JOURNAL OF MATERIALS SCIENCE 26 (1991) 3813–3816

Investigation of distribution and state of rare-earth elements in pure aluminium by method of internal friction

Part II *Influence of RE on dislocation internal friction of Al at room temperature*

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The dislocation internal friction at room temperature for 99.99% pure Al with different rare-earth (RE) contents (0–3 wt%) was measured with Ke's torsion pendulum in a vacuum. A new method is presented for accurately determining the parameters of Granato–Lück plot and processing the experiment data. It was found that at room temperature the unpinning stress of dislocations and the slope of the G–L plot increase with RE content and tend to change little when RE $\geqslant 0.1\%$. The binding energy between RE atom and dislocation, and the lattice solubility of RE in Al at room temperature were obtained according to the dependence of unpinning strain on temperature, which are about 0.19 eV and 0.0003 wt%, respectively. The existence of solid solution RE in dislocations and the lattice was confirmed and some behaviour of RE in the interior of the Al grain mentioned in part I was also proved.

1. Introduction

In Part I [1], the behaviour of RE in the grain boundary and the interior of grains from the grain boundary and the background internal friction of Al–RE specimens was discussed. It was thought that the background arises from a diffusion-controlled dislocation motion in the interior of the grain and the solid solution RE content increases with total RE content up to 0.5%, at which RE is saturated. In order to confirm these results further, the dislocation internal friction at room temperature was measured for Al–RE samples with different RE contents.

2. Experimental procedure

The apparatus and preparation of samples were described in [1]. Al–RE samples with different RE contents (0–3%) were first subjected to high-temperature annealing at 890–900 K for 3 h in a vacuum so that all the samples were in the state of complete annealing. The sample was carefully assembled in the internal friction apparatus. After more than 12 h, internal friction measurements as a function of the vibration amplitude were taken during stepwise increases in vibration amplitude at room temperature (about 283 K). The vibration frequency was about 0.6 Hz and the vacuum about 1×10^{-1} torr. Some original curves are shown in Fig. 1 in which A_0 is the reading amplitude on the scale, 3 m from the sample.

3. Processing of data and results

According to the Granato–Lück theory [2, 3], the internal friction, Q^{-1}, associated with dislocation can be expressed as

$$Q^{-1} = \Delta I + \Delta H \tag{1}$$

$$\Delta H = A_1 \rho L_N^3/(\varepsilon_0 L_c)\exp(-A_2/L_c\varepsilon_0) \tag{2}$$

in which ρ is the dislocation density, L_N the average length between two nearest strong pinning points on the dislocation, L_c the average length between two nearest weak pinning points on the dislocation, ε_0 the amplitude of strain, and A_1 and A_2 are constants. Because A_0 is directly proportional to ε_0, Equation 2 can be written as

$$\Delta H = C_3/A_0 \exp(-C_4/A_0) \tag{3}$$

For a given sample, C_3 and C_4 are constants and both are directly proportional to $1/L_c$. Inserting Equation 3 into Equation 1 we have

$$Q^{-1} = \Delta I + C_3/A_0 \exp(-C_4/A_0) \tag{4}$$

where ΔI is the internal friction independent of the vibration amplitude. Obviously, ΔI obtained by experiment should be the sum of the extra dampening caused by air, etc., and the dampening independent of A_0 in a sample. Being in a low vacuum, the former is much higher than the latter (about 10^{-5}–10^{-4} order of magnitude). Thus there are no apparent differences between the experimental ΔI values for all the samples with different RE contents (see Fig. 1). The present paper is only interested in ΔH and does not discuss ΔI, which can be approximately obtained from the curve Q^{-1} versus $\ln A_0$. Equation 4 thus becomes

$$\ln[(Q^{-1} - \Delta I)A_0] = \ln C_3 - C_4/A_0 \tag{5}$$

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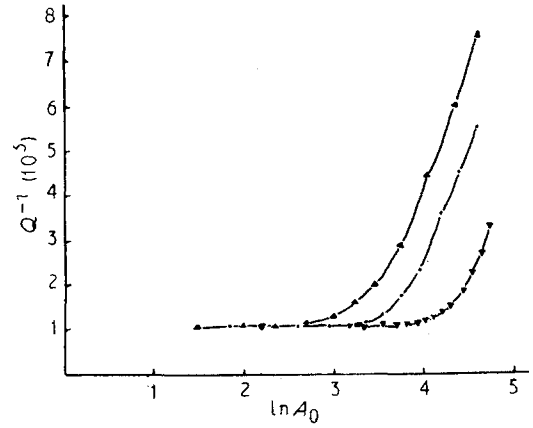

Figure 1 Original curves of Q^{-1} versus $\ln A_0$ for (▼) RE 0.0%, $l = 232$ mm, (▲) RE 0.005%, $l = 235$ mm, (●) RE = 0.097%, $l = 204$ mm. $f = \sim 0.6$ Hz.

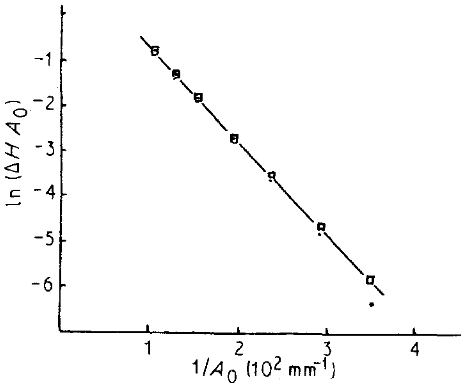

Figure 2 The G–L plot for the Al–0.1% RE specimen: (●) evaluated, (□) computed.

$\ln[(Q^{-1} - I)A_0]$ is a linear function of $1/A_0$. The corresponding plot is called a Granato–Lück (G–L) plot, which should be a straight line. But there is often deviation from the straight line in the lower amplitude region of the plot, especially in a low vacuum [2, 4, 5]. It is suggested that this may arise from inaccurate determination of ΔI. In the lower amplitude region, when ΔI changes slightly, $\ln[(Q^{-1} - \Delta I)A_0]$ will change obviously (because Q^{-1} is small), but in the higher amplitude region, it cannot be affected because Q^{-1} is much higher. From Fig. 1, ΔI for the sample A–0.1% RE can be approximately obtained, which is $\sim 1.1 \times 10^{-3}$. Its G–L plot is shown in Fig. 2 (●). The locations of the points (●) in the lower amplitude region obviously deviate from the straight line. The regression expression of Equation 5, in mathematics, is the same as the regression expression for subtracting the background internal friction mentioned in [6]. So the corresponding program was worked out by imitating the computing method given [6]. Inputting the original internal friction values and corresponding amplitude values into the computer, the optimum ΔI, the slope and intercept of the G–L plot, and the correlation coefficient of the straight line, etc., can be obtained. The corresponding values of the square symbols (□) in Fig. 2 are computations by computer, here $\Delta I = 1.06 \times 10^{-3}$. It can be seen that there is no deviation from the straight line in the lower amplitude region. The computer-processing results of original internal friction data for different samples are shown in Table I in which the unpinning amplitude, A^*, is obtained directly from the curve Q^{-1} versus $\ln A_0$ (i.e. the corresponding value of the inflection point on the curve), and l is the length of sample (cm). In addition, Peguin *et al.* [7] mentioned that when the strain of a sample is not uniform, A_0 in $\ln[(Q^{-1} - \Delta I)A_0]$ should be $A_0^{1/2}$. It has been shown by our computations that both results agree well, and there is no apparent difference.

4. Discussion

4.1. Solid solution in the interior of a grain

From Table I, because the correlation coefficients for all the samples are almost 1, it can be seen that the G–L theory is in good agreement with our experimental findings. When RE% $\geqslant 0.5$, the grain size after annealing at 890–900 K for more than 3 h is still small [1]. So the problem is complicated because of the existence of grain boundaries, but from Table I, it can still be seen that when RE% $\geqslant 0.5$ the unpinning strain (A^*/l) and the slope (C_4/l) do not change much. For samples with less than 0.5% RE, the influence of grain boundaries is small because their grain diameters are all larger than that of sample (0.9 mm). When RE% < 0.01, C_4/l and A^*/l are less than those of pure Al with no RE, and when RE% $\geqslant 0.01$, both increase with RE content up to 0.1%. C_4/l and A^*/l are directly proportional to the solute concentration in the dislocation, so it can safely be assumed that the RE part in a sample is in the solid solution state, and for small contents the percentage of solid solution RE's in the total RE content in the sample may be considered to be approximately a constant (the other part of RE in a sample, because of the purification, forms a compound or inclusions with some impurities

TABLE I Computer processing results of original data for all samples

	RE (wt %)							
	0.0	0.005	0.01	0.06	0.097	0.51	1.2	3.1
A^*/l[a]	2.03	0.59	0.90	1.11	1.33	1.30	1.23	1.19
C_4/l	16.91	5.52	7.46	8.51	9.98	8.06	8.43	6.67
R	0.9995	0.9985	0.9969	0.9982	0.9999	0.9970	0.9995	0.9994

[a] l is the length of sample (cm); A^* is the unpinning amplitude obtained directly from the curve Q^{-1} versus $\ln A_0$ (mm); R is the correlation coefficient of a straight line.

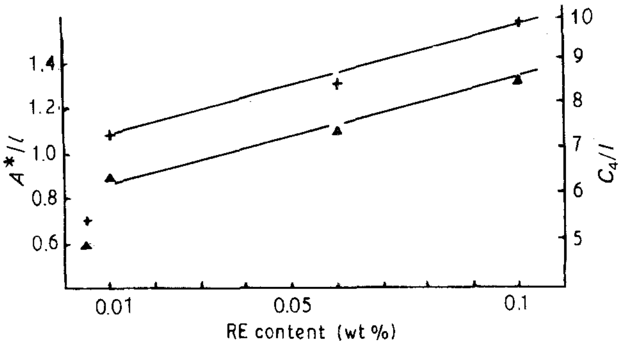

Figure 3 Plots of (▲) A^*/l and (+) C_4/l against RE content.

[1]). A plot of C_4/l or A^*/l versus RE% should give a straight line in which C_4/l or A^*/l should increase with RE content. The plots of the data in Table I are shown in Fig. 3. It can be seen that the changes in C_4/l and A^*/l with RE% agree well: in the RE content range 0.01%–0.1% both have good linear relations with RE% and when RE < 0.01% both obviously deviate from their straight lines. Thus it is known that the RE concentration in the dislocation increases with RE content up to about 0.1%, at which solid solution the RE is saturated; the solid solution RE percentage in the total RE content is less when RE% < 0.01% than that when RE is in the range 0.01%–0.1%. It may be that when RE < 0.01% solid solution RE mainly exists in the grain boundaries [1]. In addition, for A^*/l and C_4/l values for the specimen with no RE are higher than those for all other specimens (see Table I), it is found that the maximal solid solution concentration of RE in the dislocation at room temperature, is less than that of impurities in the sample with no RE in the dislocation. From the discussion of the background internal friction in Part I [1], it was concluded that when RE ⩾ 0.1% the solid solution concentration of RE in the dislocation is higher than that of impurities in the sample with no RE in the dislocation. But at room temperature, because the solubility of RE in Al will change with temperature, the conclusions deduced from discussions of the background dislocation internal friction, still hold, i.e. the solid solution RE concentration in the interior of the grain increases with RE content up to 0.1%–0.5%, at which the solid solution RE is saturated.

4.2. Binding energy and solubility

For a given specimen, its unpinning strain, ε^*, of the dislocation relates to the temperature (K). The relation between ε^* and T (absolute temperature) is given by [8]

$$\varepsilon^* = GC_0/(E\boldsymbol{b}^3)\exp(G/kT) \qquad (6a)$$

or in our case

$$A^* = LlGC_0/(rE\boldsymbol{b}^3)\exp(G/kT) \qquad (6b)$$

where L is the distance from the sample to the reading scale, l the length of a specimen, r the radius of the specimen, $\boldsymbol{b}$ the magnitude of Burgers vector, E Young's modulus, G the binding energy between solute atom and dislocation, C_0 the concentration of solute in the lattice and k Boltzmann's constant. It is easily known that for a given specimen, the change of temperature will cause a change in A^*. Equations 6a and b can be written as

$$\ln A^* = B_0 + G/kT \qquad (7a)$$

$$B_0 = \ln(LlGC_0/rE\boldsymbol{b}^3) \qquad (7b)$$

where B_0 is a constant. In the present research, the internal friction as a function of A_0 was measured for the samples Al–0.01% RE, Al–0.06% RE at different temperatures at which the measurement began after holding for several hours. The $\ln A^*$ and C_4 values at different temperatures for the two specimens are shown in Table II and the corresponding original curves of Q^{-1} as a function of $\ln A_0$ are seen in Fig. 4. Plotting $\ln A^*$ against $1/T$ from the values in Table II gives Fig. 5. It can be seen that there is a good linear relation between $\ln A^*$ and $1/T$ and the slopes of the

TABLE II $\ln A^*$ and C_4 values at different temperatures for the samples Al–0.01% RE and Al–0.06% RE

RE		Temperature (K)						
(wt %)		282	283	293	296	303	313	320
0.01	$\ln A^*$		2.60		2.23			1.70
	C_4		118.0		84.5			47.0
0.06	$\ln A^*$	3.10		2.80		2.58	2.35	
	C_4	178.6		116.0		105.0	77.48	

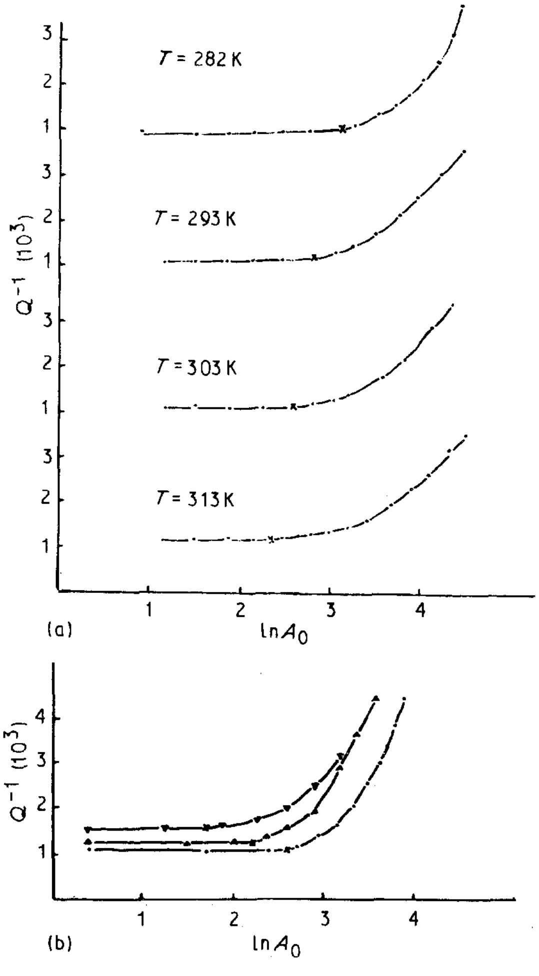

Figure 4 Q^{-1} versus $\ln A_0$ at (●) 282 K, (▲) 296 K, and (▼) 320 K for specimens (a) Al–0.06% RE, (b) Al–0.01% RE, f = 0.6 Hz.

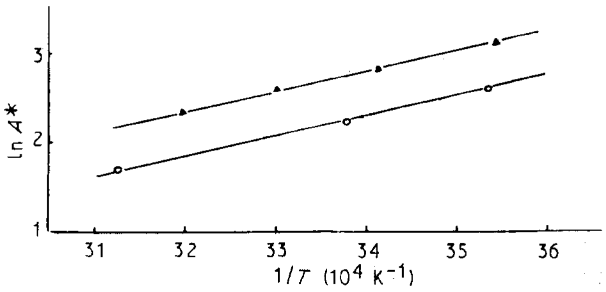

Figure 5 Plots of $\ln A^*$ as a function of T^{-1} for samples (○) Al–0.01% RE and (▲) Al–0.06% RE.

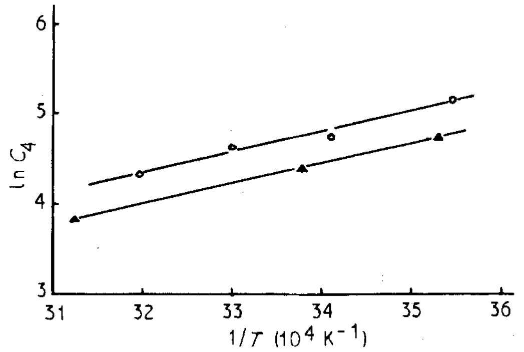

Figure 6 Plots of $\ln C_4$ as a function of T^{-1} for samples (▲) Al–0.01% RE and (○) Al–0.06% RE.

two straight lines are the same. By regression, both are 2192 for Al–0.01% RE and 2120 for Al–0.06% RE. The regression expressions for the two samples are given by

$$\ln A^* = -5.0 + 2192/T \qquad (8)$$

$$\ln A^* = -4.43 + 2120/T \qquad (9)$$

where Equation 8 is for Al–0.01% RE and Equation 9 is for Al–0.06% RE. Comparing the two expressions above with Equation 7a can give $G \sim 0.19$ eV. This G value is the binding energy between RE atoms and the dislocations at room temperature. In addition, because A^* is proportional to C_4, a plot of $\ln C_4$ against $1/T$ should also be a straight line and its slope should be the same as the slope in Fig. 5. The plots of C_4 from Table II are seen in Fig. 6; two parallel lines are also obtained whose slopes are about 2260 for Al–0.01% and 2245 for Al–0.06% by regression. The difference between the slopes in Figs 5 and 6 is within experimental error.

According to the Equations 7–9, the solid solution concentrations of RE in the lattice at room temperature for specimens Al–0.01% RE and Al–0.06% RE can be obtained by taking $E = 70.6$ GPa [9], $b = 2.86$Å [9], $L = 3$ m, $r = 0.45$ mm, $G = 0.19$ eV, $l = 150$ mm for the former and 210 mm for the latter, which are about 0.000 18 wt% and 0.000 23 wt%, respectively.

We can also obtain the solubility of RE in the Al lattice at room temperature (283 K) half-quantitatively. Obviously, the solid solution RE concentration in the sample Al–0.1% RE, in which RE is saturated, can be considered to be the solubility of RE in Al at room temperature. It was found by direct determination using Equation 6, which is about 0.000 27% RE (wt); it can also be obtained from the ratio of RE concentration for Al–0.1% RE to that for Al–0.01% RE according to Equation 6, i.e.

$$A^*1/l_1/A_2^*/l_2 = C_{01}/C_{02} \qquad (10)$$

in which subscript 1 indicates Al–0.1% RE and subscript 2 indicates Al–0.01% RE, which is also about 0.000 27 wt %. If the data for Al–0.06% RE are put into Equation 10 instead of those for Al–0.01% RE, the solubility is about 0.000 28 wt %. Thus we have the same value according to different determinations of RE solubility which is about 0.0003 wt %. This is only an evaluated value, but it is sufficient to show how small is the solubility of RE in Al at room temperature and that it is impossible to strengthen Al material using the rare-earth elements in the state of solid solution.

5. Conclusions

1. Amplitude-dependent internal friction at room temperature for Al–RE alloy can well be described by the G–L theory, the correlation coefficient of the G–L straight line for all samples being close to 1.
2. The unpinning strain of a dislocation and the slope of the G–L plot increase with RE content up to 0.1% and tend to change little when RE% > 0.1.
3. The solid solution RE concentration in the lattice at room temperature increases with increasing RE content up to 0.1% RE, at which RE is saturated.
4. The binding energy between RE atoms and dislocations is about 0.19 eV and the lattice solubility of RE in Al at room temperature is very small, only about 0.0003 wt %.

Acknowledgement

The author thanks the Guang-zhou Institute of Non-ferrous Metals for supplying all the Al-RE samples.

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Received 13 March
and accepted 6 November 1990