

High Temperature Properties of Dispersion Strengthened Materials Produced by Mechanical Alloying: Current Theoretical Understanding and Some Practical Implications

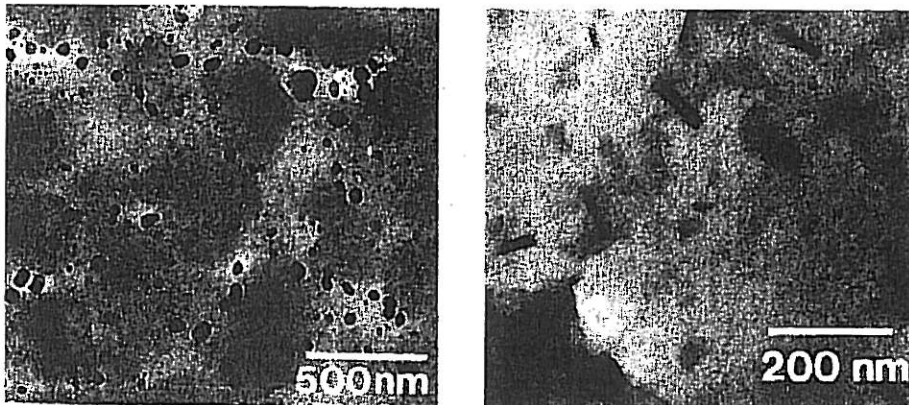
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Abstract

The typical effects of dispersoid particles and grain structure on creep of dispersion-strengthened materials are described. The current theoretical understanding of dislocation processes in these materials is reviewed extensively. It is argued that recent models based on dislocation detachment from dispersoid particles can have important practical implications for future alloy design and optimization. For creep data extrapolation, threshold stress concepts should be used with caution. Achievements and shortcomings of current models for grain boundary effects are also discussed. It is concluded that the creep behaviour of polycrystalline dispersion-strengthened materials is not fully understood and necessitates further work.

1. Introduction

Dispersion-strengthened materials for high-temperature applications were the main objective behind the development of "Mechanical Alloying" (1,2). The process as invented by Benjamin (3) involves high-energy ball milling of the starting powders together with the dispersoid particles, followed by consolidation and thermomechanical processing (for a recent review see Singer and Arzt (4)). In a similar process, termed "Reaction Milling", Jangg et al. (5-7) succeeded in forming well-distributed dispersoid particles by chemical reaction of milling additions with the powder. Typical dispersoid microstructures obtained by these two methods are shown in fig. 1.



**Fig. 1:** TEM micrographs of two modern dispersion-strengthened engineering materials; left: the nickel base superalloy Inconel MA 6000 with  $Y_2O_3$  dispersoids (small dark particles) and  $\gamma'$  precipitates (larger shadows in the background), produced by Mechanical Alloying (from Ref. 4); right: the aluminium alloy Dispal (AlC2) with  $Al_4C_3$  (dark platelets) and  $Al_2O_3$  dispersoids (light equiaxed particles), produced by Reaction Milling (from Refs. 14, 25).

The advantage of dispersion-strengthened alloys lies in the retention of useful strength up to a relatively high fraction of their melting points ( $\sim 90\%$ ) where other strengthening mechanisms, e.g. precipitation hardening or solid solution strengthening, rapidly lose their effectiveness. Compared with other processes, e.g. internal oxidation or co-precipitation, mechanical alloying techniques offer great versatility with regard to dispersoid-matrix combinations because they circumvent thermodynamic limitations imposed by the (desired) insolubility of the dispersoid material. Also dispersoids produced by mechanical alloying can be combined with other hardening phases; the prime example are oxide-dispersion strengthened (ODS) superalloys, which combine excellent high temperature strength (due to the dispersoids) with good intermediate temperature strength (due to ordered precipitates) (1-4).

The flexibility of mechanical alloying techniques has great potential for producing new dispersoid-matrix material combinations. Up to recently, the choice of dispersoid materials has been largely empirical, with much more attention being paid to thermal stability than to the efficiency of dispersoids as dislocation obstacles. As new alloy systems (e.g. aluminium, intermetallics) are now being developed for high-temperature applications, guidelines for achieving optimum dispersion strengthening would be particularly welcome. Recent attempts to model high temperature strength in dispersion strengthened materials appear to have approached a stage at which new conclusions, many of which are still tentative, for obtaining dispersoids with high efficiency and for grain structure optimization can be drawn.

The aim of this paper is therefore to review and critically discuss the current theoretical understanding of dispersion strengthening at high temperatures and to suggest conclusions for alloy design, optimization and use. Dispersoid effects are treated in section 2, while the influence of the grain structure is discussed in section 3. An extended version of this paper will be published elsewhere (8).

## 2. Effects of Dispersoid Particles on Creep Strength

### 2.1 Experimental Facts and "Threshold Stresses"

The presence of dispersoid particles causes a pronounced retardation of creep at high temperatures and comparatively low stresses; this effect is now well documented, e.g. (9-14). Two typical examples of the resulting strain rate vs. stress behaviour are shown in fig. 2. Unlike the dispersoid-free counterparts, the dispersion-strengthened materials exhibit creep rates which, on decreasing applied stress, fall extremely rapidly to (eventually immeasurable) low values. The "stress exponents", i.e. the slopes in fig. 2, which measure the stress sensitivity of the creep rate, can assume values in excess of 100. Such a behaviour is inconsistent with creep laws for conventional materials which usually exhibit stress exponents between 3 and 10. Also the temperature dependence of creep is often observed to be "abnormal" in the sense that temperature-compensated plots as in fig. 2 do not produce a common curve for different temperatures. This implies that the activation energy for creep appears to be higher than that for volume diffusion, which again is contrary to classical dislocation creep.

Because of these shortcomings a semi-empirical constitutive equation containing a "threshold stress"  $\sigma_0$  has been proposed for dispersion-strengthened materials (15-17):

$$\dot{\epsilon} = A' \frac{D_V G b}{k_B T} \left[ \frac{\sigma - \sigma_0}{G} \right]^{n'} \quad (1)$$

where  $\dot{\epsilon}$  is the creep rate,  $\sigma$  the applied stress,  $G$  the shear modulus,  $n'$  the stress exponent,  $D_V$  the lattice diffusivity,  $b$  the Burgers vector,  $k_B$  Boltzmann's constant,  $T$  the absolute temperature and  $A'$  a dimensionless constant.

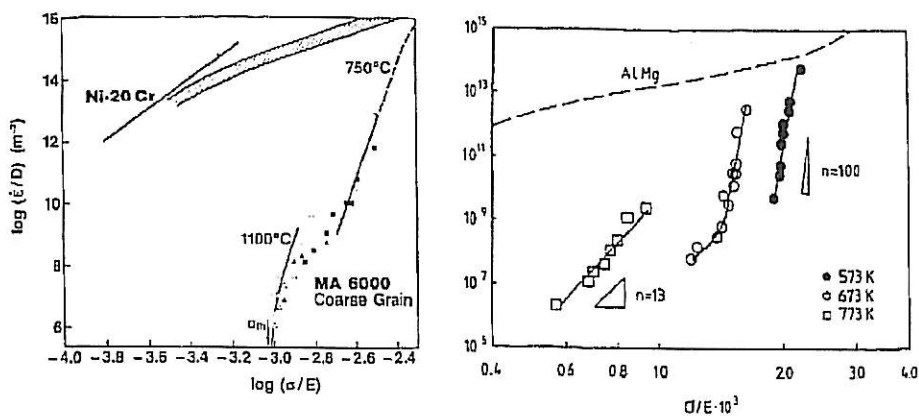


Fig 2: Typical creep behaviour of dispersion-strengthened materials; the creep rate  $\dot{\epsilon}$  is normalized by the lattice diffusivity  $D$ , the applied stress  $\sigma$  by Young's modulus  $E$ ; left: Inconel MA 6000, comparison of coarse-grain with fine-grain and dispersoid-free (Ni-20 Cr) material (4), and right: dispersion strengthened Al-4 Mg in the fine-grained condition (apparent grain boundary weakening occurs at 500 °C) (14,25).

This expression gives an arbitrarily high "apparent" stress exponent  $n=d(\log \dot{\epsilon})/d(\log \sigma)$  at stresses just above  $\sigma_0$ . By allowing  $\sigma_0$  to decrease with increasing temperature, a high "apparent" activation energy can also in principle be formally justified.

If eq. 1 is accepted as a formal representation of creep data for dispersion-strengthened materials, then an understanding of the micromechanisms of creep will hinge on a theoretical justification for  $\sigma_0$ . It must be emphasized here already that the "threshold stress"  $\sigma_0$  cannot be a true threshold for creep deformation, in the sense that at stresses below it the strain rate is identical to zero - as is suggested by the form of eq. 1. With further mechanistic background, this modified constitutive equation will be seen merely as an approximation. In fact, it will be shown in section 2.2, that recent models for the mechanism of dispersion strengthening at high temperatures lead to serious conceptual reservations concerning uncritical use of a "threshold stress" concept.

Bearing these qualifications in mind, one can still attribute some practical significance to the value of the apparent "threshold stress"  $\sigma_0$  as it characterizes the stress level at which dispersion-strengthened materials become far superior to their dispersoid-free counterparts. Further, for practical applications the "threshold stress" will be a design-limiting strength property: service stresses must always be kept below it in order to avoid occurrence of unacceptable creep deformation.

Threshold stress values have been determined by various authors for several dispersion strengthened alloy systems, including the mechanically alloyed iron-base alloy MA 956, and the nickel-base alloys MA 754, and MA 6000. Unfortunately, it is not always possible to compare these thresholds because they have been extracted from the creep data in different ways. For the sake of comparison, an attempt will be made here to evaluate "threshold stresses" from published creep data in a consistent way. In order to preclude grain boundary effects, only data for single crystals or coarse elongated grains (tested in the longitudinal

direction) are included. The data were replotted as  $(\ell/D_V)^{1/n}$  vs.  $\sigma/E$  (where  $E$  is Young's modulus) and extrapolated linearly to  $\ell = 0$ . The diffusivity normalization produced in most cases parallel lines for different temperatures, which generally improved the reliability of the extrapolations. Rather than force-fitting the data to stress exponents suggested by theoretical considerations, values typical of the dispersoid-free material were chosen: for example  $n = 4.6$  for Ni-20Cr based ODS alloys.

The results are plotted in a normalized form in fig. 3. Absolute temperatures have been divided by the melting point (or the approximate solidus temperature). The "threshold stresses" have been normalized with respect to the Orowan stress given by

$$\sigma_{Or} = \frac{0.84 M}{2\pi(1-\nu)^{1/2}} \frac{Gb}{\ell} \ln\left(\frac{r}{b}\right) \quad (2)$$

where  $M$  is the Taylor factor (or reciprocal Schmid factor for single crystals),  $\nu$  is the Poisson number ( $\nu = 0.3$ ),  $\ell$  the mean planar particle spacing, and  $r$  the mean particle radius.

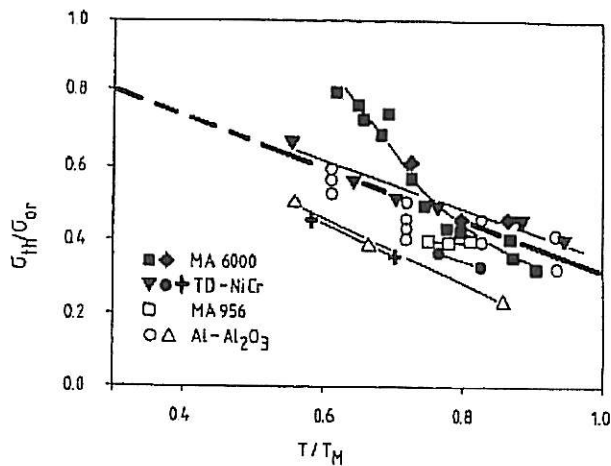


Fig. 3: Plot of experimental "threshold stresses", normalized with respect to the calculated Orowan stress, as a function of homologous temperature, for coarse-grain or single crystals materials: MA 6000 (Refs. 11,4), MA 956 (Ref. 63), Al-Al<sub>2</sub>O<sub>3</sub> (Refs. 12,13), TD-NiCr (Refs. 9,64,65).

Despite some numerical uncertainties the following observations can be made in fig. 3:

- 1) The Orowan stress is a useful normalization parameter for "threshold stresses", as it brings the data from very different materials, whose absolute "threshold stresses" differ by more than an order of magnitude, to within a factor of about 2. It appears plausible that similar mechanisms are responsible for the creep strength in these alloys.
- 2) All the threshold stresses are below the theoretical Orowan stress, and decrease with increasing temperature. These deviations from the Orowan stress are important and will need to be explained theoretically.

In summary, the "threshold stress" concept appears to have some merit for formal rationalization of the creep behaviour in dispersion strengthened alloys. Without further mechanistic background, the applicability of this concept must however remain suspect. We therefore turn to the relevant theories of dispersion strengthening at high temperatures, which will be reviewed in the following section.

## 2.2 Theoretical Understanding of Dispersoid Effects on Creep Strength

This section is entirely devoted to theoretical attempts to explain, in terms of dislocation theory, the high stress sensitivity of the creep rate ("threshold stress" behaviour) in materials with low volume fractions of dispersoids. The problem has received great attention over the past fifteen years. Early papers attempted an explanation in terms of the Orowan process and its thermal activation (18). But once the "threshold stresses" were found to be significantly lower than the Orowan stress of the particle dispersion, subsequent models focussed on the process by which dislocations can circumvent hard particles, especially by climbing around them. Following detailed transmission electron microscopy of this process, more attention has been paid recently to the interaction between dispersoid particles and dislocations. New models have been proposed which abandon the idea of climb being the rate-limiting event. The development, including latest results, is described below.

### Dislocation Climb Models

When dispersoid particles are considered as impenetrable obstacles which force gliding dislocations to climb a certain distance until they can continue to glide, a retardation of creep is predicted, but a high stress sensitivity cannot be explained in this way (19,20). The maximum stress exponent which is obtained theoretically is below  $n=4$  (21).

Only by realizing that the dislocation has to increase its line length in order to surmount the dispersoid, can a climb-related "threshold stress" be justified. This process has been modelled by Brown and Ham (22) and Shewfelt and Brown (23), who assumed that climb is "local"; this means that only the portion of the dislocation which is in close proximity with the particle-matrix interface undergoes climb while the remaining segment stays in the glide plane (fig. 4 - broken dislocation line running along particle contour).

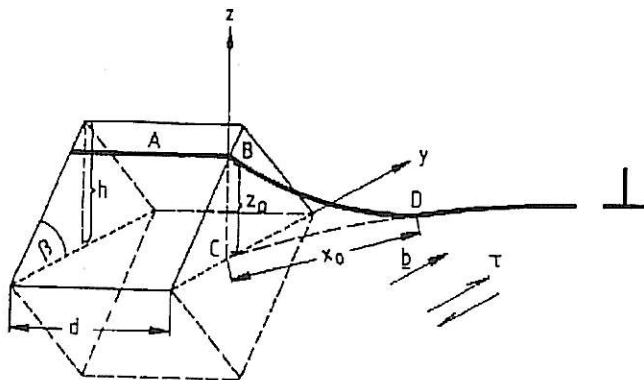


Fig. 4: Geometric assumptions for modelling climb of dislocations over dispersoids: "equilibrium climb" (28) - full line - and "local climb" (22) - broken line.

For climb over the dispersoid to occur, internal energy must be gained everywhere along the dislocation path, which requires the stress to exceed a "threshold stress"  $\sigma_{th}$  for local climb:

$$\sigma \geq \sigma_{th} = \frac{1}{2} \left[ \frac{dL}{dx} \right]_{\max} \sigma_{Or} \quad (3)$$

This threshold stress is proportional to the Orowan stress. The other important parameter is the differential of the dislocation line length  $L$  with respect to advance distance  $x$ , at the point of its maximum. This quantity is called "climb resistance". During "local climb" the dislocation profiles the particle contour and therefore the climb resistance is related to particle shape. A detailed analysis shows that the threshold stress amounts to about 70 % of the Orowan stress for cubes oriented as in fig. 4 (22), and to about 40 % for spheres (23). We note that these threshold considerations lead to values which appear to be in the same range as experimental values (cf. fig. 3). The temperature dependence however remains unexplained.

As anticipated by Shewfelt and Brown (23), there are serious objections that can be raised against the basic assumption of "local" climb. The postulate concerning dislocation shape in the vicinity of the dispersoid particle must be regarded as unduly restrictive. Lagneborg (24) has argued that local climb would be an extreme non-equilibrium process: the sharp bend in the dislocation at the point where it leaves the particle-matrix interface will in reality be rapidly relaxed by diffusion, leading to more "general" climb. Because the additional line length then required depends on the kinetics, a "back stress" is predicted which scales as the applied stress. This would result only in a retardation of creep, with the same stress exponent as for the particle-free material; such a behaviour would clearly be in contradiction with experimental data on dispersion-strengthened materials. In addition, as discussed by Rösler (25), there appears to be a flaw in the calculations, leading to gross overestimation of the diffusive fluxes.

In the meantime it has become clear that a finite, but small threshold stress must always exist: in order to thread over and under dispersoid particles in a random arrangement, a small elongation of the dislocation line is inevitable. The resulting threshold stress for "general climb" over spherical particles (23,26) amounts to negligible numerical values (less than 10 % of the Orowan stress) for low volume fractions up to 10 %. When a random particle distribution is assumed (26) and when Friedel statistics is applied (27) these values are further reduced to less than about 2 %. In conclusion, it can be said that climb-related threshold stresses are sensitive to the details of the climb process especially in one regard: the degree to which climb is localized at the particles.

In order to solve the long-standing problem of climb localization, Rösler and Arzt (28) have considered the kinetics of dislocation climb over cuboidal particles. The only assumption concerning the dislocation geometry was that of local equilibrium (fig. 4): the chemical potential for vacancies along segment  $BD$  (where it is lowered by the curvature) is set equal to the chemical potential along  $AB$  (where it is negatively biased by the applied stress). Local equilibrium can be assumed to form rapidly by short-range diffusion, while the supply of new vacancies for climb and dislocation advance typically requires diffusion over the distance of one particle spacing. This leads to a more natural dislocation configuration for which the diffusion kinetics is then evaluated.

The resulting constitutive equation for "equilibrium climb" does not contain any adjustable parameters:

$$\dot{\epsilon} = \frac{\rho G b^4}{k_B T d^3} \left[ a_p D_p + \pi D_v \ell d \right] \left[ \frac{\sigma - \sigma_{th}^r}{\sigma_{Or}} \right]^n \quad (4)$$

where the stress exponent  $n$  lies between 3 and 4, depending slightly on particle shape,  $B$  contains the particle shape,  $\rho$  is the density of mobile dislocations,  $d$  is the particle width according to fig. 4,  $\ell$  is the particle spacing,  $a_p D_p$  is the cross section of a dislocation core times its diffusivity,  $D_V$  is the volume diffusivity,  $\sigma$  the applied stress,  $\sigma_{th}$  a small threshold stress for "general" climb and  $\sigma_{0c}$  the Orowan stress.

An important result which emerges from this treatment of "equilibrium climb" is that truly "local" climb is always unstable, but the extent to which climb is localized in the vicinity of the dispersoid particle increases with applied stress. This implies that the number of vacancies required for climb (which is proportional to the length of the climbing segment) decreases as the stress increases, and therefore the dislocation velocity depends non-linearly on stress. The maximum climb-related stress exponents predicted by this model ( $n \approx 6$ ) are however far below the values typical of dispersion-strengthened materials.

In summary, it seems that the assumption of dislocation climb alone cannot explain the high stress sensitivity for creep in dispersion-strengthened materials. In other words, if the only effect of dispersoid particles consisted in forcing the dislocations to climb over them, such particles would be weak barriers to slow creep deformation at high temperatures.

#### Dislocation Detachment Models

In the search for a genuine threshold stress mechanism, attention has only recently turned to the details of the possible interactions between dispersoids and hard particles. Much insight has been gained by transmission electron microscopy of the dispersoid-dislocation configurations in creep-exposed specimens of dispersion strengthened alloys (29-31, 14, 25). The most detailed study to date has been conducted on the ODS superalloy MA 6000, which was creep-deformed at temperatures near 1000 °C and under stresses well below the Orowan stress. A typical micrograph, taken subsequently under "weak-beam" conditions by Schröder (30,31), is shown in fig. 5. The dislocation is seen in a situation where it has already surmounted a dispersoid by climb and adheres to the "departure side" of the dispersoid. Under "weak-beam" conditions, the contrast of the dislocation segment which resides in or near the dispersoid-matrix interface is often well visible.

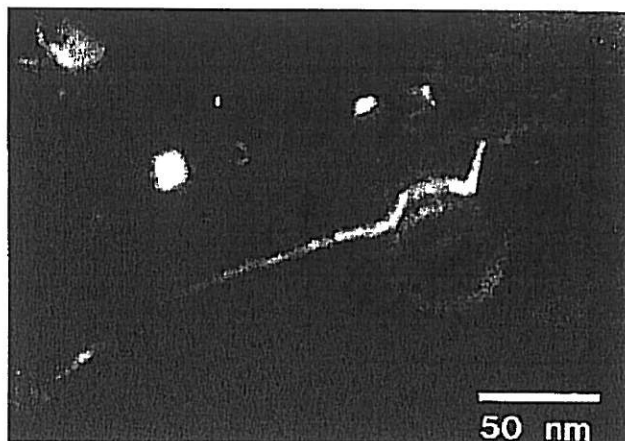


Fig. 5: TEM evidence for an attractive dislocation-dispersoid interaction during creep of Inconel MA 6000 (Refs. 30,31).



Similar, but less detailed observations were reported in the dispersion strengthened NiCr alloy MA 754 by Nardone and Tien (29), who were the first to point out the possibility of pinning on the "departure-side", and in dispersion strengthened Al by Rösler (25). While these TEM studies do by no means constitute unambiguous proof, they strongly suggest a new dislocation mechanism: particle bypassing by climbing dislocations may not be controlled by the climb process (as postulated in the early threshold stress models), but by a resistance to dislocation detachment from the particle. In other words, "threshold stresses" for creep would have to be attributed to an attractive particle-dislocation interaction.

Recent theoretical analysis confirms indeed that the assumption of an attractive particle-dislocation interaction at high temperatures is plausible. Srolovitz et al. (32) have solved the elastic problem of an edge dislocation interacting with a (cylindrical) particle, subject to the boundary conditions that both tangential tractions and gradients in the normal tractions at the particle-matrix interface are instantaneously relaxed. Such relaxation processes can be accomplished at elevated temperatures by boundary and volume diffusion, and by sliding of the phase boundary. Calculation of the characteristic relaxation times suggests that relaxation, at least of deviatoric stresses, will indeed occur quickly; therefore the dispersoid can be treated as a void (with an internal pressure corresponding to the hydrostatic component of the applied stress). The Srolovitz solutions show that at high temperatures the elastic interactions between dispersoids and dislocations become unimportant: even an infinitely stiff particle (which repels dislocations at low temperatures) can produce a strong attractive force, provided it is not coherent with the matrix. Srolovitz et al. (32) further suggest that core delocalization will occur in the interface. Therefore, the unpinning stress should be similar to that for voids, i.e. approximately equal to the Orowan stress (33).

There appears to be, however, an important discrepancy of these predictions with some TEM observations. The "weak-beam" micrograph in fig. 5 shows clearly that the identity of the dislocation is preserved in the vicinity of the particle-matrix interface and the local strain is not noticeably reduced by core spreading. Contrary to the behaviour of a void, the dispersoid evidently allows only a modest relaxation of the dislocation. That the detachment threshold can act as the decisive dislocation barrier even at small degrees of relaxation has been established by Arzt and Wilkinson (34). They treat the effects of an attractive interaction on the energetics of dislocation climb. In their model a variable attractive interaction is introduced by assigning a line tension to the dislocation segment at the particle which is lowered by a factor  $k$  ( $k < 1$ ). This parameter describes the extent to which the dislocation relaxes its energy by interaction with the particle-matrix interface. For  $k=1$ , no relaxation and, thus, no attractive interaction occurs;  $k=0$  signifies maximum interaction which is approximately realized by the presence of a void.

The authors have calculated force-distance profiles for local climb under the action of different attractive forces. The main result is that an attractive interaction causes a significant threshold stress which must be exceeded in order to detach the dislocation from the back of the particle and which is given by:

$$\sigma_d = \sqrt{1 - k^2} \sigma_{Or} \quad (5)$$

This "detachment threshold" is independent of the shape of the particle and its position with respect to the glide plane. It applies regardless of the details of the climb process which precedes detachment.

Because of the reduced line tension of the dislocation segment at the dispersoid, the "threshold stress" for local climb is lowered. This leads to an important conclusion: only a very modest attractive interaction, corresponding to a relaxation of about 6% ( $k \approx 0.94$ ), is required in order for dislocation detachment to become the event which controls the threshold stress. Because, in view of Srolovitz' calculations, some relaxation will always occur at the



interface, these results lend strong support to the detachment process as a serious candidate for a "threshold stress" mechanism.

Recently, a kinetic model for dislocation climb which allows for the effects of an attractive interaction has been developed by Arzt and Rösler (35). In order to complete the model, thermally activated dislocation detachment from dispersoids needs to be considered also. This aspect has been analyzed in detail by Rösler (25) and Rösler and Arzt (36,37). The resulting constitutive equation for "detachment-controlled creep" is given by:

$$\dot{\epsilon} = \dot{\epsilon}_0 \exp \left[ - \frac{G b^2 r}{k_B T} (1-k)^{3/2} \left(1 - \frac{\sigma}{\sigma_d}\right)^{3/2} \right] \quad (6)$$

where  $\dot{\epsilon}_0 = 3 D_V l \rho / b$  and  $\sigma_d$  is given by eq. 5.

Eq. 6 is plotted in fig. 6 for different values of  $k$ . Several features which are in qualitative agreement with experimental results can be identified: Dispersoids with a strong attractive interaction ( $k < 0.85$ ) do indeed produce a region of high stress exponent which extends over many orders of magnitude in strain rate; this gives the appearance of a "threshold stress", which falls with increasing temperature. As the interaction gets weaker (or, equivalently, if the temperature is raised), a concave curvature at low stresses becomes apparent; this corresponds to a loss of strength which would not be expected by linear extrapolation of the data from higher strain rates. It is also clear from fig. 6 that there is no "universal" stress exponent for dispersion-strengthened materials; rather the stress sensitivity depends on temperature, stress and dislocation-dispersoid interaction.

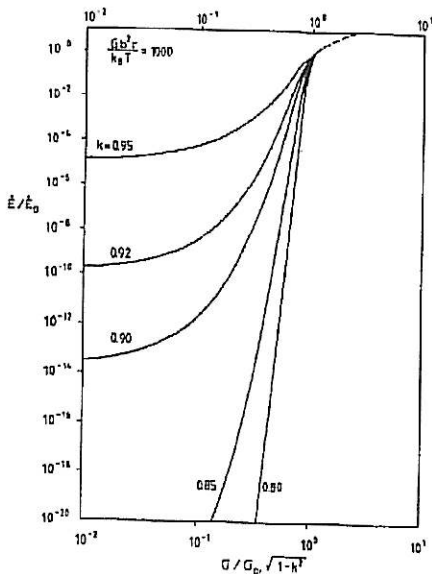


Fig. 6: Theoretical prediction of the creep rate (normalized) as a function of stress (normalized) on the basis of thermally activated dislocation detachment from attractive dispersoids (eq. 6), as a function of interaction parameter  $k$  (from Ref. 37).

When the interaction parameter  $k$  and the pre-exponential factor are adjusted, eq. 6 can be made to describe the creep behaviour of several dispersion strengthened materials extremely

well. Extensive data evaluation shows that the value of  $k$ , obtained by fitting the theory to the creep data, varies characteristically with the type of dispersoid (table 1): carbides in aluminium seem to be most efficient in attracting dislocations, while oxides appear less suitable (25,8,37). Also the rapidly solidified Al-Fe-Ce alloy studied by Yaney et al. (38,39) fits well into the theoretical scheme. Its "anomalous" creep behaviour can be fully described by assigning a weak interaction effect ( $k=0.95$ ), again independent of temperature, to the intermetallic precipitates. This interpretation is also consistent with Yaney et al. (38), who have suggested that the loss of strength at higher temperatures cannot be attributed to thermal instability of the microstructure.

Material	Reference	Dispersoid	$k$
AlC2	25, 14	$Al_2O_3 + Al_4C_3$	0.75
AlC0	25, 14	$Al_2O_3$	0.84
AlMg4C1	25, 14	$MgO + Al_4C_3$	0.85
Al-Fe-Ce	38	intermetallic	0.95
MA 6000	4	$Y_2O_3$	0.92

Table 1: Interaction parameter  $k$  for different materials, determined by fitting eq. 6 to experimental creep data (25).

### 2.3 Practical Implications

On the basis of the detachment model described above several conclusions can be drawn, some of which must necessarily remain speculative at the present state. Above all, the "threshold stress" concept, when applied uncritically, can be seriously misleading. Contrary to the implications of the local climb models there exists no significant minimum "threshold stress" (apart from the small threshold for general climb) that must be exceeded in order to sustain creep deformation. Laboratory creep rates may indeed give the impression of a "threshold stress" behaviour but eventually, at sufficiently low strain rates, an upward curvature of the curves is inevitable (fig. 6). Until now such a loss in strength has been attributed alternatively to the dissolution, coarsening or deformability of dispersoid particles (in TD-NiCr single crystals (9), and Al (13)) or to the onset of grain boundary sliding or damage formation (MA 754, Ref. 10, and Al, Ref. 14).

For the purpose of life-time prediction, it must be realized that extrapolations from laboratory data to low creep rates can be seriously in error. Only materials with highly attractive dispersoids ( $k < 0.8$ ) would permit linear extrapolation. The value of  $k$ , which is therefore important, can be estimated, by a method proposed by Rösler (25), from the values of the apparent stress exponent and the apparent activation energy for creep. In conclusion, even in cases where near-"threshold stress" behaviour is observed, it seems warranted, in the interest of clarity, to use the term "pseudo-threshold" instead.

The practical perspectives in terms of alloy design are enticing but still vague. Given a certain usable volume fraction of dispersoid (which is limited by minimum ductility requirements), the "detachment model" predicts that there should be an optimum particle size (and spacing). Physically, this comes about because (i) the probability of thermally activated detachment is raised for small dispersoids, and (ii) large particles are associated, at a given volume fraction, with a low Orowan stress and hence a small athermal detachment stress  $\sigma_d$ . This further implies that the creep of strength of alloys with initial particle size below this optimum value would be insensitive to moderate particle coarsening.

Furthermore, "detachment" discriminates different dispersoids by the degree of dislocation relaxation, suggesting that the properties of the dispersoid-matrix interface should be of criti-

cal importance. Interestingly, table 1 suggests tentatively that particles dispersed by mechanical alloying tend to be more efficient than those produced by (rapid) solidification. It remains to be seen whether concepts such as interface modification by segregation alloying can be put to use in order to further improve the efficiency of dispersoids.

### 3. Grain Boundary Effects on Creep Deformation and Fracture

The previous considerations, which concerned the role of dispersoid particles in impeding the motion of lattice dislocations, can be strictly valid only for single crystals, in which grain boundaries do not contribute to creep deformation. Grain boundary weakening is of particular importance for polycrystalline dispersion-strengthened materials which as engineering materials are subjected to moderate stresses at high homologous temperatures. Because of processing limitations, single crystals of dispersion strengthened materials are difficult, if not impossible, to produce in useful dimensions. Therefore the effects of grain boundaries on deformation and fracture have received considerable attention in the literature; the understanding is however still incomplete as will be shown below.

#### 3.1 Some Experimental Observations

In general, grain boundaries can lower the high-temperature strength in at least three ways: i) stress-induced vacancy accumulation leads to cavity formation on grain boundaries transverse to an applied tensile stress and, subsequently, to premature intergranular failure; ii) grain boundary sliding results in stress concentrations which accelerate dislocation creep; and iii) by acting as vacancy sinks and sources, grain boundaries promote additional grain deformation due to diffusional creep ("Nabarro-Herring creep").

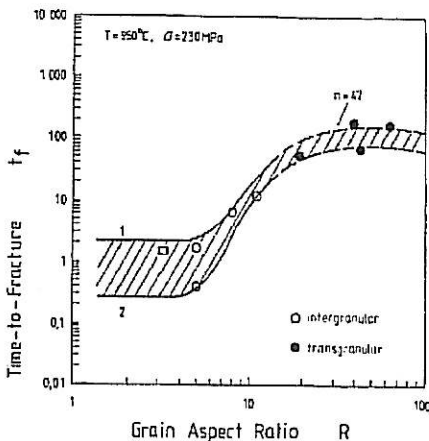


Fig. 7: Creep rupture time of coarse grained Inconel MA 6000 as a function of grain aspect ratio after recrystallization by zone annealing. Shaded area shows range of theoretical predictions based on eq. 7 (from Ref. 42).

In order to suppress premature fracture, nickel-base ODS alloys are commonly recrystallized by zone annealing to obtain an extremely elongated, coarse grain structure (40). Fig. 7 shows that the decisive parameter is the grain aspect ratio (GAR), see also (41). Fine grains, which constitute recrystallization defects and do not necessarily differ in chemical composition from the otherwise coarse-grained matrix, are particularly susceptible to the formation of creep damage. They reduce creep life by acting as starting points for transgranular cracks (42). The property degradation which results from this mechanism has been found to be even more aggravated under combined creep-fatigue loading (43,44).

An important corollary of the severe vulnerability of grain boundaries in ODS materials is a pronounced anisotropy of tensile creep properties. While (short term) tensile strength (45) and short-term compressive creep strength (11) are relatively insensitive to loading direction with respect to the extrusion axis (grain elongation), tensile creep strength is substantially reduced in the transverse directions (46) (fig. 8). The reason is again the occurrence of premature intergranular fracture at the longitudinal grain boundaries, with a concomitant severe reduction in ductility. At low stresses creep rates are affected even under compressive transverse loading, which leads to considerable deformation below the "threshold stress" (46). This type of loading produces  $\gamma'$ -free diffusion zones on grain boundaries parallel to the compression axis (fig. 8), which has been interpreted as being indicative of a contribution by diffusion creep. What is atypical of diffusion creep, however, is the high stress exponent that applies in this regime. Similar evidence for diffusion creep has been found earlier by Whittenberger (47).

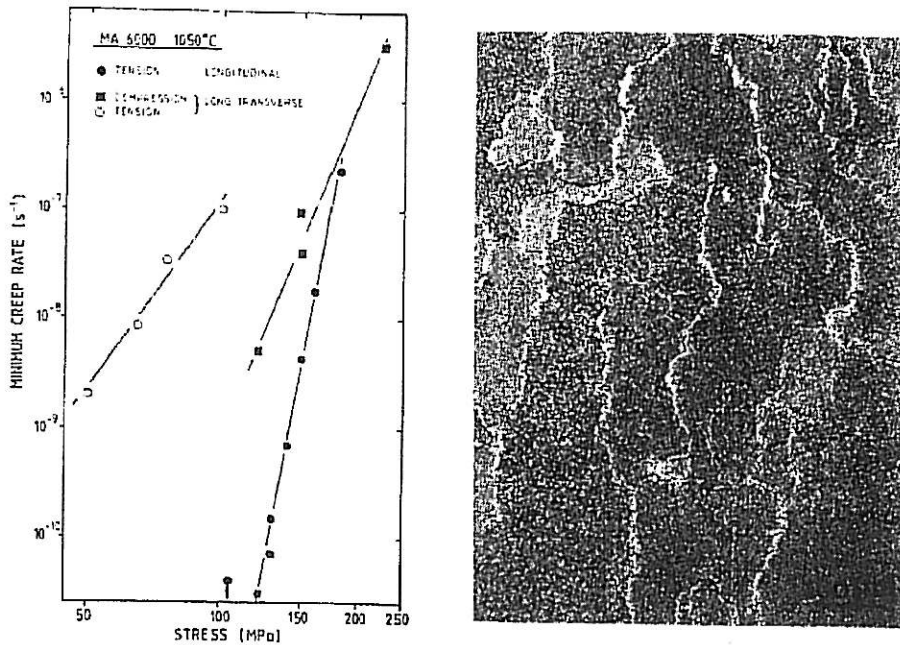


Fig. 8: Results of creep tests with Inconel MA 6000 in the long transverse direction (from Ref. 46): left: comparison of creep rates, under tension and compression with longitudinal data (tension); right: evidence for diffusion creep in the form of precipitate-free zones on a transverse section after compressive loading in the long transverse direction (stress axis vertical).

Grain size per se is very important: for the rate of creep deformation in nickel-base ODS alloys; fine-grain (as-extruded) material exhibits creep strengths at least an order of magnitude lower than recrystallized material as is seen in fig. 9. By contrast, the difference in creep strength between fine-grained and recrystallized Al-Al<sub>2</sub>O<sub>3</sub> appears to be much smaller ( $\approx 30\%$ ) at a similar homologous temperature. A grain coarsening treatment may thus be less advantageous in the latter case.

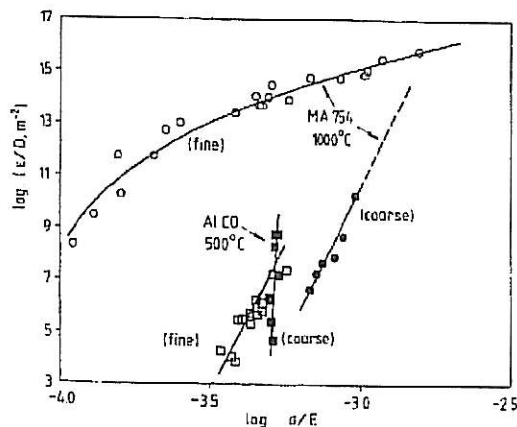


Fig. 9: The effect of grain size on the creep strength of Ni-base ODS alloy MA 754 (data from Ref. 48) and of Al-Al<sub>2</sub>O<sub>3</sub> (data from Refs. 25,49,50).

### 3.2 Theoretical Understanding of Grain Boundary Effects

Models for the growth of creep cavities in elongated grain structures typical of some modern dispersion-strengthened materials have been developed by Arzt (51), Arzt and Singer (52), Stephens and Nix (53), and Zeizinger and Arzt (42). In view of the high resistance to dislocation creep, all models assume pore growth to occur by diffusion; this is supported by observations of particle-free zones in the ligaments between creep cavities (42). The theories differ in the type of accommodation process necessary for assuring compatibility between the deformation of damaged and undamaged grains. When sliding along longitudinal grain boundaries provides sufficient accommodation, a dependence on the grain aspect ratio arises naturally from the coupling of grain boundary sliding and cavity growth, between which the applied stress is distributed. A shortcoming of this approach is the linear stress dependence of the creep rate. Also, sliding could not be detected in model experiments (42), which casts some doubt on the applicability of the model to alloys like MA 6000.

Another possibility for damage accommodation is by dislocation creep, enhanced by the shedding of load from cavitating grain boundaries to the adjacent intact grains. A numerical solution for this case by Stephens and Nix (53) gives a strong dependence of the rupture time on GAR. The experimental strain-rate/stress dependence of MA 754 with uniform fibre grain morphology is well described by this model, except at low stresses where creep strength is consistently overestimated. Zeizinger and Arzt (42) take a similar modelling approach which yields the following analytical equation for the rupture time as a function of grain aspect ratio  $R$ :

$$t_f = \frac{0.085 k_B T \lambda^3}{\delta D_b \Omega \sigma} \left[ 1 - \left(1 - \frac{1}{R}\right)^n \right] + \frac{\epsilon_B}{\dot{\epsilon}_u} \left(1 - \frac{1}{R}\right)^n \quad (7)$$

where  $\lambda$  is the cavity spacing,  $\epsilon_B$  the total strain due to void growth and  $\dot{\epsilon}_u$  the creep rate of uncavitated material. This equation, which was derived on the basis of a model by Cocks and Ashby (54) for equiaxed grains, is found to describe the GAR-dependence of the rupture

time in MA 6000 well. Also the poor rupture properties in the transverse direction can be understood because a low GAR applies in this case.

In the light of these investigations it appears that the GAR-dependent "threshold stresses for diffusional creep" reported by Whittenberger (55) could also be a consequence of the coupling between grain boundary damage processes and accommodation by dislocation creep with a pseudo-threshold stress. As the GAR increases, the accommodation process becomes increasingly more important and the material exhibits a larger fraction of the pseudo-threshold for matrix creep.

We now turn to the case in which adherence of the grain boundaries is maintained during creep, but shear stresses are relaxed by grain boundary sliding. This mechanism has originally been invoked to explain the inferior creep strength of low-GAR grain structures (56). Detailed modelling of the coupling between grain boundary sliding and dislocation creep (57) shows, however, that the stress concentrations resulting from sliding are quite small: the loss in creep strength due to grain boundary sliding is predicted to be only about 20 %. Apart from a narrow transition region, the stress exponent of the dislocation creep process is retained down to low strain rates. The only experimental results which do not immediately contradict this prediction seem to be the creep data for dispersion-strengthened aluminium measured by Rösler et al. (50) and Joos (49) (fig. 9). The grain boundaries in this material apparently do not contribute to premature creep fracture, which is in contrast to MA 6000. In MA 754 the strength difference between fine and coarse grain material (fig. 9) is much higher and cannot be interpreted in terms of grain boundary sliding alone (as discussed by Nix (48)). A possible explanation for this apparent difference may be connected with the lack of texture in fine-grained MA 754 as opposed to the Al alloy which has a strong  $\langle 111 \rangle$  texture (49).

Finally, diffusion creep can occur in fine-grained materials, which requires grain boundaries both, to act as vacancy sinks and sources, and to slide. Several models have been put forward to predict the effect of grain boundary dispersoids on diffusional creep. One type of model, pioneered by Ashby (58) and detailed by Arzt et al. (59), considers the emission and absorption of vacancies at the grain boundaries (the "interface reaction") as the rate-limiting process; this idea is supported by atomistic studies of grain boundaries which suggest a fairly well-defined structure: vacancies can be produced or absorbed only at grain boundary dislocations which have to move non-conservatively in the grain boundary plane. Because these dislocations are pinned by grain boundary particles, a minimum "threshold stress" for diffusional creep is predicted which is equal to the Orowan stress or the "local climb" stress for boundary dislocations. A difficulty in applying this model lies in the assumption that must be made with regard to the Burgers vector of the boundary dislocation; it is only clear that it is smaller than for lattice dislocations. In analogy to dislocation creep, an attractive interaction between boundary dislocations and particles could also play a role, as suggested by TEM studies of grain boundary dislocations interacting with particles (60). But this effect has not been included in the models so far.

In another type of model it is assumed that the grain boundaries, but not the particle-matrix interfaces, act as perfect sources and sinks for vacancies. A stress concentration then builds up at the particles, which must be relaxed before creep can continue. Relaxation can occur by the nucleation of lattice dislocation loops (61), or alternatively, defect loops can be nucleated in the particle-matrix interface (62). Both processes give rise to a "threshold stress" for diffusion creep.

It is difficult to ascertain how important the role of diffusion creep is in the engineering alloys considered in this review. The experimental evidence for diffusion creep is limited to occasional observation of particle-free zones (e.g. 46,55). The stress dependence of the creep rate however is always found to be non-linear, but lower than in coarse-grained material. Such intermediate stress exponents ( $n = .12...16$ ) are difficult to justify theoretically. It is clear



from the absolute magnitude of the creep rates that diffusion creep is suppressed or strongly inhibited in the fine-grained dispersion-strengthened Al alloys.

### 3.3 Practical Conclusions

Grain boundaries can be extremely vulnerable microstructural elements in dispersion-strengthened high-temperature materials. The effect of GAR on creep rupture time of nickel-base alloys is well established and understood. It follows that, in order to minimize the detrimental influence of grain boundaries, a high GAR in the loading direction must be achieved. Also the generally poor properties in the transverse directions are due to the presence of grain boundaries and could possibly be improved in one transverse direction by increasing the GAR in that direction as well. Diffusion creep apparently places a lower bound on the creep rates in the transverse directions; this effect can be minimized by increasing the width of the elongated grains. Since this will tend to lower the overall GAR in the longitudinal direction, an optimum balance between longitudinal and transverse properties would need to be struck for a given application. Fine-grain regions should be eliminated as far as possible in order to exploit the full potential of dispersion-strengthening with regard to creep and high-temperature fatigue properties. Single crystals of course would in principle be an ideal solution.

The advantages of grain coarsening appear to be much smaller for dispersion-strengthened aluminium than for nickel alloys. Therefore the use of fine-grained materials for high-temperature applications may be feasible in some cases. The reasons for this difference in grain boundary behaviour are not understood. Clearly more scientific and technological work on the creep behaviour of extremely fine-grained dispersion-strengthened alloys is urgently necessary.

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