HIGH TEMPERATURE STRENGTH OF ODS SUPERALLOYS DUE TO DISPERSOID-DISLOCATION INTERACTION

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ABSTRACT. The microstructure of the oxide-dispersion strengthened (ODS) superalloy Inconel MA 6000 was studied by TEM after creep and high-temperature low-cycle fatigue. The micrographs suggest that an attractive interaction between dislocations and dispersoid particles exists at high temperatures, which makes the detachment of the dislocations from the dispersoids the strength-determining process. These observations are shown to be compatible with a newly developed theory incorporating an attractive interaction in the treatment of particle bypass by dislocation climb. Preliminary comparison between theoretical predictions and experimentally measured "threshold stresses" for creep exhibits promising agreement.

1. INTRODUCTION

The high-temperature mechanical behaviour of oxide-dispersion strengthened (ODS) alloys is characterized by a strong stress sensitivity: when stressed below a certain threshold value, the creep rates become negligible and the rupture lives exceedingly large, see e.g. /1-5/. While a distinct yield stress at room temperature can be attributed to the onset of particle bypass by dislocations according to the Orowan mechanism, threshold stresses at high temperatures - which enable the dislocations to circumvent hard particles by climbing around them - are more difficult to understand. At present the exact details of this mechanism, and its correlation with experimentally measured threshold stresses, are still unclear. In order to predict high temperature creep strength from the properties of the particle dispersion, and to improve it in a rational way, such knowledge would be essential.

Brown and Ham /6/ and subsequently Shewfelt and Brown /7/ have put forward theoretical models which suggest 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 requires an increase in dislocation line length whose energy must be supplied by the remote shear stress. A computer simulation of a dislocation passing through a random array of hard particles showed that deformation does not occur below a threshold stress in tension given by:

$$\sigma_{th} = 0.32 \quad M \, \frac{G\,b}{2\,\lambda} \tag{1}$$

G is the shear modulus of the matrix, b the magnitude of a lattice Burgers vector, 2λ the mean planar spacing of the particles, and M the Taylor factor (for polycrystals) or the reciprocal of the Schmid factor (for single crystals). A similar result was obtained analytically by Arzt and Ashby /8/ and other climb models have been developed along similar lines /9-11/.

From these considerations it generally follows that the assumption of "local climb" explains a threshold stress which amounts to roughly 0.4 times the "Orowan stress" defined by:

$$\sigma_{Or} = 0.84 \quad M \, \frac{G\,b}{2\,\lambda} \tag{2}$$

The model of local climb has been criticized /12/ on the grounds that a dislocation will tend to avoid points of extremely high curvature which appear during local climb at the particle/ matrix interface. Instead, the dislocation will leave its glide plane at all locations up to a certain distance from the particle. Because the length of the climbing segment is expected to increase with decreasing strain rate (or stress), a "threshold stress" which scales with the applied stress is predicted. The consequence of this "general climb" model is that for steady-state deformation a true threshold stress should not exist. Yet, in order to pass through a volume with a random array of dispersoids, the dislocation has to adopt a certain minimum curvature even when undergoing "general climb". Therefore a threshold stress of finite value always exists in the presence of hard particles but its numerical value is at least one order of magnitude below that for local climb /8, 9, 13/. Therefore the dilemma that remains in explaining threshold stresses in ODS alloys on the basis of current climb models is the following: General climb yields numerical values much too low compared with experimental results, while the physical justification for "local climb" rests on shaky ground.

As part of the COST 501 programme on ODS superalloys, the microstructure of Inconel MA 6000 after creep and high-temperature low-cycle fatigue (LCF) exposure has been studied in detail, with particular attention being paid to the dislocation configurations in the vicinity of the dispersoids. In this paper the surprising results of these investigations will be described and discussed in the light of a newly developed theory for the dispersoid-dislocation interaction at high temperatures. In short, there is strong evidence suggesting that the strengthening effect of the dispersoids, at high temperatures, is not due to the dislocations being forced to circumvent the disersoids by climb, but due to an attractive interaction which makes dislocation detachment from the dispersoid the strength-determining process. Some implications for maximizing threshold stresses in ODS alloys on the basis of these results will be discussed.

2. EXPERIMENTAL

The study was carried out on Inconel MA 6000, a γ'-forming ODS superalloy with a nominal composition as shown in TABLE I. The incoherent Y_2O_3 dispersoids, which are nearly spherical, have a mean diameter of about 30 nm, with a distribution ranging from about 10 to 70 nm, and a mean spacing of about 100 nm. At room temperature, they produce significant misfit strains in the surrounding matrix, which can however be considered to be relaxed at creep temperatures /14, 15/.

TABLE I
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	.05	.01	.15	1.1 (2.5 vol.-%)

Constant load compression tests were conducted on cylindrical specimens (3 mm in diameter, 6 mm in height), with the stress axis perpendicular to the grain elongation. Test temperatures were 850, 950 and 1050°C, and the stresses amounted to about half the temperature-corrected Orowan stress. After a test duration of about 2h, giving a strain of roughly 1.5 %, the specimens were cooled under load. For comparison, stress rupture specimens provided by COST 501 collaborators were also analyzed. Furthermore, the microstructure of specimens subjected to LCF was examined.

Foils for transmission electron microscopy were prepared by thinning in a Tenupol jet polisher at -28°C and 40 V, using an electrolyte of ethanol with 7.5 % perchloric acid. The microstructural observation were carried out with a JEOL 200 CX transmission electron microscope. Using a weak-beam technique in addition to bright-field imaging, a clear dislocation contrast in the vicinity of the dispersoid-matrix interface could be obtained in most micrographs /16/.

3. DISLOCATION CONFIGURATIONS IN THE VICINITY OF DISPERSOIDS

Dislocations were found to be very inhomogeneously distributed in the crept and fatigued specimens. Large dislocation-free regions alternated with zones of significant dislocation density, which was always low enough to allow the interaction of single dislocations with the dispersoids to be investigated.

Typical micrographs are shown in figs. 1 and 2, from which the following striking features have been identified as being representative for the dislocation configurations developed during slow high-temperature deformation (see also /16/):

a) The identity of the dislocations in (or near) the dispersoid-matrix interface is preserved in all but a few instances; core spreading therefore affects only a minor component of the distortion field.

b) The dislocations are bent to a high curvature at the points where they leave the dispersoid-matrix interface, suggesting an attractive interaction between the dislocations and the dispersoids.

c) The dislocations in the matrix and the segments at the dispersoids are almost always curved in the same direction; this observation indicates that the dislocations have already surmounted the dispersoids by climb.

Qualitatively the same features were found at all three temperatures studied and also in the stress rupture specimens. In fatigued samples, a dislocation network which is anchored at dispersoids is seen to develop (fig.3). Again, on close inspection an attractive interaction between dislocations and dispersoids becomes evident (fig.4).

What is most striking in the micrographs is that most of them capture the dislocations *after* they have climbed over the dispersoids. This fact is strong evidence for an unexpected strengthening mechanism: not the climb process itself controls the average dislocation velocity, but the process of overcoming the attractive interaction with the dispersoid particles. How this detachment process can provide an additional mechanism for a threshold stress at high temperatures, is the subject of the following theoretical considerations.

4. THEORY FOR THE EFFECT OF AN ATTRACTIVE INTERACTION ON DISLOCATION CLIMB OVER DISPERSOIDS

The microscopic observations summarized in the preceding section necessitate the incorporation of a dispersoid-dislocation attraction in the models for dislocation climb. The attractive interaction is easily modelled by assigning a reduced line tension $k \cdot E$ to the dislocation segment in the vicinity of the particle, compared with the

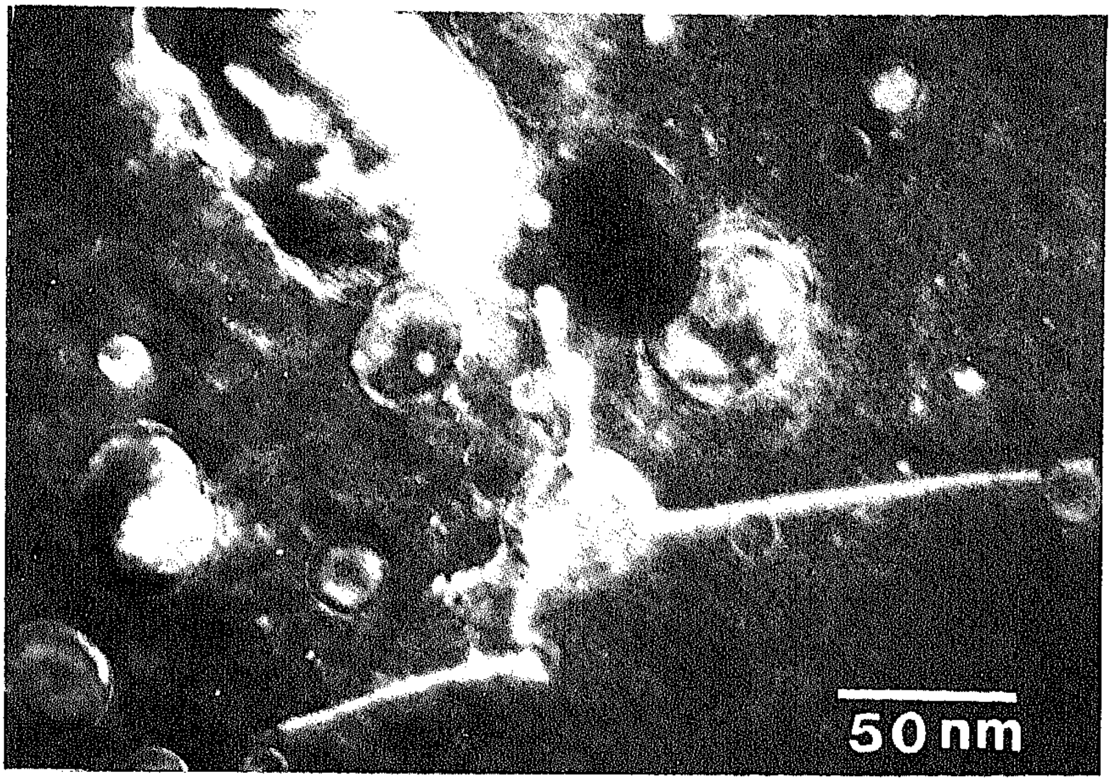

Figure 1. A weak-beam TEM image of a dislocation interacting with a dispersoid in a specimen crept at 1050 °C.

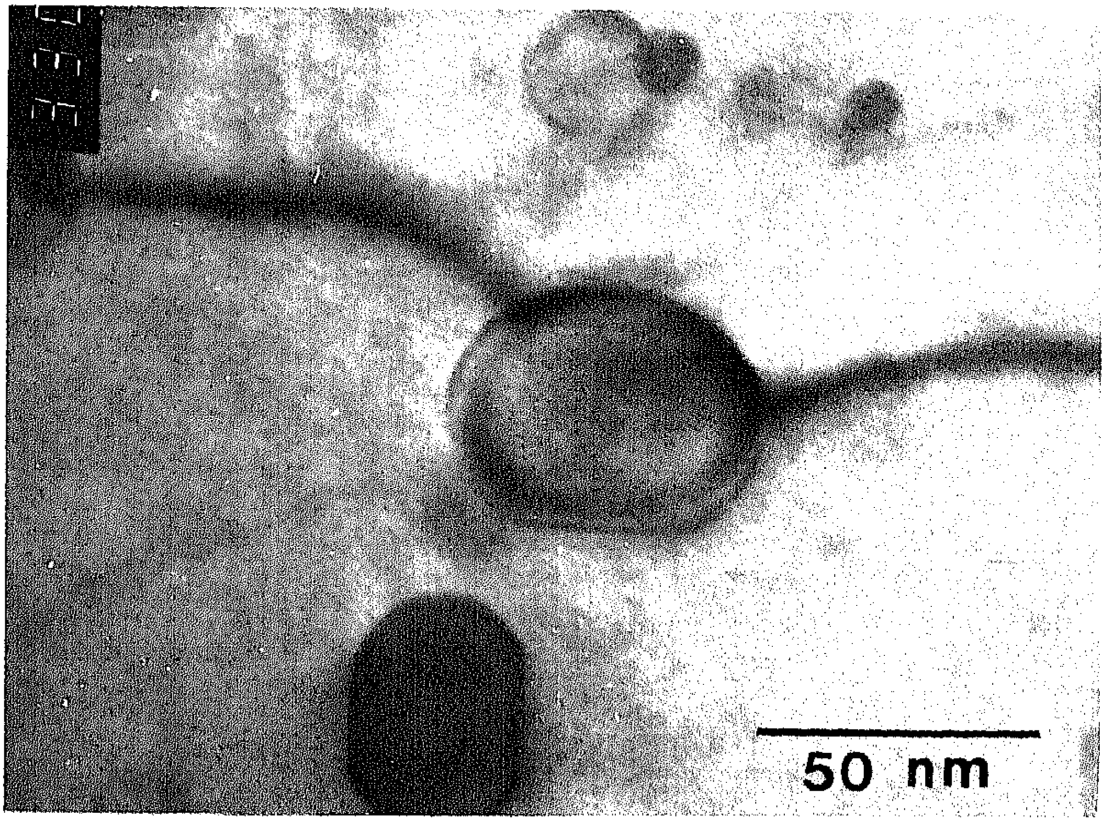

Figure 2. Dispersoid-dislocation interaction in bright-field imaging prior to dislocation detachment.

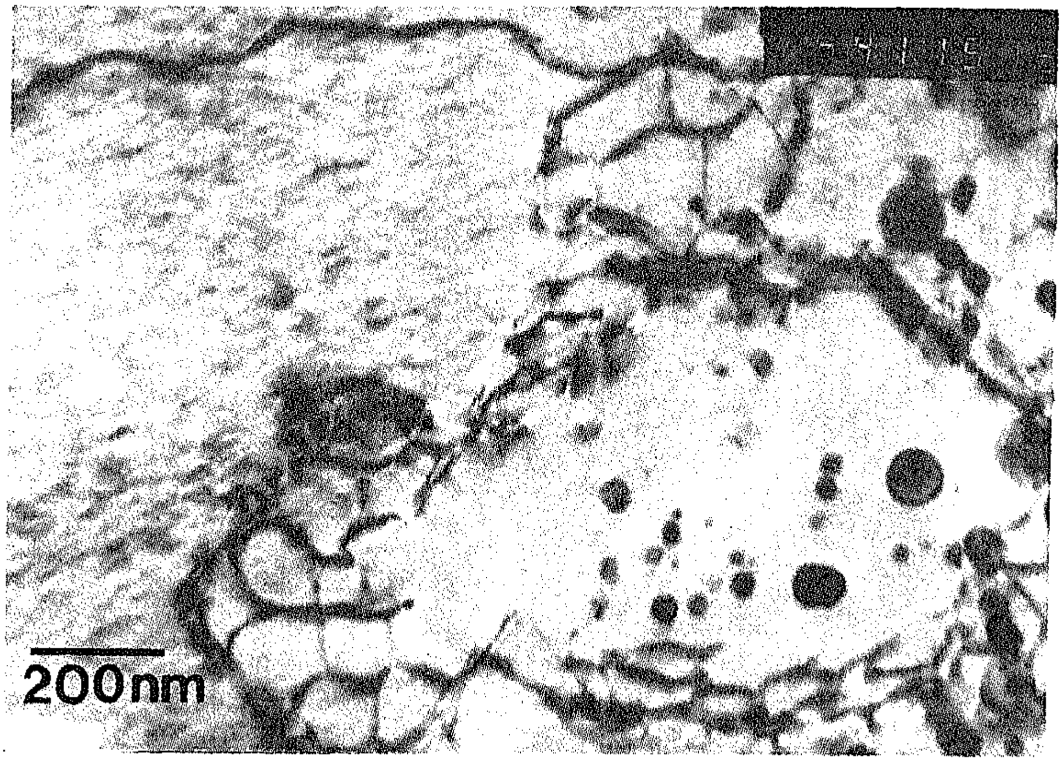

Figure 3. Dislocation network formed during low-cycle fatigue at 1050 °C.

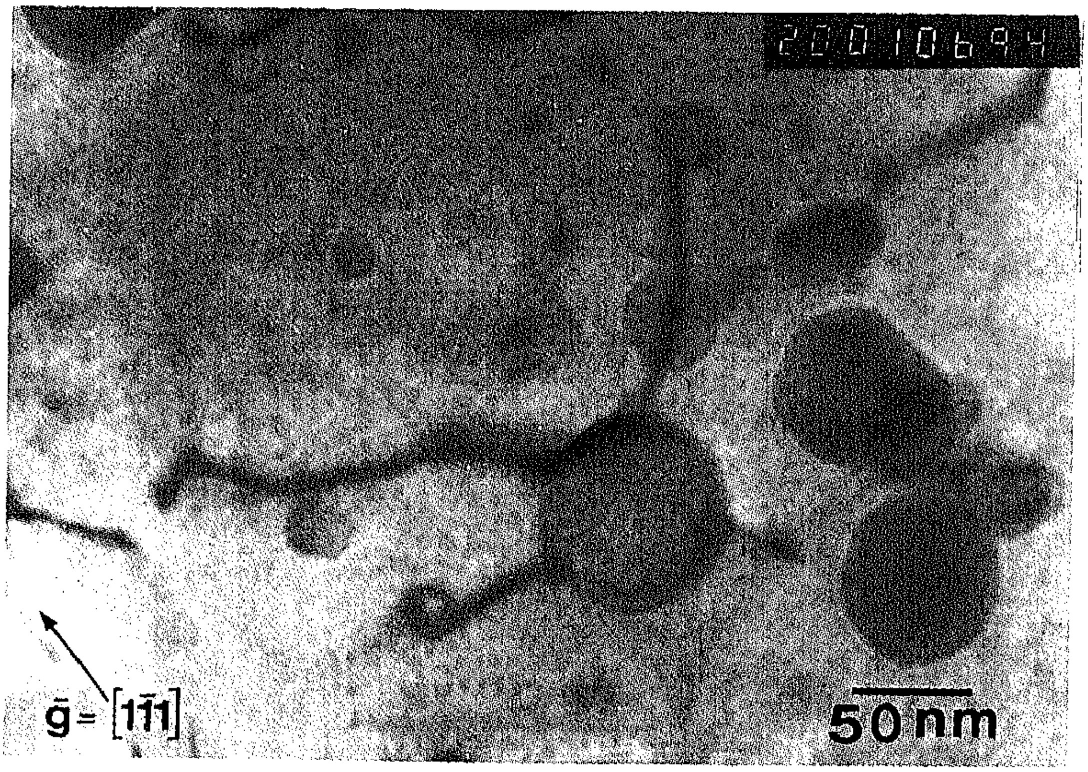

Figure 4. Attractive dislocation - dispersoid interaction in a fatigued specimen.

line energy E of the dislocation in the matrix. The parameter k is a "relaxation factor" and takes on values between 0 and 1. We postpone discussion of possible mechanisms for line energy reduction until Section 5.

When the dislocation now rides up over the dispersoid particle on its arrival side as shown schematically in fig. 5, bulk dislocation of higher line energy is exchanged for a segment of lower energy, on the one hand, while, on the other, the total line length is increasing; the converse is true when the dislocation has passed the top of the particle. The total energy change has to be equated to the work done by the advancing dislocation, giving a complicated but analytical expression for the stress necessary for climb to continue at any given point in the dispersoid-matrix interface. This calculation has recently been carried out in detail by Arzt and Wilkinson /17/. In the interest of brevity a simplified account will be given here which contains all the important features of the thorough analysis.

First we address the question of how the climb threshold expressed in eq. 1 is modified by the assumption of reduced dislocation line energy at the dispersoid. Because the energy increment due to extra dislocation line created at the dispersoid scales with k, the new climb threshold is obtained, to a good approximation, by multiplying eq. 1 by k:

$$\sigma_c(k) = 0.32 \; k \; M \frac{G\,b}{2\,\lambda} \qquad (3)$$

At low values of k, signifying a strong attractive interaction, this threshold will become negligible (in fact for k=0 the dislocation can gain energy by riding up the dispersoid, which now behaves just like a void). But now a second threshold will become important: that for detachment of the dislocation from the dispersoid. This new threshold arises because bulk dislocation has to be recreated at the expense of only a low-energy segment at the dispersoid. Its magnitude, which is independent of particle size, is given by (see Appendix):

$$\sigma_d(k) = M \frac{G\,b}{2\,\lambda}\sqrt{1 - k^2} \qquad (4)$$

The overall threshold stress for dislocation bypass is simply the larger value of the two stresses given in eqs. 3 and 4. These thresholds are plotted in fig. 6 for Inconel MA 6000 at a temperature of 950 °C as a function of k. It is apparent that only a weak attractive interaction, corresponding to a line energy reduction of less than 10 %, is required before the barrier governing the threshold stress changes from climb on the arrival side of the dispersoid to the detachment process on the departure side.

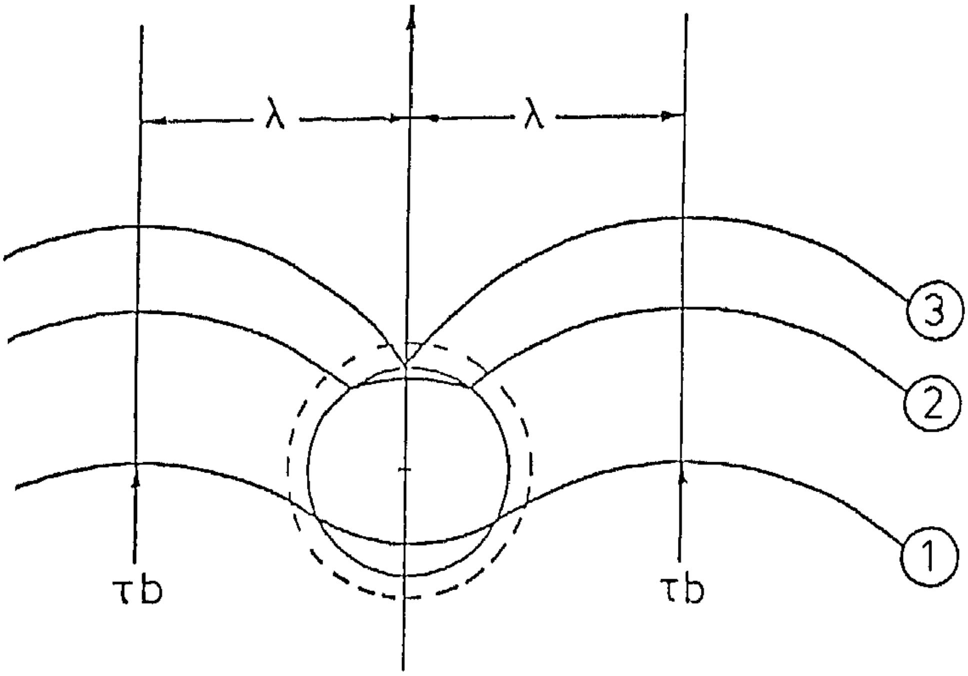

Figure 5. The geometry of particle - dislocation interaction during local climb. The dislocation, moving from bottom to top, is shown in three positions:
at "1" it is climbing up on the arrival side, "2" is a typical configuration on the backside, "3" is at the point of detachment.

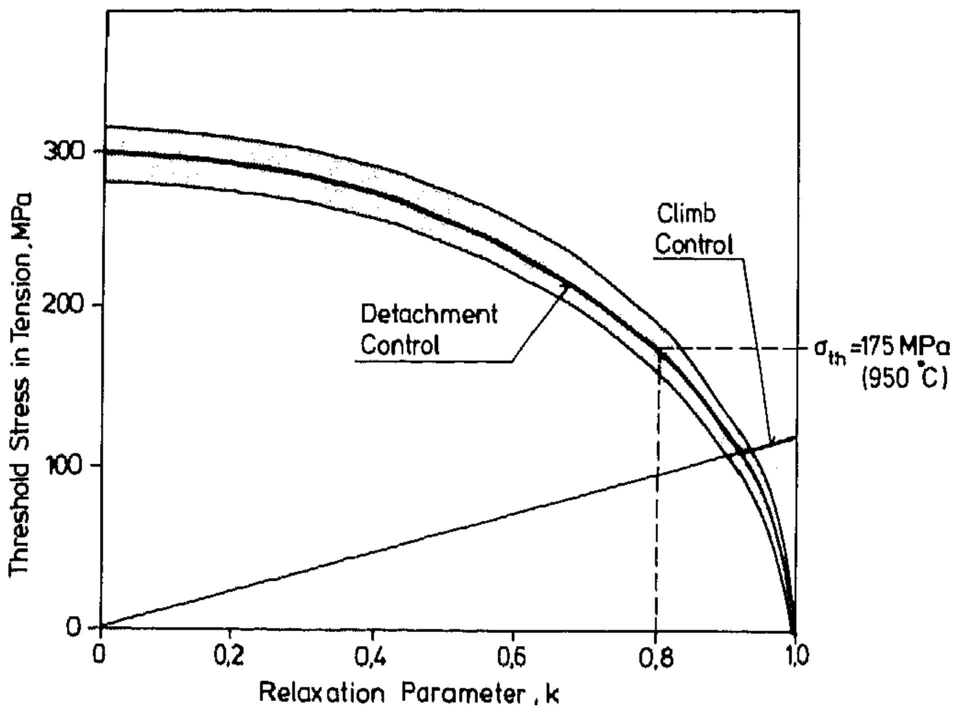

Figure 6. The range of the theoretical detachment and climb threshold for MA 6000 at 950°C, as a function of k. On the right, the approximate experimental threshold at 950°C is marked.

5. DISCUSSION

The micrographs in figs. 1 and 2 show unambiguously that an attractive interaction exists between dispersoids and dislocations. Similar observations were reported on Inconel MA 754 by Nardone et al. /18, 19/, but their micrographs, taken in bright field only, did not reveal the exact dislocation configuration in the vicinity of the dispersoids. Studying a γ'-forming ODS superalloy such as MA 6000 offers an additional advantage: the reprecipitation of γ' during cooling from test temperature enables the high-temperature configurations to remain stabilized against elastic "springback" on subsequent unloading at room temperature. Therefore we can be fairly confident that the configurations observed in the TEM are representative of the deformation mechanism at high temperatures.

The mechanism leading to a dispersoid-dislocation attraction is still open to discussion, because classically a hard particle would be expected to repel dislocations. Srolovitz et al. /20, 21/ have presented a possible explanation which is based on the following idea: a particle-matrix interface which is unable to sustain shear tractions at high temperatures enables the dislocation to relax the shear component of its strain field, thereby lowering its line energy. In some pictures the dislocation contrast at the dispersoid is indeed lost suggesting that such a relaxation process has occurred. Since in most of our micrographs,however, the dislocation contrast is well visible in the vicinity of the dispersoids, any core relaxation as postulated by Srolovitz et al. must be insignificant in these cases. Here the theoretical considerations presented in Section 4 and in the Appendix provide the "missing link": indeed only a minor reduction in line energy, which would be undetectable in the dislocation contrast, is required to ensure detachment control and, thus, dislocation configurations of the type observed.

The final objective of our study would be to establish a correlation between the observed configurations and experimentally measured creep or stress rupture strength. Because the dispersoid-dislocation interaction is responsible for the high-temperature, low-stress, long-term strength, which is time-consuming and expensive to measure extensively, such a correlation would be particularly rewarding. While this task is still the object of current work, the following preliminary comparison looks promising: On the right-hand side ordinate of fig. 6 the measured threshold stress for stress rupture, i.e. the stress below which rupture times well exceed 10^4h /22/, is indicated for the temperature of 950 °C. The plot indicates that this strength level lies indeed in the range of detachment control, with a corresponding k-value of roughly 0.8 . Quantitative evaluation of the dislocation configurations using the rigorous interaction model in /17/ seems to confirm independently that k values do predominantly lie in this range /14/. Values greater than about 0.8 have not been encountered, which is compatible with our theory: if k were close to 1, then detachment would cease to be rate-controlling, and the dislocation would have to be observed on the arrival side where it feels the climb threshold instead. Obviously, these and further questions, including the temperature dependence of k, merit close scrutiny, and further work long these lines is in progress.

6. CONCLUSIONS

1. Detailed TEM investigations carried out on crept and fatigued specimens of the oxide-dispersion strengthened superalloy Inconel MA 6000 confirm that an attractive interaction exists between lattice dislocations and the incoherent oxide dispersoids at high temperatures. The detachment of the dislocations from the particles appears to be the rate-limiting step in the creep deformation of this alloy.

2. A model for the detachment of dislocations from the dispersoids has been developed which indicates indeed that the threshold stress in ODS superalloys is not determined by dislocation climb over the dispersoid but by their detachment, provided a moderate attractive interaction occurs.

3. The detachment process gives rise to a reliable high temperature threshold stress which affects the low-stress, long-term creep properties of MA 6000. Its magnitude is independent of the size of the dispersoids and depends only on their spacing and on the relaxation of a dislocation at the dispersoid-matrix interface. This mechanism should be considered when aiming at an exploitation of the full potential of dispersion strengthening in high temperature alloys.

7. ACKNOWLEDGEMENTS

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9. APPENDIX: CALCULATION OF THE DETACHMENT THRESHOLD

The detachment threshold stress is derived by considering the energy balance at the point where the length of the dislocation segment in contact with the dispersoid shrinks to zero (see fig. 7). Equate the work done by the dislocation during an infinitesimal advance by ds to the detachment point with the energy required for exchanging a dislocation segment of length dl_1 and energy $k \cdot E$ by a segment of length dl_2 in the matrix:

$$2 \tau b \lambda \, ds = 2 E (dl_2 - k \, dl_1) \qquad \text{(A1)}$$

Here τ is the applied shear stress, b the Burgers vector of a lattice dislocation, 2λ the dispersoid separation. From simple geometry it follows that at the point of departure:

$$(dl_2)^2 = (dl_1)^2 + (ds)^2 \qquad \text{(A2)}$$

and

$$ds = dl_2 \cdot \sin\phi = dl_2 \cdot \frac{\lambda}{R} \qquad \text{(A3)}$$

where R is the radius of curvature to which the dislocation in the matrix is bent by the action of the applied stress, which is usually expressed as:

(A4) $$R = \frac{G\,b}{2\,\tau}$$

Inserting these equations in eq. A1 and rearranging gives the following simple result for the detachment threshold in tension:

(A5) $$\sigma_d = M\,\frac{G\,b}{2\,\lambda}\sqrt{1 - k^2}$$

This detachment stress is determined only by the energy balance at the point of departure, and is therefore independent of the exact trajectory along which the dislocation moves over the dispersoid. It is further independent of the dispersoid size.

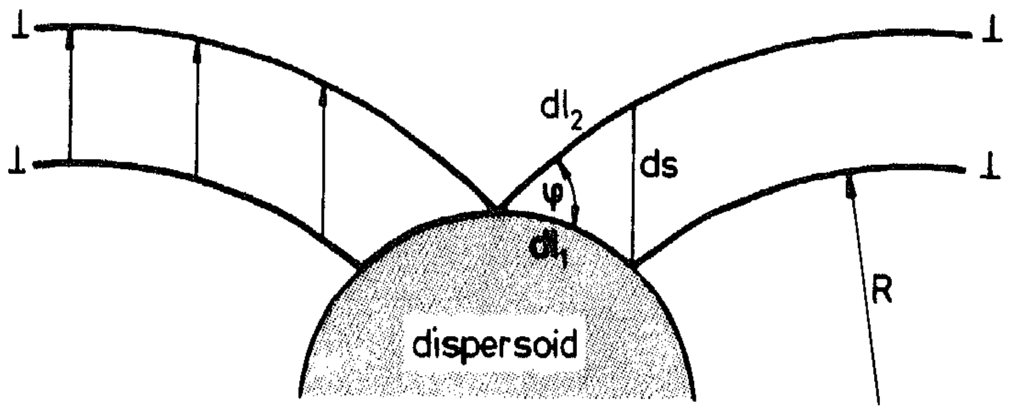

Figure 7. Dislocation geometry at the point of detachment from a dispersoid.