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# Study by mechanical spectroscopy of the dislocation substructure in an aluminium matrix composite reinforced by alumina fibres

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**Abstract**

The main contributions to the high temperature internal friction spectrum of aluminium composites are found to originate in Al matrix dislocations. Following internal friction results, it is concluded that the reinforcement: (a) acts as a substructure size controller, improving its resistance to coarsening; (b) leads to a heterogeneous substructure where tangled dislocations (near the fibres) coexist with dislocations in more relaxed configurations; (c) generates internal stresses that affect dislocation mobility.

## 1. Introduction

The mechanical loss spectrum of aluminium between 300 K and 800 K, at frequencies of about 1 Hz, may be generally described by two relaxation maxima superimposed on a background increasing exponentially with increasing temperature. These three contributions are shown to originate in relaxation mechanisms involving thermally activated dislocation motion.

The lower-temperature maximum $M_1$ located at (0.4–0.5) $T_m$ (where $T_m$ denotes the melting temperature) is found in samples exhibiting intragranular dislocations in non-relaxed configurations (tangles) [1]. It disappears after annealing at 700 K in coincidence with substructure evolution towards coarser, more relaxed, states. The origin of this maximum has been proposed to be the stress-induced dislocation motion controlled by the dragging of point defects.

The higher-temperature maximum $M_2$ is found at about (0.6–0.7) $T_m$. It is agreed that $M_2$ originates in dislocation relaxation but, while some authors propose that intragranular dislocations are responsible for the relaxation [2], others associate the maximum with a mechanism involving dislocations in the grain boundary zone [3].

In previous work [4] it was found that Al matrix composites reinforced by short non-oriented fibres exhibit internal damping spectra characterized by maxima in roughly the same temperature ranges as those found in monolithic Al. It was concluded that between 300 K and 800 K the main contribution to dissipation arises in the A1 matrix, *i.e.* involve matrix defects.

Nevertheless, the spectra of the composites showed new features associated with the presence of the fibres such as a lower damping level in the whole temperature range, a large hysteresis (damping measured during heating is different from that measured during cooling) and a strong dependence on the rate of temperature change ($\dot{T}$ effect).

The fact that the main contributions to the composite spectrum originate in Al matrix dislocations allows us to study the characteristics of such dislocation substructure in the presence of fibres by internal friction measurements. From the modifications introduced by fibres to the different relaxations observed, information about dislocation mobility in the confined highly stressed matrix between fibres may be obtained.

In this paper, results concerning the effect of an increasing volume fraction of fibres $V_f$ on the shear modulus $G$ and on the internal friction (IF) spectrum of Saffil Al composites are presented. Special attention is paid to the effect of the fibres on the substructure coarsening during annealings.

## 2. Experimental procedures

Composite samples containing 0.1, 0.2 and 0.3 volume fraction of alumina (Saffil) fibres, produced by squeeze casting (SQC), were provided by Alusuisse-Lonza AG. The matrix composition is 99.998 wt.% Al with Si (0.0014 wt.%), Mg (0.0010 wt.%) and Zn (0.0019 wt.%) as principal impurities. Monolithic aluminium samples obtained by the same processes as the composites were also provided. After infiltration at 1050 K, samples were slow cooled to room temperature and machined to cylinders 2 mm in diameter and 55 mm in length.


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IF measurements were made as a function of the temperature, in an inverted torsion pendulum, by the method of free oscillation decay. The strain amplitude was $\epsilon = 5\times10^{-5}$, and heating or cooling rates were 2 K $\mathrm{min}^{-1}$.

Three annealing treatments were selected in order to investigate the evolution of the IF spectrum. They were performed *in situ* in the pendulum in the following sequence: (i) the as-machined specimen was fast cooled to 100 K, (ii) heated to 600 K, annealed for 30 min at 600 K and cooled to 100 K, (iii) heated to 700 K, annealed for 3 h at 700 K and cooled to 100 K, (iv) heated to 800 K, annealed for 3 h at 800 K and cooled to 100 K. After annealings (iii) and (iv) the reproducibility of the resulting spectra was investigated by performing five cycles between 100 K and the respective annealing temperature. In both cases, the resultant spectra were highly reproducible.

## 3. Results

Figure 1 shows the IF spectrum of monolithic Al measured at $f_{RT} = 2$ Hz. During the first heating a maximum appears at 400 K ($M_1$); during subsequent cooling after 30 min at 600 K another maximum is found at 520 K ($M_2$) while $M_1$ has strongly attenuated. After 30 min at 700 K only $M_2$ may be detected. This maximum shows an increased height and has shifted to 560 K. From these results, it may be concluded that in monolithic Al the annealing imposed on the specimen promotes the evolution of the dislocation substructure from tangled to more relaxed configurations, having longer mean free loop lengths.

Figure 2 shows the IF reproducible spectrum and modulus $G$ for the SQC 20% composite, measured after 3 h at 800 K, at $f_{RT} = 2.3$ Hz. Their main features are: (a) a marked heating–cooling hysteresis in damping and in modulus, and (b) that both IF maxima ($M_1$ at 460 K and $M_2$ at about 600 K) are simultaneously observed even after high temperature annealing. These results indicate that a certain proportion of non-relaxed dislocations remains in the matrix even after high temperature annealing and cooling.

This improved stability of the maximum $M_1$ was further investigated. Figure 3 shows the evolution of $M_1$ in an SQC 10% ($f_{RT} = 2.2$ Hz) composite during progressive annealing. It may be observed that $M_1$ first develops during annealing at 700 K and then its height reduces after 3 h at 800 K. Changes in peak temperature are difficult to establish. This behaviour is found in all the composites studied.

Figure 4 shows the spectra measured during cooling after 3 h at 800 K in composite specimens containing different volume fractions of fibres. As $V_f$ increases, the maxima $M_1$ and $M_2$ shift to lower temperatures and decrease in height. This behaviour is also found after annealing for 3 h at 700 K.

Finally, Fig. 5 shows the composite shear modulus $G$ as a function of volume fraction of fibres. A linear increase in modulus with increasing $V_f$ is found. For comparison, the values predicted by the mean-field model [5] are also plotted.

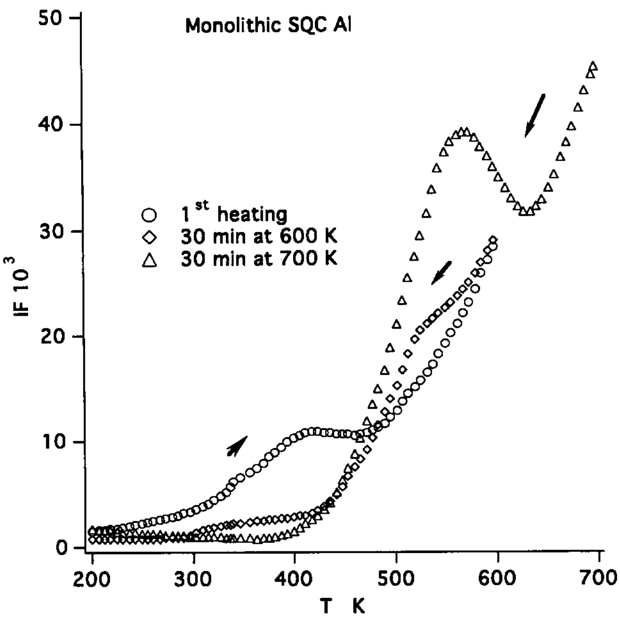


Fig. 1. Effect of annealing on IF in SQC monolithic Al. After 30 min at 700 K $M_1$ originating in tangled dislocations is not observable.

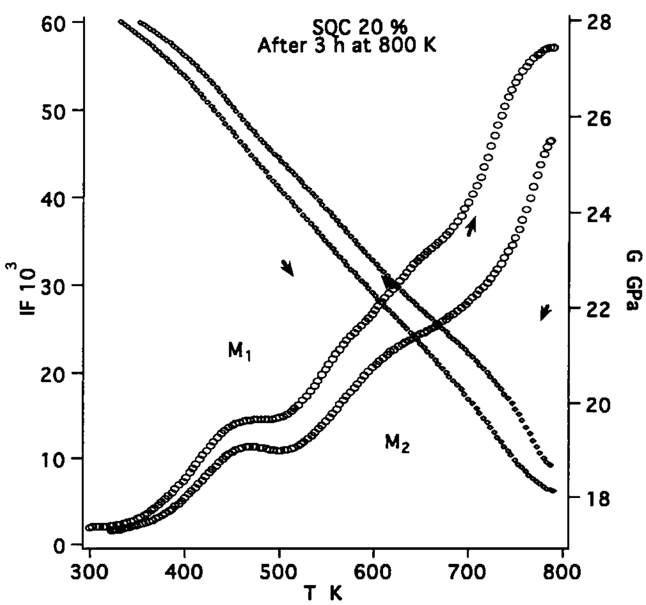


Fig. 2. Reproducible IF and modulus $G$ in an SQC 20% composite after 3 h at 800 K. Both local maxima $M_1$ and $M_2$ are observable.

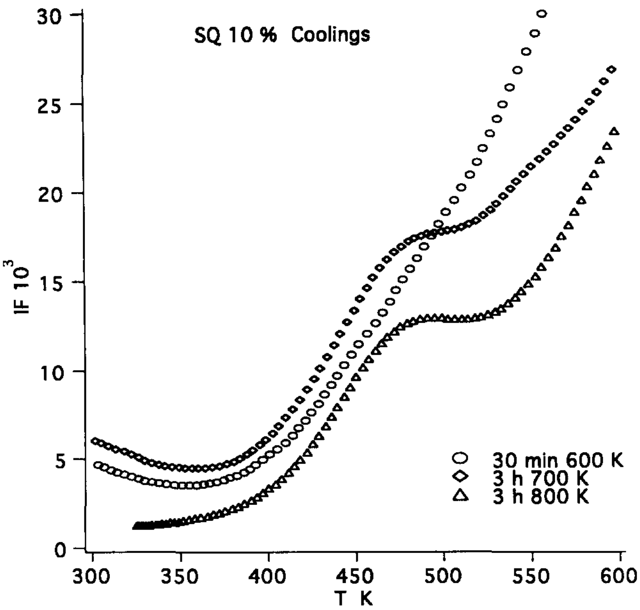


Fig. 3. Effect of progressive annealing on $M_1$ detected in an SQC 10% composite. Its height first increases and then decreases.

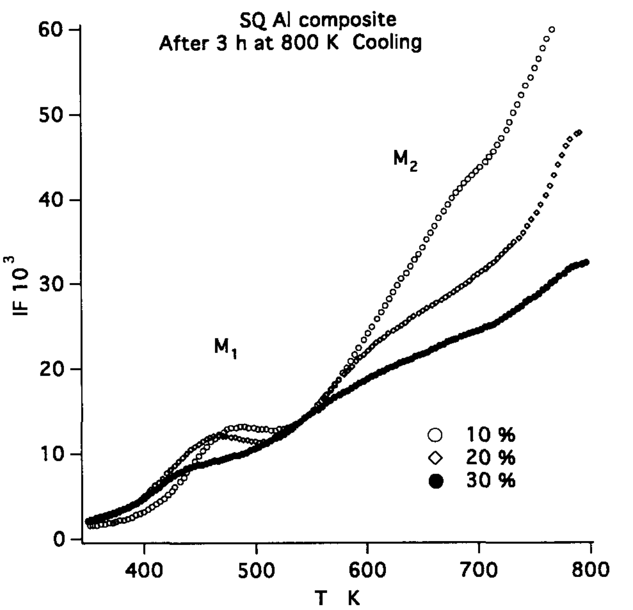


Fig. 4. Effect of fibres on the IF composite spectrum. IF maxima attenuate and shift to lower temperatures as $V_f$ increases.

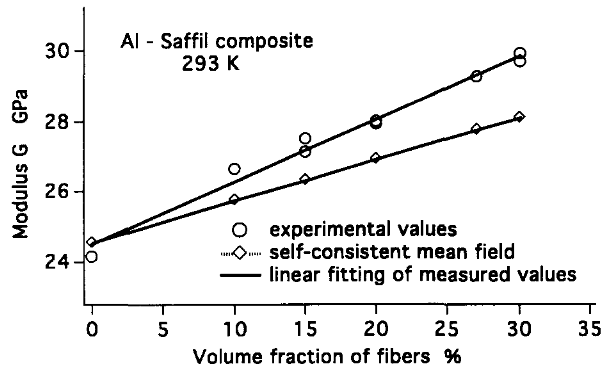


Fig. 5. Modulus $G$ as a function of fibre content in SQC as-received composites, measured at room temperature (293 K) during the first heating.

## 4. Discussion and conclusions

The results described before confirm that, to a first approximation, the internal friction spectrum of the studied composite may be considered as a matrix (Al) spectrum, modified by the presence of fibres.

The modifications introduced by fibres to the matrix spectrum may be summarized as follows: (a) lower IF levels after high temperature annealing; (b) large and reproducible heating–cooling hysteresis; (c) a stronger $\dot{T}$ effect, particularly above 400 K; (d) coexistence of both relaxation peaks, even after high temperature annealing; and (e) changes in the internal friction peak parameters such as peak heights, temperatures and/or apparent activation energies.

The first modification (a) may be explained by considering the reinforcement as a three-dimensional array of obstacles controlling substructure size, leading to a smaller dislocation mean loop length, and improving resistance to coarsening.

Modifications (b), (c) and (d) may be considered as having a common origin. As a consequence of the different values of coefficient of thermal expansion between matrix and fibres, large internal stresses $\sigma_{th}$ develop in the matrix when the temperature changes. Depending on their level and on the temperature such internal stresses may be partly relaxed by different mechanisms [6]. At high temperature and low $\sigma_{th}$ diffusional and dislocation creep are likely to occur while at low temperature and high internal stress plastic relaxation takes place, *i.e.* dislocations are generated in the matrix near the fibres.

In our experience, the IF measurement imposes on the specimen a cycle with thermal amplitude large enough to induce local values of $\sigma_{th}$ close to the matrix yield strength $\sigma_y$ and dislocation generation becomes competitive as a stress-relaxation mechanism. This is likely to occur during cooling below 400 K. On the other hand, in the high temperature part of the cycle (above 400 K) dislocation creep becomes predominant.

In this way, not only is the fresh dislocation density not the same during cooling and heating, but the internal stress states are also different. This explains the observed hysteresis and the strong $\dot{T}$ effect detected.

Concerning the observation of the low-temperature peak $M_1$, even after high temperature annealing, it may also be explained by the presence of these thermal unrelaxed dislocations near fibres. The coexistence of both peaks after high temperature annealing suggests that in the composite the dislocation substructure is not homogeneous.

The observed changes in peak heights and temperatures as the volume fraction of fibres increases presents a more complex situation. Let us consider the maximum $M_1$ as originated in dislocation motion controlled by the dragging of point defects. In this case, the relaxation strength $\Delta$ and relaxation time $\tau$ are given by

$$\Delta \propto \Lambda L^2$$

$$\tau \propto L^2 \exp[(H-\sigma v)/kT]$$

where $L$ is the mean loop length between hard pinning points, $\Lambda$ is the mobile dislocation density, $H$ is the activation energy at $\sigma = 0$ and $v$ is the activation volume. In the equation for $\tau$, $\sigma$ is an effective stress acting on the dislocation line which may be considered as the result of the applied stress $\sigma_a$ and the thermal stress, proportional to the volume fraction of fibres [6]:

$$\sigma = \sigma_a + \sigma_{th} = \sigma_a + \chi V_f$$

Then the apparent activation energy depends on $V_f$ and so on peak temperature. The simultaneous decrease in peak height and temperature as $V_f$ increases suggests that fibres also affect the mean dislocation loop length $L$ in non-relaxed configurations. It may be concluded that changes in the peak parameters are the result of both thermal stresses and substructure modifications introduced by fibres.

Further investigation of the dependence of the parameters of this IF peak on the volumetric fraction of fibres will provide a more complete description of the process of generation, movement and reorganization of thermal dislocations in the regions close to the fibres.

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