

Internal friction in Mg-4Li alloy

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Abstract

Anelastic behaviour of the Mg-4wt.%Li has been studied using acoustic methods. Strain dependence of the logarithmic decrement was measured at room temperature after the thermal treatment of samples. Activation volume for dislocation motion has been estimated depending on the annealing temperature. Temperature relaxation spectrum of the internal friction was measured in the temperature interval from room temperature up to 380 °C at three frequencies in the multi-frequentional mode. Two internal friction peaks appeared in the first heating sub-circuit. While the low temperature peak vanished during the first cooling, the high temperature peak remained stable during three heating cooling loops. The high temperature peak has been ascribed to the grain boundary sliding.

Key words: magnesium alloy, damping, internal friction, dislocation motion, grain boundary sliding

1. Introduction

Magnesium-lithium alloys are the lightest metallic structural materials. Pure magnesium and some magnesium alloys are well known as damping materials [1], material damping [2] using magnesium alloys will be a prominent method to control vibration and noise in vehicles and instruments. It is inferred that the damping properties of a material depend on the material microstructure [3]. Magnesium-lithium alloys have relatively bad mechanical properties. One possibility how to improve the mechanical properties is Hall-Petch strengthening, i.e. preparation of the material with the small grain sizes. In general, the strength of magnesium-lithium alloys rapidly decreases at elevated temperatures. The primary factor is recognised to be related to the occurrence of grain boundary sliding. Dislocations and grain boundary sliding are very effective sources of the internal friction. Such study can help to develop the creep resistant alloys in which the grain boundary sliding will be restricted [4, 5], or superplastic alloys with the easy grain boundary sliding [6, 7].

1.1. Dislocation damping

If a material containing dislocations is submitted to a harmonic applied stress $\sigma = \sigma_0 \sin \omega t$ with an angular frequency $\omega = 2\pi f$, one can define the mechanical loss factor η as

$$\eta = \frac{1}{2\pi} \frac{\Delta W_{\text{diss}}}{W_{\text{max}}}, \quad (1)$$

where ΔW_{diss} is the mechanical energy dissipated in one cycle of the applied stress, and W_{max} is the maximum mechanical energy stored on it. In the case of an anelastic dislocation strain ε_d

$$\Delta W_{\text{diss}} = \oint \varepsilon_d d\sigma. \quad (2)$$

The maximum stored energy can be well approximated by the maximum elastic stored energy

$$W_{\text{max}} = \int_0^{\sigma_0} \sigma d\varepsilon_{\text{el}} = \frac{1}{2} J_{\text{el}} \sigma_0^2, \quad (3)$$

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where J_{el} is the elastic compliance related to the shear modulus $G^{-1} = J_{el}$. The mechanical loss factor is

$$\eta = \frac{1}{\pi J_{el} \sigma_0^2} \oint \varepsilon_d d\sigma. \quad (4)$$

The logarithmic decrement, δ , as another damping quantity, is given as

$$\delta = \frac{1}{n} \ln \frac{A_i}{A_{i+n}}, \quad (5)$$

where A_i and A_{i+n} are the amplitudes of the i -th cycle and $(i + n)$ -th cycle, respectively, separated by n periods of the free vibrations of the specimen. Between these damping quantities the following relationship holds $\pi\eta = \delta$ (for small δ).

2. Experimental

Extruded Mg12Li alloy (nominal concentration 12 at.%, i.e. 3.7 wt.% of Li) was used in this study. Damping measurements were carried out on bending beams (80 mm long with thickness of 3 mm) in vacuum (about 30 Pa) at ambient temperature. The damping was obtained by the measurement of the logarithmic decrement of free vibration by a vibrating apparatus. The beam was fixed on one side and a permanent magnet was fastened on the opposite side. The beam was excited to resonance vibrations by a sinusoidal current flowing in a field coil. After reaching definite amplitude the coil current was switched off by a PC and the declining mechanical vibration was stored in the PC via an ADC measuring the voltage induced by the moving permanent magnet. Using these data the decrement δ of the free vibration was determined by the PC according to exponential law

$$A(t) = A_0 \exp(-\delta \cdot t / \tau_p). \quad (6)$$

In Eq. (6), A_0 is the required amplitude, t is time and τ_p is the period of vibrations. The samples were sequentially annealed at increasing temperatures up to 400 °C for 15 min and then quenched into water of ambient temperature. The measurements of δ were carried out at room temperature.

Samples used for internal friction spectra measurements were machined from the bulk die cast material in the form of cantilever beams with dimension of $3 \times 10 \times 32 \text{ mm}^3$. Internal friction spectra were measured with a dynamic mechanical thermal analyser (DMTA) (DMA 2980 TA Instruments) in the forced vibration single cantilever mode while heating and cooling. The heating rate as well as the cooling rate was 1 K/min. Three measurement frequencies, 1,

5, and 50 Hz, were used. Throughout the measurements the strain amplitude was 3.3×10^{-5} . The energy dissipation was measured by the mechanical loss $\tan \varphi$ (φ is the loss angle). There is a simple relationship between all quantities characterising the damping $\eta = \delta / \pi = \tan \varphi$.

3. Results and discussion

3.1. Amplitude dependent internal friction

The amplitude dependence of the logarithmic decrement for Mg-4Li alloy sequentially thermally cycled at increasing temperature is introduced in Figs. 1a and 1b. (The logarithmic decrement is plotted against the logarithm of the maximum strain amplitude.) The strain dependence of the logarithmic decrement can be divided into a strain independent and a strain dependent component:

$$\delta = \delta_0 + \delta_H(\varepsilon), \quad (7)$$

δ_0 is the amplitude independent component (or slightly dependent), found at low amplitudes. The component δ_H depends on the strain amplitude ε and it is usually due to interaction between dislocations and foreign atoms (or their small clusters). The critical strain ε_{cr} at which the logarithmic decrement becomes amplitude dependent may be used to calculate the effective critical stress amplitude $\sigma_{cr} = E\varepsilon_{cr}$, where E is the Young's modulus.

The critical strain ε_{cr} estimated from the curves in Figs. 1a,b is plotted in Fig. 2 against the annealing temperature. It is seen that the critical strain slightly increases with increasing temperature up to 100 °C, then rapidly decreases. Values estimated for temperatures 240–300 °C are approximately the same. It is interesting to note that the temperature dependence of the yield stress exhibits the similar local maximum in the vicinity of 70 °C as it can be seen from Fig. 3. This maximum has been interpreted due to the interaction of dislocations with solute atoms [8]. The strain amplitude dependence of the logarithmic decrement suggests dislocation unpinning processes. Damping behaviour of the alloy thermally cycled to various upper temperatures can be attributed to the interaction between dislocations and point defects including small clusters of foreign atoms. The strong strain dependence of δ shown in Figs. 1a,b may be explained using the Granato-Lücke theory of dislocation damping [9]. The dislocation structure is assumed to consist of segments of L_N along which weak pinning points are randomly distributed. The mean distance between the weak pinning points is ℓ with $\ell \ll L_N$. The mean total density of dislocations is ρ . A periodic stress

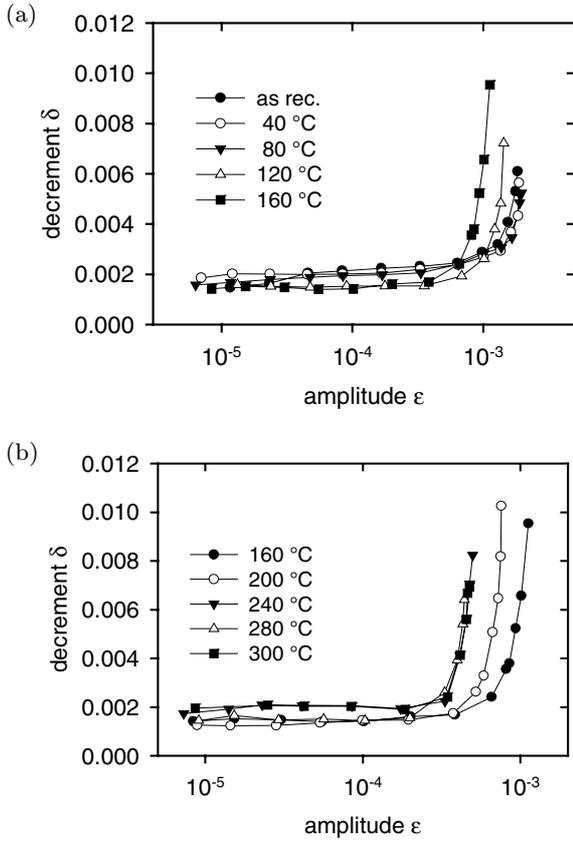


Fig. 1. Strain amplitude dependence of logarithmic decrement estimated for lower annealing temperatures (a), strain amplitude dependence of logarithmic decrement estimated for higher annealing temperatures (b).

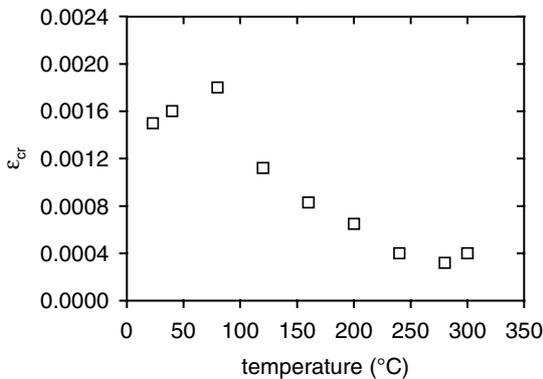


Fig. 2. Critical strain amplitude depending on the annealing temperature.

$\sigma = \sigma_0 \sin \omega t$ is applied. At $T = 0$ K and at sufficiently high stress the dislocations are able to break-away from the weak pinning points and remain anchored at the strong pinning points. The stress required for the break-away of dislocations depends strongly on the statistic distribution of the pinning points. With

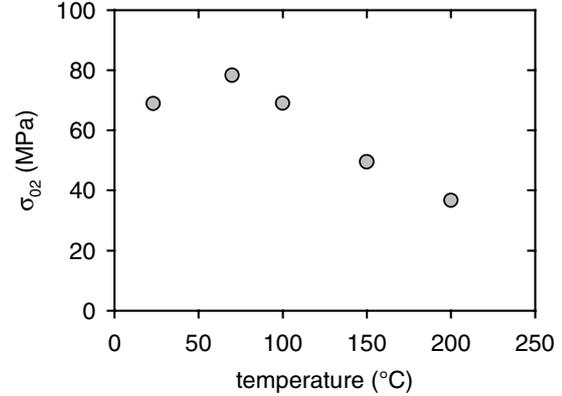


Fig. 3. Temperature dependence of the yield stress.

increasing temperature, for $T > 0$ K, the stress for unpinning decreases, because the break-away process is thermally activated. The stress necessary for break-away of dislocation loops σ_{cr} at a certain temperature T is given by [10]

$$\sigma_{cr} = \sigma_M \left[1 - \left(\frac{kT}{U_1} \ln A \right)^{\frac{2}{3}} \right] \quad (8)$$

with

$$A = \frac{2 \nu \sigma_M}{3 \omega \sigma_0} \left(\frac{kT}{U_1} \right)^{\frac{2}{3}}, \quad (9)$$

σ_M is the break-away stress in the pure mechanical process, kT has its usual meaning and ν is the dislocation frequency. For a double loop with the loop length ℓ_1 and ℓ_2 the break-away occurs under the stress

$$\sigma_M = \frac{2F_m}{b(\ell_1 + \ell_2)}. \quad (10)$$

Here F_m is the maximum binding force between a dislocation and a pinning point. $U_1 = 4/3(F_m^3/\Phi)^{1/2}$, where Φ is a constant. Considering that the activation energy U_1 is approximately the same (all measurements were performed at room temperature), the critical stress (strain) depends only on the dislocation segment length ℓ . The observed decrease of the critical strain at temperatures between 100 and 240 °C indicates elongation of ℓ . Solubility of Li in Mg is relatively high, about 15 at.%. Then the concentration 4 wt.% (12 at.%) is in the primary solubility region and the material studied is a solid solution. After thermal treatment at increasing temperature, Li atoms may diffuse to dislocation lines and form clusters. Lower number of pinning points leads to the higher mean values of ℓ . This process has a saturation character.

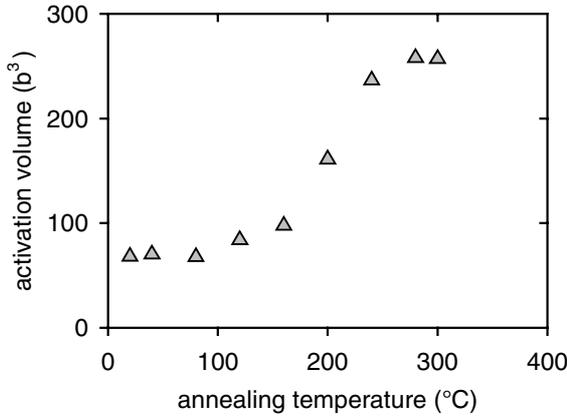


Fig. 4. Annealing temperature dependence of the activation volume.

Curves obtained for temperatures 240–320°C are practically the same.

Pequin et al. [11] developed an idea originally mentioned by Mason [12] to find a relationship between the damping at higher amplitudes and dislocation strain. It follows from the consideration of the thermally activated overcoming of local barriers in the slip plane by dislocations. When the stress carried by sonic waves achieves the micro-yield value ($\sigma_i = E\varepsilon_i$) the amplitude dependence of decrement can be expressed by the following equation

$$\delta_p = \frac{A_1}{\varepsilon\Omega} \exp [B(\varepsilon - \varepsilon_i)], \quad (11)$$

where A_1 and B are done as

$$A_1 = \frac{2\rho b\nu L}{\pi\omega} \exp(-\Delta H/kT), \quad B = \alpha \frac{VG}{kT}.$$

In Eq. (11) G is the shear modulus, ρ the dislocation density and L the activation distance, ν is the dislocation frequency, α is a constant ≈ 0.5 , ω is the vibration frequency, Ω is a constant between 0.5 and 1 and ε_i is the strain amplitude at which dislocation loops start operating in the slip plane. By fitting of the Eq. (11) to experimental curves it is possible to estimate the constant B , which is connected with the activation volume V . The estimated values of the activation volume V as a function of the upper temperature of the thermal treatment are plotted in Fig. 4. The values of the activation volume in the order of 10^2 [b³] were also estimated in plastic deformation region by the other method [13] and they are typical for magnesium and magnesium alloys polycrystals. Considering geometrical interpretation of the activation volume

$$V = bdl = bA^*, \quad (12)$$

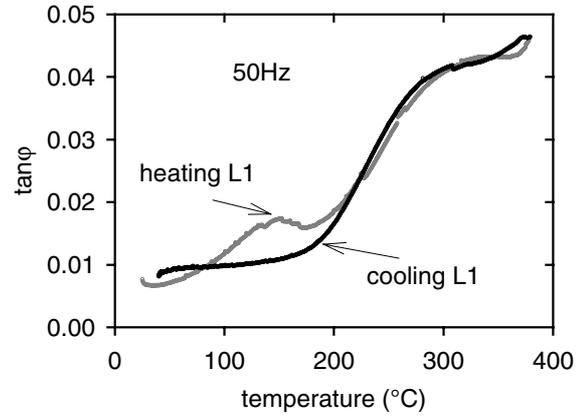


Fig. 5. Temperature dependence of $\tan \varphi$ measured for 50 Hz in the first run while heating and cooling.

where the energy barrier has a width of d and ℓ is the length of the dislocation segment between barriers (A^* is the activation area). An increase in the activation volume with increasing upper temperature of the thermal treatment is due to increase of the dislocation segments length. Migration of solute atoms and formation of solute atoms clusters leads to elongation of dislocation segments. Therefore, an increase of the activation volume V should be occurred.

3.2. Temperature dependent internal friction

Figure 5 shows the temperature dependence of the mechanical loss $\tan \varphi$ measured at 50 Hz while heating and cooling. Two internal friction peaks are visible at the curve: the peak between 100 and 200°C and the high temperature peak at about 300°C (see Fig. 5). During the first cooling sub-circuit the low temperature peak vanished while the peak observed at higher temperatures did not change. This peak remained stable also during heating and subsequent cooling in two next measurements. The temperature dependence of $\tan \varphi$ measured for three frequencies in the heating part of the second heating sub-circuit is introduced in Fig. 6. Frequency shift of the peak is well visible. The internal friction peaks are assumed to be imposed by a background IF_b expressed by

$$IF_b = A_b + B_b \exp(-C_b/kT), \quad (13)$$

where A_b , B_b , and C_b are constants. After subtracting the background by using a fitting program PeakFit, the maximum temperature T_p was estimated for the high temperature relaxation peak. The internal friction peak appears at the condition $\omega\tau = 1$ [14], with

$$\omega\tau = \omega\tau_0 \exp(\Delta H/kT), \quad (14)$$

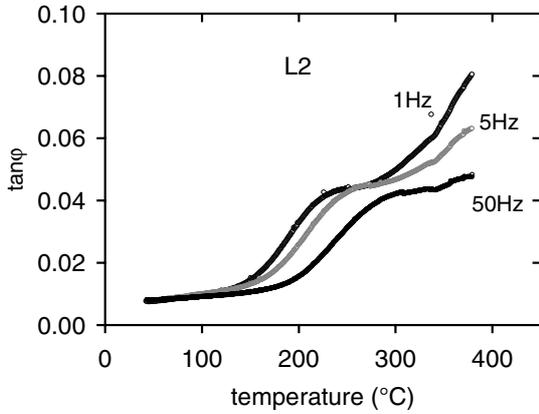


Fig. 6. Temperature dependence of $\tan \varphi$ measured for three frequencies.

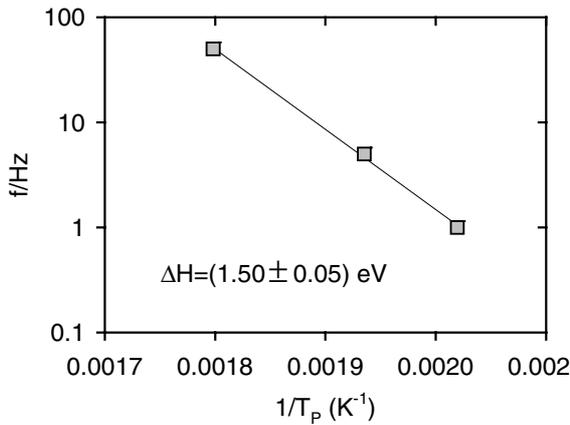


Fig. 7. Arrhenius plot constructed from the peaks position introduced in Fig. 6.

where ω is the angular frequency ($= 2\pi f$, f is the measuring frequency), τ is the mean relaxation time, τ_0 is the pre-exponential factor, and ΔH is the mean activation enthalpy. Figure 7 shows the semi-logarithmic plot of the frequency versus reciprocal value of the peak temperature T_P , so-called Arrhenius plot. The value of the activation energy was estimated to be (1.5 ± 0.05) eV. This peak may be described to the grain boundary sliding. An existence of the high temperature peak in magnesium has been reported by Kê [15]. He ascribed this peak as being related to the grain boundary relaxation.

Grain boundaries in the material with smaller grain size contain dense network of overlapping grain boundary dislocations. Grain boundary sliding is realized by the slip and climb (providing a maintenance of vacancy sources and sinks) of grain boundary dislocations. Since both modes of dislocation

motion must occur simultaneously, the slower one will control the grain boundary sliding. The climb mode involves jog formation and grain boundary diffusion and both modes may be affected by the interaction with impurities segregated in grain boundaries. According to Lakki et al. [16] grain boundary dislocation segments vibrate under applied cyclic stress, restoring force K is assumed to be from the elasticity of the limiting grains at triple junctions. According to this theory, the relaxation strength for small grains does not depend on the grain size d :

$$(\tan \varphi)_{\max} = \frac{G\rho_s b^2}{C}, \quad (15)$$

where ρ_s is the dislocation grain boundary surface density (total dislocation length per unit of grain boundary area), G is the shear modulus and C is a constant. For larger grains it is possible to write

$$(\tan \varphi)_{\max} = \frac{G\rho_s b^2}{c_2} \frac{1}{d}. \quad (16)$$

The relaxation time in this model is

$$\tau = \frac{kT}{b^2 C_j D K} \exp\left(\frac{\Delta H_j + \Delta H_{GB}}{kT}\right) = \tau_0 \exp\left(\frac{\Delta H}{kT}\right), \quad (17)$$

where C_j is the linear density of jogs along the dislocation line, D is the diffusion coefficient, ΔH_j , ΔH_{GB} are the activation enthalpies for the jog formation and the grain boundary diffusion, respectively. The activation enthalpy for grain boundary diffusion in the coarse grained magnesium is 0.95 eV [17]. Comparing with the estimated activation enthalpy value of 1.50 eV, the possible jog formation energy should be approximately 0.55 eV, which seems to be a reasonable value.

4. Conclusions

The activation volume estimated from the strain amplitude dependences of the logarithmic decrement increases with the annealing temperature. This increase is very probably a consequence of the solute atoms clustering. Two peaks have been observed in the temperature relaxation spectrum. Broader peak detected between 100 and 200 °C vanished after the first heating. The high temperature peak is much more stable. Its position in the temperature scale and the height did not change during three heating cooling loops. The activation enthalpy, estimated from the Arrhenius diagram, indicates that the grain boundary sliding is responsible for the occurrence of the high temperature peak.

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