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WEAK BEAM STUDIES OF DISLOCATION/DISPERSOID INTERACTION IN AN ODS SUPERALLOY

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Introduction

Oxide dispersion strengthened (ODS) superalloys are promising candidate materials for high temperature applications (above 1000 °C), for example as gas turbine blades /1/. Their outstanding high temperature strength is due to a fine dispersion of stable incoherent particles, which act as non-shearable barriers to dislocation movement up to high temperatures. The exact mechanism of this dispersoid/dislocation interaction is unclear at present.

In principle, high temperatures enable the dislocations to surmount the oxide particles by thermally-activated climb. A "threshold stress" for creep was calculated by Brown and Ham /2/ and Shewfelt and Brown /3/ under the assumption that the dislocations climb "locally" at the particles, i.e. that climb is confined to the region in or near the particle/matrix interface. This process should lead to high dislocation curvatures at those points where the dislocation leaves the particle/matrix interface. In contrast, it has been argued by Lagneborg /4/ among others that the line tension of the dislocation will always tend to avoid points of such high curvature. According to this argument the dislocation is more likely to leave its glide plane at all locations up to a certain distance from the dispersoid; this is called "general climb". The actual dislocation geometry is important for understanding the high-temperature strength achievable by dispersion strengthening: general climb requires stresses about an order of magnitude lower than does local climb /5/.

In all of these models, the particles are just treated as impenetrable obstacles forcing the dislocations to climb around them, with no concern for the nature of the particle/dislocation interaction. According to calculations by Srolovitz et al. /6,7/, incoherent hard dispersoids can exert an attractive force on dislocations at high temperatures; the reason is that the particle/matrix interface, unable to sustain shear tractions, relaxes parts of the dislocation's stress field - to what extent is at present unknown. Recent TEM investigations by Nardone and Tien /8,9/ in an ODS superalloy do in fact give the impression that the dislocations stick to the "departure" side of the oxide particles. The evidence, however, is scarce and the dislocation contrast in the vicinity of the dispersoid appears somewhat ambiguous.

One reason for this is that bright field TEM micrographs are usually insufficient to reveal the exact dislocation configuration in the vicinity of the dispersoids. This note presents dark field micrographs which show, owing to the weak-beam technique employed, more clearly the dispersoid/dislocation configurations and shed more light on the mechanism of dispersion strengthening in an ODS superalloy at high temperatures.

Experimental

For our investigations, the γ'-forming ODS superalloy Inconel MA 6000 (for the composition according to INCOMAP see TABLE 1) was crept, and analyzed by TEM. Its oxide dispersion has been characterized in detail /10/. The dispersoids are nearly spherical with a mean diameter of about 30 nm and a size distribution as shown in fig. 1; their mean spacing is about 100 nm.

Constant load compression tests were carried out at temperatures high enough for the oxide dispersion, rather than the γ' precipitates, to be strength-determining. The stresses were deliberately kept below the Orowan stress, as calculated with the equations given by Brown and Ham /2/ and Lund and Nix /11/ (see fig. 2).

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TABLE 1

Nominal Chemical Composition of Inconel MA 6000 (in wt-%)

Ni	Cr	W	Mo	Al	Ti	Ta	C	B	Zr	Y_2O_3
69	15	4	2	4.5	2.5	2.0	0.05	0.01	0.15	1.1 (2.5 vol-%)

We used cylindrical samples (3 mm in diameter, 6 mm in height), with the stress axis perpendicular to the extrusion direction of the bar. After a test duration of about 2 h, which resulted in a strain of about 1.5 %, the specimens were cooled under load. For comparison, stress rupture specimens (950 °C, 220 MPa) in which the load had dropped to zero during fracture at temperature were also analyzed.

TEM investigations

TEM foils were thinned in a Tenupol jet polisher at -28 °C and 40 V, using an etchant of ethanol with 7.5 % perchloric acid. The microstructural observations were carried out on a JEOL 200 CX transmission electron microscope. The dislocations were imaged in the dark field mode under weak-beam conditions /12/, with the (4g)-reflection on the Ewald sphere and the weak (1g)-reflection in the optical axis of the microscope. The image was focussed in bright field and then the beam-deflecting system was switched to dark field mode. Exposure times of about 30 s were required, which called for high specimen and image stability.

Observations of dispersoid/dislocation configurations

In the crept specimens the distribution of dislocations appeared to be very inhomogeneous. Large dislocation-free regions alternated with regions of appreciable dislocation density, which was always low enough, however, to allow the interaction of single dislocations with the incoherent disperoids to be studied. These detailed investigations required magnifications of about 300000 times.

The merit of the weak-beam technique is illustrated in fig. 3. While bright field imaging leaves the exact position of the dislocation segment at the dispersoid largely open to speculation (fig. 3a), the weak-beam image displays a clear dislocation contrast even in the vicinity of the particle (fig. 3b).

The peculiar configuration of fig. 3b was by no means a singular observation. Where a dislocation came in contact with a dispersoid, a similar configuration was often seen (see further examples in figs. 4 and 5). From these repeated observations the following typical features emerged:

1) The identity of the dislocation is preserved in (or near) the dispersoid/matrix interface. Core spreading, if it occurs at all, affects only a component of the distortion field.

2) The dislocation is bent to a high curvature at the points where it leaves the dispersoid/matrix interface. This reflects exactly the geometry postulated in the "local climb" models /2,3/.

3) The curvatures of the dislocation in the matrix and of the segment at the dispersoid have the same sign. This suggests that the dislocations have already surmounted the particles by climb and are at a late stage of the climb process.

4) Hardly ever has a dislocation at the beginning of climb, with the segment's curvature opposite to that of the free dislocation arm, been observed.

Qualitatively the same features were found in the stress-rupture specimens.

Discussion

The evidence presented above makes a strong case for the "local climb" process as modelled by Shewfelt and Brown /3/. The criticism voiced against it on the grounds that points of high dislocation curvature would be unstable is weakened substantially by the actual observation of such "high-energy" configurations. Under our experimental conditions, "general" climb can be ruled out.

The assumption of local climb is, however, insufficient to explain all our observations. The micrographs give the impression of an attractive interaction between the dislocations and the oxide particles, as was suggested earlier on more ambiguous microstructural evidence /8,9/. Such a conclusion merits closer scrutiny: in principle, the possibility that the dislocation segment has not yet reached the original glide plane and is therefore not yet mobile, cannot be discarded altogether. But the following considerations lend support to the attraction hypothesis: suppose that local climb occurs without any attractive force, then according to the Shewfelt-Brown model the dislocation should be spending more time climbing "up hill" (where dL/dx, the line length increment, is positive) than "down hill" (where dL/dx becomes negative). In contrast, an attractive force, which may well be favouring the "local" nature of climb, could inhibit the process of dislocation break-away after climb has been completed. Our micrographs, showing exclusively the configuration at (or towards) the end of the climb process, clearly support the assumption of local climb under mutual attraction.

In our view, it is also significant that a small particle is just as effective a dislocation barrier as a larger one (see fig. 4). The theory /3/ predicts that the time for by-pass by climb scales as R^2 (for bulk diffusion) or R^3 (for dislocation core diffusion) allowing the smaller particle in fig. 4 to be surmounted in about 1/30 to 1/10 of the time required for by-pass of the larger one. Yet, at both particles the dislocation has advanced to roughly the same position. Further, the time calculated for by-pass according to eq. 8 in /3/ is several orders of magnitude smaller than that permitted on the basis of the strain rate measured in creep specimens under the same conditions /10/.

All this suggests that in our alloy and under the experimental conditions studied the climb process does not determine the average dislocation velocity or the strength. Dislocation detachment from the incoherent dispersoid seems to be more important, and a model for the resulting "threshold stress" will be published elsewhere /13/.

The origin of the dispersoid/dislocation attraction could lie in the relaxation of the dislocation stress field, but the observation of well-defined dislocation contrast at the dispersoids precludes any large-scale core spreading at the interface. This spreading and its temperature dependence are the objects of current work.

A final word of caution is in order: we cannot be absolutely certain that the dislocation configurations as observed in the TEM are identical to those effecting deformation at high temperatures. In view of the insensitivity of the configurations to the cooling and unloading procedure (cooling under load in the compression tests vs. unloading at temperature in the stress rupture tests) we are confident that the modifications induced are not critical. On the contrary, the presence of 50 vol-% ordered γ' in this alloy would seem to stabilize single dislocations against spring back during unloading of the compression samples at room temperature, which adds an extra degree of confidence in the validity of our observations.

Conclusions

TEM studies employing the weak-beam technique have revealed interesting details of the interaction of dislocations with incoherent dispersoids in crept specimens of the ODS superalloy Inconel MA 6000:

1. A clear dislocation contrast is produced even in (or near) the dispersoid/matrix interface, with no signs of extensive core spreading.
2. The micrographs repeatedly confirm the presence of points of high dislocation curvature where the dislocation leaves the particle/matrix interface - a hitherto much disputed feature typical of particle by-pass by "local" climb.
3. The evidence supports the concept of an attractive interaction between dislocations and the dispersoids, which seems to determine the high-temperature strength of ODS alloys.

Acknowledgement

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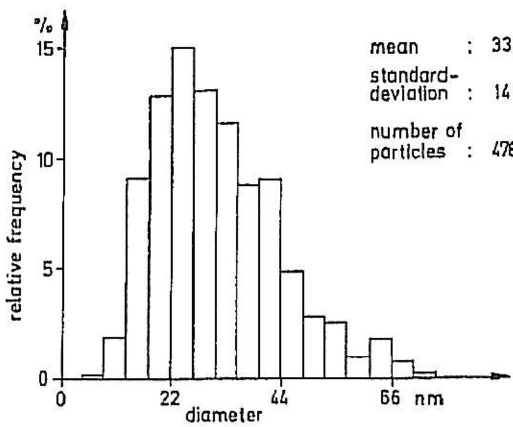


FIG. 1

Size distribution of oxide dispersoids in Inconel MA 6000.

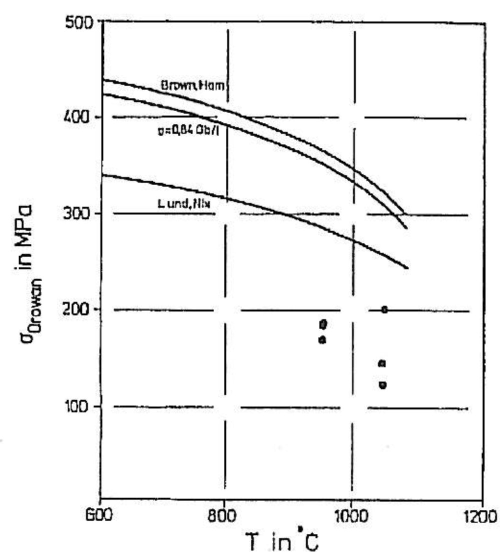


FIG. 2

Stress and temperature during compression tests (circles), compared with the Orowan stress calculated by the equations of Brown and Ham /2/ and Lund and Nix /11/ (lines).

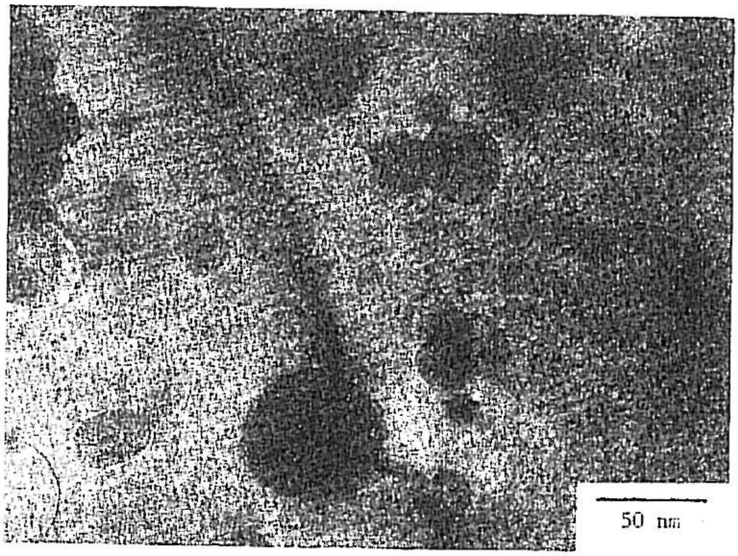


FIG. 3a
Bright-field image of a dislocation at an oxide dispersoid. The dislocation contrast at the dispersoid remains ambiguous.

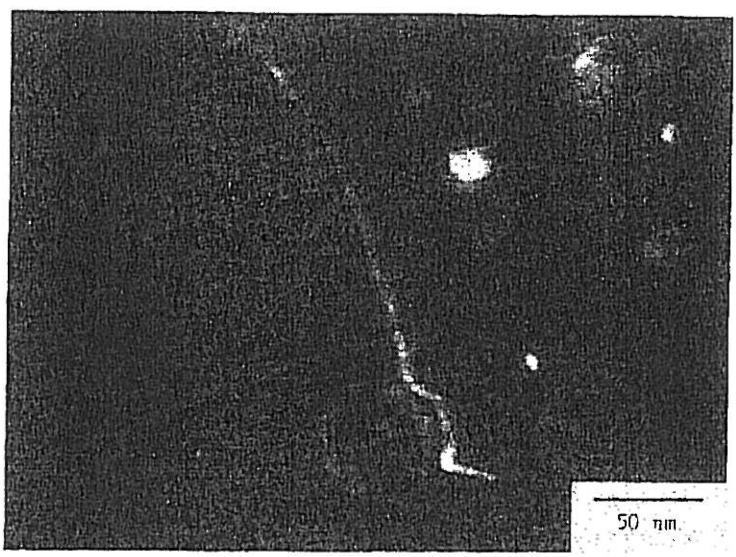
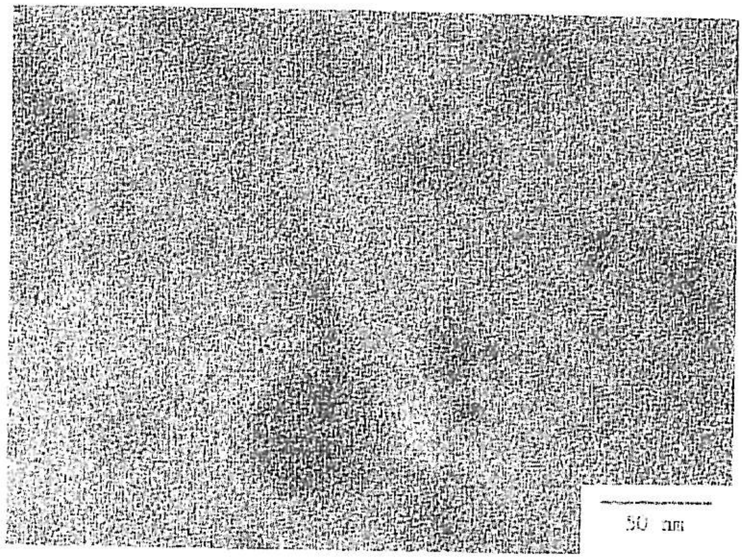


FIG. 3b
Weak-beam image of the same configuration as in fig. 3a. Note the clear dislocation contrast at the dispersoid.



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Bright-field image of a dislocation at an oxide dispersoid. The dislocation contrast at the dispersoid remains ambiguous.

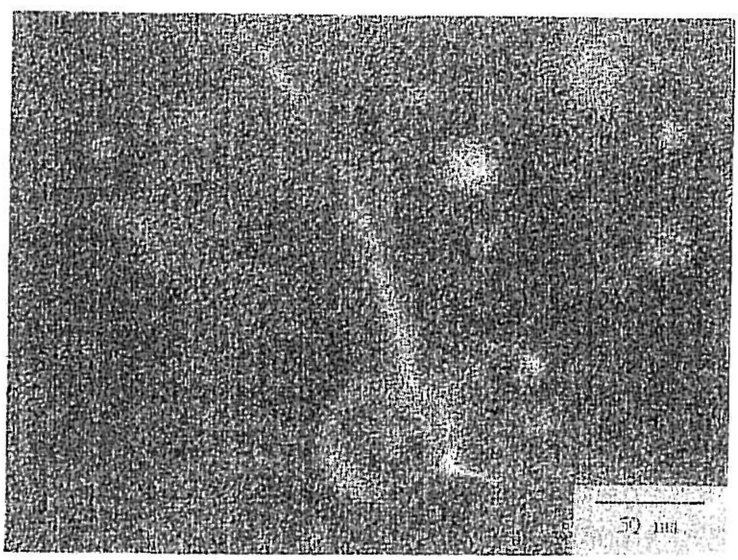


FIG. 3b

Weak-beam image of the same configuration as in fig. 3a. Note the clear dislocation contrast at the dispersoid.

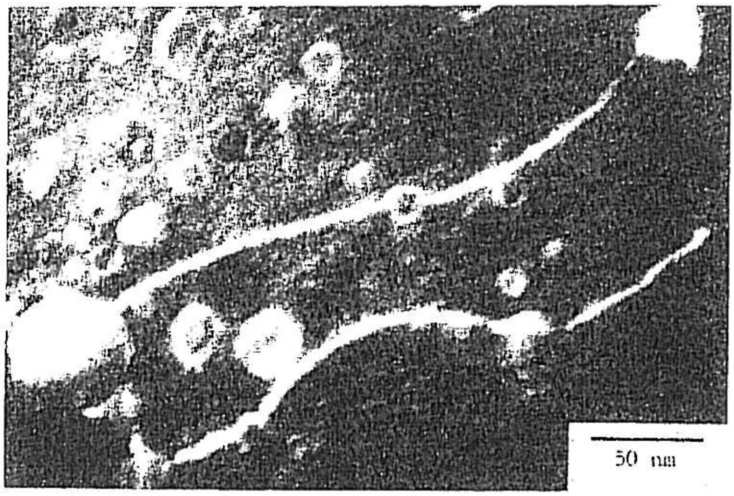


FIG. 4
Weak-beam image of dislocations held back, at the same time, by oxide dispersoids of different size.

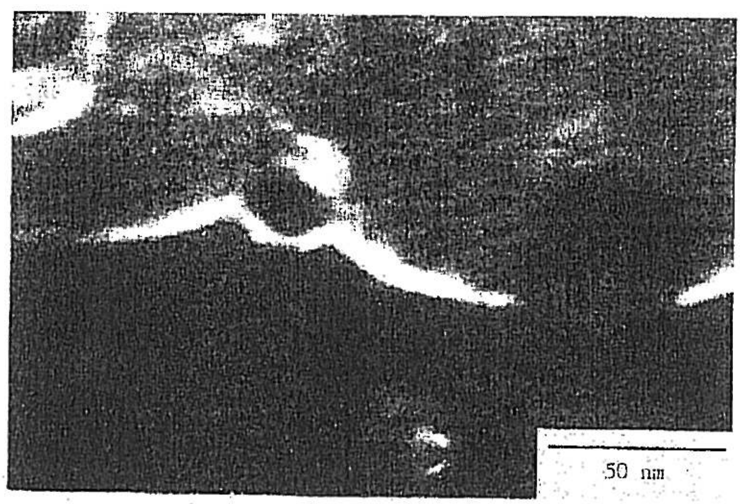
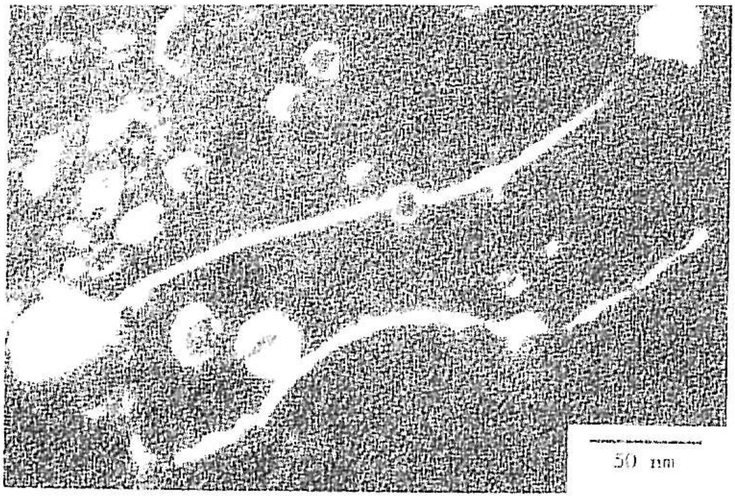


FIG. 5
Another weak-beam image of the dislocation/dispersoid configuration.



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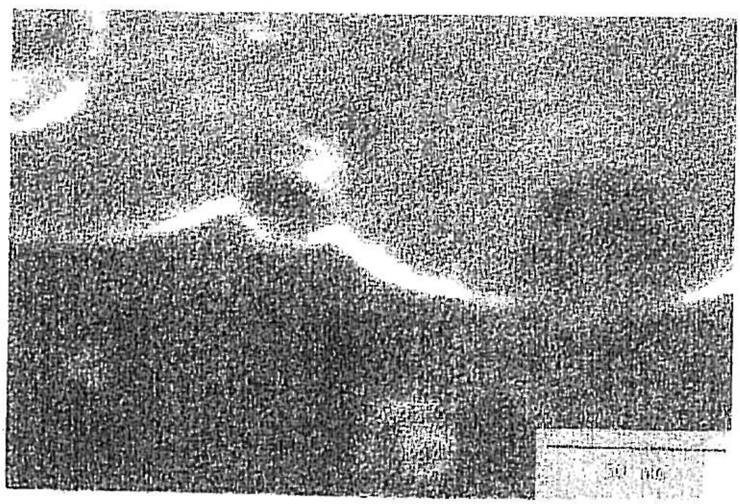

FIG. 5
Another weak-beam image of the dislocation/dispersoid configuration.