INFLUENCE OF THERMAL TREATMENT ON INTERNAL FRICTION OF AM20 MAGNESIUM ALLOY

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Precipitation processes in magnesium alloy AM20 have been studied. Homogenised alloy was annealed step by step at increasing upper temperature of the thermal cycle. Amplitude dependences of decrement and modulus defect were measured after each annealing step. While amplitude independent part of decrement was not influenced by the thermal treatment, the amplitude dependent part varies with the annealing temperature. Results were analysed according the Granato and Lücke theory of thermally activated break away of dislocation segments from solute atoms. Precipitation process(-es) decrease(s) number of solute atoms and increase(s) the length of free dislocation segments which are mainly responsible for damping behaviour of the alloy. Precipitation process (-es) occur(s) in the temperature interval from 200 to 320 °C. These results were confirmed by stress relaxation tests.

Key words: internal friction, dislocations, precipitation, magnesium alloys

1. Introduction

Attempts to improve the performance of existing magnesium alloys, and to develop new alloys, have to date been restricted by a lack of understanding of precipitation behaviour, alloy, microstructures, deformation behaviour, strengthening mechanisms, and microstructural factors that are most important in controlling precipitation, strength and mechanical properties [1–3]. The most popularly used magnesium alloys are those based essentially on the Mg-Al system. The most widely used magnesium alloys are based on the Mg-Al-Mn system (AM alloys). The AM alloys exhibit good mechanical properties. The AM20, AM50 and AM60 alloys (with 2, 5 and 6 wt.% of Al, respectively, and 0.2–0.5 wt.% Mn) form a series

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of high-purity alloys with reduced iron content. An addition of manganese is required to control the corrosion behaviour. AM50 and AM60 alloys are used for applications where good ductility and high fracture toughness are required. The improved properties arise because of a reduction in the amount of $Mg_{17}Al_{12}$ phase around grain boundaries [4]. The precipitation processes in these alloys appear to involve solely the formation of the equilibrium phase $Mg_{17}Al_{12}$, which is associated with the diffusivity of aluminium in magnesium. Internal friction as a tool to study precipitation processes in alloys was used in several studies [5–7]. Usually the temperature dependence of internal friction was measured. Similar attempts were performed in composites. Amplitude dependence of decrement was applied to study generation of new dislocations and precipitation processes in composites [6–10].

In this paper we present indirect experimental evidence of precipitation process in the homogenised magnesium alloy AM 20 after thermal treatment at increasing temperature. Non-destructive internal friction method and stress relaxation test have been used.

1.1 Mechanical damping

If a material containing dislocations is submitted to a harmonic applied stress $\sigma = \sigma_0 \sin \omega t$ with an angular frequency $\omega = 2\pi f$ (t is time, f frequency and σ_0 stress amplitude), one can define the mechanical loss factor η as

$$\eta = \frac{1}{2\pi} \frac{\Delta W_{\mathrm{diss}}}{W_{\max}}, \tag{1}$$

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},

$$\Delta W_{\mathrm{diss}} = \oint \varepsilon_{\mathrm{d}} \mathrm{d}\sigma. \tag{2}$$

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

$$W_{\max} = \int_0^{\sigma_0} \sigma \mathrm{d}\varepsilon_{\mathrm{el}} = \frac{1}{2} J_{\mathrm{el}} \sigma_0^2, \tag{3}$$

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

$$\eta = \frac{1}{\pi J_{\mathrm{el}} \sigma_0^2} \oint \varepsilon_{\mathrm{d}} \mathrm{d}\sigma. \tag{4}$$

1.2 Stress relaxation

In stress relaxation (SR) tests strain is constant and then the stress $\sigma(t)$ as a function of time t is recorded. Assuming a relaxation process that depends on a single relaxation time, the stress is given by [11]

$$\sigma(t) = \varepsilon_0[E_\mathrm{R} + \Delta E \exp\left(\frac{-t}{\tau}\right)], \tag{5}$$

where E_R is the unrelaxed modulus, ε_0 strain amplitude, ΔE modulus defect and τ time constant.

At relatively high homologous temperatures, damping of most materials rises continuously to high values. This phenomenon known as high temperature background is highly structurally sensitive. Schoeck et al. [12] considered that the high temperature background is produced by a broad spectrum of diffusion controlled relaxation (involving dislocations moving in a viscous manner). They derived the relationship between the quality factor Q and temperature as

$$Q^{-1} = \frac{A}{[\omega \exp{(\Delta H_0/kT)}]^n}, \tag{6}$$

where n and A are constants, A is related to dislocation structure, ΔH_0 is the true activation enthalpy of the controlling mechanism, k is the Boltzmann constant and T absolute temperature. In the experiments, the apparent activation enthalpy, $n\Delta H_0$, is measured from the temperature dependence of the internal friction. The value of the apparent activation enthalpy is meaningless unless n is determined by the frequency dependence of the internal friction. ΔH_0 was found to be close to the activation enthalpy for self-diffusion ΔH_v [13].

2. Experimental procedure

AM20 magnesium alloy with nominal composition Mg-2wt.%Al-0.4wt.%Mn was prepared by squeeze casting method. Homogenisation thermal treatment was performed in an argon protecting atmosphere at 413 ℃ for 18 h.

Damping measurements were carried out on bending beams (80 mm long with thickness of 3 mm) in a 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 bending beams fixed at one end and a permanent magnet attached at the free end dipping into a coil system were contactlessly excited to mechanical resonance of their fundamental vibration (clamped-free-bar with end loading) by an alternating magnetic field. This was realised by a closed feedback loop consisting of an excitation coil, an induction coil and a power AC amplifier.

When the amplifier was cut from the coil system at given amplitude by relays, the free decaying vibration was measured via the effective alternating voltage induced by the moving permanent magnet into the induction coil. It has to be stressed that this method allows the measurement of amplitude-dependent internal friction for a single individual decay of free vibration. After reaching a 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 [14, 15]. Using these data the decrement δ of the free vibration was determined by the PC according to exponential decay of the amplitude A:

$$A(t) = A_0 \exp(-\delta t/T), \tag{7}$$

where T is the period of vibrations. The reversible stress relaxation was performed with an electronic balance that allows one to apply a certain strain to the bending beam by lifting or lowering the scale of the balance. The time dependent change of the stress $\Delta\sigma$ in the beam is proportional to the change of the weight that is read out. Strain is constant and Eq. (9) is valid:

$$\frac{\Delta\sigma}{\sigma} = \frac{\Delta E}{E} = \frac{\Delta G_{\mathrm{w}}}{G_{\mathrm{w}}} = \frac{G_{\mathrm{w}}(t) - G_{\mathrm{w}}(t_1)}{G_{\mathrm{w}}(t_1)}, \tag{8}$$

where $\Delta E/E$ is the modulus defect and G_{w} is the weight. The stress relaxation $\Delta\sigma/\sigma$ (or $\Delta E/E$) was determined over the time period $t_1 = 3$ s to $t_2 = 3600$ s. To exclude effects due to thermal variations the test temperature was kept constant at 31 ± 0.02 ℃. A more detailed description of the experimental procedure is given elsewhere [16, 17].

The samples were sequentially annealed at increasing temperatures up to 400 ℃ for 15 min and then quenched into water of ambient temperature. The step of temperature was 20 ℃. Measurements of the amplitude dependence of decrement were performed at room temperature after each step of thermal treatment.

3. Results and discussion

3.1 Damping measurements

Amplitude dependence of decrement δ estimated for sample in the condition after homogenisation treatment is introduced in Fig. 1.

The curve has a typical course with an amplitude independent region measured for lower strains and amplitude dependent part measured for higher strain amplitudes, i.e. δ can be expressed as a sum of two components,

$$\delta = \delta_0 + \delta_{\mathrm{H}}(\varepsilon), \tag{9}$$

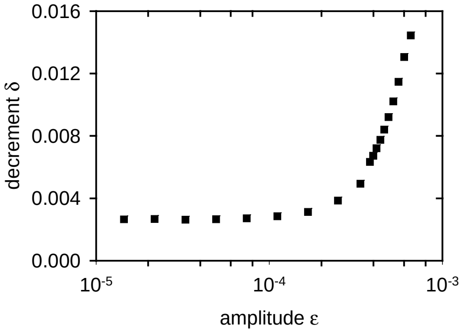

Fig. 1. Amplitude dependence of decrement obtained for sample after homogenisation.

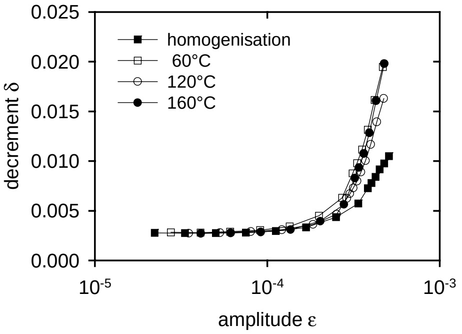

Fig. 2. Amplitude dependence of decrement measured for annealed sample up to 160 °C.

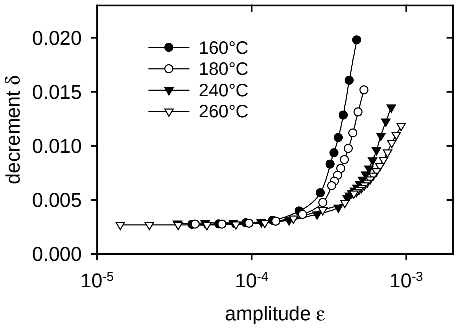

Fig. 3. Amplitude dependence of decrement measured for annealed sample up to 260 °C.

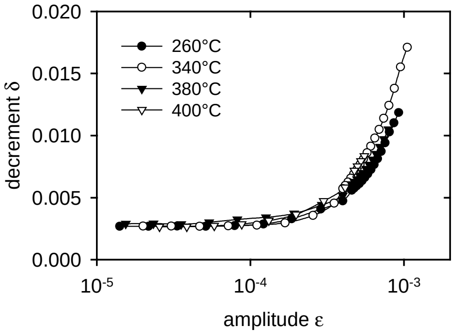

Fig. 4. Variations of decrement for sample annealed at temperatures higher than 260 °C.

where δ_0 is the amplitude strain independent component for low strain amplitudes and δ_H is the strain amplitude dependent component of δ. While the amplitude independent component does not depend or weakly depends on the thermal treatment temperature, the values of δ_H vary with upper temperature of the annealing cycle. It can be seen in Fig. 2 (only selected curves are introduced). The amplitude dependent component of decrement δ_H increases with increasing annealing temperature up to 160 °C. Following decrease of decrement is observed for higher annealing temperatures, as seen in Fig. 3.

Further thermal treating eventuated to decrease of decrement as it is seen in Fig. 4. Variations of decrement for sample annealed at temperatures higher than

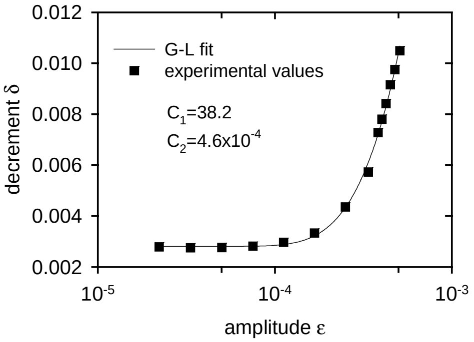

Fig. 5. Granato-Lücke fit performed for the dependence introduced in Fig. 1.

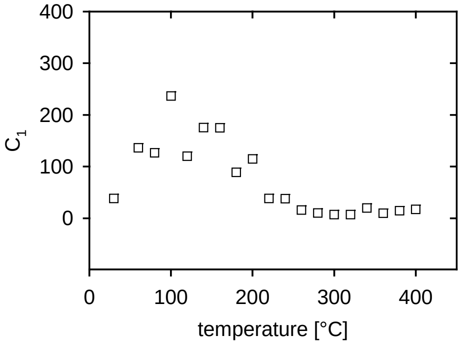

Fig. 6. Annealing temperature dependence of C_1 parameter.

260 °C are not significant as it is demonstrated in Fig. 4.

Presented experimental data indicate microstructure changes in the sample. While amplitude independent component of decrement may be changed by various influences (i. e. inhomogeneities in the material, dislocations, grain boundaries, thermoelastic effect), the amplitude dependent component is due to presence of dislocations in the material. The strain amplitude dependence of the logarithmic decrement suggests dislocation unpinning processes. The differences in the damping behaviour of specimens thermally treated to various upper temperatures can be attributed to the interaction between dislocations and foreign atoms including their small clusters.

Considering the dislocation structure to consist of segments of L_{N} along which weak pinning points are randomly distributed. The mean distance between two weak pinning points is ℓ with $\ell \ll L_{\mathrm{N}}$. The mean total density of dislocations is ρ. At $T = 0$ and at sufficiently high stress, dislocations are able to break-away from the weak pinning points. Only ends of the longer segments L_{N} are assumed to be unbreakable pinning points. The stress required for break-away of dislocations is determined by the largest double loop in a segment and it depends on the distribution of the pinning points. With increasing temperatures, the stress decreases because the break-away process is thermally activated [18]. At higher temperatures the break-away can occur at lower stresses than at $T = 0$, but higher activation energies are required because the break-away is simultaneous from several neighbouring pinning points. At high temperatures and low frequencies, the stress dependence of δ_{H} can be expressed as [18],

$$\delta_{\mathrm{H}} = \frac{\rho L_{\mathrm{N}}}{6}\frac{\nu}{\omega}\left(\frac{3\pi kT}{2U_0}\right)^{1/2}\left(\frac{\ell^3\sigma_0^2}{U_0 G}\right)^{1/2}\exp\left[-\frac{4}{3}\frac{U_0}{kT}\left(\frac{U_0 G}{\ell^3}\right)^{1/2}\frac{1}{\sigma_0}\right], \tag{10}$$

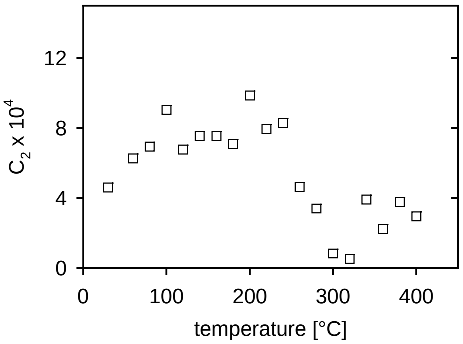

Fig. 7. Annealing temperature dependence of C_2 parameter.

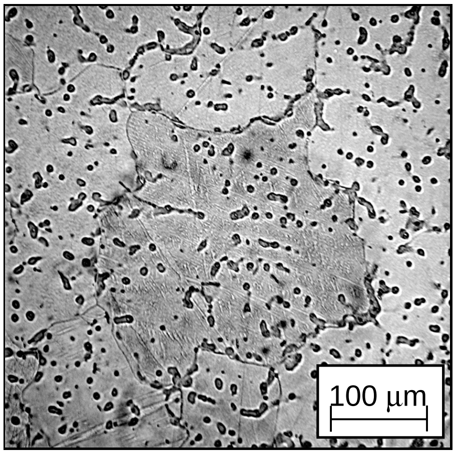

Fig. 8. Microstructure of AM20 alloy with $Mg_{17}Al_{12}$ particles.

where G is the shear modulus, σ_0 is the amplitude of the applied stress and ω its frequency, ν the dislocation frequency, U_0 is the activation energy, k is the Boltzmann constant. This relationship has a similar form as the original formula given by Granato and Lücke [19, 20]. The δ_{H} component depends exponentially on the stress amplitude. The experimental data were analysed using Eq. (10) in the form $\delta = \delta_0 + C_1\varepsilon \exp(-C_2/\varepsilon)$. Example of such fitting procedure is introduced in Fig. 5. Values of C_1 parameter depending on annealing temperature are introduced in Fig. 6 and values of C_2 parameter in Fig. 7. Discussion of both parameters can be lead only qualitatively, because we do not know other quantities in Eq. (10), as dislocation density, the length of the longer dislocation segments L_{N}, the dislocation frequency or the activation energy U_0. Discussion of the C_2 parameter is simpler because it depends only on the length of the small dislocation segments ℓ and the activation energy U_0. Assuming that the activation energy is in the first approximation the same (all measurements were performed at room temperature) we may attribute that the observed decrease of both parameters at temperatures between 200–320 ℃ is due to precipitation in the alloy. $Mg_{17}Al_{12}$ intermetallic phase particles are formed as it is demonstrated in Fig. 8. Weak pinning points (solute atoms) form precipitates and effective length ℓ of dislocation segments increases. The C_2 parameter is proportional to $C_2 \propto \ell^{-2/3}$, then, with increasing annealing temperature the values of C_2 parameter decrease. Above 320 ℃, the C_2 slightly increases; it can be caused by several influences: decreased dislocation density ρ, thermal stresses and the interaction of dislocations with precipitates. Observed increase of C_2 as well as C_1 parameter at temperatures up to 160 ℃ could be caused by the segregation of solute atoms at dislocations. Such processes were observed at the plastic deformation of various Mg alloys [21–24]. The strain ageing phenomena are a consequence of dislocation-solute atom interaction.

3.2 Stress relaxation

The anelastic relaxation processes were studied on specimens thermally treated for damping measurements. Immediately after damping measurement, specimens were put into the balance and the stress relaxation was measured. Stress drops $\sigma(t)$ against time are presented in Fig. 9 for various states of the annealed specimens.

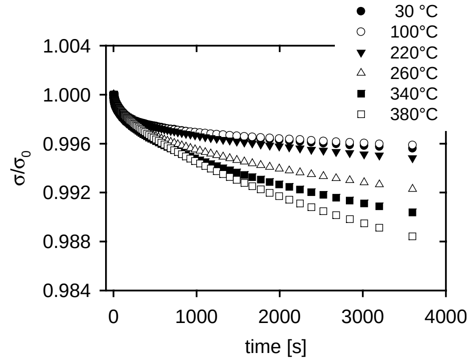

Fig. 9. Stress relaxation obtained for increasing upper temperature of thermal treatment.

After annealing treatment at increasing temperature, the amount of the stress relaxation after 3600 s increases with increasing temperature up to 200℃, then it rapidly decreases. Considering that the high temperature damping is produced by an activation energy spectrum [12], the principle of superposition should be considered and hence

$$\sigma(t) = \varepsilon_0 \left[E_{\mathrm{R}} + \sum_i \Delta E_i \exp\left(-\frac{t}{\tau_i}\right) \right], \tag{11}$$

if relaxation processes are independent on each other. Lower energies are responsible for the faster relaxation (i.e. shorter relaxation times) processes, while for the slower (i.e. longer times) processes, higher activation energies are involved. These combined processes give rise to a continuous monotonous relaxation that does not reach its saturation state.

The relation between SR and specific damping factor Q^{-1} is given by the equation

$$Q^{-1}(\omega) = \left(\frac{\partial \ln \sigma}{\partial \ln t}\right)_{t=1/\omega}. \tag{12}$$

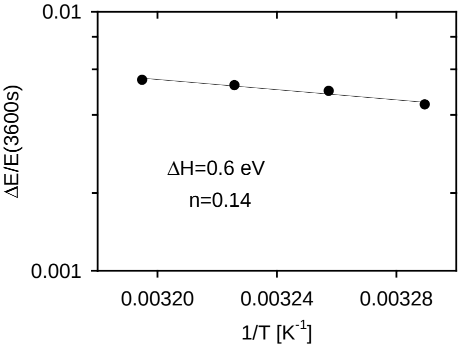

Fig 10. Arrhenius plot constructed for modulus defect $\Delta E/E$, obtained for 3600 s.

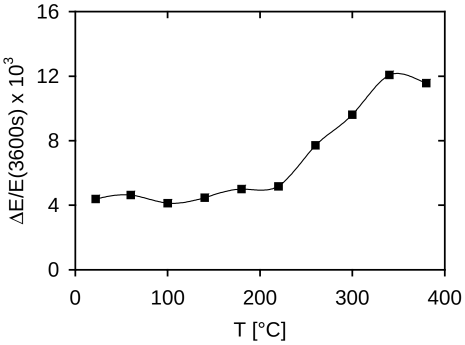

Fig. 11. Annealing temperature dependence of modulus defect obtained for 3600 s.

Using (12), Eq. (6) can be rewritten into the following equation:

$$\frac{\Delta E}{E} = \frac{E\left(t_1\right) - E\left(t\right)}{E\left(t_1\right)} = \frac{\sigma(t_1) - \sigma(t)}{\sigma(t_1)} = K_{\mathrm{R}}\left(t^n - t_1^n\right)\exp\left(\frac{-nU_0}{kT}\right) = K\left(t^n - t_1^n\right). \tag{13}$$

In Eq. (13) K_{R} and n are constants, which depend on viscous movement of dislocations. If $n = 1$, the process is described by single relaxation time. Theoretical models [25, 26] introduce $n = 0.5$. U_0 is the activation energy for the thermally activated dislocation motion. t_1 is time of the first measurement after load change. The time dependences of the modulus defect were fitted according to Eq. (13) and the value of n parameter was estimated. An apparent activation energy $U = nU_0$ was found from the temperature dependence (Arrhenius plot) of the modulus defect for 3600 s (Fig. 10) to be 0.6 eV. Such value of the activation energy seems to be reasonable for dislocation processes. The modulus defect should be according to Eq. (13) proportional to time and $K = K_{\mathrm{R}}\exp(-nU_0/kT)$. Parameter K_{R} in Eq. (13) should be, according to the theory, proportional to the dislocation density and square of the length of the shorter dislocation segments, i. e. $K_{\mathrm{R}} \propto \rho\ell^2$. With increasing annealing temperature the length of shorter dislocation segments increases also due to precipitation process than the modulus defect should increase too. It was really observed as it is demonstrated in Fig. 11.

4. Conclusions

From the internal friction measurements it can be concluded that changes in the microstructure of AM20 magnesium alloy occur practically in the whole annealing temperature region. In the first interval from room temperature up to 200 °C migrating solute atoms segregate at dislocation lines. Thermal treatment at higher temperatures from 200 to about 320 ℃ leads to precipitation of $Mg_{17}Al_{12}$ intermetallic phase particles. Activation energy was estimated from the temperature dependence of the modulus defect to be 0.6 eV. This activation energy can be described to the thermally activated dislocation process, supported very probably by the diffusion.

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