This leaves the effects of grain shape and size on the initiation and propagation of fracture on our list. Three such effects can be imagined. First, the nucleation of creep cavities on transverse boundaries will become more improbable as the density of grain boundary surface perpendicular to the stress decreases. The importance of this effect is difficult to estimate, but since we have found copious cavitation in specimens with low GAR, void nucleation is not thought to be the controlling process.

Second, the very last stage of intergranular fracture is dominated by the pull-out of individual grains, much like fibre pull-out in composite materials. The applied stress is then carried only by the longitudinal boundaries, which slide at a rate determined by the shear traction, which in turn decreases with increasing GAR. However, the pull-out stage occupies such a negligible portion of the total rupture life that it would be unreasonable to explain a grain shape effect in this way.

The third, and in our view most important effect is related to the accumulation of creep damage by growth of intergranular voids on transverse boundaries. Several mechanisms for void growth exist - for a recent review see (12): diffusion processes, which require accommodation by grain boundary sliding or matrix deformation; power-law creep; and coupled processes. If matrix deformation is difficult (as in MA 6000), it is reasonable to assume that cavity growth occurs by diffusion and the necessary accommodation is achieved by sliding of the elongated grains along the longitudinal grain boundaries. In the limit of sliding-controlled cavity growth a grain shape dependence of the rupture life follows in a natural way (13, 14). This point will be expanded in the following.

A Model for Grain Sliding Controlled Creep Cavitation

We assume that the grains behave like undeformable fibres or deform homogeneously without influencing the fracture process at grain boundaries. The grain separation in the longitudinal direction increases as damage in the form of cavities is developed on transverse boundaries. This produces local incompatibilities between neighbouring grains; for cavity growth to continue, accommodation by localized sliding of the grains has to occur (fig. 4). Because some fraction of the applied stress is dissipated by this sliding process, the stress σ_{CG} driving cavity growth will be less than the applied stress σ:

$$\sigma = \sigma_{CG} + 2\,\tau_{GB}\,\mathrm{GAR} \tag{1}$$

where τ_{GB} is the shear stress driving grain boundary sliding and GAR the grain aspect ratio. Compatibility requires the displacement rates due to cavity growth ($\dot{u}_{CG}$) and that due to sliding ($\dot{u}_{GB}$) to match:

$$\dot{u}_{CG} = \dot{u}_{GB} \tag{2}$$

Suppose that both cavity growth and sliding are controlled by grain boundary diffusion, which leads to the following approximate equations (12, 6):

$$\dot{u}_{CG} = \frac{C_1\ \delta D_b \Omega\ (\sigma_{CG} - \sigma_0)}{kT\ \ \lambda^2} \tag{3}$$

$$\dot{u}_{GB} = \frac{C_2\ \delta D_b \Omega\ (\tau_{GB} - \tau_0)}{kT\ \ h^2} \tag{4}$$

Here δD_b is the boundary width times its diffusivity, Ω the atomic volume, λ the planar spacing of the cavities in the grain boundaries, h the average height of boundary steps or serrations, and C_1 and C_2 constants of order 1..10. Also included in the equations are a threshold stress σ_0 for cavity growth and a threshold shear stress τ_0 for grain boundary sliding, both of which may arise in particle-strengthened materials because of the influence of hard particles on the vacancy sink and source action of grain boundaries (10, 15, 16).

The resulting strain rate for cavity growth coupled with grain boundary sliding is the solution of eqs. 1 to 4, related to the grain length L:

$$\dot{\varepsilon}_{coupled} = \frac{C_2\ \delta D_b\ \Omega}{kT\ h^2\ L} \left(\frac{\sigma - \sigma_0 - 2\,\tau_0\,\mathrm{GAR}}{\lambda^2/h^2 + 2\,\mathrm{GAR}} \right) \tag{5}$$

This is an equation for grain boundary sliding with an effective shear stress given by the expression in brackets. It has two simple limits: when

$$\lambda^2/h^2 \gg 2\,\mathrm{GAR} \tag{6}$$

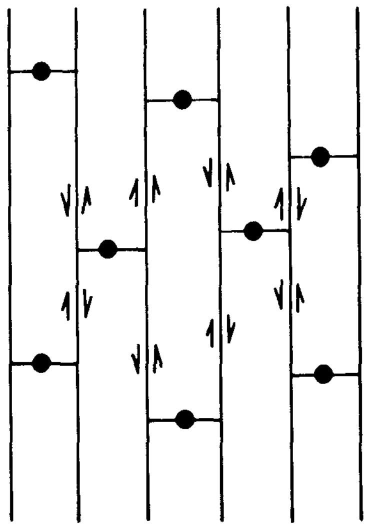

Figure 4 - Cavity growth on transverse boundaries produces incompatibilities between neighbouring grains which have to be eliminated by local grain boundary sliding in the longitudinal direction

(i.e. equiaxed grains, few cavity nuclei, smooth boundaries), cavity growth is uninhibited by grain boundary sliding. If, on the other hand,

$$\lambda^2 / h^2 \quad << \quad 2 \text{ GAR} \tag{7}$$

(elongated grains, many cavities, stepped boundaries), cavity growth is limited by the rate of grain boundary sliding. The strain rate which is then produced by the formation of damage is given by:

$$\dot{\varepsilon} = \frac{C_2 \; \delta D_b \; \Omega \; (\sigma - \sigma_0 - 2 \; \tau_0 \; \text{GAR})}{2 \; kT \; h^2 \; L \; \text{GAR}} \tag{8}$$

This equation is independent of the particular mechanism of cavity growth; it holds whenever grain boundary sliding is the controlling process.

The rupture time is now estimated as the time to reach a critical amount of strain due to cavity growth (12):

$$\varepsilon_f = 0.2 \; \lambda / L \tag{9}$$

Thus the time-to-fracture t_f is obtained:

$$t_f \simeq \frac{kT \; h^2 \; \lambda \;\; \text{GAR}}{\delta D_b \; \Omega \; (\sigma - \sigma_0 \; - 2 \; \tau_0 \; \text{GAR})} \tag{10}$$

When applying this concept of boundary sliding controlled damage accumulation to our experiments, we have to ask whether it is reasonable to assume that grain sliding was the rate-controlling process. While this possibility is usually dismissed in equiaxed grains, the requirement stated in eq. 7 is more easily met in elongated grains with a high GAR and an irregular boundary shape, both of which are characteristic features of the microstructure of commercial MA 6000.

Interpretation of Experimental Results

In the light of the above analysis the following picture of the influence of grain shape on stress rupture of MA 6000 can be developed (fig. 5). At low GAR, the grains slide readily past each other and intergranular fracture by cavity growth is unimpeded. As the GAR increases, the shear stress in the longitudinal boundaries, which is caused by cavity growth on transverse boundaries, decreases towards the threshold stress for sliding. The limiting GAR at which sliding is suppressed and transgranular fracture supervenes is predicted by eqs. 8 and 10:

$$GAR_{transition} = \frac{\sigma - \sigma_0}{2\ \tau_0} \simeq \frac{\sigma}{2\ \tau_0} \qquad (11)$$

Comparing the actual value with this prediction, we find that a threshold stress $\tau_0 \simeq 6$ MPa is required to obtain agreement. Such a magnitude is indeed realistic: results of bicrystal sliding experiments by Petkovic-Luton et al. (17) on Incoloy MA 956, a ferritic superalloy containing the same oxide dispersion, suggest, when replotted with appropriate axes, a threshold of about 5 MPa. Furthermore, a theoretical estimate, based on a formalism developed for describing the interaction of grain boundary dislocations with hard particles (10), yields a value of the same order of magnitude.

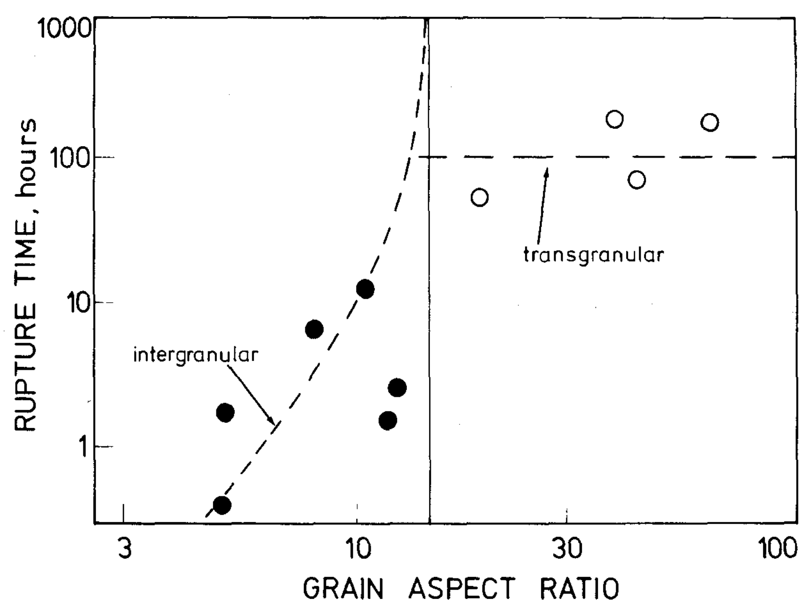

Figure 5 - Interpretation of our experimental results (see text above for details)

There is further evidence for such a threshold stress in the data obtained on several ODS alloys by Whittenberger (5), who finds a linear dependence of the threshold on the GAR. The results are in perfect quantitative agreement with the prediction of eq. 8, with $\tau_0 = 9$ MPa and $\sigma_0 = 6$ MPa.

Conclusion

The results of our stress rupture tests on Inconel MA 6000 with widely varying GAR confirm earlier conclusions that a minimum GAR (roughly between 15 and 20) is needed in order to take full advantage of the dispersion strengthening. The explanation given in this paper is based on the idea that the accumulation of creep damage on transverse boundaries, and hence the rupture time, can be controlled by an accommodation process, i.e. mutual sliding displacements between neighbouring grains in the longitudinal direction. Both an elongated grain shape and boundary serrations contribute to the retardation of this process, and thus to a prolongation of creep life. This appears to be the most natural explanation for the strong influence of the GAR on rupture time and fracture mode. Under transverse loading, accommodation of the local displacements due to damage accumulation is unnecessary, and cavity growth is uninhibited; this can account for the inferior creep properties in that direction.

The oxide dispersoid plays a dual role in the deformation and fracture of ODS alloys: not only does it strengthen the grain interiors, but it can also inhibit the absorption and emission of vacancies at grain boundaries. This latter effect implies a threshold stress for grain boundary sliding by diffusion, which translates into a threshold stress in tension proportional to the GAR. At GAR values greater than a critical value, the shear stress driving sliding falls below the threshold and intergranular fracture is suppressed. It is this low-GAR regime which has to be avoided in practical applications.

Our model calculations lead to equations for the coupling between diffusion-controlled sliding and cavity growth; they could in principle be extended to include other mechanisms like power-law creep. To decide whether this is necessary, further experimental work is in progress with the aims to clarify the micromechanisms of damage accumulation, sliding, and transgranular fracture, and to quantify the importance of the effects which have been treated theoretically in this paper.

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