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LETTER TO THE EDITOR

An investigation of the purification of cerium in aluminium by the method of internal friction

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Abstract. In this letter, the relation between the purification effect and measurements of internal friction is established, including the grain boundary and dislocation internal friction. Corresponding internal frictions were measured for 4N pure aluminium samples with cerium content 0–0.1 wt.% (four in number) in Ke's vacuum torsion pendulum. From the changes of the grain boundary internal friction peak height, the unpinning stress, and the slope of the GL plot with Ce content, it has been shown that there are different Ce purification effects in the grain boundary and in the interior of the grain of Al; a small amount of Ce (<0.01%) has a significant purification effect, but somewhat higher Ce content will decrease this effect because of segregation, especially in the grain boundary.

We know that rare earth (RE) elements are, chemically, very active and easily form compounds with many other elements [1], and can decrease the solute concentration in a solid solution, which is called the purification effect of REs. In this letter, the purification effect of the RE element cerium in 4N pure Al is investigated by the method of internal friction, including measurements of grain boundary and room temperature dislocation internal frictions, after a detailed discussion of the relation between the purification effect and measurements of internal friction.

We first discuss the relation between purification and internal friction. Because of purification, the solute (or impurity) concentration in parent materials should decrease, and the concentration in grain boundaries and dislocations should also be reduced, which means that the extent of segregation of solutes will be decreased after addition of some element with a purification effect. Therefore, under the same heat treatment conditions, the grain size should be larger due to the purification effect, because the existence of impurities in the grain boundary will hinder the migration of the grain boundary [2]; in addition, the distance between the nearest weak pinning points on a dislocation line will be greater. All these factors can be described in terms of internal friction.

We now discuss the internal friction associated with the grain boundary. It has been systematically investigated by Ke [3] and in some previous work [4–6]. The existence of impurities in the grain boundary will decrease the grain boundary internal friction peak height [4, 5]. According to the grain boundary dislocation network model [6–8], a decrease in the concentration of impurities in the grain boundary will cause the distance between the two nearest weak pinning points to be greater, and so the grain boundary glide distance longer, which will result in increasing grain boundary peak height or relaxation strength.

We now consider the internal friction associated with a dislocation. According to the Granato-Lück theory [9, 10], the internal friction Q^{-1} associated with a dislocation can be expressed as

$$Q^{-1} = \Delta I + \Delta H \quad (1)$$

$$\Delta H = \left(\frac{A_1 \rho L_N^3}{\varepsilon_0 L_C} \right) \exp\left(\frac{-A_2}{L_C \varepsilon_0} \right) \quad (2)$$

in which ΔI is the internal friction, independent of the vibration amplitude, ρ is the dislocation density, L_N is the average length between two nearest strong pinning points on the dislocation line, L_C is the average length between two nearest weak pinning points on the dislocation line, ε_0 is the amplitude of the strain, and A_1 and A_2 are constants. Equation (2) can be changed into

$$\ln(\Delta H \varepsilon_0) = \ln\left(\frac{A_1 \rho L_N^3}{L_C} \right) - \frac{A_2}{L_C \varepsilon_0} \quad (3)$$

$\ln(\Delta H \varepsilon_0)$ is a linear function of $1/\varepsilon_0$. The corresponding plot is called a Granato-Lück (GL) plot, which should be a straight line. A_2/L_C is the slope of the GL plot. Obviously, a small slope value corresponds to large value of L_C and low impurity concentrations on the dislocation line. In addition, purification will cause the unpinning strain amplitude ε_0^* to become larger. If the RE purification effect occurs in Al, the internal friction measurements should coincide with the deduction above.

We will now give details of the experiments. Four 4N Al samples with different Ce contents (0.01%, 0.1% weight) were cold drawn to a diameter of 0.9 mm for internal friction measurement. For measurement of the grain boundary internal friction, the samples were, *in situ*, annealed at 720 K for 2 h in Ke's torsional pendulum in a vacuum of about 5×10^{-4} Torr, and this was followed by internal friction measurement with the temperature decreasing at about 1 K min^{-1} ; for measurement of the dislocation internal friction, the samples were first annealed at high temperature (890–900 K) for 3 h in a vacuum so that the grain size became larger than the sample's diameter (which eliminates the effect of the grain boundary on the internal friction). The sample was carefully assembled in Ke's torsional pendulum and left for more than 12 h before measurement of internal friction as a function of the vibration amplitude at room temperature (about 283 K) and in a vacuum of 1×10^{-1} Torr. The vibration frequency was about 0.6 Hz.

We now present the results for the grain boundary internal friction. Figure 1 shows the original curves of internal friction versus temperature for all samples. The internal friction peak for the sample Al–0.005% Ce is higher than that for the sample with no Ce, but for the samples with 0.01% Ce, or more, the peak heights are lower than that of the sample free from Ce. By the method presented in [11], we can accurately subtract the background from the original curves and obtain some related parameters, which are given in table 1.

We now report the results for the dislocation internal friction. Some original curves of Q^{-1} versus the natural logarithm of the reading of vibration amplitude A_0 can be seen in figure 2. Because A_0 is directly proportional to ε_0 , equation (3) can be written as

$$\ln[(Q^{-1} - \Delta I)A_0] = \ln C_3 - C_4/A_0 \quad (4)$$

For a given sample, C_3 and C_4 are constants directly proportional to $1/L_C$, and C_4 is also the slope of the GL plot. In this letter, we are only interested in ΔH and do not discuss

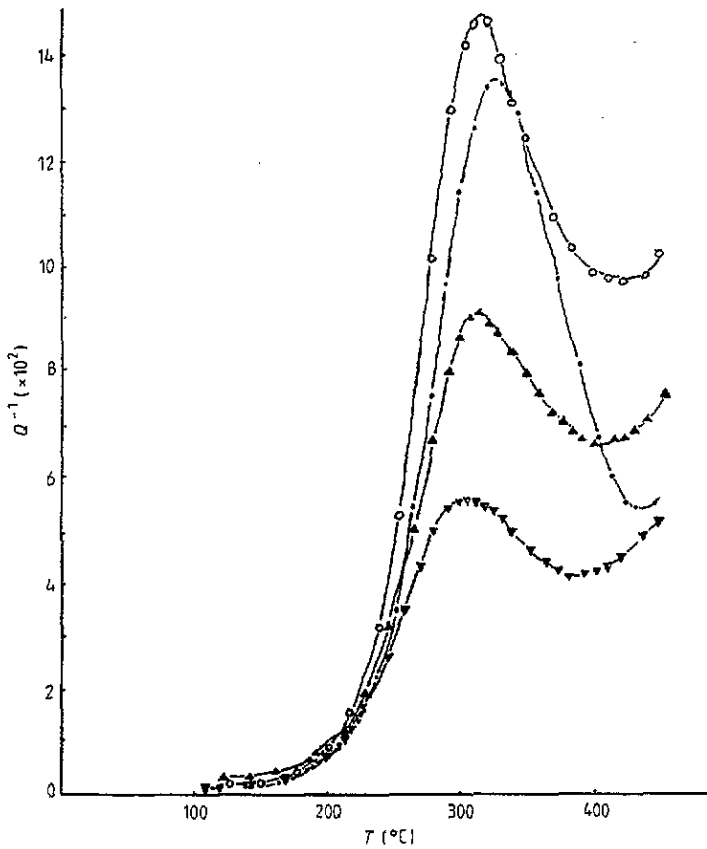


Figure 1. Original curves of grain boundary internal friction Q^{-1} versus temperature T for: Al (●); Al-0.005% Ce (○); Al-0.01% Ce (▲); Al-0.1% Ce (▼); (720 K for 2 h, decreasing temperature: 1 K min^{-1} , $f \approx 5 \text{ Hz}$.)

Table 1. Grain boundary internal friction parameters and grain sizes: Q_{max}^{-1} , peak height; Δ_M , relaxation strength; d , average grain diameter in cross-section.

Ce (wt.%)	0.0	0.005	0.01	0.1
$Q_{\text{max}}^{-1} (10^2)$	11.31	12.34	6.88	3.81
Δ_M	0.532	0.625	0.352	0.170
$d (10^2 \text{ nm})$	20	32	16	11

ΔI , which can be approximately obtained from the curve of Q^{-1} versus $\ln A_0$, and has no apparent differences for all samples measured in a low vacuum (1×10^{-1} Torr). According to (4), $\ln[(Q^{-1} - \Delta I)A_0]$ is a linear function of $1/A_0$. The corresponding plot is called a GL plot, and should be a straight line, but inaccurate determination of ΔI will cause deviation from the straight line in the lower-amplitude region of the plot, especially in the low vacuum in the present work [4,5]. The regression expression of (4), is mathematically the same as the regression expression for subtracting the background internal friction mentioned in [11].

So the corresponding program was worked out by imitating the computing method given in [11], and with the help of a computer, the optimum ΔI value, the slope and intercept of the GL plot, and the correlation coefficient of the straight line etc can be obtained for all samples. The computer processing results are shown in table 2 in which the reading of unpinning amplitude A^* , directly proportional to the unpinning strain amplitude ε_0 , is obtained directly from the curve of Q^{-1} versus $\ln A_0$ (i.e. the corresponding amplitude value of the inflection point of the curve). All correlation coefficients are close to one. In addition, Peguin *et al* [12] mentioned that when the strain of a sample is not uniform, A_0 in $\ln[(Q^{-1} - \Delta I)A_0]$ should be replaced by $\sqrt{\langle A_0^2 \rangle}$. It has been shown by our computations the two results agree well, and there is no apparent difference.

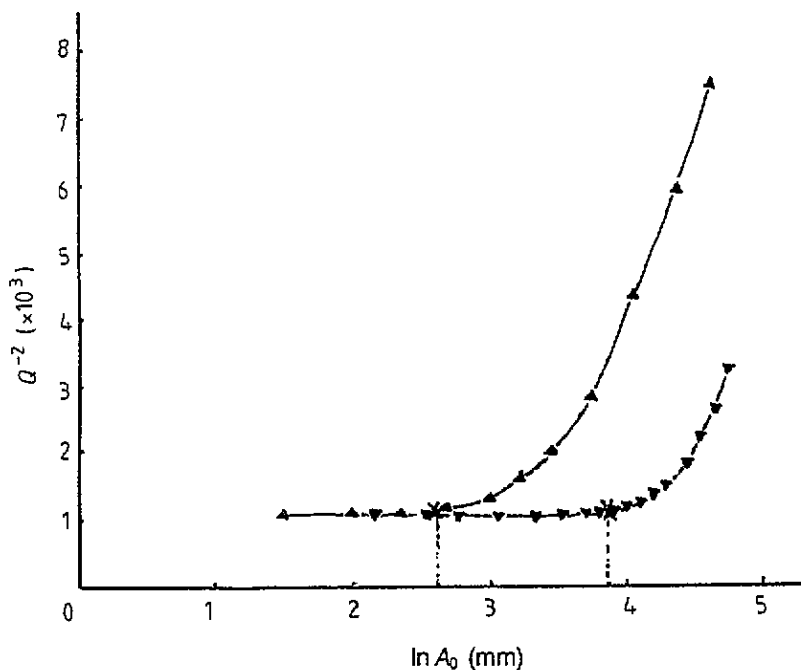


Figure 2. Curves of Q^{-1} versus natural logarithm of the reading of vibration amplitude A_0 (mm) for the following samples: Ce 0.0%, $l = 23.2$ cm (▼); Ce 0.005%, $l = 23.5$ cm (▲). $f \simeq 0.6$ Hz, $T = 283$ K, vacuum 1×10^{-1} Torr. (x, inflection point of the curve.)

Table 2. Computer processing results of GL plots for all samples. l is the length of the sample (cm); R is the correlation coefficient of the straight line.

Ce (wt.%)	0.0	0.005	0.01	0.1
A^*/l	2.03	0.59	0.90	1.33
C_4/l	16.91	5.52	7.46	9.98
R	0.9995	0.9985	0.9969	0.9999

We now briefly discuss the purification in the grain boundary. The addition of Ce at 0.005%, because of the purification effect, results in a decrease in the total concentration of impurities in Al, an increase in the pinning distance of solute atoms in the grain boundary dislocation network, and easier grain boundary migration during annealing, which cause a greater peak height Q_{\max}^{-1} or relaxation strength Δ_M and grain size than those of the sample with no Ce (see table 1). For the samples with Ce content $\geq 0.01\%$, the segregation of Ce itself in the grain boundary causes the relaxation strength and grain size to decrease (see table 1), which we will discuss in detail in another paper [13].

We now move on to the purification in the interior of the grain. For a given specimen, the reading of unpinning amplitude A^* is related to the concentration C_0 of solute in the lattice, the absolute temperature T and the binding energy G between the solute atom and the dislocation, as given by [14]

$$A^* = \frac{L_0 l G C_0}{r E b^3} \exp\left(\frac{G}{kT}\right) \quad (5)$$

where L_0 is the distance from the sample to the reading scale, r the radius of the sample, b the magnitude of the Burgers vector, E Young's modulus and k Boltzmann's constant. The slope of the GL plot is directly proportional to $1/L_C$, which is directly proportional to C_0 . So, A^*/l is directly proportional to C_4/l . Table 2 shows that both have the same tendency of change with percentage of Ce and that the values for the sample with no Ce are maximal. In addition, the binding energy between the Ce atom and the dislocation should be greater than that for some common impurities, Cu, Fe, Mn, Mg etc, in Al. So we know that when the Ce content is $\leq 0.1\%$, the total amount of solute in Al-Ce samples is less than that in Al with no Ce; this results from the purification effect of Ce. The sample with 0.005% Ce assumes the greatest purification effect (the lowest A^*/l and C_4/l values correspond to the lowest total amount of solutes). When the Ce content is $>0.005\%$, due to the increase in the concentration of Ce in the lattice and the segregation of Ce atoms in dislocations, the purification effect decreases gradually but still exists with increasing Ce content up to 0.1%.

We have reached the following conclusions.

- (i) Ce has an obvious purification effect in Al.
- (ii) A small amount of Ce ($<0.01\%$) has the greatest purification effect, but more Ce will decrease this effect.
- (iii) There are different purification effects of Ce in the grain boundary and in the grain. The purification effect is counteracted by the increase in solid solution Ce when the Ce content is $\geq 0.01\%$ for the grain boundary, and when the Ce content is $>0.1\%$ for the grain interior.

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