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Internal friction sensitivity to precipitation in Al-12 wt% Mg alloy

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ABSTRACT

In order to improve the understanding of the sensibility of the internal friction phenomena to different stages of precipitation in Al-Mg alloys, the ageing effect of a supersaturated solid solution on the Temperature Dependent Internal Friction (TDIF) spectra has been studied. The research showed that the dissolution of the β phase and the transfer of Mg atoms to a solid solution and the formation of β′ metastable phase have an effect on internal friction. Thus, it was proved that the precipitation or the dissolution of β phase plays an important role in the anelasticity of Al-Mg alloys. A detailed TDIF analysis revealed the sensibility of the internal friction technique to the formation of semi-coherent and non-coherent phases (β′ and β) but it is not sensitive enough to the formation of coherent phases (GP zones). Several observations on the nature and the shape of the precipitates are also discussed.

1. Introduction

A continuous precipitation occurs in Al-Mg alloys and has an important influence on physical and mechanical alloy properties. The comprehension of all these phenomena is really complicated because mechanisms and kinetics of these transformations result from various internal and external causes rather than one factor. The ageing behavior of supersaturated solution has been extensively studied and a four-stage process is reported as follows [1–3]:

sss α → GP zones → β″ → β′ → β

where sss α is the supersaturated solid solution, GP is Guinier-Preston zones (also indicated as δ″), β″ (so-called GP zones 2 or ordered GP zones) is an $L1_2$ ordered coherent phase (composition Al_3Mg), β′ is a semi-coherent hexagonal (a = 1.002 nm, c = 1.636 nm) intermediate phase (composition Al_3Mg_2), and β is the non-coherent stable phase (Al_3Mg_2) having a complex f.c.c structure (a = 2.824 nm).

Several studies show the effectiveness of different experimental techniques to follow precipitation reactions [3–18].The study of internal friction during ageing of Al-5% Mg and Al-8% Mg alloys showed the existence of a damping peak due to the presence of the β′ semi-coherent phase precipitates [4]. For the first time, the study of Al-8 wt% Mg and Cu-15 wt% In alloys shows similarity between continuous and discontinuous precipitations: an interesting lattice parameter variation was found; it contains characteristics of both precipitations: the continuous variation and the simultaneous existence of two lattice parameters during a certain ageing time in the same alloy [5]. Precipitation sequence and changes in micro-hardness measurements of Al-8 wt% Mg and Al-12 wt% Mg alloys have been investigated by differential scanning calorimetry (DSC) and other techniques (as microhardness measurements) [6]. The scan has shown that the hardening is caused by the formation of intermediate particles (Al_3Mg_2) that precipitate at temperatures above the reversion temperature of GP zones. The decomposition of the Al-Mg supersaturated solid solution is a four-stage process and Transmission electron microscopy (TEM) examinations show the presence of GP zones, β′, and β particles of different morphologies and confirm the DSC investigations in Al-8 wt% Mg alloy [7]. The study of the effect of prior cold work on the precipitation, recrystallization reactions, and phase transformation of an Al-12 wt% Mg alloy, has shown the difficulty of following the interaction between precipitation and recrystallization reactions and their reciprocal effects in such a supersaturated solid solution [8]. It was shown that the contribution of β′ and/or β phases to the expansion of the matrix is more important than the contraction due to the diminution of the lattice parameter [9].

It is well known that the IF method can be very sensitive to some structural transitions [10]. In the case of hardening alloys, three internal friction sources come into consideration: the movement of dislocations and their possible interactions with other defects, the movement or reorganization of point defects in solid solution, and non-elastic effects due to precipitation [11]. Nilson [12] observed on Al-Mg alloys (at low frequency ~ 1 Hz) a stable peak at about 150 °C and an instable one at a lower temperature. Belson et al. [13] showed that this peak is not due to the β phase precipitation. Dey and Quader [14] remarked on annealed Al-7.5 wt% Mg alloy that the IF peak did not appear clearly.

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Moreover, after quenching and ageing at 150 °C, there are three peaks towards 40, 80, and 120 °C. A Zener peak (H = 1.09 eV and $\tau_0 \approx 10^{-14}$ s) was detected in Al-12 wt% Mg alloy after quenching [15]. According to these authors, the first peak corresponds to a relaxation phenomenon involving clusters of atoms in solution and the third one would be a Zener-type relaxation (i.e., of reorientation under stress of atoms in solution). According to Shoeck's theory [16], the coherent precipitates cannot be the cause of an internal friction peak; only the relaxations in the interfaces can cause an IF peak. It is demonstrated that the most probable mechanism of the influence of the β-phase precipitation on the relaxation strength is a decrease in the mobility of grain boundaries at a temperature below the solvus one [17]. Stress in a material lowers the energy barrier for the dislocation motion and also creates new dislocations [18]. In this paper we attempt to explain some unsettled questions related to the ageing effect on IF peaks in order to compare it with other experimental techniques and to explore the IF sensitivity to the formation of different phases.

2. Material and Methods

The Al-12 wt% Mg alloy is used for this study. Chemical analysis of this alloy shows an Mg content of 11.95 wt%. In order to obtain homogeneous supersaturated solid solutions, the samples were annealed at 430 °C for 17 h and quenched in iced water. Isothermal ageing of the supersaturated solid solutions at 150 and 250 °C under argon, were then carried out for different time periods to form β′ and β phases.

The Temperature Dependent Internal Friction (TDIF) was measured by two dynamic mechanical analyzers DMA1000 Metravib and DMA Q800 TA Instrument, both in mode of forced binding vibrations, on samples of 65 × 5 × 4 mm^3 at the heating rate of 10 °C/min (Metravib) and 2 °C/min (TA Instruments) and maximum deformation amplitudes $\varepsilon_0 = 1 \times 10^{-4}$ ($\varepsilon = \varepsilon_0\cos(\omega t + \varphi)$), where $\omega = 2\pi f$ is the circular frequency of vibrations; φ is the loss angle between the applied cyclic stress and the resulting deformation; and $\tan\varphi \cong Q^{-1}$ where Q^{-1} is the inverse quality factor or internal friction (IF), i.e. damping. All studied samples were tested at the same set of frequencies of forced vibrations of 1 and 10 Hz (Metravib) and 0.3, 1, 3, 10, and 30 Hz (TA Instrument).

DSC measurements were carried out under argon with a Setaram DSC 131. The samples were in a disc shape, 3 mm in diameter and 1.5 mm in thickness. The thermal cycle applied consisted of heating from room temperature to 430 °C with a heating rate of 10 °C/min.

The dilatometric measurements were performed and computer-controlled with a NETZSCH DIL 402 C differential dilatometer under a protective atmosphere of pure argon. The samples for these measurements were in parallelepiped shape of 25 × 5 × 5 mm^3. The thermal cycle applied consisted of heating from room temperature to 430 °C with a heating rate of 10 °C/min.

All the experiments were repeated twice to confirm the obtained results.

The microstructure of this alloy was investigated using a scanning electron microscopy (SEM). After different heat treatments, the samples were prepared for a scanning electronic microscopy examination. The SEM observations were performed by Jeol JSM-6700F field emission (FEG) equipped with an EDS with a working voltage of 0.5 kV to 30 kV.

3. Results

3.1. Mechanical Spectroscopy Analysis

The IF behavior of the quenched Al-12 wt% Mg alloy is presented in Fig. 1, which shows similar curves shape for two frequencies (1 and 10 Hz) of forced bending vibrations: the presence of a background damping and a peak-like effect at about 385 °C. For measuring frequency of 10 Hz the damping background is lower because it is thermally activated and therefore shifted to higher temperature. The peak-like effect at about 385 °C is the result of dissolution of precipitated particles, which leads to easier grain boundary (GB) sliding and becomes possible only when a dissolution process is finished. The peak shape and consequently its height, earlier observed in the Al-Mg alloys with different Mg content [4,9,14,17], depends on frequency. Belson et al. [13] attributed the IF peak (H = 1.4 eV and $\tau_0 = 10^{-17}$ s) in alloys with Mg contents of up to 12% to the reorientation of substitutional atom pairs in solid solution, that is, to the mechanism of Zener relaxation. Until 200 °C, the IF spectrum reveals nothing particular while between 200 and 400 °C, when the main part of the transition effects take place, in particular the precipitation of the β′ and β phases, some effects appear. At about 200 °C one can observe a significant increase in the dislocation mobility accompanied with a maximum at about 385 °C. The temperature position of the 385 °C IF- peak practically does not depend on measuring frequency underlying the fact that this effect is rather due to a certain structural transition. As discussed above, this structural transition is dissolution of β phase. In turn it leads to unlocking of GB sliding which is thermally activated relaxation effect. Consequently, above 385 °C we can observe in the experimental curves the remaining high-temperature part of the GB relaxation while

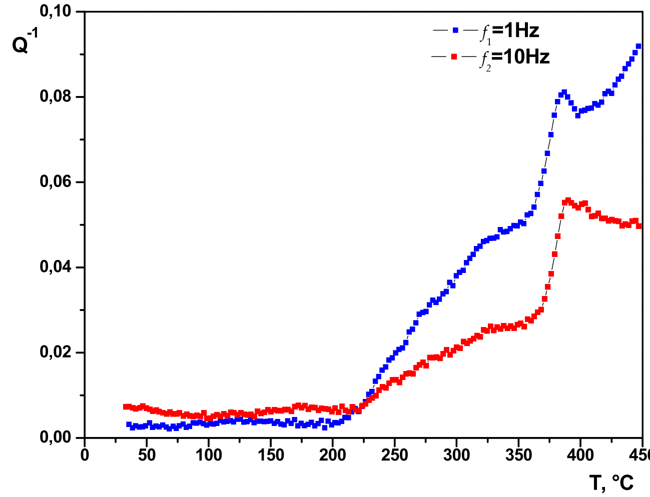

Fig. 1. The temperature dependence of internal friction (Q^{-1}) of water quenched from 430 °C Al-12 wt% Mg specimen for frequencies 1 and 10 Hz, heating rate 10 °C/min, and maximum amplitude of deformation of 1 × 10^{-4}.

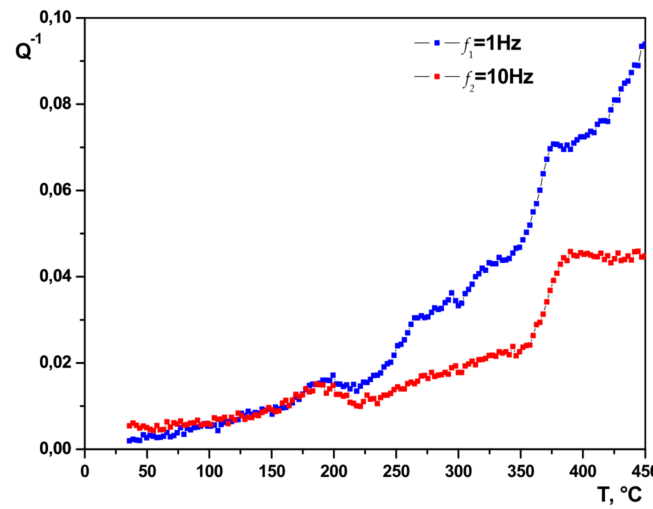

Fig. 2. The temperature dependence of internal friction (Q^{-1}) of water quenched from 430 °C and aged at 150 °C for 25 h Al-12 wt% Mg alloy for frequencies 1 and 10 Hz, heating rate 10 °C/min, and maximum amplitude of deformation of 1 × 10^{-4}.