Creep and Fracture of Engineering Materials and Structures

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B. Wilshire and R.W. Evans

Department of Materials Engineering
University College, Swansea

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THE MECHANISM OF DISPERSION STRENGTHENING IN HIGH TEMPERATURE ALLOYS

E. Arzt, J. Rösler and J.H. Schröder

Max-Planck-Institut für Metallforschung
Institut für Werkstoffwissenschaften
Seestrasse 92, D 7000 Stuttgart 1, FRG

SUMMARY- The creep behaviour of dispersion strengthened alloys is characterized by the existence of a "threshold stress" below which the rate of deformation is negligible. Unlike classical models for the room-temperature strength, the mechanism of which has been well examined, satisfactory explanations for the high-temperature strength are still lacking. In the present paper an attempt is made to solve the essential conceptual problems which are central to an understanding of dispersion strengthening at high temperatures. A kinetic model for dislocation climb over dispersoid particles is presented, with the following new features: i) the dislocation shape in the vicinity of the particle is determined by the condition of constant chemical potential for vacancies along the climbing dislocation segment, and ii) the effect of an attractive interaction between dislocation and particle on the climb process is included. An attractive force is shown to affect the climb and bypass process in two distinct ways: Above a certain stress, purely local climb becomes a stable mechanism because the interaction can support a sharp dislocation bend; and a threshold stress has to be exceeded in order to detach the dislocation from the particle over which it has just climbed. The assumption of an attractive interaction is supported by a detailed TEM study of the dislocation configurations in a crept ODS superalloy; the evidence for this is described and discussed. It is concluded that repulsive dispersoid particles of low volume fraction are inefficient obstacles to dislocation motion at high temperatures because they are easily circumvented by climb. To achieve strength at very high temperatures, at which diffusive processes are rapid, an attractive particle-dislocation interaction is indispensable.

1. INTRODUCTION

Incorporation of incoherent, non-shearable dispersoids is an effective way of improving the mechanical properties of metallic materials at high temperatures (1, 2). This principle has been applied in the production of a number of dis-persion strengthened materials, including Ni, Fe, Al, Pt, Ti, Cu and their alloys. The creep rates of such alloys exhibit generally a high stress sensitivity with unusually high stress exponents. This behaviour is best rationalized by defining a "threshold stress" below which the rate of creep deformation is negligible (3-8). The threshold is also reflected in the stress rupture behaviour of dispersion-strengthened materials: there are indications that in an ODS superalloy, for example, rupture times increase drastically when the applied stress is below a certain "threshold" value (9).

A clear understanding of the mechanisms which cause such a threshold stress would be highly desirable for the following reasons: When designing with dispersion-strengthened materials for high temperature applications, information about the potential long-term reliability of the "threshold" is important. Also, when optimizing or developing new dispersion-strengthened alloys, first principles could provide useful guide lines for estimating the effectiveness of certain matrix-dispersoid combinations. Finally, from a scientific point of view, a clarification of the mechanisms by which dispersoids impede the motion of dislocations would be an important contribution; unlike the behaviour at room temperature, dislocation-particle interactions at high temperatures are by no means fully understood.

Treatment of the dislocation kinetics is complicated by the fact that at high temperatures dislocations can circumvent hard particles by climb. In the models which have been put forward the length over which the dislocation climbs is a critical parameter. Some authors, e.g. (10, 11), postulate that the dislocation profiles the particle closely, remaining in its glide plane everywhere in between the particles (fig. 1a): this process has been termed "local climb". It is important to note that local climb leads to sharp bends of the dislocation at the points where it meets the particle. A different approach is made in (12, 13), where the dislocation is allowed to "unravel", i.e. to reduce the high curvature at these bends. The resulting climb process is called "general" or "extensive" (fig. 1b).

A criterion for the exact dislocation shape which should be used in the calculations is not available. This is a severe shortcoming because the magnitude of the threshold stress predicted theoretically depends on the amount of extra dislocation line length that has to be created to allow particles to be overcome by climb (14). For "general" climb the thresholds are unrealistically small (13, 14, 7). Threshold effects of the type which is observed in dispersion-strengthened alloys thus cannot be explained in terms of the energetics of "general" climb. By contrast, models for "local" climb give correct order-of-magnitude estimates of creep thresholds, but their physical soundness is questionable: the sharp bend in the dislocation, which is instrumental in producing the threshold, can be rapidly smoothed out by diffusion. This point has been

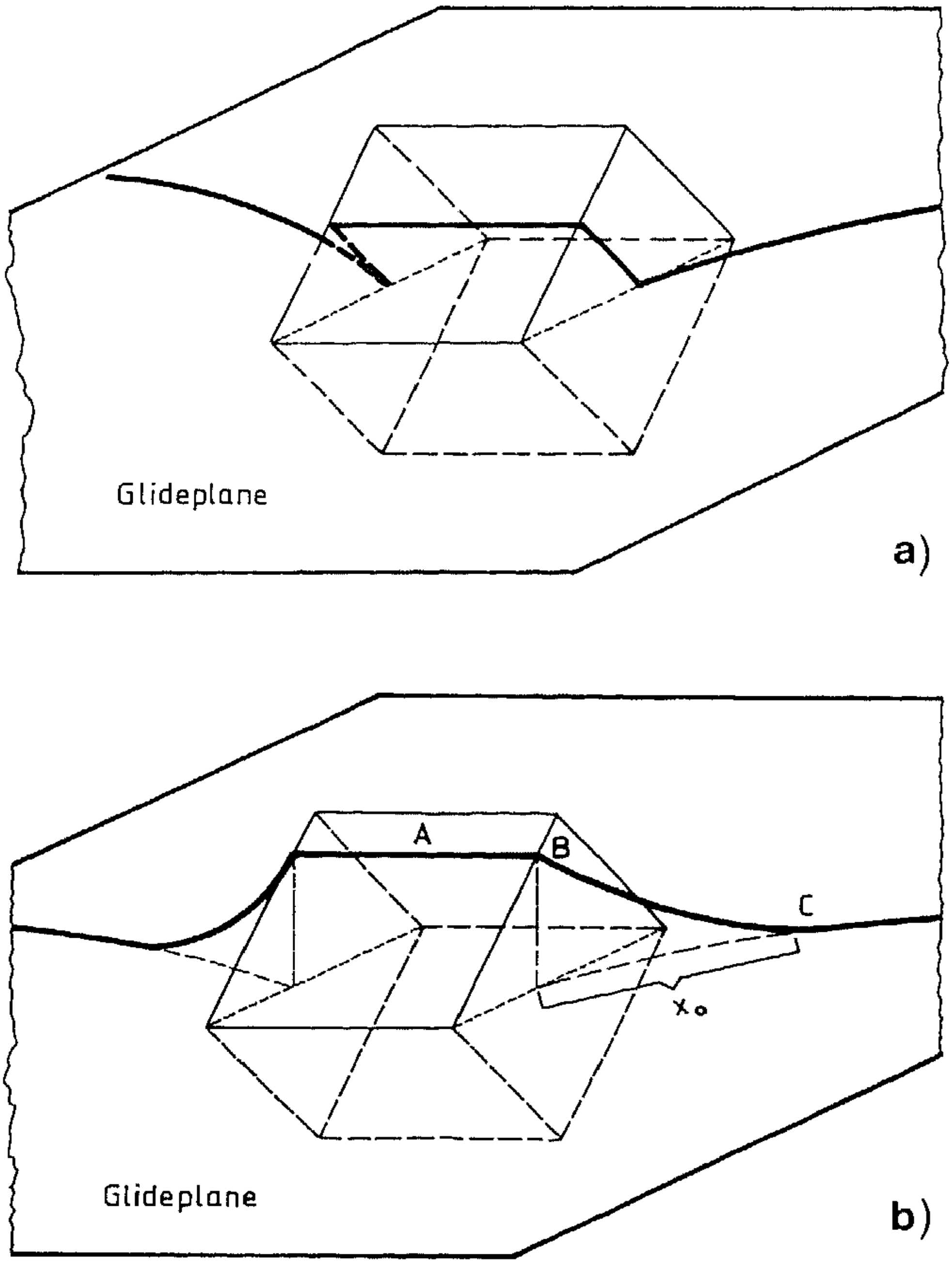

FIG. 1: Two model geometries of a dislocation climbing over a dispersoid particle: a) "local" climb, with sharp dislocation bends, and b) "general" climb during which the sharp bends "unravel". The equilibrium unravelling distance x_o increases with decreasing stress (section 3.1).

made clear by Lagneborg (12). In summary, none of the current climb models can account for the creep behaviour of dispersion strengthened high temperature alloys.

Progress can be made by considering the interaction between dislocations and particles more closely. While at room temperature elastically hard particles should repel dislocations, it has been shown, e.g. (15), that at high temperatures an attractive interaction may exist instead. The reasons are that diffusion relaxes both shear and hydrostatic stresses imposed on the particle by the approaching dislocation, and that a phase boundary must be considered as "slipping" at high temperatures. The dislocation can then lower its energy as a result of the interaction with the particle, which after complete relaxation behaves elastically just like a void.

The present paper examines the effect that an attractive interaction would have on the climb process and on the creep threshold. The results of TEM observations of dislocation-particle configurations in a crept ODS superalloy are described; they lend experimental support to the assumption of an attractive interaction. A kinetic model for dislocation climb is then presented, which incorporates an attractive interaction and does not rely on arbitrary assumptions concerning the dislocation shape in the vicinity of the particle. Finally, general conclusions for optimum alloy design, derived from these findings, are briefly discussed.

2. OBSERVATIONS OF DISLOCATION-PARTICLE CONFIGURATIONS

2.1 Experimental

The alloy on which these studies were performed was Inconel MA6000, an oxide-dispersion strengthened nickel-base superalloy. The dispersion consists nominally of Y_2O_3, with a mean diameter of 30nm and a mean particle spacing of about 100 nm (giving a volume fraction of about 2.5 %). A coarse elongated grain structure, which is typical of such alloys, minimizes the role of grain boundaries during deformation.

Constant load compression tests were carried out at temperatures up to 1050 °C and at stresses corresponding to about half the Orowan stress due to the yttria dispersion. After a test duration of up to 10 h the specimens were cooled to room temperature under load so that the dislocations were stabilized by reprecipitation of γ' particles. TEM foils were prepared by mechanical and electrolytical thinning, using an etchant of ethanol with 7.5 % percloric acid. In a JEOL 200 CX transmission electron microscope, dislocations were imaged in bright field and weak-beam mode. Further details can be found in (16).

2.2 Microscopic Observations

Dislocation densities in the crept specimens were always low enough to allow the interaction of an individual dislocation with the particles to be observed. One of the resulting, typical micrographs is shown in fig. 2. On it, as on about 50 more micrographs taken in this study, the following features can be identified:

a) The dislocation segments in the matrix and at the particle are bent in the same direction, indicating that the dislocation has already surmounted the particle by climb and is just about to detach itself from the particle.

b) The dislocations are bent to high curvatures at the points where they enter the particle-matrix interface. This corresponds to the geometry postulated in the local climb models.

c) The identity of the dislocation is preserved in (or near) the particle-matrix interface, suggesting only slight relaxation or core spreading.

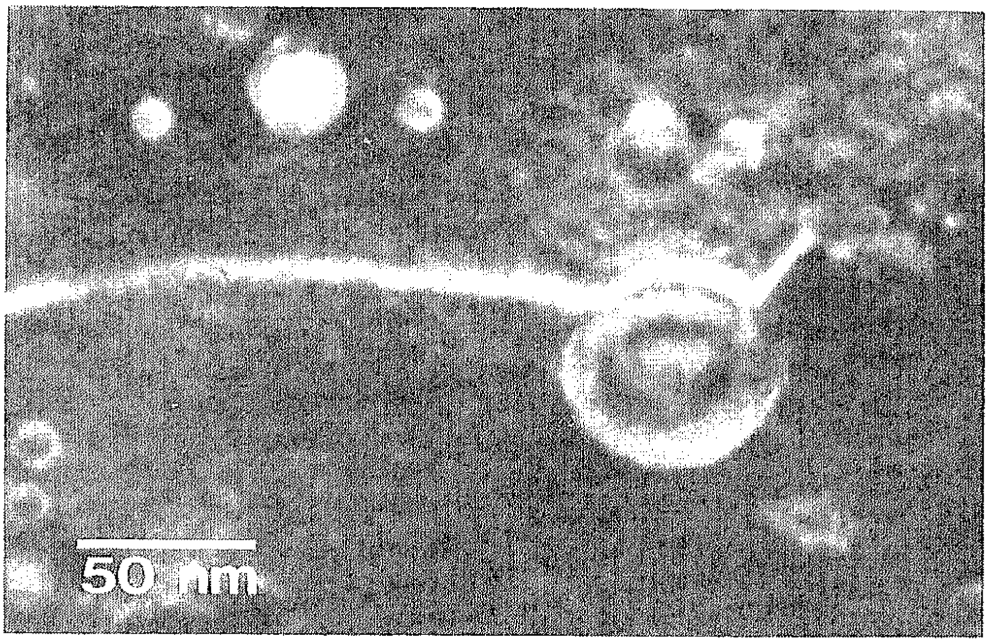

FIG. 2: Example of a weak-beam TEM micrograph of a crept ODS superalloy, showing the attractive interaction between a dispersoid particle and a dislocation climbing over it.

The micrographs constitute ample evidence for an attractive particle-dislocation interaction. They confirm earlier findings on a similar alloy, with less convincing experimental techniques (17, 18). Because it is known that the particles produce substantial misfit strains in the matrix owing to differential thermal contraction (19), in-situ heating experiments were also conducted to preclude artefacts related to the cooling process. At temperatures up to 650 °C the same type of dislocation configuration was found.

The sharp bends of the dislocations suggest that climb is local; it will be shown below that local climb is indeed a consequence of the attractive interaction, because the dislocation will attempt to maximize its line length at the particle. The fact that almost all dislocations were captured in this typical configuration on the "departure" side of the particle is a strong indication that dislocation movement is controlled by the detachment from the particles rather than by the climb process itself.

3. EFFECTS OF AN ATTRACTIVE INTERACTION ON CLIMB

We now ask how an attractive interaction between particles and dislocations climbing around them influences the creep behaviour, and whether the conclusion regarding detachment control is at all reasonable. For this purpose we first treat briefly the kinetics of dislocation climb over "non-interacting" particles. Subsequently, the effects of an attractive interaction on the climb process and on the resulting threshold stress are modelled. The details of the calculations are complicated and will be given elsewhere (20).

3.1 Kinetics of Climb Over "Non-Interacting" Particles

For simplicity we consider a dispersion of cube-shaped particles of low volume fraction (as in fig. 1). The particles are assumed to exert only a short-range repulsive force on the dislocations. When a shear stress below the Orowan stress is applied, a dislocation can glide a short distance until it is pinned by the particles. At low temperatures dislocation motion would stop here and no further deformation would be possible.

Thermal activation however allows the particle to be bypassed by dislocation climb. As the dislocation rides up the particle, it leaves the glide plane up to a certain distance (x_o) from the particle (fig. 1b). Rather than imposing a shape on the dislocation, we determine the extent to which climb is localized at the particle from an equilibrium condition: we let the dislocation "unravel" by diffusion, until the driving force for inserting further vacancies at B-C (due to the remaining curvature) becomes equal to that at A-B (due to the applied stress). This condition of minimum energy distinguishes the present model in principle from earlier local climb theories (10, 11), but also in detail from a previous approach to general climb by Lagneborg (12).

The resulting equilibrium configuration is a function of the applied stress: high stresses can support a high dislocation curvature along BD, leading to localized climb (with small x_0), while at low stresses the climbing dislocation segment extends further out. Purely local climb with $x_0=0$, however, cannot be stable under the equilibrium condition.

Given the equilibrium shape of the dislocation, the bypass times for a dislocation as a function of applied stress have been calculated from diffusion theory. The resulting creep rates can be fitted with high accuracy to an equation of the following form (20):

$$\dot{\epsilon} = B \; C_i \; \left(\frac{\sigma}{\sigma_{Or}} \right)^n \tag{1}$$

where B containes size and shape of the particles, C_i is a kinetic constant proportional to the diffusivity, σ_{Or} is the Orowan stress (which can often be identified with the room temperature strength), and σ the applied stress. A typical log $\dot{\epsilon}$-log σ plot is shown in fig. 3.

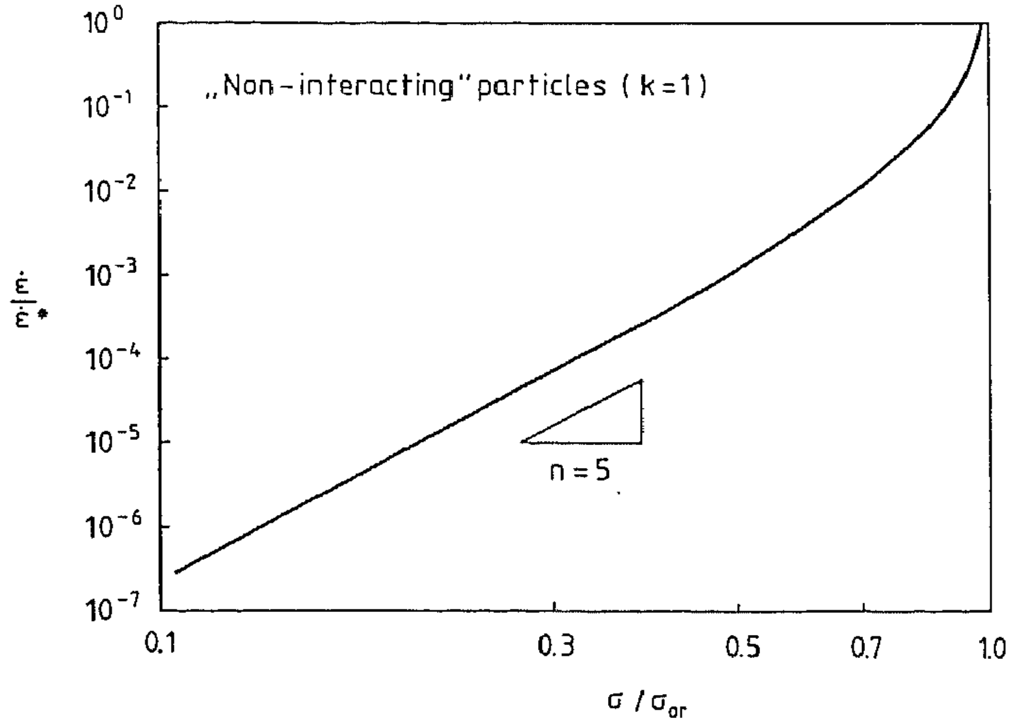

FIG. 3: Calculated strain rate vs. applied stress (in units of the Orowan stress) due to a dislocation climbing over a "non-interacting" particle. Because climb is more localized at higher stresses, a power-law (with n $\approx$ 5) appears. The curve reflects a transition to Orowan bowing at very high stresses. Note that no (significant) threshold stress arises.

Although the rate of stress-induced diffusion is usually linearly proportional to the stress, a "natural" power-law with stress exponent n arises here. The reason lies in the localization of climb at higher stresses (as described before): fewer vacancies are required to support localized climb, making the creep rate increase with stress in an over-linear manner. The value of n, which depends slightly on the particle parameters, lies in the range 5...6. This is the maximum stress sensitivity that can be obtained for creep controlled by climb over "non-interacting" particles.

Because no significant threshold stress arises under these conditions, it must be concluded that "non-interacting" particles are not very efficient obstacles to dislocation motion at high temperatures. They can retard but not suppress creep (at practically significant stress levels). The results of the model calculations are therefore clearly incompatible with the high stress sensitivities typical of dispersion-strengthened alloys. Hence explanations for the existence of threshold stresses must be sought in an additional mechanism.

3.2 Detachment Threshold Due to Attractive Interaction

The microstructural evidence presented in section 2 points to the need to allow for an attractive interaction between dislocations and particles when calculating the bypass time of individual dislocations at particles. One important effect has been considered previously (21): detachment of dislocations, rather than the climb process itself can become the rate-determining process. The reasons are summarized below.

An attractive interaction is modelled by assigning a lower line energy T' to the dislocation segment in the vicinity of the particle:

$$T' = k \cdot T \qquad (2)$$

T is the line energy of a dislocation remote from the particle. k can be thought of as a "relaxation factor": it describes the extent to which the dislocation is still localized after strain relaxation has occurred in the particle-matrix interface. For "non-interacting" particles (k=1) the creep behaviour reduces to that described in section 3.1. Voids, on the other hand, would lead to maximum relaxation, with k values approaching zero. Depending on the amount of relaxation that is possible at a given particle, k can in general assume values intermediate between 0 and 1.

In order to detach a dislocation from such a particle, a threshold stress must be exceeded. A full analysis of the energetics of dislocation climb under these conditions shows that it amounts to (21):

$$\sigma_{th} = \sigma_{Or} \sqrt{1 - k^2} \tag{3}$$

The magnitude of this threshold is plotted in fig. 4 as a function of k. Also included in the diagram is the (hypothetical) threshold for local climb. It is apparent that even if local climb were accepted as a viable mechanism, it would determine the threshold stress only when the interaction strength is negligible. Once the relaxation exceeds only about 5 % ($k \leq 0.95$), the detachment event dominates the dislocation kinetics. If general climb, the opposite extreme, is considered, then a significant threshold is produced only by the detachment process. In other words: alloys which show a threshold behaviour can only derive it from an attractive interaction, while in the absence of a measurable threshold - as for example in alloys strengthened by coherent precipitates - any attractive interaction must be vanishingly small.

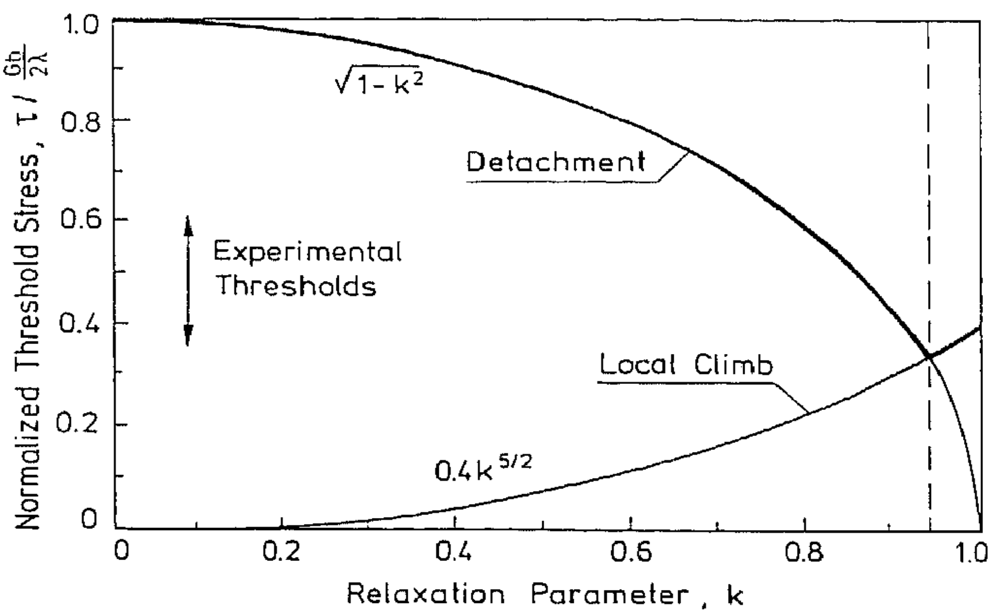

FIG. 4: Threshold stresses (normalized by $Gb/2\lambda$) due to dislocation detachment and to local climb, as a function of k. Provided a small particle-dislocation interaction exists, detachment controls the threshold stress.

The detachment threshold is a reliable limit to dislocation motion at all temperatures. Because the critical configuration which determines its magnitude is reached only at the very point of detachment (fig. 5a), it is independent of particle size and shape, and it applies irrespective of the details of the climb process. It is not affected by the position of the glide plane with respect to the particle it intersects. There seem to be only two ways in which this threshold could be circumvented: particle dragging by the travelling dislocations or thermally activated dislocation detachment. Both of these processes however are limited to extremely small particle sizes; they do pose a potential limit to the fineness of an efficient particle dispersion.

3.3 Stability of Local Climb Due to an Attractive Interaction

Besides introducing a threshold stress for dislocation detachment, an attractive interaction also has an effect on the equilibrium dislocation shape at the particle: because of the discontinuity in line energy a sharp bend of the dislocation is now stable, and local climb becomes possible. The bend allows the dislocation to maximize its length in the particle-matrix interface, where its line energy is reduced. Minimum energy obtains when the contact angle γ reaches an equilibrium value γ_o for which:

$$\cos \gamma_o = k \tag{4}$$

γ_o is equivalent to the "wetting angle" that is formed when two surfaces of different specific energy intersect. For "non-

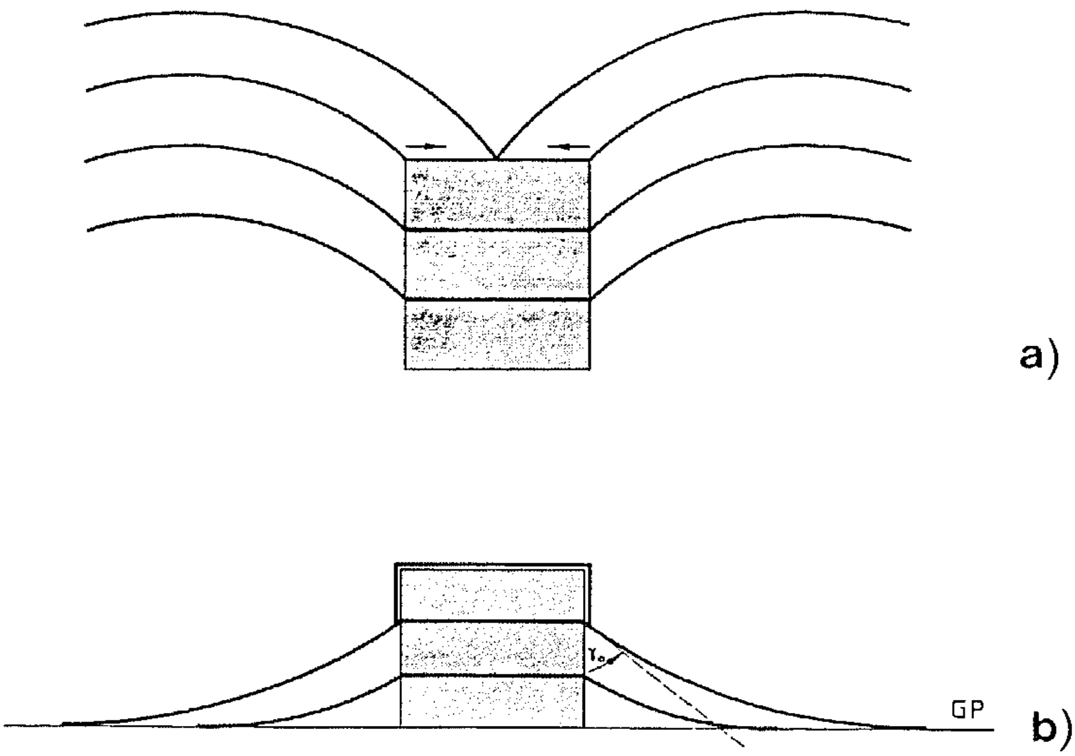

FIG. 5: The effects of an attractive interaction between dispersoid particles and dislocations on the climb and by-pass process: a) a threshold stress for detachment of the dislocation from the particle is introduced, and b) a transition from general to local climb occurs once the equilibrium angle (γ_o) is reached. Both processes are incorporated in the model.

interacting" particles (k=1) this condition reduces to a tangent criterion which rules out local climb. If $k < 1$, then the dislocation will unravel until $\gamma = \gamma_0$ (fig. 5b). Then further vacancies are accepted only by the segment at the particle, and a transition to local climb takes place.

The point at which this transition occurs, depends on the angle γ at a given applied stress as a function of dislocation position. The full analysis (20) suggests three distinct climb regimes. For high stresses:

$$\sigma \gtrsim k\,\sigma_{Or} \tag{5}$$

climb can be considered as local from the very beginning because the equilibrium angle forms early in the climb process. At sufficiently low stresses,

$$\sigma \lesssim 0.7\,k\,\sigma_{Or} \tag{6}$$

climb is general throughout because the equilibrium angle is never reached. In the intermediate stress range bounded by eqs. 5 and 6 a transition from general to local climb occurs in the process.

4. DISCUSSION

An attractive interaction between particles and dislocations climbing around them has been shown to have two separate effects on the climb and by-pass process (illustrated in fig. 5): i) it introduces an athermal threshold stress for dislocation detachment from the particle after climb is completed, and ii) it stabilizes local climb at sufficiently high stresses. Both effects lead to a highly stress-sensitive dislocation velocity, as is seen in fig. 6. The diagram shows the results of calculations of the kinetics, based on vacancy diffusion, for an attractive particle; for comparison also the "non-interacting" case (k=1) is included.

The exact shape of these curves depends somewhat on the particle parameters, but the cross-over between the two curves in fig. 6 is a typical feature. It arises from the combined effect of the detachment stress (at which the dislocation velocity is truncated) and of climb localization (to support local climb, fewer vacancies are required, which speeds up the rate at the high stress end).

It is readily apparent from fig. 6 that the assumption of an attractive interaction has great potential for explaining the creep behaviour of dispersion strengthened materials. Of course, the simplifications with regard to particle shape and the neglect of the statistics of the particle distribution can alter the details of the curves. But their shape is generally in at least qualitative agreement with experiment. A more thorough comparison would require more information on the relaxation process and the resulting value of k. As a limiting case, the interaction of a dislocation with a void can be considered. An estimate for the relaxation at a void (22) gives $k \approx 0.2$ ($k = 0$ is never reached because part of the elastic field of the dislocation remains even if the core has entered the void). According to equation 3, the threshold stress is then still $\sigma_{th} \approx \sigma_{Or}$. That voids produce a strengthening effect of this magni-tude is confirmed by the results of a recent computer analysis (23).

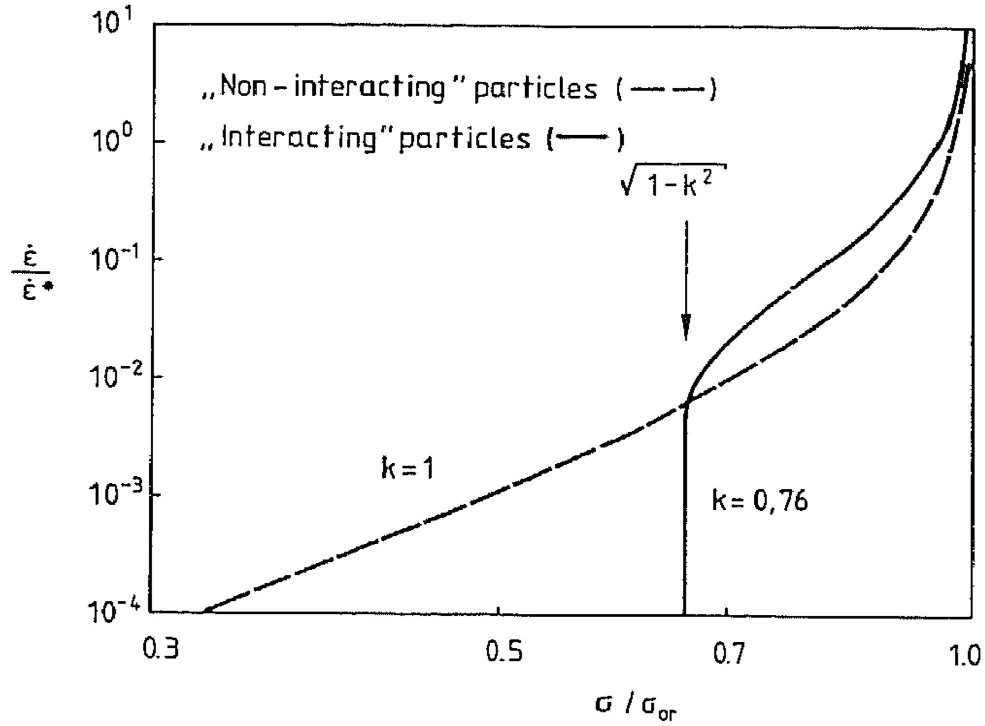

FIG. 6: Calculated creep rate (normalized) vs. applied stress (normalized) for a relaxation parameter k=0.76, compared with the behaviour predicted for "non-interacting" particles (dashed line). The curve reflects both effects illustrated in fig. 5: the threshold truncates the creep rate at low stresses, and localization of climb speeds up climb at higher stresses.

When the evidence for thresholds in a number of different dispersion strengthened materials is compiled (22), an "average" threshold emerges roughly at $\sigma_{th} \approx 0.4 \ldots\ldots 0.6\ \sigma_{Or}$ - a "give-away" of about 50 % compared to the maximum that would be achievable. This suggests that the dispersoid particles do not quite behave like voids, and the relaxation of the dislocation is far from complete. This conclusion is supported by the weak-beam micrograph of section 2, where the dislocation at the particle produces a clearly visible contrast. The experimental thresholds would require k $\approx$ 0.8....0.9. Such a minor relaxation would in fact be invisible by conventional TEM techniques. Thus the mechanism of dispersion strengthening as proposed in this paper is consistent with TEM observations.

The exact mechanism of dislocation relaxation at the particle is expected to depend on the structure of the phase boundary between particle and matrix. This aspect clearly requires further detailed work. A provisional guide line for ensuring optimum dispersion strengthening in high temperature materials can, however, be given at this stage. Because "non-interacting" particles are easily overcome by dislocations at high temperatures, the particle-matrix interface should be optimized to allow a strong interaction effect in the form of relaxation. The final goal should be a dispersion of voids, possibly filled with a gas which is insoluble in the matrix to prevent coarsening. They should be closely spaced to produce large Orowan stresses, but sufficiently large to avoid being dragged along by the dislocations. With such a dispersion it can be expected that the threshold stresses could be raised to about twice the values which are typical of present-day dispersion strengthened materials.

5. CONCLUSIONS

1 - Threshold stresses for creep of dispersion strengthened alloys can be explained only in terms of an attractive interaction between dislocations and dispersoid particles. "Non-interacting" particles would be inefficient obstacles to climbing dislocations.

2 - An attractive interaction arises at high temperatures because of dislocation relaxation in the vicinity of the particle-matrix interface. This relaxation, and thus the amount of dispersion strengthening, should be optimal for closely spaced soft particles or voids. The exact nature of the attractive interaction requires further work on the dislocation and atomistic level.

3 - The mechanism of dispersion strengthening at high temperatures as it is proposed here is consistent with TEM observations of dislocation structures in crept ODS superalloys. The theoretical considerations suggest that it must also be operative in other dispersion strengthened systems.

6. ACKNOWLEDGEMENT

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