

# Internal friction due to G–P zones in pure Al–16 wt % Ag and Al–16 wt % Ag–0.2 wt % Fe–0.1 wt % Si alloys

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A study has been made of an internal friction peak which occurs due to the precipitation of Guinier–Preston zones from two supersaturated Al–Ag alloys. The peak observed was interpreted as being due to relaxation around the zones of different elastic constants from that of the Al matrix. Differences in internal friction characteristics between specimens heated at 100 and 200 °C were attributed to the reversion process occurring around 200 °C in pure alloy. The height of the relaxation peak and the level of internal friction background were found to be highly affected by the presence of Fe and Si impurities in the doped alloy.

## 1. Introduction

The precipitation processes in aluminium-rich Al–Ag alloys has been extensively investigated and the basic precipitation sequence has been established [1] to be spherical Guinier–Preston (G–P) zones (Ag-rich particles, possibly ordered) [2] →  $\gamma'$  plates (the metastable form of the hexagonal close packed  $\xi$ -AlAg<sub>2</sub> phase) [3] →  $\gamma$  (the stable form of the  $\xi$  phase formed by continuous precipitation) [4]. Reversion of some G–P zones during heating at temperatures where nucleation of  $\gamma'$  plates occurs, has been also reported by some authors [1, 5–7].

Measurements of microstructure-sensitive properties such as microhardness, electrical resistivity and internal friction (IF) were frequently used to trace the precipitation sequence in Al–Ag alloys. The apparent disagreement of results concerning the transformation stage, G–P zones →  $\gamma'$  precipitates, may explain the difficulty of accounting for the IF evolution during precipitation in this system. Moreover, explanations of the origin of the anelastic behaviour seems to be contradictory. Schaller and Benoit [7] explained the origin of the IF peaks they found in some Al–Ag alloys by considering the Zener relaxation (the stress-induced orientation of elastic dipoles consisting of point defect configurations) to be the only mechanism responsible for the anelastic behaviour observed. Accordingly, they claimed that the model of Schoeck and Bisogni [8, 9], based on relaxation at precipitate interfaces, should be refuted. On the other hand, the work of Monzen *et al.* [10], Mori *et al.* [11] and Okabe *et al.* [12] gave theoretical and experimental evidence for interfacial relaxation caused by diffusion around a second-phase particle. Because, in an Al–Ag alloy, the number and size of G–P zones can be easily governed through appropriate heat treatment, IF peaks related to the formation, growth and transformation of these zones were thought to provide other possibilities for the interpretation of the relaxa-

tion phenomenon during the precipitation processes. Hence, the present work was undertaken to shed more light on the origin of the anelastic behaviour in this alloy.

The effect of small additions of other elements to the binary alloy, on the relaxation phenomena has also been investigated using an Al–Ag alloy containing small amounts of Fe and Si impurities.

## 2. Experimental procedure

Two Al–4.5 at % (16 wt %)–Ag alloys were used in the present investigation. One was made from high-purity Al (99.995%), while the other was made from commercial Al containing mainly Fe and Si as impurities. High-purity (99.99) Ag was used to prepare both alloys by melting in air. The ingots were given a homogenizing vacuum anneal for 3 days at 500 °C. Analytical examination showed that the pure Al–Ag alloy had 16 wt % Ag with traces of Cu, Mg and Mn, while the doped alloy had the following composition (wt %): Ag 16.0, Fe 0.20, Si 0.10, Mg 0.013, Zn 0.006, Ti 0.005, Mn 0.004, Cu 0.003.

The homogenized alloys were either drawn in the form of wires 0.5 mm diameter for IF experiments, or cold-rolled into 0.2 mm thick foils to be used for electron microscope (EM) investigation. Both wire and foil specimens were solution heat treated in vacuum ( $10^{-5}$  torr; 1 torr =  $1.333 \times 10^2$  Pa) for a standard time of 4 h at  $T_s = 520$  °C and then water quenched at 25 °C. Some of the quenched specimens were subsequently heated at precipitation temperatures of 100 and 200 °C for precipitation times of 5, 25 and 60 min. Internal friction measurements were carried out using a torsional pendulum with electromagnetic excitation and optical read-out. The frequency of vibrations was  $\sim 0.8$  Hz and the internal friction was proved to be amplitude independent up to a maximum strain amplitude of  $2 \times 10^{-4}$ . For EM

examination, thin foils were prepared by the window technique using a cold (approximately  $-10^{\circ}\text{C}$ ) electrolyte composed of 15% perchloric acid, 15% glycerol and 70% ethyl alcohol. A Zeiss microscope, type EM10, operating at 80 kV was used.

### 3. Results

Internal friction,  $Q^{-1}$ , was measured isochronally as a function of temperature in the range  $25\text{--}400^{\circ}\text{C}$  every  $20^{\circ}\text{C}$  at a constant frequency of 0.8 Hz at a constant heating rate of  $2^{\circ}\text{C min}^{-1}$ .

#### 3.1. "Pure" Al-16% Ag alloy

The evolution of IF of this alloy obtained during isochronal treatment is shown in Fig. 1 as plots of  $\Delta Q^{-1}/Q_0^{-1} = (Q_T^{-1} - Q_0^{-1})/Q_0^{-1}$  against temperature for wire specimens of different degrees of precipitation.  $Q_T^{-1}$  is the value of IF at temperature,  $T(^{\circ}\text{C})$  whereas  $Q_0^{-1}$  its value at room temperature. From Fig. 1 it is clear that:

1. general features of IF curves are common, they all increase with the increasing temperature, then a peak is observed followed by the high-temperature IF increase;
2. the IF background of the specimens heated at 100 and  $200^{\circ}\text{C}$  clearly showed a significant separation

between them. For both specimens the background showed an increase with increasing precipitation time;

3. the dependence of the peak height on precipitation time is given in Fig. 2. For all precipitation times, the IF peak heights for specimens heated at  $100^{\circ}\text{C}$  were found to be larger than that of specimens heated at  $200^{\circ}\text{C}$ . While the increase in precipitation time has no effect on peak height of specimens heated at  $200^{\circ}\text{C}$ , specimens heated at  $100^{\circ}\text{C}$  showed an increase in peak height with increasing precipitation time.

#### 3.2. Al-16% Ag-0.2% Fe-0.1% Si alloy

The effect of Fe and Si addition on the precipitation process could be predicted by comparing the results of IF measurements of specimens of the doped alloy obtained under the same conditions with those of the "pure" Al-Ag alloy. Fig. 3 shows the behaviour of IF of specimens heated at 100 and  $200^{\circ}\text{C}$ . Although the general behaviour was found to be similar to that of "pure" alloy, a very steep increase in damping was noticed. Even so, as in the case of "pure" alloy, the peak height of specimens heated at  $100^{\circ}\text{C}$  showed generally higher values for all precipitation times (Fig. 4). However, the difference in peak heights between the two types of specimen was not as large as in the case of the "pure" alloy (see Fig. 2). A slight shift in

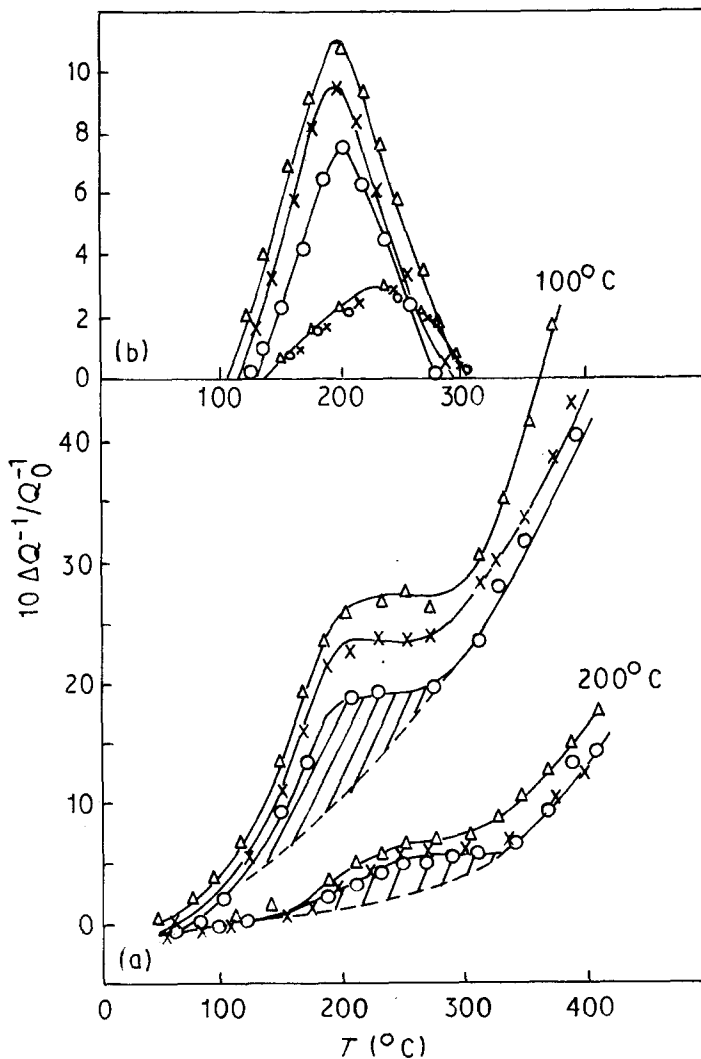


Figure 1 (a) Variation of internal friction with temperature of "pure" Al-Ag alloy for specimens heated at 100 and  $200^{\circ}\text{C}$  for precipitation times: (○) 5, (×) 25, (△) 60 min. (b) Background-subtracted peaks of the curves shown in (a).

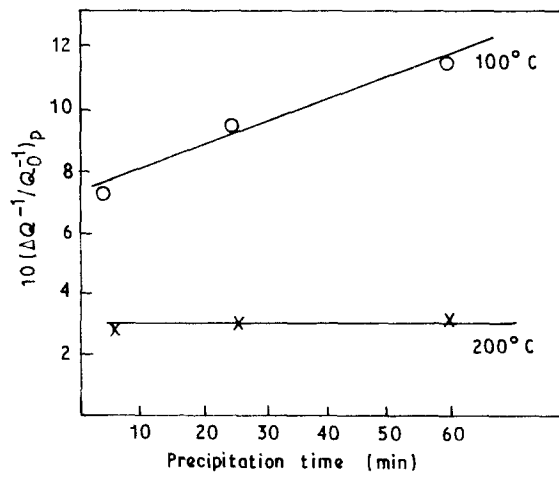


Figure 2 Variation of the peak height with precipitation time of specimens of the "pure" Al-Ag alloy heated at 100 and 200°C.

peak position towards lower temperature was obtained in specimens heated at 100°C with increasing precipitation time; such a variation was not observed for specimens heated at 200°C. Results of high-temperature damping indicated, as in the case of "pure" alloy, that specimens heated at 100°C were characterized by a higher level of IF background, compared to those heated at 200°C. For specimens heated at 100°C, this background IF however, was

found to be less sensitive to variation in precipitation time. On the other hand, specimens heated at 200°C showed not only a stronger dependence on precipitation time but also a decrease in IF background as a result of the increase in precipitation time.

#### 4. Discussion

##### 4.1. "Pure" binary Al-Ag alloy

##### 4.1.1. Relaxation caused by diffusion around large G-P zones

Electron micrographs taken from our specimens showed that heating alloy specimens at 100°C for different lengths of time gave rise to large G-P zones (Fig. 5a). Hence, the peak observed at ~200°C is thought to be related to the grown G-P zones and not to a supersaturated solid solution surrounding the zones as has already been reported [6]. Because the peak height increased on heating at 100°C for longer times (see Fig. 2), it is reasonable to consider this peak as being due to anelastic effects caused by diffusion around the Ag-rich particles (i.e. zones). This might be true in view of the fact that larger particles correspond to relatively longer times of precipitation at 100°C (assuming in this case that the number of zones remains unchanged). In view of a largely depleted matrix (as a result of the formation of large-sized and/or large number of zones), the observed peak cannot be

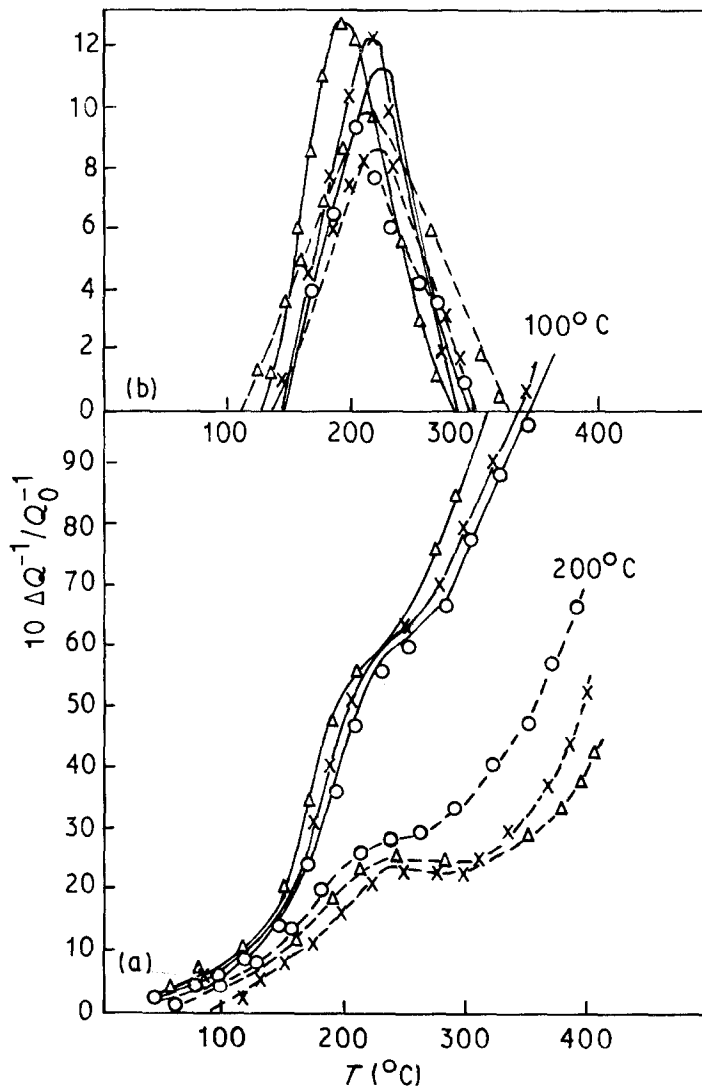


Figure 3 (a) Variation of internal friction with temperature of doped Al-Ag alloy for specimens heated at 100 and 200°C for precipitation times: (○) 5, (×) 25, (△) 60 min. (b) Background-subtracted peaks of the curves shown in (a).

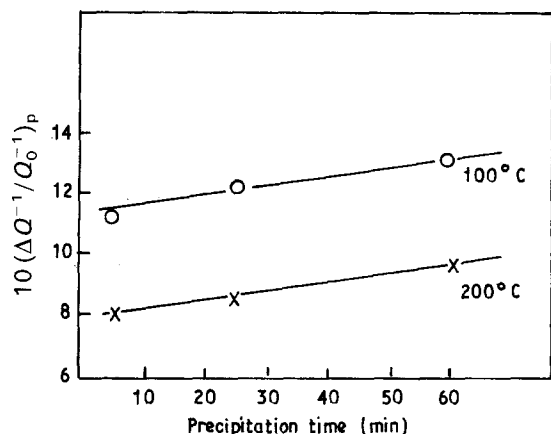


Figure 4 Variation of the peak height with precipitation time of specimens of the doped Al-Ag alloy heated at 100 and 200 °C.

ascribed to a Zener relaxation caused by enriched solid solution as has been claimed previously [6, 7, 13]. The Zener relaxation is attributed to local ordering in the solid solution induced by the applied stress. The occurrence of this effect in a homogenous solid solution apparently requires a substantial difference in the size of the solvent and solute atoms [14], and a large concentration of solute (of the order of 10 at %). In view of the fact that in the Al-Ag alloy the difference in atomic radii of Al and Ag is only 1% and due to the relatively low concentration used (4.5 at %), it is reasonable to conclude that the observed peaks in the present work are not Zener relaxation peaks.

Based on the concept of an elastic isotropic continuum, Eshelby [15, 16] and Schoeck [8] considered the case of coherent particles present in a matrix with different elastic constants. They showed that the interaction energy responsible for the evolution of IF peaks is proportional to the volume fraction of the particles. If G-P Zones are considered as inclusions present in the Al matrix in the coherent state and composed mainly of Ag atoms, then the presently observed IF peaks might occur through a diffusion-controlled change of volume and shape of the inclusions with a resulting change in interaction energy.

#### 4.1.2. Relaxation caused by diffusion around small G-P zones

The reversion of zones and the associated nucleation of precipitates are considered here. Partial reversion of G-P zones previously formed in specimens aged at moderately high temperature is known [6, 7, 17, 18] to occur around 200 °C for "pure" Al-Ag alloys and in the vicinity of  $\gamma'$  nuclei. Reversion of G-P zones and the nucleation of  $\gamma'$  precipitates are two processes which occur nearly simultaneously. Thus a number of small platelets of  $\gamma'$  precipitates were observed along with the partially reverted zones, (Fig. 5b). The lower volume fraction of G-P zones obtained under the reversion conditions was reflected in a noticeably smaller peak height.

Considering the relation  $\tau = \tau_0 e^{E/kT_p}$ , the shift in peak position,  $T_p$ , towards higher temperature (from  $\sim 200$  to  $\sim 250$  °C, see Fig. 1) was found to be consistent with the interfacial relaxation theory [8-12] which predicted a relaxation time,  $\tau$ , proportional to the third power of particle size. Accordingly, partially reverted zones of smaller size caused a shift in peak position towards a higher temperature.

In view of the lower relaxation strength and the shift towards higher  $T_p$ , as observed for specimens heated at 200 °C, it is less probable that the peak obtained at  $\sim 250$  °C is due to relaxation caused by diffusion around  $\gamma'$  platelets.

#### 4.2. Binary alloy with Fe and Si (doped Al-Ag alloy): effect of Fe and Si impurities on relaxation

The noticeable increase in IF of this alloy (see Fig. 3), could be simply related to the existence of Fe and Si impurities and their effect on the nucleation of the G-P zones which form the relaxation units in action. The apparent activation energy for vacancy formation is known [19] to be greatly reduced by small additions of impurities. Thus, during quench and subsequent heating, and due to binding [20] between vacancies

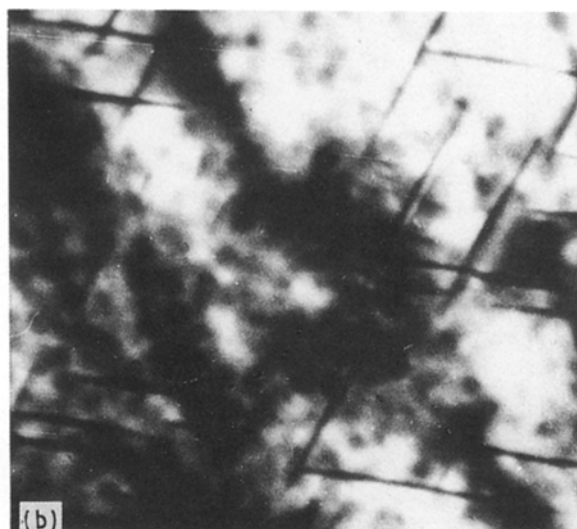
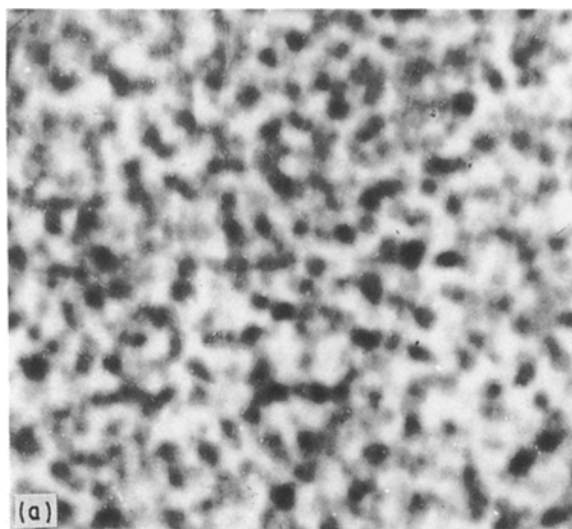


Figure 5 Electron micrograph of "pure" Al-Ag specimens heated for 1 h at: (a) 100 °C, showing large G-P zones, and (b) 200 °C, showing reversion of zones along with the formation of  $\gamma'$  platelets,  $\times 150000$ .

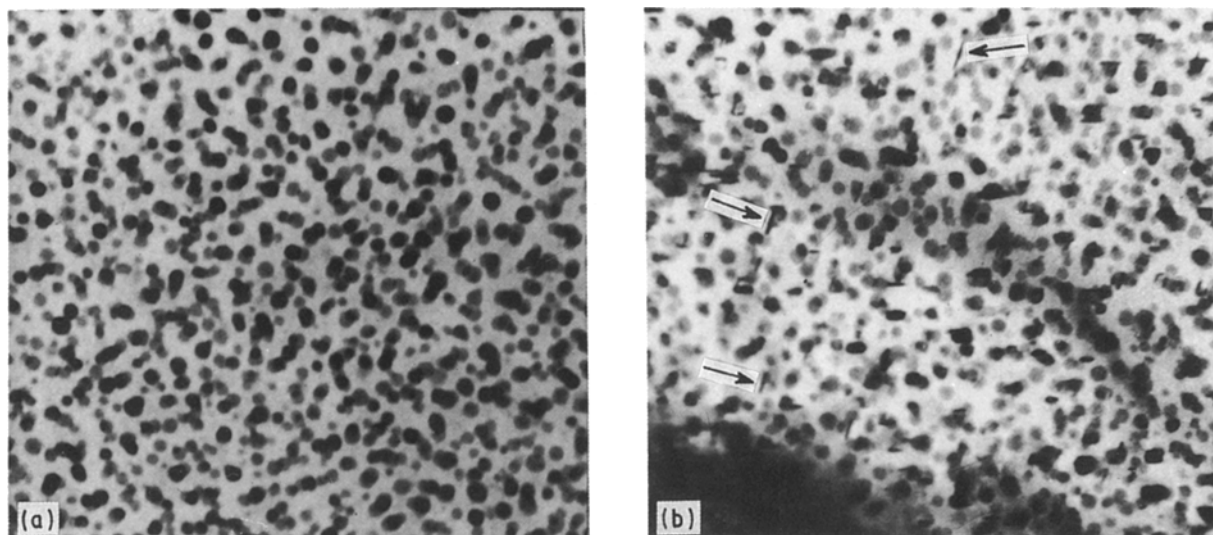


Figure 6 Electron micrograph of doped Al-Ag specimens heated for 1 h at: (a) 100 °C, showing G-P zones, and (b) 200 °C, showing the mostly stabilized zones, along with the formation of few minute  $\gamma'$  platelets (arrowed),  $\times 130\,000$ .

and both Fe and Si solutes, vacancy-impurity clusters are likely to be formed. These clusters might act as nucleation sites for the formation of a large number of small zones (Fig. 6a) leading to higher IF peaks. Consistent with the observations obtained for the "pure" alloy, the changes in peak height (Fig. 4) indicated that the volume fraction of zones increased with time of precipitation.

Unlike specimens of "pure" alloy, electron microscope investigation of specimens heated at 200 °C before IF measurement (Fig. 6b) showed that reversion of G-P zones was mostly inhibited in the doped alloy, and only few very small  $\gamma'$  platelets formed. Such inhibition of zone reversion may be considered as a growth stabilization that eventually resulted in zones of volume fraction which were not very much less than that obtained in specimens heated at 100 °C. In other words, when G-P zones in the doped alloy were mostly stabilized, the peak height was moderately lowered, while in the "pure" alloy where zone reversion took place, lowering of the peak height was striking. This may demonstrate evidence for the possibility of obtaining relaxation peaks due to diffusion around G-P zones.

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