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High temperature damping in dispersion-strengthened aluminium alloys

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Abstract

Dispersion-strengthened alloys are promising candidates for application at high homologous temperatures. Mechanical loss measurements were carried out on a dispersion-strengthened Al alloy with about 10 vol.% of incoherent Al_2O_3 and Al_4C_3 particles. We observe a loss maximum at about 590 K for a frequency $f=1$ Hz which exhibits an exceptionally high activation enthalpy ($H=2.2$ eV) compared with that for self-diffusion ($H_D=1.48$ eV). The peak height increases with oscillation frequency. Additionally, transmission electron microscopy studies were carried out for a correlation of the loss spectrum with the microstructure. To explain the experimental results, a relaxation model is proposed which considers jog nucleation on climbing edge dislocations as the time-limiting mechanism.

1. Introduction

Powder metallurgically produced dispersion-strengthened alloys are promising candidates for application at high homologous temperatures [1]. The basic physical mechanisms which lead to the extraordinarily high creep resistance have been studied extensively in recent years [2]. To obtain further insight into these mechanisms, we applied mechanical loss measurements to dispersion-strengthened aluminium alloys. In the present alloy up to 10 vol.% of small incoherent Al_2O_3 and Al_4C_3 particles about 30–40 nm in size are dispersed in an Al matrix of technical purity. Examinations of the dislocation arrangement of dispersion-strengthened Al alloys have shown that the loss spectra are connected with the dislocation arrangement in the Al matrix [3, 4]. Other mechanisms resulting from processes within the particles or at the particle–matrix interface may be disregarded.

In alloys with a low volume fraction of particles we observe four loss maxima, whereas alloys with a high volume fraction exhibit only three maxima [3, 4]. In this paper we present results on such an alloy with about 10 vol.% of dispersoids (designated AlC2; see Table 1). We have concentrated on the third maximum, which exhibits the highest peak temperature $T_p \approx 590$ K (1 Hz), and its correlation with the dislocation microstructure. For this purpose the samples were also investigated by transmission electron microscopy (TEM). We propose a relaxation model which involves jog pair nucleation on climbing dislocation segments which are anchored at the particles.

TABLE 1. Chemical composition (in weight per cent) and calculated dispersion volume fraction f_v (in volume per cent) of the alloy AlC2

Al	Si	Fe	Mg	Cu	C	O	f_v
Balance	0.05	0.168	<0.005	0.002	2.05	0.80	9.03

2. Experimental details

The *dispersion-strengthened aluminium alloy* (tradename DISPAL) was supplied by PEAK (Radevormwald, Germany) in the form of extruded rods 12 mm in diameter. Samples for the mechanical loss measurements with dimensions of $5\times1\times50$ mm^3 were prepared by spark cutting. To avoid changes in the microstructure during the measurements, all samples were pre-annealed at 823 K for 2 h. The chemical composition of the material was determined by wet chemistry (see Table 1). The volume fraction of dispersoids, f_v, was calculated by assuming that carbon and oxygen are present as Al_4C_3 and Al_2O_3 [1].

The *microstructure* was mainly investigated with a Jeol 200CX transmission electron microscope. After grinding to 100 μm, the samples were thinned at $T=243$ K with an electrolyte containing one part HNO_3 and

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two parts CH_3OH by applying a voltage of 30–35 V. The size of the dispersoids was determined from bright field images with a semiautomatic image analyser. Weak beam images were produced for the study of dislocation structures.

The *mechanical loss measurements* were carried out with an automatic, computer-controlled, inverted torsion apparatus from which the inertia parts of the pendulum had been removed [5]. The loss angle $\varphi \propto Q^{-1}$, *i.e.* the angle between applied stress and resulting strain, was measured with forced vibrations at constant frequency and the temperature was increased at a mean heating rate $\dot{T}$ between 0.6 and 4 K min^{-1}. The applied maximum shear strain γ_{max} was varied between 2×10^{-5} and 4×10^{-5}. The measured loss spectra were analysed by fitting with a superposition of Debye maxima [6]. A background damping consisting of a superposition of a linear and an exponential term was subtracted before applying the non-linear regression procedure.

The activation enthalpy H and the inverse attempt frequency τ_∞ of the relaxation process were determined using the peak-temperature-shift-with-frequency technique, since the relaxation time τ obeys the Arrhenius equation

$$\tau=\tau_\infty \exp\left(\frac{H}{kT}\right) \qquad (1)$$

3. Results

On transverse planes, *i.e.* vertical to the extrusion axis, AlC2 has an equiaxed grain structure (grain size $d\approx1.5$ μm) and most of the grains are separated by high angle boundaries (Fig. 1). In the longitudinal direction, *i.e.* parallel to the extrusion axis, the grains are elongated by a factor of about 1.5. The dislocation density ρ was estimated as about 10^{14} m^{-2} (Fig. 2). Most of the dislocations are in contact with particles, which act as firm pinning points (Fig. 2) and thus stabilize the dislocation structure.

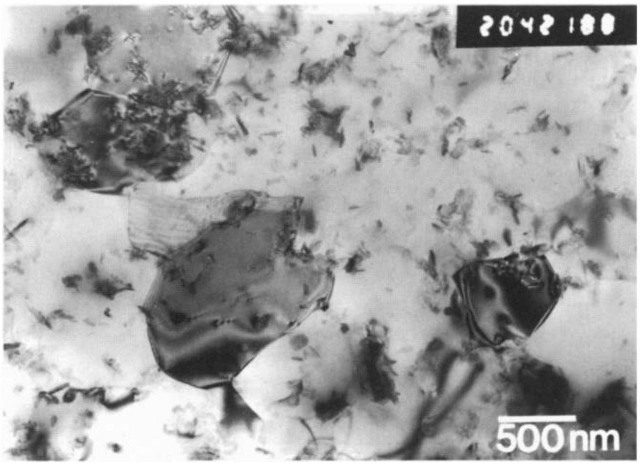

Fig. 1. TEM image of AlC2 vertical to the extrusion direction showing the grain structure.

Figure 3 shows measurements of Q^{-1} *vs.* T for AlC2 at four frequencies (f=0.02, 0.1, 1 and 10 Hz). The loss spectra show three maxima which are designated P1, P2 and P3 [3]. With increasing frequency they are shifted to higher temperatures. In this paper we concentrate on the high temperature maximum P3. From the frequency shift of the maximum P3 we obtain $H=2.2\pm0.2$ eV and $\tau_\infty=8.6\times10^{-21\pm1}$ s. The maximum P3 exhibits an interesting property: as can be seen in Fig. 3, the peak height increases with increasing measuring frequency. Fitting with broadened Debye maxima corresponding to a Fuoss–Kirkwood distribution of re-

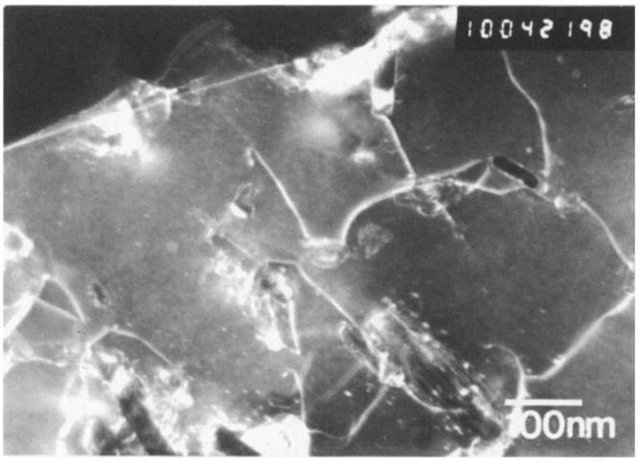

Fig. 2. TEM image of AlC2 vertical to the extrusion direction showing the dislocation structure and dispersoid arrangement.

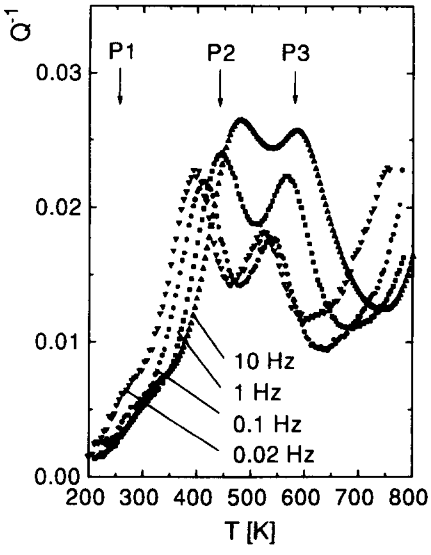

Fig. 3. Mechanical loss spectra $Q^{-1}(T)$ for AlC2 (f=0.02, 0.1, 1 and 10 Hz; $\dot{T}\approx2$ K min^{-1}; $\gamma_{max}\approx4\times10^{-5}$).

laxation times gives the following values of the relaxation strength: $\Delta=(7.4, 6.8, 9.2, 14)\times 10^{-2}$ in order of increasing frequency (0.02, 0.1, 1, 10 Hz). We found that the maximum P3 is not sensitive to variations in the heating rate ($\dot{T}=0.6$–4 K min^{-1}) or maximum shear strain ($\gamma_{\max}=(2$–$4)\times 10^{-5}$) [3, 4]. It is smaller in alloys with a higher volume fraction of dispersed particles [3, 4].

4. Discussion

The maximum P3 has two unusual properties: the activation enthalpy controlling the mechanical loss is substantially higher ($H=2.2$ eV) than that for self-diffusion ($H_D=1.48$ eV [7]) and the relaxation strength increases with frequency. Since the maximum is not detected in Al alloys without particles, a direct particle mechanism [8] or mechanisms involving the interaction between particles and dislocations or grain boundaries [9] must be operative. However, since the relaxation strength decreases with increasing volume fraction of dispersoids, a direct particle mechanism may be excluded [3]. To our knowledge, there exists no model which could explain the high activation enthalpy by interaction of grain boundaries with particles. In the following we propose a dislocation model [10] which involves the climbing of edge dislocations by jog pair nucleation [11]. As will be shown, this model is able to take into account the rather high activation enthalpies.

We propose that the maximum P3 is due to additional climbing of dislocation segments between the dispersoids which act as firm pinning points. To simplify the analysis, we consider only edge dislocations which are fixed at the particles. The energy $2W_J$ of a jog pair of width x is [12]

$$2W_J \approx \frac{\mu b^2 h}{2\pi(1-\nu)} - \frac{\mu b^2 h^2}{8\pi x(1-\nu)} \tag{2}$$

where μ is the shear modulus, ν is the Poisson ratio, b is the Burgers vector and h is the height of the jog (Fig. 4). A possible contribution of the jog entropy to the free energy is neglected. Therefore the jog pair nucleation rate per unit length [11] may be expressed as [3]

$$J=\frac{\sigma b h}{a^2 kT} D_J \exp\left(-\frac{2W_J}{kT}\right) \tag{3}$$

where σ is the external (applied) stress. The calculation of the diffusivity D_J of a jog yields for the steady state condition, with the assumptions that the diffusion of vacancies from the jog via the dislocation to the particle is fast and that the particle surface acts as a perfect source [3],

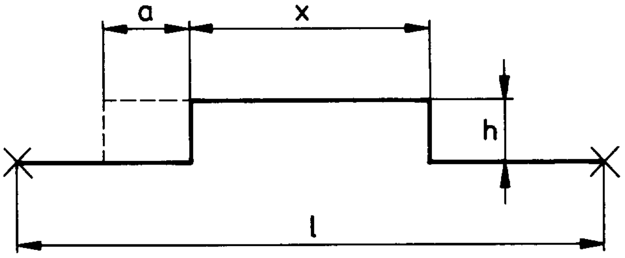

Fig. 4. Assumed configuration for calculation of the jog pair nucleation rate. Crosses signify the pinning points at the particles, the heavy line is the schematic dislocation contour, h is the distance between glide planes, a is the distance between possible jog positions and x is the width of a jog pair.

$$D_J=\frac{4\pi a}{hb} D_S \frac{r_0 R}{R-r_0} \tag{4}$$

where r_0 is the particle radius, R is the outer cut-off radius, D_S is the self-diffusion in the bulk and a is the lateral displacement of a jog due to the insertion of one vacancy. For calculating the velocity v of the dislocation, two different critical cases can be considered [13, 14].

(i) The average distance X_J swept out by a jog pair along the dislocation,

$$X_J=2a \exp\left(\frac{W_J}{kT}\right) \tag{5}$$

is much smaller then the length l of the dislocation segments between the pinning points, *i.e.* $X_J \ll l$. With the assumption of a constant jog density, the dislocation velocity v may then be expressed as [13, 14]

$$v=\frac{2\sigma b h^2}{akT} D_J \exp\left(-\frac{W_J}{kT}\right) \tag{6}$$

(ii) For the other case, $X_J \gg l$, the velocity v may be expressed as [13, 14]

$$v=hlJ=\frac{\sigma b h^2 l}{a^2 kT} D_J \exp\left(-\frac{2W_J}{kT}\right) \tag{7}$$

Furthermore, the relaxation time may be calculated from v and the area swept out by the dislocation [14]. For $X_J \ll l$ (case (i)) we obtain

$$\tau=\frac{akT}{T_L h^2}\frac{1}{D_J}\frac{l^2}{4}\exp\left(\frac{W_J}{kT}\right) \tag{8}$$

and for $X_J \gg l$ (case (ii))

$$\tau=\frac{a^2kT}{T_Lh^2}\frac{1}{D_J}\,l\,\exp\left(\frac{2W_J}{kT}\right) \tag{9}$$

T_L is the line tension approximated as $T_L=\mu b^2/2$. The mean pinning length is approximated by the mean planar particle distance $l=l_p=2r_0(\pi/6f_v)^{0.5}$, where $2r_0$ is the particle size. For the temperature of the maximum P3 ($T_{P3}=600$ K) the value of the distance X_J is calculated from eqn. (5) as $X_J=700$ nm. This is considerably larger than the mean planar distance between particles in AlC2, $l_p=96$ nm. Therefore case (ii) should be applicable. Inserting the values of Table 2 in eqn. (9) and comparing with eqn. (1) gives for AlC2 $\tau_\infty=3.4\times10^{-18}$ s and $H=2.23$ eV. The activation enthalpy is composed of the activation enthalpy for self-diffusion and the free energy of a jog pair, *i.e.* $H=H_D+2W_J$. The values for H and τ_∞ are in good agreement with the experimental values ($\tau_\infty=8.6\times10^{-21}$ s, $H=2.2$ eV). The relaxation strength for this mechanism may be estimated from $\Delta\approx\frac{1}{10}\rho l^2$ as $\Delta\approx0.1$. The measured values of the relaxation strength (0.07–0.14) are of the order of magnitude expected for dislocation relaxation.

Analogously to the Bordoni maximum, we expect that a "Paré condition"

$$2W_J\leqslant\sigma_i bhl \tag{10}$$

has to be fulfilled for the appearance of the maximum P3. From eqn. (10) we may estimate the necessary internal stress σ_i, for which we obtain $\sigma_i=18$ MPa. This stress acts in Al over a distance of 100 nm from an edge dislocation.

The frequency and temperature dependences of the relaxation strength of the maximum P3 may be interpreted by a relaxation model which was proposed by Mi *et al.* [16] for oxygen diffusion in a high temperature superconductor ($YBa_2Ca_3O_{6+x}$). This model is based on an asymmetric double-well potential.

The starting point of our consideration is two different pinned dislocation configurations with different energy levels. Position A represents the bowed state, where the dislocation has climbed, and G is the ground state. If eqn. (10) is not fulfilled for a dislocation, the energy difference between positions A and G is $2\Delta E=2W_J-\sigma_i bhl$. The activation hill for the movement from G to A is the free-energy separation curve for jog pair nucleation.

TABLE 2. Values of parameters used for calculation of τ in eqn. (9)

Parameter	Value	Reference
μ	26.6 GPa	[15]
ν	0.347	[15]
a	0.249 nm	
h	0.233 nm	
b	0.286 nm	
D_S	$1.7\cdot10^{-4}\exp\left(\frac{-1.48}{kT}\right)$ m^2 s^{-1}	[7]

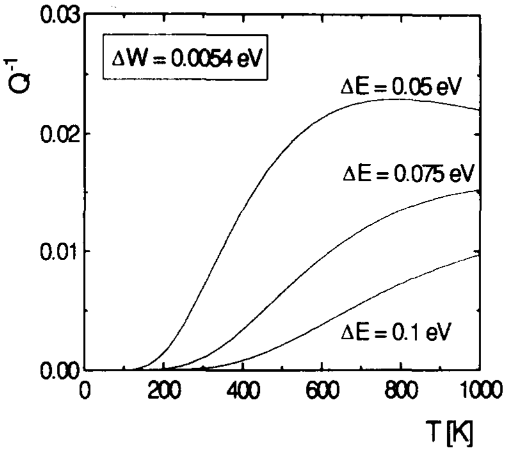

Fig. 5. Relaxation strength as a function of temperature for $\rho bh/(\sigma/G)=1$ and various ΔE values.

According to Mi *et al.* [16], the relaxation strength for an asymmetric double-well potential can be estimated as [3]

$$\Delta=\frac{\gamma_{an}(t=\infty)}{\gamma_{el}}=\frac{\rho bh}{\sigma/G}\left(\frac{\exp[(\Delta E+\Delta W)/kT]}{2\cosh[(\Delta E+\Delta W)/kT]}-\frac{\exp(2\Delta E/kT)}{1+\exp(2\Delta E/kT)}\right) \tag{11}$$

where $\Delta W=\sigma bhl/2$ is the distortion of the energy levels due to an external shear stress. Calculations for various values of ΔE are shown in Fig. 5, in which the relaxation strength is plotted as a function of temperature. Obviously the relaxation strength increases with increasing frequency for larger values of ΔE. Thus our observation of an increase in the peak P3 with frequency may be explained, at least qualitatively, by taking into account an unbalanced "Paré condition".

5. Conclusions

The dislocation model which we have proposed for the loss maximum P3 (about 590 K) in dispersion-strengthened aluminium alloys is able to explain the essential experimental features: (i) the unusually high activation enthalpy, being larger than that for self-diffusion in Al; (ii) the increase in the relaxation strength (maximum height) with measurement frequency.

Dislocation motion proceeds by nucleation of jog pairs and climbing of dislocations which are anchored at the dispersoid particles. The dispersoids are responsible for the stabilization of the almost random arrangement of dislocations with high density up to high homologous temperatures. This is believed to be the origin of the superior high damping properties of dispersion-strengthened Al alloys at elevated temperatures. A study of the implications of the proposed jog nucleation–climbing model for the creep behaviour of dispersion-strengthened alloys will be published in a forthcoming paper.

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