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Creep in Dispersion Strengthened Aluminium Alloys at High Temperatures – A Model Based Approach

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Abstract

The mechanism of dispersion strengthening has been studied in dispersion-strengthened aluminium alloys produced by reaction milling. The beneficial effect of second phase particles is usually attributed to the existence of a "threshold stress", below which particles cannot be surmounted by climbing dislocations. But, as will be shown below, predictions of this "threshold stress" concept are widely inconsistent with the experimentally observed creep behaviour. A new constitutive equation is proposed which is based on TEM observations suggesting thermally activated dislocation detachment from the particles to be rate controlling. Creep in several dispersion-strengthened alloys can be consistently described with this concept, which also points to ways of optimizing these alloys.

1. Introduction

Dispersion-strengthened aluminium alloys exhibit excellent creep properties at temperatures as high as 0.8 T_m. Thus they are promising candidates for applications above about 300 °C where precipitation hardened alloys rapidly lose their strength because of microstructural degradation. Although the beneficial effect of a finely distributed, thermally stable dispersion of particles has been well known since the pioneering work of Irmann (1) and Zeerleder (2) it is still debatable why dispersoids can retard the motion of dislocations so effectively. Up to now it is widely believed that "local climb" as proposed by Brown and Ham (3) and Shewfelt and Brown (4) is the rate controlling process causing a threshold stress below which creep should be negligible. In the past, serious objections have been raised against this model. Lagneborg (5) has argued that "local climb" should be an energetically unstable process. A recent analysis of the climb kinetics by Rösler and Arzt (6) does indeed show that climb should never be truly local and that dispersoids should be only weak obstacles to the motion of dislocations at high temperatures if the climb process itself were rate controlling.

An alternative mechanism for the decisive dislocation process was suggested by Srolovitz et al. (7,8). They showed that dislocations can be attracted to the dispersoid-particles at high temperatures because of diffusional relaxation of the stress field at the particle-matrix interface. Arzt and Wilkinson (9) subsequently showed that a significant "threshold stress" σ_d must be exceeded in order to detach dislocations from attractive particles. The kinetics of the detachment process was calculated by Rösler (10) and Rösler and Arzt (11), who propose a new constitutive equation for creep in dispersion strengthened alloys.

The question whether dislocation climb or dislocation detachment is rate limiting is of great practical significance, because the respective models lead to totally different predictions regarding the optimum design of dispersion-strengthened high-temperature alloys. It is the aim of the present paper to analyse the creep data of dispersion-strengthened aluminium alloys in the light of the new constitutive equation. Consequences for the optimum design of these alloys will then be drawn from these results. The application of the concept to other alloys is described in (11-13).

2. The Creep Models

As illustrated in fig. 1 the current dislocation models propose different rate-controlling deformation mechanisms. In the case of the more "classical" concept, "local climb" at the front
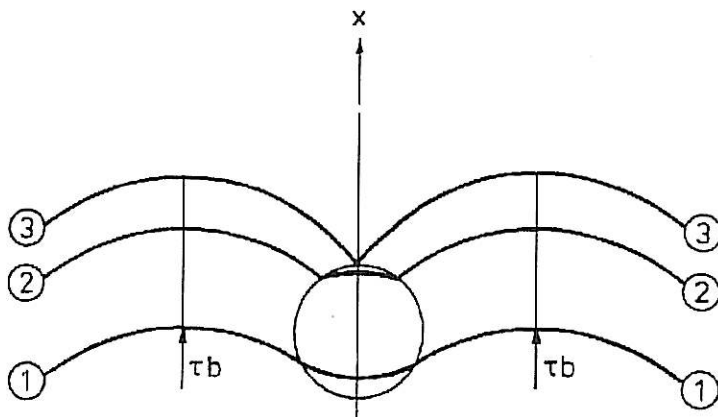
Fig. 1: Schematic illustration of the proposed rate controlling deformation mechanisms for the different creep models; 1: local climb of the dislocation over the dispersoid; 2+3: dislocation detachment from the disperoid.

side of the particle is believed to be rate-limiting. This means that only the dislocation segment at the particle is allowed to climb, whereas the dislocation arms stay in the glide plane. From this geometrical assumption a threshold stress σ_{th} results, which is about 70 % of the Orowan stress for cubes (3) and 40% for spheres (4). Thus the semi-empirical power-law equation for creep can be corrected by introducing an effective stress $\sigma - \sigma_{th}$:

$$\dot{\epsilon}/D = A' \cdot \frac{Gb}{k_B T} \left[\frac{\sigma - \sigma_{th}}{G} \right]^n \qquad (1)$$

with creep rate $\dot{\epsilon}$, diffusivity D, shear modulus G, Burgers vector b, dimensionless constant A' and thermal energy $k_B T$. The following predictions regarding the creep behaviour of dispersion-strengthened materials arise from eq. (1):

- The activation energy for creep should be that for volume diffusion. Thus the creep data should be temperature-independent when the normalized strain rate $\dot{\epsilon}/D$ is plotted versus the normalized stress σ/G.
- The influence of the second phase particles should be only of geometrical nature, with the value of σ_{th} depending on the interparticle spacing but not on temperature (except through G). The creep strength is predicted to be independent of the crystal structure and interfacial properties of non-shearable particles.

Because it is difficult to rationalize from a theoretical standpoint why dislocations should climb locally (5,6), attention has focussed on the detachment process as also illustrated in fig. 1. In fact it has been shown by Arzt and Wilkinson (9) that only a slight dislocation line energy relaxation of about 6 % at the particle is necessary to shift the rate controlling mechanism from local climb to the detachment process. This point of turn over would be shifted towards even smaller relaxations if more realistic climb geometries were assumed. The kinetics of the detachment process, which is thermally activated, was recently quantified by Rösler (10) and Rösler and Arzt (11), who derive a new constitutive equation for creep in dispersion-strengthened materials:

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

with $\dot{\epsilon}_0 = 6\rho\lambda D/b$. Here ρ is the dislocation density, 2λ the interparticle spacing, and r the particle radius. The numerator in the exponential term is the activation energy $\dot{E}_d$ necessary to detach the dislocation from the back of the particle.

Two important parameters appear in eq. (2). First, the parameter k defined by

$$k = \frac{E_p}{E_m} \qquad (3)$$

where E_m and E_p are the dislocation line energies in the matrix and at the particle respectively. Maximum creep strength is obtained for $k \to 0$ i.e. for maximum relaxation at the particle, whereas the detachment barrier vanishes for $k \to 1$ (= no relaxation). That the creep strength is a strong function of k is illustrated in fig. 2, where the calculated creep rate $\dot{\epsilon}/\dot{\epsilon}_0$ is plotted versus σ/σ_d. A remarkable transition in the creep behaviour occurs at $k \approx 0.9$. For $k < 0.9$ high stress exponents up to insignificant small creep rates are predicted whereas for $k > 0.9$ the creep strength is predicted to degrade significantly at low strain rates and/or high temperatures, resulting in low values of the stress exponent n.
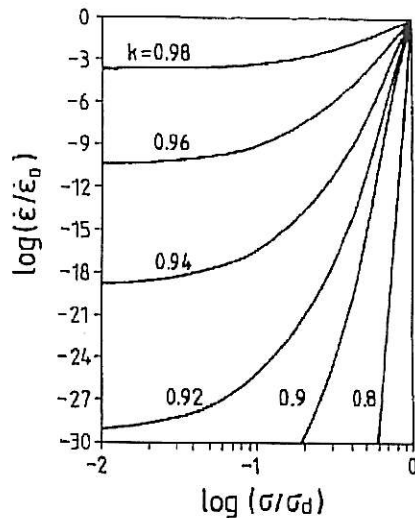

Fig. 2: Calculated stress dependence of $(\dot{\epsilon}/\dot{\epsilon}_0)$ as a function of the interaction parameter k with $Gb^2r/k_B T = 3000$.

The second parameter is the athermal detachment stress σ_d. For stresses $\sigma \geq \sigma_d$ the dislocation can be detached spontaneoulsy from the particle back. As shown by Arzt and Wilkinson (9) the athermal detachment stress is related to the Orowan stress σ_0 by

$$\sigma_d = \sigma_0 \cdot \sqrt{1-k^2} \qquad (4)$$

From the concept of thermally activated detachment the following predictions regarding the creep strength of dispersion hardened materials can be made:

- The apparent activation energy for creep should be $Q_{app} = Q_V + E_d$ (Q_V: activation energy for volume diffusion; E_d: activation energy for the detachment process). Thus the creep strength is expected to be temperature dependent even when plotting $\dot{\epsilon}/D$ versus σ/E.
- The creep strength should be a strong function of the interaction parameter k (fig. 2) and thus of the particle-matrix interfacial properties.
- Because, at a given volume fraction f_V, the athermal detachment stress σ_d is a function of the particle radius r it follows that an optimum particle radius should exist (10,11):

$$r_{opt} = 0.3 \frac{b \cdot \sqrt{1-k^2} \cdot \sqrt{f_V}}{\sigma/E} \qquad (5)$$

These predictions differ substantially from those of the local climb concept in that the interfacial properties, i.e. the particle/matrix combination, are now expected to become important and that an intermediate rather then a minimum diameter should be optimum for non-shearable particles. In the following section an attempt will be made to distinguish between the two models by analyzing experimental creep data in the light of eqs. 1 and 2.

3. Analysis of Creep Data

The alloys investigated in detail were Al-2.2% C-0.8% O (subsequently referred to as "AlC2") and Al-4% Mg-1.4% C-2.6% O ("AlMgC1") (all percentages by weight). Both materials were produced by reaction milling (14,15). The dispersoids in AlC2 are about 8 vol%-Al_4C_3 and 1.5 vol% Al_2O_3, with an average particle size of 43 nm (see also fig. 3). The alloy AlMgC1 is hardened by about 5.2 vol% Al_4C_3 and 4.9 vol% MgO, with a typical particle size of 34 nm (Ref. 10); the magnesium is completely transformed into MgO and is no longer in solid solution. The creep properties of the two alloys are displayed in figs. 4 and 5.
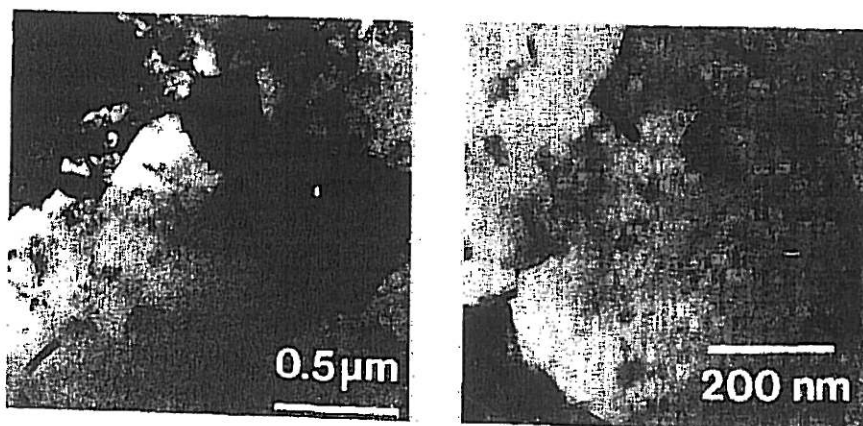

Fig. 3: Micrographs of reaction milled Al-2.2wt% C-0.8 wt% O (alloy "AlC2") showing the fine grained structure (left) and the distribution of carbide and oxide dispersoids (right).
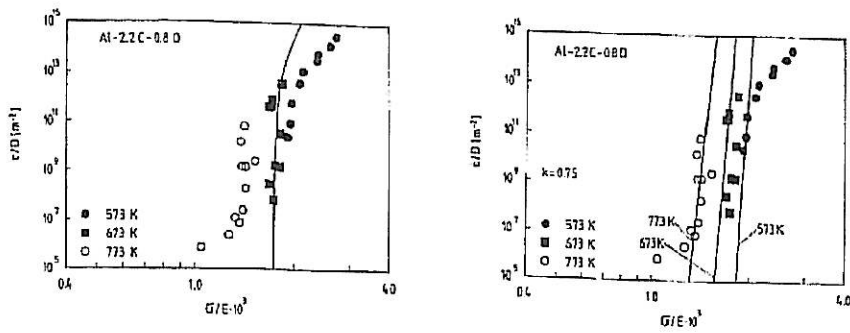
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Fig. 4: Creep data of Al-2.2 wt% C-0.8wt% O (alloy AlC2);
left: comparison with the model calculations (solid line) due to the threshold stress concept;
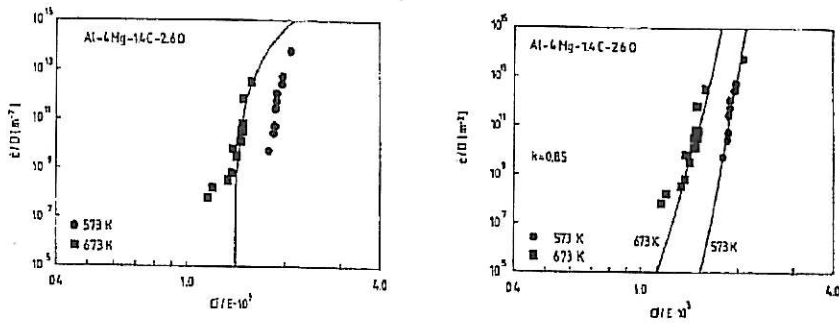right: comparison with the model calculations (solid lines) due to the detachment concept.

Fig. 5: Creep data of Al4 wt% Mg-1.4 wt% C-2.6 wt% O (alloy AlMgC1);
left: comparison with the model calculations (solid line) due to the threshold stress concept;
right: comparison with the model calculations (solid lines) due to the detachment concept.

A striking feature of the creep data is the extremely high stress sensitivity, especially in the case of AlC2 at low normalized strain rates. In this regime creep is controlled by the inter-action of single dislocations with dispersoids, whereas at high strain rates ($\dot{\varepsilon}/D \geq 10^{12}$ m^{-2}) the formation of dislocation networks is observed. The strong drop in the creep strength at the highest temperature and lowest creep rates was shown to be due to the fine grain size (10,16). Since it is possible to optimize the grain structure by means of secondary recrystalli-zation at least for low particle volume fractions (16), this does not seem to be a principal limitation. Thus we will focus our attention on the direct strengthening contribution of the second phase particles, noting that the models introduced in section 2 were not designed to explain the creep behaviour at the highest and lowest normalized strain rates.

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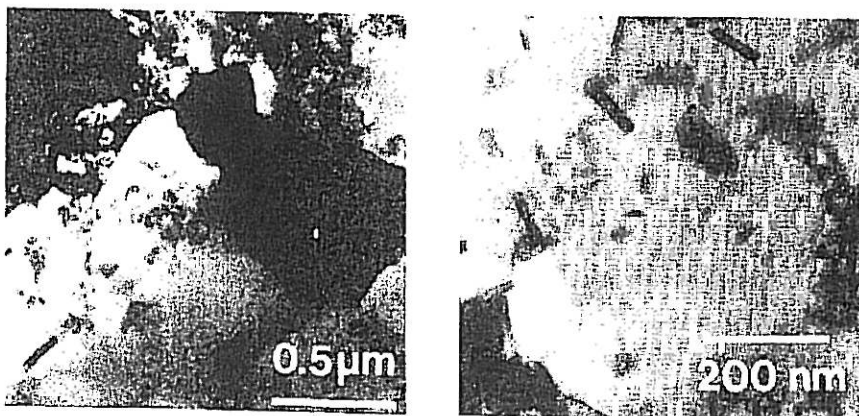

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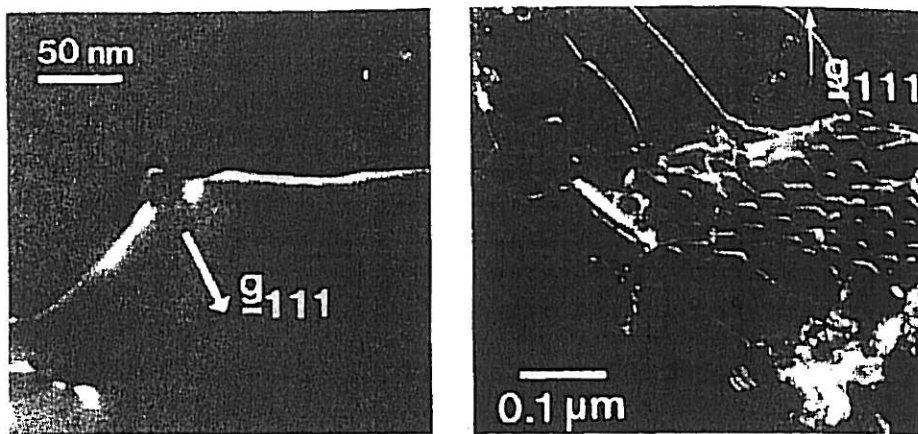
Fig. 6: Typical dislocation configurations after creep deformation (material: AlC2);
left: single dislocation–dispersoid interaction is rate controlling at low strain rates;
right: formation of dislocation networks at high strain rates $\dot{\epsilon}/D \geq 10^{12}$ m^{-2}.

It is remarkable in figs. 4 and 5 that the creep data do not lie on single curves despite modulus and diffusivity correction. This shows that the activation energy for creep is higher than that for volume diffusion. Thus the classical threshold stress concept fails to describe the temperature dependence properly.

The theory of thermally activated dislocation detachment gives a natural explanation for the strong temperature dependence because the activation energy Q_{app} is the sum of the activation energy for volume diffusion and that for the detachment process. Knowing Q_{app} and the stress exponent n_{app} the interaction parameter k can be calculated (10,11). It is seen in fig. 4 that the experimental creep behaviour of AlC2 is well described by assuming a line energy relaxation of 25% at the second phase particles (k=0.75). Applying the same analysis to alloy AlMgC1, k=0.85 is found, which corresponds to a line energy relaxation of 15%.

These results suggest that carbide particles should be more effective barriers to the motion of dislocations at high temperatures than magnesium oxide. In fact the 400 °C creep strength of AlC2 is about 20 % higher than that of AlMgC1 although the mean interparticle spacing is about 24 % less in the latter case (10) (77 nm instead of 101 nm). It would be hard to rationalize the inferior creep properties of AlMgC1 with a climb threshold concept according to which σ_{th} should be inversely proportional to the particle spacing.

The detachment model is further supported by the fact that it explains even the creep behaviour of Al–8Fe–4Ce (17), in which dispersoids are formed by rapid solidification. Its creep behaviour differs from a threshold like behaviour in that the stress exponent declines strongly with decreasing normalized strain rate $\dot{\epsilon}/D$ and a pronounced temperature dependence is observed (fig. 7). One would tend to invoke microstructural instabilities as being responsible for the remarkable loss in strength, but as was shown by Yaney and Nix (17) this contribution is only of minor importance. Instead the concept of thermally activated dislocation detachment offers a natural explanation for this peculiar behaviour. By assuming a very weak particle dislocation attraction (only 5 % line energy relaxation, k=0.95), both the stress and the temperature dependence of the creep rate are satisfactorily described (fig. 7). Although specula-

tive, since detailed TEM investigations are lacking for this type of material, this remarkable result suggests that thermally activated dislocation detachment may be the rate controlling deformation mechanism also in these materials. It is evident from fig. 7 that one would seriously overestimate the creep strength by extrapolating the data points obtained at low temperature by means of the threshold stress concept.
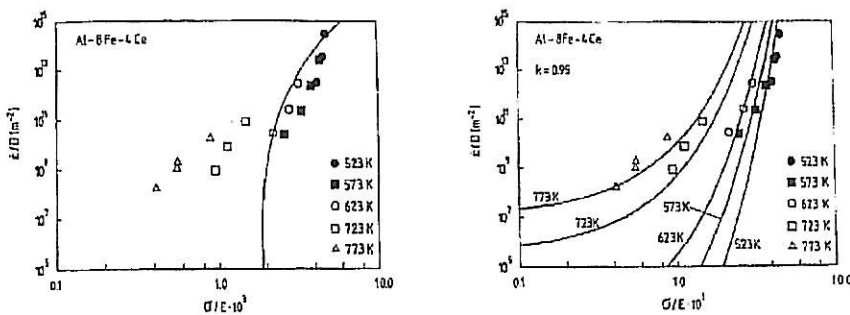

Fig. 7: Creep data of Al-8Fe-4Ce (data from Yaney and Nix (17));
left: comparison with the model calculations (solid line) due to the threshold stress concept;
right: comparison with the model calculations (solid lines) due to the detachment concept.

As is discussed by Rösler (10) and Rösler and Arzt (11) the correlation between model predictions and experimental data is not fully quantitative in that unrealistically high values of the pre-exponential factor in eq. 2 have to be assumed to fit the data. In fact very satisfactory correspondence has been obtained in analyzing single crystal data (11). Thus it seems likely that there is an additional contribution from other deformation mechanisms like diffusional creep to the overall creep behaviour. Because the value of the relaxation factor is insensitive to the magnitude of Q_{app} the conclusions stated above are nevertheless still valid.

4. Consequences for the Design of Dispersion Strengthened Alloys

From the new constitutive equation for creep (eq. 2) two major requirements for optimum high temperature strength can be deduced. Firstly the interaction parameter k, which appears as a new material parameter, should be as low as possible. Its magnitude must in some way depend on the interfacial properties and should be characteristic for a given particle-matrix combination. Maximum interaction strength was found for the system Al/Al_4C_3 (k=0.75) followed by Al/MgO (k=0.85) and Al/Al_xFe_yCe (k=0.95). Thus Al alloys strengthened by carbide particles seem to have the best potential for applications at very high temperatures. The above analysis suggests that intermetallic particles produced by rapid solidification are less efficient than ceramic particles incorporated by mechanical alloying techniques. Examination of further creep data is required to see whether this observation is part of a general trend.

The second requirement regards particle size. Given a certain particle volume fraction, the detachment model predicts that there should be an optimum particle radius, while the climb model would demand a small spacing. Inserting typical stress levels in eq. (5) one finds that
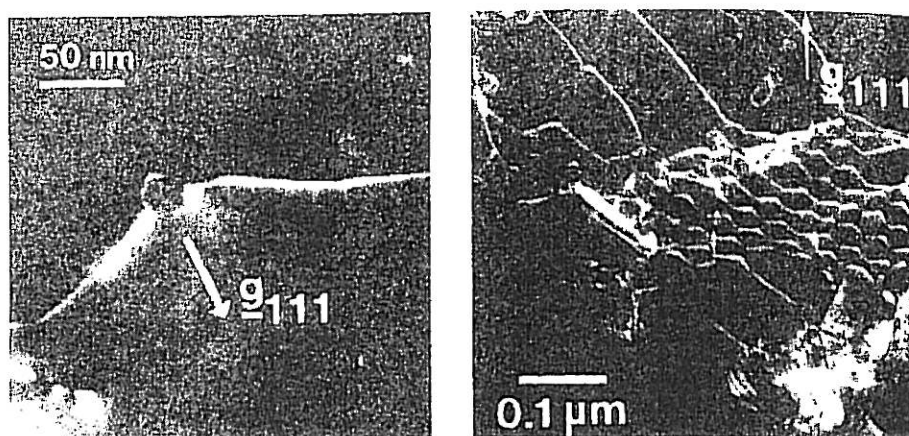
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the optimum particle diameter lies generally between 10 nm and 30 nm. Because actual spacings are generally in or above this range, there seems to be some further potential for increasing the creep strength by particle refinement.

5. Conclusions

- The comparison of experimental creep data with theoretical predictions strongly suggests that dislocation creep in dispersion-strengthened aluminium alloys is controlled by thermally activated dislocation detachment from the dispersoids and not by a climb-related "threshold stress".
- An optimum dispersoid diameter is predicted which lies typically in the range of 10 - 30 nm. Further refinement of the dispersion would result in inferior creep properties because of enhanced detachment kinetics.
- Two "quality parameters" (k and σ_d) for the dispersion have been defined. For the purpose of alloy screening, they can be readily obtained from a limited number (about five) of creep tests.

6. Acknowledgements

7. References

(1) R. Irmann: Techn. Rundsch., 41, (1949) 19.
(2) A. v. Zeerleder: Z. Metallkd., 41, (1950) 228.
(3) L.M. Brown, R.K. Ham: in "Strengthening Methods in Crystals", A. Kelly, R.B. Nicholson, eds., Applied Science Publishers, London, (1971) p. 126.
(4) R.S.W. Shewfelt, L.M. Brown: Phil. Mag., 35, (1977) 945.
(5) R. Lagneborg: Scripta Met., 7 (1973) 605.
(6) J. Rösler, E. Arzt: Acta Met., 36 (1988) 1043.
(7) D.J. Srolovitz, R.A. Petkovic-Luton, M.J. Luton: Acta Met., 31 (1983) 2151.
(8) D.J. Srolovitz, M.J. Luton, R.A. Petkovic-Luton, D.M. Barnett, W.D. Nix: Acta Met, 32 (1984) 1079.
(9) E. Arzt, D.S. Wilkinson: Acta Met., 34 (1986) 1893.
(10) J. Rösler: Ph.D. Thesis, University of Stuttgart (1988).
(11) J. Rösler, E. Arzt: to be published.
(12) E. Arzt: this volume.
(13) E. Arzt: Res Mechanica, in press.
(14) G. Jangg, F. Kutner, G. Korb: Aluminium, 51 (1975) 641.
(15) G. Jangg, F. Kutner, G. Korb: Powder Met. Int., 9 (1977) 24.
(16) R. Joos: Diploma Thesis, University of Stuttgart (1988).
(17) D.L. Yaney, W.D. Nix: Met. Trans. A., 18 (1987) 893.