

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

Part I *Influence of RE on grain boundary internal friction of aluminium*

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This paper is the first part of an investigation of the distribution and the state of rare earth (RE) elements in aluminium by the method of internal friction. The grain boundary internal friction was measured for 4N pure aluminium samples with various RE contents (0 to 3%, nine in number) with a high vacuum Ke torsion pendulum. It has been found that the peak height and the background decrease with increasing RE content up to 0.5%, and that when RE content $> 0.5\%$ the peak height changes little but the background rapidly increases with RE content. A series of grain boundary internal friction parameters were obtained with the help of conventional and new methods. The various behaviours of RE in aluminium were discussed from the effect of RE content on the relaxation strength Δ and the activation energy H . The relation between the parameter β_H , which is a measurement of the distribution width of H , and RE content was inferred.

1. Introduction

In recent years, the use of rare earth (RE) elements in aluminium and its alloys has been becoming increasingly extensive [1]. Little is known, however, about the effect of the mechanisms of RE in aluminium. Obviously, it is especially necessary to know the distribution and the state of RE in aluminium. From the fact that the differences between the RE atom and aluminium atom in diameter and electronegativity are considerable [2], it is known that the solubility of RE in aluminium is very small, however, reliable values of the solubility have not been obtained. The unavoidable existence of the defects (grain boundary and dislocation etc) in the interior of metals and the much bigger diameter of RE atoms means that it can be shown that the RE atom in the state of solid solution, if any, should have a preference for segregating in grain boundaries and dislocations. When the RE content is high, most of RE elements will exist in an Al-RE eutectic compound or intermetallic compound in the grain boundary, which has been confirmed when RE% > 1 [3].

Since internal friction is especially sensitive to impurities we use this method to investigate the distribution and the state of RE in aluminium. Much work about various internal friction behaviours of aluminium and its alloys has been done but little about internal friction of Al-RE alloys. The present paper studies, in particular, the influence of RE on grain boundary internal friction of aluminium and discusses the behaviour of RE in aluminium.

2. Samples and experimental procedure

Nine 4N aluminium samples with different RE contents (1: 0.0%; 2: 0.005%; 3: 0.01%; 4: 0.06%; 5: 0.097%; 6: 0.51%; 7: 1.0%; 8: 1.2%; 9: 3.0% in weight) were kindly supplied by Guangzhou Institute of Non-ferrous Metal Research. These samples are all in cold-drawn states with diameters of 1.2 mm. The rare earth contained in the samples is the mischmetal which contains about 40% Ce, 20% La, 10% Nd in weight (the rest: other rare earth elements). These specimens were redrawn to a diameter of 0.9 mm for our internal friction measurement by Ke's torsional pendulum in a vacuum of about 5×10^{-4} Torr. Specimens for measurement are about 300 mm long, and annealed at 720 K for 2 h in the internal friction apparatus. Measurements were carried out with the temperature decreasing about 1°C min^{-1} .

3. Experimental results

The original grain boundary internal friction curves for the specimens 1 to 9 were obtained as shown in Figs 1 and 3. It is shown that when RE% > 0.5 the background internal friction increases rapidly with RE content and that when RE% ≤ 0.5 the opposite is true. The background internal friction of the original curves in Figs 1 and 3 was subtracted according to a new method [4]. Corresponding curves are seen in Figs 2 and 4. The activation energy values were found from the shift of each point on the curve (after subtraction of the background) with vibrating frequency

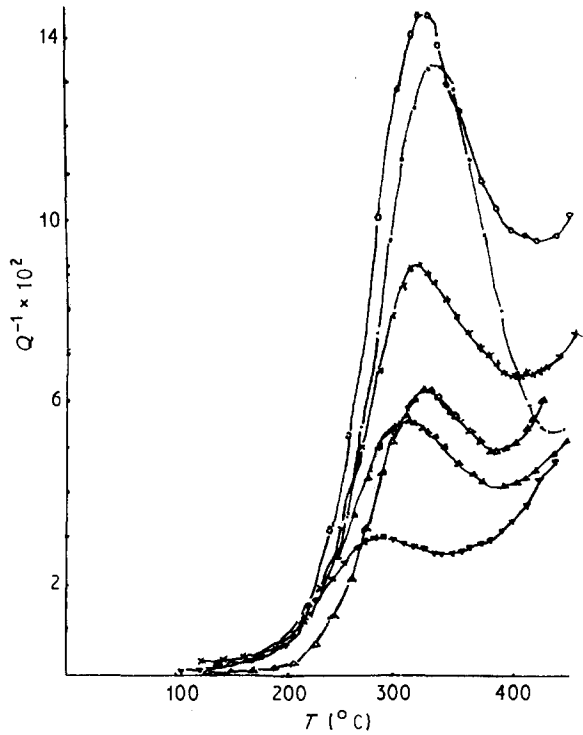


Figure 1 Original curves of grain boundary internal friction for specimens 1 to 6 (● 1, ○ 2, × 3, △ 4, ▲ 5, ▼ 6) 450°C for 2 h, $f \sim 0.5$ Hz.

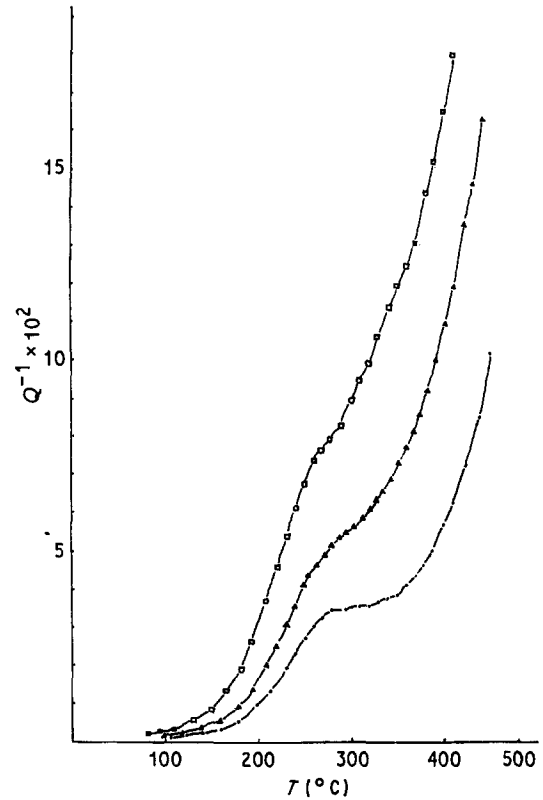


Figure 3 Q^{-1} against T experimental curves for specimens 7 to 9 (□ 9, ▲ 8, ● 7).

along reciprocal temperature axis [5]. The peak height Q_p^{-1} and peak temperature T_p can be obtained directly from Figs 2 and 4. The frequency factor can be determined from the relation $\omega\tau = 1$ when $T = T_p$ in which ω is the circular frequency and τ the relaxation time. Under the condition of the knowledge of the activation energy, the relaxation strength Δ and $\beta(T_p)$, which is a measurement of the distribution width of the relaxation time τ around T_p , can be obtained from Nowick's method [6]. These results are shown in Table I. In addition, with the help of the method in

which various parameters can be determined only from one curve after subtraction of the background internal friction [4], the values of the corresponding parameters mentioned above were obtained as shown in Table II. It can be seen from both tables that both accord well and some parameter values in Table II cannot be found by the conventional method.

Specimens 7 to 9 have very high background internal friction after annealing at 720 K for 2 h. Specimen 8 was chosen for further study. Fig. 5 shows its internal friction curve after additional high temperature annealing at 890 K for 7 h in a vacuum (named 8-2, and 8-1 for 720 K + 2 h). The background decreases considerably but the peak height changes a

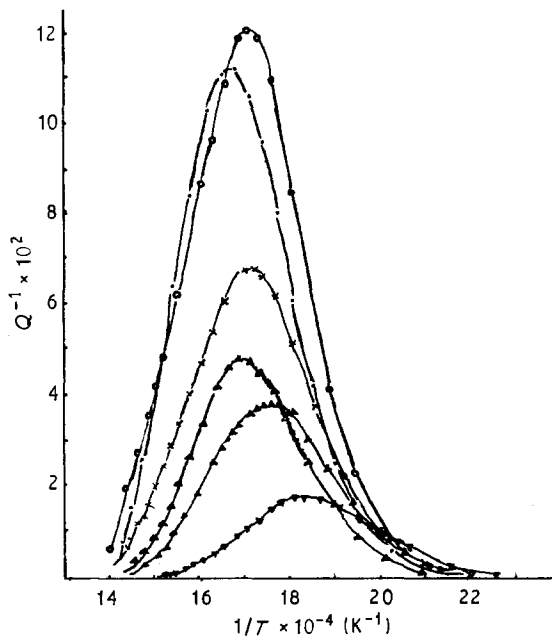


Figure 2 Q^{-1} against $1/T$ curves obtained from Fig. 1 after subtraction of background. $f \sim 0.5$ Hz. Symbols as in Fig. 1.

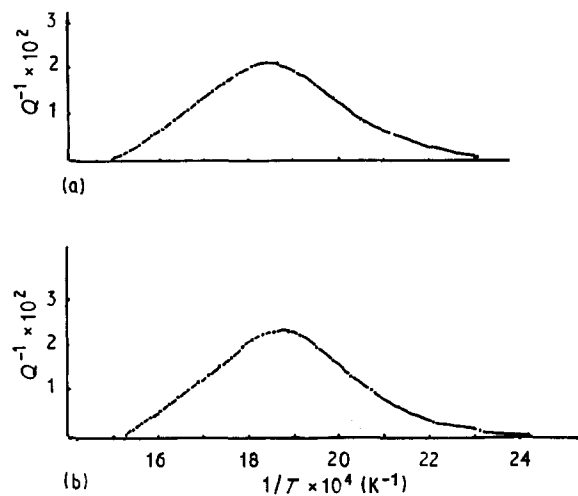


Figure 4 Q^{-1} against $1/T$ curves obtained from Fig. 3 after subtraction of background. $f \sim 0.5$ Hz. (a) 7, (b) 8-1.

TABLE I Grain boundary internal friction parameters obtained by conventional methods

Specimen	H (kJ mol ⁻¹)	$\tau_0 \times 10^{16}$	$\beta(T_p)$	T_p (K)	$Q_{\max}^{-1} \times 10^2$	$\bar{d} \times 10^2$ (mm)	Δ	RE (wt %)
1	160.76 ± 8.0	30.9	2.73	598	11.20	20	0.550	0.0
2	162.60 ± 8.0	10.7	2.85	585	12.08	32	0.629	0.005
3	169.37 ± 10	2.17	3.33	583	6.83	16	0.357	0.01
4	217.78 ± 15	1.24 × 10 ⁻⁴	4.00	598	5.03	6.3	0.295	0.06
5	159.26 ± 12	6.43	3.00	570	3.80	11	0.169	0.097
6-1	143.79 ± 7.0	57.5	2.85	547	1.75	2.0	0.072	0.51
6-2	161.43 ± 8.0	7.36 × 10 ⁻¹	3.13	562	2.16	18	0.096	0.51
7	162.85 ± 8.0	6.17 × 10 ⁻¹	3.77	543	2.13	1.2	0.109	1.0
8-1	162.18 ± 8.0	4.83 × 10 ⁻¹	3.71	536	2.10	0.78	0.106	1.2
8-2	160.51 ± 8.0	8.59 × 10 ⁻¹	3.29	541	1.63	2.4	0.074	1.2
9				532	3.00	0.55		3.0

\bar{d} is the average grain diameter in section, 6-2: 720 K 2h + 910 K 3h, 8-2: 720 K 2h + 890 K 7h.

TABLE II Grain boundary internal friction parameters obtained by a new method

Specimen	H_m (kJ mol ⁻¹)	$\tau_{0m} \times 10^{16}$	β_0	β_H	T_p (K)	$\beta(T_p)$	$\tan \phi(T_p) \times 10^2$	Δ
1	160.76 ± 5.0	39.6	-0.16 ± 0.05	1685 ± 10	597.9 ± 0.5	2.66	11.31	0.532
2	157.63 ± 5.0	51.1	2.39 ± 0.05	262 ± 10	585.5 ± 0.3	2.84	12.34	0.625
3	169.37 ± 5.0	2.88	2.59 ± 0.05	406 ± 10	581.9 ± 0.3	3.29	6.88	0.352
4	212.47 ± 5.0	4.06 × 10 ⁻⁴	1.70 ± 0.05	1221 ± 10	596.0 ± 0.3	3.75	5.04	0.277
5	160.51 ± 5.0	5.39	2.04 ± 0.05	553 ± 10	569.0 ± 0.3	3.02	3.81	0.170
6-1	147.30 ± 5.0	19.6	-1.50 ± 0.05	2392 ± 10	544.4 ± 0.3	2.90	1.75	0.073
6-2	161.85 ± 5.0	2.41	-0.95 ± 0.05	2189 ± 10	561.8 ± 0.3	2.94	2.16	0.092
7	163.69 ± 5.0	5.39 × 10 ⁻¹	1.96 ± 0.05	1025 ± 10	543.4 ± 0.3	3.85	2.14	0.112
8-1	162.18 ± 5.0	4.91 × 10 ⁻¹	1.98 ± 0.05	822 ± 10	535.5 ± 0.3	3.52	2.11	0.102
8-2	160.5 ± 5.0	8.94 × 10 ⁻¹	0.99 ± 0.05	1226 ± 10	541.4 ± 0.3	3.25	1.65	0.074

little, the peak temperature slightly shifts to a higher temperature. The metallographic inspection showed that the grain size has not grown obviously and its diameter in section is 0.024 mm (0.0078 mm for specimen 8-1). It may be suggested that the slight increase in peak temperature results from the growth of grain size [7]. The different parameter values for specimens 7 to 9 were obtained in the way mentioned above and are shown in Tables I and II.

The grain size diameter (in section) for all samples is seen in Table I. After additional high temperature annealing of specimen 5 at 890 K for 7 h and specimen 6 at 910 K for 3 h (named 6-2, and 6-1 for 720 K + 2 h) in a vacuum, it has been found that the former's grain size is larger than the specimen's diameter and the height of grain boundary internal friction peak decreases considerably, but the latter is

about 0.18 mm, namely has not grown obviously and its internal friction curve accords with that of specimen 6-1. It seems that the growth effect of grain at high temperature can well be prevented when RE% > 0.5.

4. Discussion

4.1. Influence of RE on aluminium grain boundary internal friction

It is seen from Figs 2 and 4 and Tables I and II that RE strongly influences the grain boundary internal friction of aluminium at the peak height, the relaxation strength and the activation energy when RE content is not more than 0.5%. When RE % > 0.5 the influence of RE is almost saturated.

4.1.1. Peak height

Many dislocation models have been put forward for the grain boundary structure [8-10]. Briefly, it was thought that there exists a dislocation network in the grain boundary and the dislocation can move under stress leading to the slide of grain boundary, and that this motion of the dislocation includes glide and climb [9]. The concept of grain boundary dislocation can explain our experimental phenomena.

The big atom size means that there is a strong tendency of RE atoms to segregate to the grain boundary. More and more RE atoms will preferably be distributed in the grain boundary with the increase in RE content, which, under stress, will prevent the motion of grain boundary dislocations, reduce the

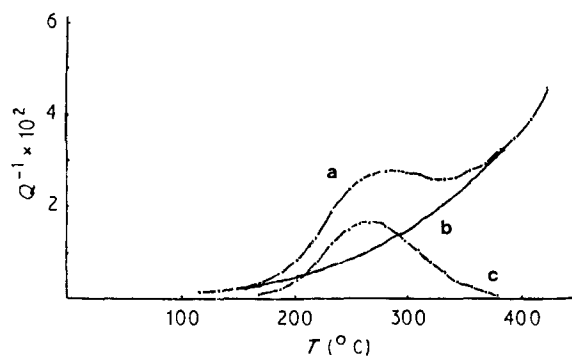


Figure 5 Q^{-1} against T curves for specimen 8-2. (720 K 2h + 890 K 7h) (a) original curve, b theoretical background, c after subtraction of background) $f \sim 0.5$ Hz.

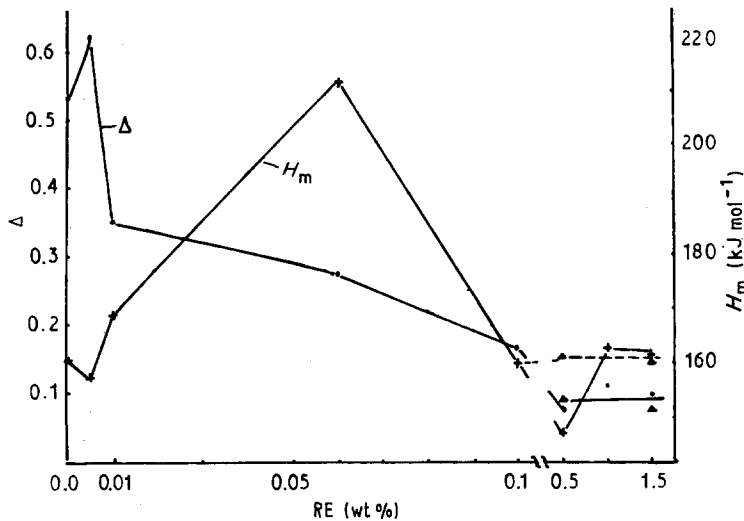


Figure 6 Effect of RE content on the relaxation strength Δ and the activation energy H_m . (● Δ 720 K, 2 h; Δ Δ 720 K 2 h + 890 K 3 h; + H_m 720 K, 2 h; \blacktriangle H_m 720 K 2 h + 890 K 3 h).

moving distance of them and decrease the sliding distance of grain boundaries, thus the peak height or the relaxation strength Δ will rapidly decrease with RE content, which agrees with the experimental facts as shown in Fig. 6 which is plotted from the data in Table II. It can be seen from Fig. 6 that the relaxation strength value is higher for specimen 2 than for specimen 1 with no RE. The RE in aluminium has purifying and the segregating effects [1]. The added RE may partly form inclusions or compounds with some non-solute or solute in aluminium. As a result the impurity content in the state of solid solution decreases. The other part of RE, naturally, has a segregating effect but the purifying effect will dominate in both effects of RE when RE content is small or when RE% < 0.01, which makes the total solid solution solute content in aluminium, hence, in the grain boundary decrease. In addition, the solute atoms in the grain boundary can prevent the grain from growing [11]. It can be deduced from the discussion above that specimen 1 with no RE content should be smaller in grain size than specimen 2 with 0.005% RE content after the same heat treatment because of the higher total solid solution solute content in the former, which has been proved by metallographic inspection. The former is about 0.2 mm, and the latter 0.32 mm (see Table I). Meanwhile, the decrease in solid solution solute content in the grain boundary will make the slide of the grain boundary under alternative stress easier and increase the movable distance of the dislocation on the grain boundary, i.e. the activation energy will be smaller and the relaxation strength greater (see Fig. 6). It can also be seen from Fig. 6 that when RE% \geq 0.01 the relaxation strength is smaller than that of specimen 1, i.e. when the RE content is about 0.01% the segregating effect of RE in grain boundaries has been dominant and the segregation extent of RE in the grain boundary has been greater than that of impurity atoms in specimen 1 with no RE in grain boundary. Thus the relaxation strength decreases with RE content when RE% \geq 0.01 and up to 0.5% solid solution RE in the grain boundary is saturated. When RE% > 0.5 the Al-RE eutectic compound increases with RE content but the solid solution RE content does not, therefore, the peak height or the relaxation

strength changes little or increases slightly (the phase boundary may have a contribution to the grain boundary internal friction peak height [12-14]).

The segregating effect of RE content means that when RE% \geq 0.01 the grain size becomes smaller with RE content increase. Especially when RE% > 0.5 the grain size decreases more markedly due to the increase in Al-RE eutectic compound.

4.1.2. The relaxation time τ

τ includes the most probable values τ_{0m} and H_m of the frequent factor τ_0 and the activation energy H , and β_0 and β_H [4] which are the measurements of the distribution widths of $\ln \tau_0$ and H respectively.

4.1.2.1. τ_{0m} . For all specimens, the τ_0 values obtained by the conventional method (see Table I) and the τ_{0m} values obtained by a new method [4] (see Table II) were in accord with the order of magnitude. The differences between the different specimens in the τ_{0m} or τ_0 value are, however, considerable. The relaxation time is directly proportional to the grain size for Al [7, 15], thus the effect of grain size should be eliminated by dividing τ_0 or τ_{0m} by \bar{d} , which is the average grain section diameter. The results are seen in Table III. It can be shown that the τ_{0m}/\bar{d} or τ_0/\bar{d} values for most specimens agree to within an order of magnitude except that specimen 4 is obviously different. On this basis, it is known that the RE content has little effect on the τ_{0m}/\bar{d} values despite the apparent effect on the τ_{0m} values, which may be related to the small solubility of RE content in aluminium.

4.1.2.2. β . β , which is a measurement of the distribution width of $\ln \tau$ and relates to temperature T [4], includes the joint contribution of the distributions of τ_0 and H . It is difficult to separately discuss the distribution of τ_0 or H only from the β value at a temperature. All specimen's β_0 and β_H values, were obtained with the help of the new method [4] (see Table II). The distribution of τ_0 contains distributions of both Debye frequency and the activation entropy,

TABLE III The frequent factors after eliminating the affect of grain size

Specimen	1	2	3	4	5	6-1	6-1	7	8-1	8-1
$\tau_0/\bar{d} \times 10^{15}$	15.5	3.34	1.36	1.97×10^{-4}	5.85	2.88×10^2	0.41	5.14	6.19	3.58
$\tau_{0m}/\bar{d} \times 10^{15}$	19.8	16.0	1.80	6.44×10^{-4}	4.90	98.0	1.34	4.49	6.29	3.73

means that it is also difficult to discuss both respectively only from β_0 values and it will not be discussed here but only β_H the measurement of the distribution width of H is discussed as follows.

From Fig. 6 it is seen that the most probable activation energy H_m changes with RE content. Statistically, the concentration fluctuates even for an homogeneous solid solution. This manifests a possibility of the distribution of the activation energy H . It is suggested that there is an increase in the thickness of the grain boundary where there are RE atoms in the state of solid solution, and that all the grain boundary areas in a specimen are divided into many parts which contain n atoms and in which the RE concentration (in the state of solid solution) is expressed as C . There are different RE concentrations for different parts thus there exists the distribution of RE concentration C in the grain boundary. The average solid solution RE concentration over all parts is represented by C_m . Meanwhile, taking into account the interaction between RE and aluminium atoms, it can easily be found [16] that the distribution function of solid solution RE concentration in grain boundaries, because of the fluctuation, is subjected to Gaussian distribution. B , which is a measurement of the distribution width of RE concentration in the grain boundary, is given by

$$B = \left[n \left(\frac{-ZW}{kT} + \frac{1}{2C_m(1 - C_m)} \right) \right]^{-\frac{1}{2}} \quad (1)$$

$$W = W_{\text{REAl}} - (W_{\text{RERE}} + W_{\text{AlAl}})/2$$

where W_{REAl} , W_{RERE} and W_{AlAl} are the binding energies between the atoms denoted by the subscripts, Z the coordination number and k Boltzmann's constant. There is no harm in suggesting that the fluctuation of the activation energy relates to that of the RE concentration, then it is easily shown that when RE% is small there is an approximate linear relation between β_H and the square root of RE content (see Appendix) i.e. $\beta_H \propto \sqrt{\text{RE}\%}$. To verify this three specimens 2, 3, 4 were chosen whose RE contents are the smallest, the

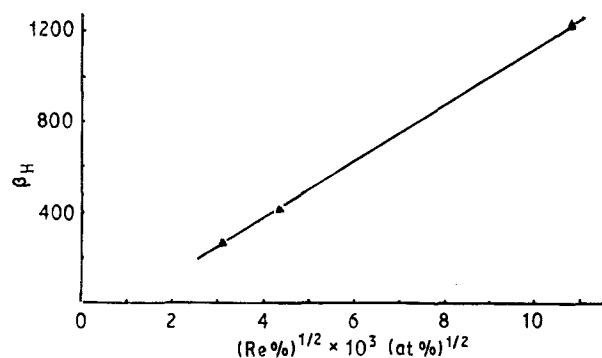


Figure 7 β_H as a function of RE content.

curve of β_H against $\sqrt{\text{RE}\%}$ was plotted from the data in Table II (Fig. 7). A good linear relation exists between β_H and $\sqrt{\text{RE}\%}$ when RE% is not more than 0.06. Thereby it is verified that the distribution of the activation energy arises from the concentration fluctuation of solid solution RE in the grain boundary. From the data in Table II it is shown that when RE > 0.5%, because of saturation, β_H changes little with increase in RE content.

4.2. The influence of RE content on the background

It is generally thought that the background internal friction arises from the interior of grains and its value directly relates to the degree of cold work or dislocation density. The background is very high for the specimen whose annealing is incomplete [17]. On the basis of the fact that the true activation energy controlling the background process was found to be equal to that of self-diffusion of atoms [18], it has been pointed out [15] that the background results from a diffusion-controlled dislocation motion. This involves the bowing of a dislocation segment which is pinned at its ends and is surrounded by an atmosphere of solute atoms. For a sample containing N_v dislocations per unit volume, the relaxation strength Δ is given by the approximate relation [15]

$$\Delta = N_v L^3 / 18 \quad (2)$$

where L is the average dislocation segment length. It may be seen from Fig. 1 that the change tendency of the background with RE% is the same as that of the grain boundary peak height when RE% \leq 0.5. When RE% > 0.5 the increase rate of the background with the temperature rapidly increases with RE content (see Fig. 3). These phenomena can well be explained from the model above.

When RE% < 0.01, because the purification effect of RE dominates, the pinning points at the dislocation are reduced and L becomes longer. Thus the background for specimen 2 is higher than that for specimen 1. As mentioned above, there is a strong tendency for RE atoms to pin dislocations. With the increase in RE content, more and more RE atoms will pin the dislocations in the interior of grains and L becomes shorter (for fully annealed samples, N_v is approximately equal). Thereby the background decreases with RE content increase when RE% \geq 0.01 up to 0.5%. When RE% = 0.5 the solid solution RE is saturated and the concentration at dislocations is up to the greatest and L is the shortest thus the background is a minimum (see Equation 2).

It may also be seen from Figs 1 and 2 that the changes of the peak height and the background with RE content have a difference: for the peak height,

when RE% ≥ 0.01 , it is lower than that of specimen 1 but for the background, only when RE% ≥ 0.06 . This shows that RE atoms segregate more strongly in grain boundaries than in dislocations in the interior of grains, which arises from large RE atomic radius.

The Al-RE eutectic compound will increase with RE content when RE content is more than 0.5%. In cold deforming the Al-RE eutectic compound is crushed and dislocations move around the compound. Obviously, the dislocation density is higher at the same degree of deformation compared with the samples with no compound. In addition, the Al-RE eutectic compound would increase the resistance to the motion of dislocations in annealing at high temperature and make the recrystallization rate slow. The background for samples 7 and 8-1 and especially sample 9 is very high. As mentioned above, incomplete annealing can result in a high background [17]. It can thus be suggested that the deformation of these three samples, after annealing at 720 K for 2 h, has not yet been eliminated completely and the recrystallization has not been finished, which causes the high background. After annealing at higher temperature, the background obviously decreases because of further recrystallization (see Fig. 5). Comparing specimen 8-2 with specimen 6-1, both internal friction curves are similar not only in the background but also in the peak height (see Figs 5 and 1). This also confirms the above viewpoints when RE% ≥ 0.5 the solid solution RE in the grain boundary and the interior of grains is saturated, this is shown in that under the condition of complete annealing the peak height and the background change little with RE content when RE% ≥ 0.5 ; the deformation cannot be eliminated completely for the treatment at 720 K for 2 h when RE% > 0.5 ; incomplete recrystallization is the cause of high background internal friction, i.e. RE can increase the recrystallization temperature of aluminium.

5. Conclusions

The conclusions are as follows.

(1) When RE% < 0.01 , the purification effect of RE dominates, which leads to the increases in the internal friction peak height, the background and the grain size.

(2) When RE% ≥ 0.01 , RE atoms have a preference for segregating in grain boundaries and dislocations, which causes the grain boundary internal friction peak height and the background to lower, and grain refining with RE content to increase up to 0.5%.

(3) When RE% > 0.5 , the solid solution RE is saturated and Al-RE eutectic compound appears leading to the increase in recrystallization temperature of aluminium, which gives rise to little change in grain boundary internal friction peak height and high background when annealed at 720 K for 2 h.

(4) The distribution of activation energy associated with grain boundaries results from that of the solid solution RE concentration in grain boundaries. There exists an approximate linear relation between β_H and $\sqrt{\text{RE}\%}$ when RE% ≤ 0.06 .

Appendix

Assume that the distribution of H relates to the fluctuation of RE concentration C in the grain boundary, i.e. $H = f(C)$ and $H_m = f(C_m)$ in which the subscript m means most probable. At C_m , we have

$$H - H_m = A(C - C_m) \quad (\text{A1})$$

$$A = \left. \frac{dH}{dC} \right|_{C=C_m}$$

As mentioned, C is of Gaussian distribution. From a knowledge of probability theory and Equation 1 it is easily shown that H also obeys Gaussian distribution and β_H , which is a measurement of the distribution width of H , is given by

$$\beta_H = |A|B = \frac{|A|}{\sqrt{n}} \left(-\frac{ZW}{kT} + \frac{1}{2C_m(1 - C_m)} \right)^{-\frac{1}{2}} \quad (\text{A2})$$

When C_m is small, the term ZW/kT can be ignored. It does not harm to estimate the approximate values: let $T = 300$ K, $C_m = 0.1\% = 0.001$, $Z = 10$ (being less close-packed in grain boundaries), $W = 0.1$ eV [19], $ZW/kT \approx 40$ and $1/(2C_m(1 - C_m)) \approx 500$ thus ZW/kT can be ignored. We have

$$\beta_H \approx \frac{|A|}{\sqrt{n}} \left(\frac{1}{2C_m(1 - C_m)} \right)^{-\frac{1}{2}} \quad (\text{A3})$$

Assume that RE in aluminium exists, according to some proportion, in the state of solid solution and, if any, in the form of compound which is formed by RE with non-RE impurity in aluminium. The proportion is approximately considered a constant, which is expressed as D , when the range of RE content is small. The relation between solid solution RE concentration C_1 and the total RE composition RE% can be written as

$$C_1 = D \text{RE}\% \quad (\text{A4})$$

The relation between the C_m , which is also the solid solution RE concentration in grain boundaries, and C_1 is

$$C_m = C_1 \exp(\Delta G/RT) \quad (\text{A5})$$

where R is the gas constant, ΔG the mole free energy difference of RE in the interior of grain and the grain boundary, thus

$$C_m = F \text{RE}\% \quad (\text{A6})$$

$$F = D \exp(\Delta G/RT)$$

For different samples (small change of RE%) under the same conditions, F can be thought of as a constant. Substituting Equation A6 into Equation A3 we have

$$\beta_H \approx \frac{|A|}{\sqrt{n}} \left(\frac{1}{2F \text{RE}\%(1 - F \text{RE}\%)} \right)^{-\frac{1}{2}}$$

when RE% is very small

$$\beta_H \approx P \text{RE}\% \quad (\text{A7})$$

$$P = \frac{|A|}{\sqrt{n}} (2F)^{1/2}$$

For a small range of RE composition P is approximately a constant, so there is a linear relation between β_H and $\sqrt{\text{RE}\%}$, i.e. $\beta_H \propto (\text{RE}\%)^{1/2}$.

Note that in Equation A7, only the change of β_H with RE% is considered. It is seen from Fig. 7 that β_H is not zero when the RE composition is extrapolated to zero. Even in 100% Al the density of atoms in grain boundaries cannot everywhere be the same, thus β_H should not be zero.

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