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Relation between fabrication parameters and grain boundary internal friction peaks in commercial Al–Mg–Si alloys

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Abstract

The polycrystalline Al–Mg–Si alloys (Al alloy 6063) have an internal friction peak at 480 K (1 Hz) attributed to grain boundary relaxation in the presence of particles. The aim of this work is to check the influence of certain fabrication parameters (the alloy chemical composition, the extrusion temperature and the homogenization conditions) on the grain boundary internal friction peaks of a special set of alloys.

The relaxation strength increases with increasing Si content. This effect is due to an enhancement of Si grain boundary precipitation. We do not observe the dependence on the excess Si (with respect to the stoichiometric chemical composition) predicted by others authors. The extrusion temperature indicates a separation of the alloys into different groups obeying different β and Si grain boundary precipitation. The homogenization parameters do not seem to be important in this case.

1. Introduction

The Al–Mg–Si alloys, treated to reach the peak-aged condition (maximum in the hardness *vs.* aging time plot for the optimum aging temperature) show mechanical properties that are extremely sensitive to microstructure. The desired high matrix strength may be achieved in samples with different compositions after thermomechanical treatments, leading to the precipitation of the intermediate phase β'' [1] inside the grains. However, for a given dispersion of hardening precipitates inside the grains, fracture may be transgranular or intergranular depending on other microstructural features such as grain boundary precipitation.

The polycrystalline Al–Mg–Si alloys (Al alloy 6063) exhibit an internal friction peak at 480 K (about 1 Hz) which has been attributed [2] to grain boundary relaxation in the presence of precipitates by analogy with the pure Al or Al–Si grain boundary peak. The mechanism proposed [3] involves glide and climb of dislocations in the grain boundary zone. In ref. 3, the conditions for the observation of the two internal friction peaks predicted by Mori *et al.* [4] (one at a lower temperature than in the pure metal for effective blocking precipitates and another at a higher temperature when stress is relaxed by diffusional accommodation around the precipitates) were analysed for the case of aluminium. The conclusion in ref. 3 was that in Al alloys the model based on a continuous sliding mechanism leading to the lower temperature peak could be applied only when particles are large (1 μm) and closely spaced. The presence of the higher temperature peak is conditioned by the stability of the precipitates. Only one peak is expected in the case of smaller particles and, in this situation, dislocation models become necessary to describe the effect of particles. In these conditions, peak characteristics depend on the distribution geometry, the local diffusion (at the boundary or in the matrix near the precipitates) and the nature of the precipitate–matrix interface.

In this work the grain boundary internal friction peak is investigated in a special set of samples produced in a research program [5] by Aluminio Argentino S.A.

2. Experimental procedure

The compositions and thermomechanical treatments of the specimens are shown in Table 1.

After extrusion, all samples were cooled at 10 °C min^{-1} to room temperature, naturally aged for 6 h and then artificially aged for 8 h at 175 ± 1 °C, to reach the peak-aged condition.

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TABLE 1. Fabrication parameters of alloys

Sample	Mg (wt.%)	Si (wt.%)	Excess Si (wt.%)	Fe (wt.%)	T_h (°C)	t_h (h)	V_c (°C h^{-1})	T_{extr} (°C)	d (μm)
5	0.58	0.50	0.16	0.17	540	4	650	430	42
6	0.46	0.46	0.19	0.17	580	4	650	430	38
8	0.56	0.38	0.06	0.17	580	8	650	430	34
10	0.58	0.50	0.16	0.17	580	4	180	480	42
12	0.46	0.31	0.04	0.17	580	8	180	480	41
13	0.56	0.38	0.06	0.17	540	4	650	480	44
15	0.46	0.46	0.19	0.17	540	4	650	480	57

T_h, homogenization temperature; t_h, homogenization time; V_c, cool rate after homogenization; T_{extr}, extrusion temperature; d, grain size measured with standard method [6].

Samples were machined from ingots of 16 mm diameter to cylinders of 2.6 mm diameter and 50 mm length, with heads of 3.2 mm diameter and 5 mm length.

Internal friction and period measurements were made in an automatic inverted torsion pendulum, oscillating at constant amplitude, at frequencies of about 2 Hz and at a maximal strain amplitude of 2.7×10^{-4}. The internal friction signal was calibrated by free oscillation decay in the peak range. The spectra were obtained during controlled heating from room temperature to 573 K at 1.5 K min^{-1}.

Grain size determinations were made in finely polished (0.25 μm diamond paste) and electrochemically polished surfaces (5% HBF_4 in water), observed with polarized light.

Special care was taken in grain size determinations by the standard method [6] using optical micrographs as shown in Fig. 1, for example.

3. Results

Figure 2 shows an example of our internal friction results. Curve 1 is the resulting internal friction spectrum as a function of temperature and curve 2 the square of the frequency calculated from the period measurement. They both correspond to sample 15.

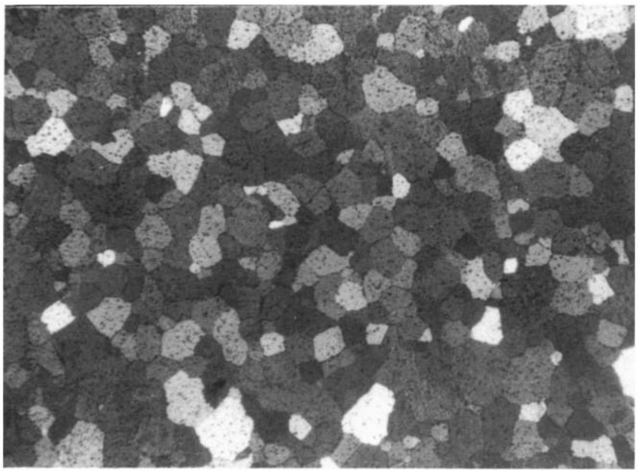

Fig. 1. Optical micrograph of sample 15 showing grain structure (d=57 μm). (Magnification, 50×.)

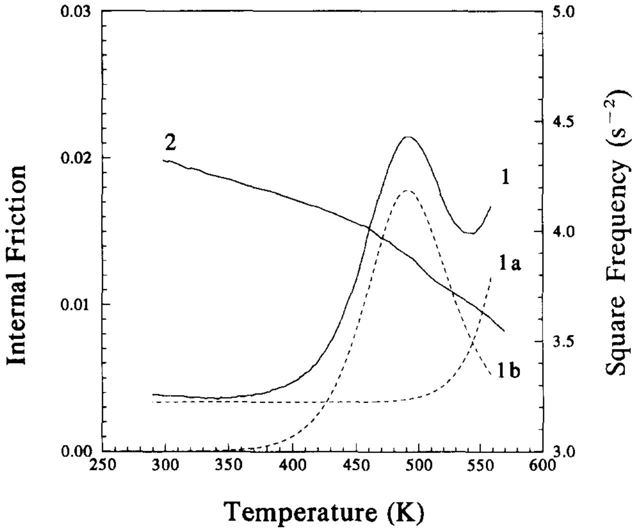

Fig. 2. Example of internal friction measurement and square frequency calculation during the first heating of sample 15 as received from the factory ($\dot{T}$=1.5 K min^{-1}): curve 1, internal friction spectrum; curve 1a, background subtracted; curve 1b, grain boundary peak; curve 2, square frequency.

The spectrum is analysed in terms of two contributions: a background increasing exponentially with increasing temperature (curve 1a) and an internal friction peak (curve 1b) about 2.8 times wider than a Debye peak. For more details concerning this spectral analysis, see ref. 2.

The internal friction peak is found at about 490 K (about 2 Hz) in all the samples. Its activation energy is 140 kJ mol^{-1}. These values are in agreement with those reported in ref. 2.

In order to compare the results of different samples, normalization to the average grain size (d_0=50 μm) was applied to the relaxation strength ($\Delta d/d_0$) where d is the grain size and Δ the relaxation strength obtained from the height ϕ_{max} of the peak from the relation

$$\tan \phi_{\max} = \frac{\Delta}{2(1+\Delta)^{1/2}}$$

No correlation between the internal friction spectrum and the homogenization variables (T_h, t_h and V_e; see Table 1) considered here could be established, in agreement with the results reported by Daroqui and Pampillo [5] for the main metallurgical properties of these alloys. As the age-hardening treatment is the same for all the samples, only the extrusion temperature and composition are retained as the processing variables that influence the final properties.

The two extrusion temperatures considered in this work (430 and 480 °C) are below the solvus temperatures for Si and β-Mg_2Si equilibrium precipitates of all the samples. Therefore precipitation is likely to occur, especially in the grain boundaries, during hot forming.

During extrusion at the higher temperature (480 °C), the undercooling is lower and in addition the mechanical work locally raises the metal temperature above the solvus, leading to a lower density of β-Mg_2Si precipitates. Predominantly small Si particles are expected in grain boundaries. These particles grow during subsequent artificial aging.

When extrusion is carried out at the lower temperature, the equilibrium precipitate β-Mg_2Si becomes competitive and, in addition to Si, larger β particles form at the grain boundaries as well as inside the grains. Further annealing during artificial aging makes Si precipitates grow by the addition of Mg and Si atoms.

So, the two extrusion temperatures lead to different precipitation states in the grain boundary zone in the peak-aged condition. The extrusion at 480 °C promotes a fine dispersion of fine precipitates (preferentially Si), while the extrusion at 430 °C gives predominantly β-Mg_2Si precipitates larger in size.

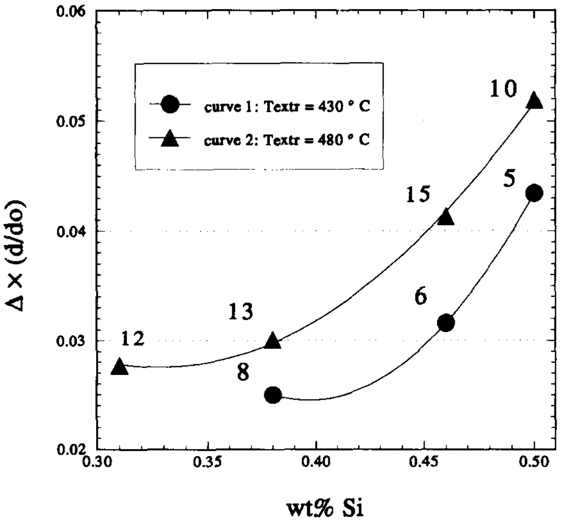

Fig. 3. Grain-size-normalized relaxation strength for all the samples as received as a function of Si content taken from spectra measured as in Fig. 2 (frequency, about 2 Hz; $\epsilon = 2.7 \times 10^{-4}$; $\dot{T} = 1.5$ K min^{-1}).

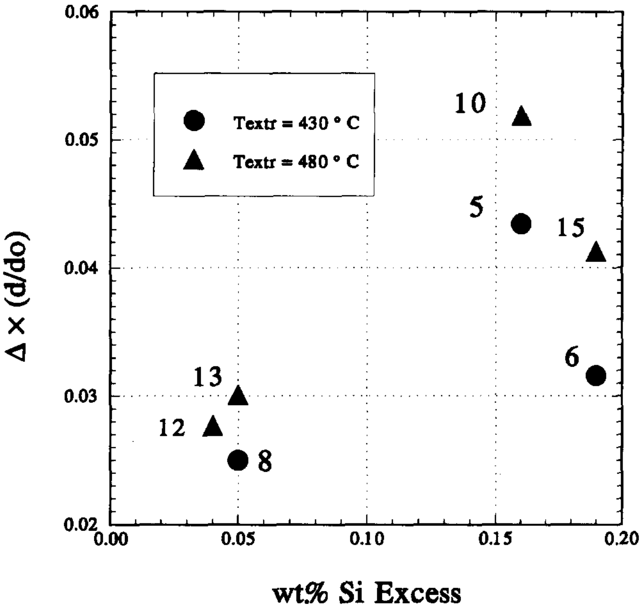

Fig. 4. Grain-size-normalized relaxation strength for all the samples as received as a function of excess Si content taken from spectra measured as in Fig. 2 (frequency, about 2 Hz; $\epsilon = 2.7 \times 10^{-4}$; $\dot{T} = 1.5$ K min^{-1}).

The other processing variable as well as the composition also determines the amount and nature of grain boundary precipitates in the peak-aged condition. The influences of the nominal content of Si and the excess Si in relation to the stoichiometric composition have been investigated in this paper.

Figure 3 shows the normalized relaxation strength as a function of the nominal Si content of the alloys. Two facts are observed.

(1) A continuous increase in relaxation strength is shown for both extrusion temperatures (curves 1 and 2), as the Si content increases.

(2) For a given Si content, specimens extruded at the lower temperature (curve 1) exhibit a smaller relaxation strength than those extruded at the higher temperature (curve 2).

From these internal friction results and microstructural considerations, the following may be concluded.

(a) The relaxation strength strongly depends on the precipitation state in the grain boundary zone.

(b) The relaxation strength is reduced by larger β-Mg_2Si equilibrium precipitates (promoted by extrusion at low temperature).

(c) The relaxation strength increases as the amount of smaller Si-rich precipitates increases (extrusion at higher temperature; higher Si content).

The Si excess content relative to stoichiometry is often mentioned as the principal parameter controlling the metallurgical properties of this alloy [1].

In Fig. 4 the same magnitudes are plotted as a function of the excess Si content of the alloy, referred to the stoichiometric relation 1.73 Mg:Si. Again, relaxation strength values are systematically lower for the specimens extruded at the lower temperature and a similar behaviour is found for these two temperatures. There is not a simple dependence of these magnitudes on the excess Si. This can be explained by taking into account that silicon is actually present in the intermetallic Al–Fe–Si, in the β'' needles (1Mg:1Si), and in the β' and β phases. In consequence, a calculation of the true silicon excess must be more complicated.

4. Discussion

Between the fabrication parameters, the homogenization conditions (T_h, t_h and V_e as given in Table 1) have importance only from a commercial point of view; higher temperatures mean shorter homogenization times. Our results are in agreement with those of Daroqui and Pampillo [5] in the sense that they make no noticeable difference to the metallurgical properties. Therefore we shall pay attention only to the extrusion temperature and the chemical composition. In order to do this, some considerations of the internal friction peak and precipitation in grain boundaries are made.

The effect of β-Mg_2Si precipitated in grain boundaries on the internal friction peak may be explained on the basis of the model developed by Mori *et al.* [4]. These workers propose a model of grain boundary sliding in the presence of blocking particles. According to this, the effect of particles on the grain boundary peak is to decrease its relaxation strength and to shift the peak temperature to lower values, provided that no diffusional stress relaxation takes place at the matrix–particle interface. The β-Mg_2Si precipitates appearing during extrusion at low temperature are this kind of blocking particle because they are coarse and their interface with the matrix has a relatively good resistance to decohesion.

Concerning the effect of Si precipitates in grain boundaries on the peak height, it may be explained in terms of the picture proposed in ref. 3, applicable to grain boundary relaxation in the presence of smaller particles (0.1–0.6 μm), having a low resistance to decohesion (high interfacial energy). Silicon precipitates present in these alloys exhibit these characteristics. When they nucleate and grow in the boundary region, they locally change the mobile dislocation density in the boundary and lead to an increased relaxation strength.

In a study carried out on an Al–11.8%Si alloy containing eutectic silicon particles, Zhou *et al.* [7] arrive at the conclusion that the increase observed in the internal friction with increasing temperature (in the range 400–550 K) is linked with thermal stresses originated in the thermal expansion mismatch between matrix and particles. Because thermal stresses can be relaxed by microplastic strain around particles, the local increase in the density of dislocations can be responsible for internal friction behaviour. This conclusion is in agreement with our interpretation given above about the Si precipitation role in the internal friction.

The fact that the density of Si-rich precipitates in the grain boundary and therefore the relaxation strength depend on the total content of Si rather than on the excess Si may be explained by considering that the nucleation of these precipitates takes place in a solid solution with oversaturation given by the total amount of free silicon in the matrix.

Many workers agree that the drop in ductility often observed in the peak-aged condition is associated with an increased proportion of intergranular fracture. In particular, Si-rich precipitates nucleated in the grain boundary region (the boundaries themselves and the precipitate-free zone near them) are thought to be responsible for boundary weakening [8, 9], leading to early crack development.

Our monotonically increasing internal friction with increase in the Si content is similar to the increase in the percentage of intergranular fracture reported by Acuña and Daroqui [10]. These workers studied the impact load fracture behaviour of samples 12, 10 and others after homogenization (30 min at 520 °C), quenching in water, natural aging at 20 °C and artificial aging for 8 h at 175 °C. In our set of alloys, samples 12 and 10 represent low and high Si contents respectively.

They observed from sample 12 to sample 10 that, as the nominal Si content increases (meaning a higher volume fraction of Si-rich precipitates in the grain boundary zone), the tendency to intergranular fracture increases and the global ductility drops. In this way, the proportion of intergranular fracture is directly related to the internal friction peak height.

5. Conclusions

In this work, the influence of the fabrication parameters on the grain boundary internal friction peak of some special Al–Mg–Si alloys (Al alloy 6063) has been investigated.

Among the variables considered, the composition and extrusion temperature are the most important in determining peak height. They both influence the relaxation strength by promoting different precipitation reactions in the boundary zone.

β-Mg_2Si precipitates reduce and Si-rich precipitates increase the relaxation strength, the former by blocking grain boundary relaxation and the latter by increasing locally the density of dislocations responsible for relaxation.

References

1 L.F. Mondolfo, *Aluminium Alloys: Structure and Properties,* Butterworth, London, 1979, p. 566.
2 S.E. Urreta de Pereyra, H. Bertorello and A.A. Ghilarducci, *Phys. Status Solidi A, 108* (1988) 577.
3 S.E. Urreta de Pereyra, E. Carreño Morelli and A.A. Ghilarducci, *Scr. Metall., 23* (1989) 1691.
4 T. Mori, M. Koda, R. Monzen and T. Mura, *Acta Metall., 31* (1983) 275.
5 F. Daroqui and C. Pampillo, *Bol. Acad. Nac. de Cienc., Córdoba, Argentina, 54* (1980) 259.
6 *ASTM Standard E 112,* 1974.
7 X. Zhou, R. Fougeres and A. Vincent, *Mater. Sci. Forum, 119–121* (1991) 371.
8 A.K. Busby, L. Edwards and J.W. Martin, *Mater. Sci. Technol., 2* (1986) 363.
9 G. Scharf and B. Grzemba, *Aluminium, 58* (7) (1982) 391.
10 R. Acuña and F. Daroqui, *Aluminium, 59* (1983) 598.