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Thermal and mechanical stability of Mg based nanocomposite studied by internal friction measurements

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Abstract

Magnesium matrix composites show improved wear resistance, enhanced strength and creep resistance in comparison with their monolithic counterparts. On the other hand they keep low density and good machinability. Internal friction measurements are suitable tool to detect changes in the microstructure of thermally or mechanically loaded composites. Samples from pure magnesium reinforced with zirconia nanoparticles were thermally cycled between room temperature and increasing upper temperature of thermal cycle. After thermal cycling amplitude dependence of decrement was measured. Very high values of the logarithmic decrement were ascribed to the poor binding between the matrix and ceramic nanoparticles. The influence of cyclic bending on the damping behaviour of the same nanocomposite was determined at room temperature. Measured decrease of the resonant frequency indicates the stiffness loss as a function of cycling. Observed decrease of amplitude independent component of decrement at the end of the sample life time is due to increase of the dislocation density. These dislocations can be absorbed by the interface. Crack deflection along an interface is followed by the separation of the particle/matrix interface.

Key words: magnesium nanocomposite, internal friction, dislocations, bending test, fatigue

1. Introduction

New promising magnesium materials may be produced using nanosized ceramic particles as reinforcement. We may call these materials as nanocomposites. Mg based nanocomposites may be fabricated by mixing and comilling of microscaled metal powder with nanoscaled particles followed by hot consolidations [1, 2] or by friction stir processing [3]. These methods offer a good route to incorporate ceramic particles into the magnesium (and Mg alloys) matrix in order to form bulk composites. These composites may be attractive in applications because of their high strength and low density characteristics. In practice, materials with good mechanical properties and with good ability to suppress vibrations are needed. One way to reduce the vibrations is to use high-damping materials. Various methods can be used to determine the damping characteristics. In resonance experiments (the frequency is of the order of the resonant frequency), the decay of free vibrations of the system is measured after excitation. The logarithmic decrement δ which expresses the reduction in amplitude of vibration of a freely decaying system during one cycle is defined as

$$\delta = \ln(A_n/A_{n+1}), \quad (1)$$

where A_n and A_{n+1} are the amplitudes of a free decay of vibrations after n cycles and $(n+1)$ cycles, respectively. Measurement of the logarithmic decrement at small strains (stresses) is a sensitive method determining the microstructure of the material. Damping (internal friction) as a function of temperature and/or

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frequency has been used to identify several atomic processes in materials.

The objective of the present paper is to estimate the influence of thermal and mechanical cycling on the damping behaviour of magnesium/ZrO_2 nanocomposite. The logarithmic decrement is used as a measure of the damping.

2. Experimental procedure

Experimental material for this study has been prepared using commercially available microcrystalline Mg powder with the particle diameter of about 20 μm. ZrO_2 nanoparticles with a characteristic size of 14 nm were prepared by evaporation with the pulsed radiation of a 1000 W Nd:YAD laser. The zirconia nanopowder and Mg micropowder were mixed together in an asymmetrically moved mixer for 8 h, then the mixture was milled together for 1 h, then pre-compressed in a uniaxial press and subsequently hot-extruded at 450 ℃ and 650 MPa. The original more or less uniaxial grains changed into very asymmetrical elongated grains with the long axis in the extrusion direction. ZrO_2 nanoparticles, or their agglomerates are located in the grain boundaries of the resultant material.

Test specimens for internal friction measurements were machined as bending beams (85 mm long with a thickness of 3 mm and 10 mm width). The specimens fixed at one end were excited into resonance by a permanent magnet fixed on the free side of the bending beam and a sinusoidal alternating magnetic field. The resonant frequency was about 130–140 Hz. The damping was measured as the logarithmic decrement δ of the free decay of the vibrating beam. The strain amplitude dependences of the logarithmic decrement, so called internal friction curves, were measured. The specimens were sequentially annealed at increasing temperatures up to 550 ℃ for 0.5 h and then quenched into water of ambient temperature. Annealing at higher temperatures was performed in an argon atmosphere to avoid oxidation. The internal friction measurements were carried out immediately after quenching at room temperature in vacuum (about 30 Pa).

Cyclic deformation (fatigue) was realised by the controlled bending loading of the beam samples in the similar apparatus as used for the damping measurements automatically controlling the amplitude and number of vibrations. Samples were cycled a certain time at the maximum amplitude. From the time and frequency, the number of cycles was calculated. Immediately after cycling the strain amplitude dependence of the decrement was measured. Because of different apparatus setting in comparison with the previous case the resonant frequency of the system was about 60 Hz. The end of the fatigue life of the sample was manifested by the rapid decrease of the resonant frequency. The sample was cycled up to the sample failure. It was reached at $N = 1.03 \times 10^8$ cycles.

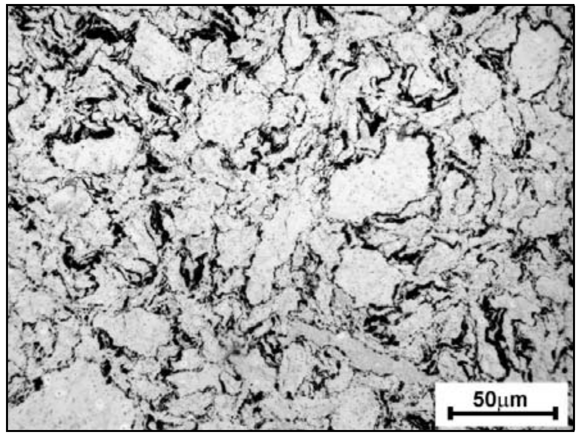

Fig. 1. The microstructure of as-extruded Mg+3vol.%ZrO_2 nanocomposite.

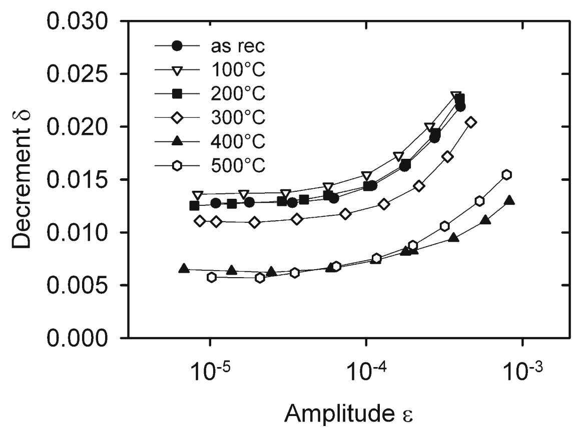

Fig. 2. Amplitude dependences of decrement measured at room temperature after thermal cycling at increasing upper temperature of the thermal cycle.

3. Results

Figure 1 shows an optical micrograph of the as prepared nanocomposite. The grain size estimated in the cross section exhibits about 3 μm and in the extrusion direction about 10 μm. The distribution of the nanoparticles was not homogenous. The nanoparticles or their agglomerates were located mainly in the grain boundaries of the resultant material.

Figure 2 shows the plots of the logarithmic decrement against the logarithm of the maximum strain amplitude before and after one thermal cycle between room temperature and increasing upper temperature of the thermal cycle.

For many metallic materials, the strain dependence of the damping capacity can be divided into a strain independent and a strain dependent component. In the case of the logarithmic decrement, the experimentally found results may be expressed as:

$$\delta = \delta_0 + \delta_{\mathrm{H}}(\varepsilon), \tag{2}$$

where δ_0 is the amplitude independent component, found at low strain amplitudes. The component δ_{H} depends on the strain amplitude ε and it is usually caused by dislocation depinning from weak pinning points in the material. The critical strain $\varepsilon_{\mathrm{cr}}$ at which the logarithmic decrement becomes amplitude dependent may be used to calculate the effective critical stress amplitude σ_{C} corresponding to the micro yield stress according to the equation

$$\sigma_{\mathrm{C}} = E\varepsilon_{\mathrm{cr}}, \tag{3}$$

where E is the Young's modulus. From Fig. 2 it is seen that the applied thermal treatment influences previously the amplitude independent component δ_0, while the amplitude dependent component δ_{H} varies with the upper temperature of the thermal cycle only slightly.

The logarithmic decrement δ plotted against number of bending cycles for two values of the strain amplitude is introduced in Fig. 3. It can be seen that the decrement increases with increasing number of cycles for both amplitudes up to 2.4 $\times$ 10^6 cycles, then slightly decreases, up to 2.9 $\times$ 10^7 cycles. Further cycling decreases the decrement rapidly.

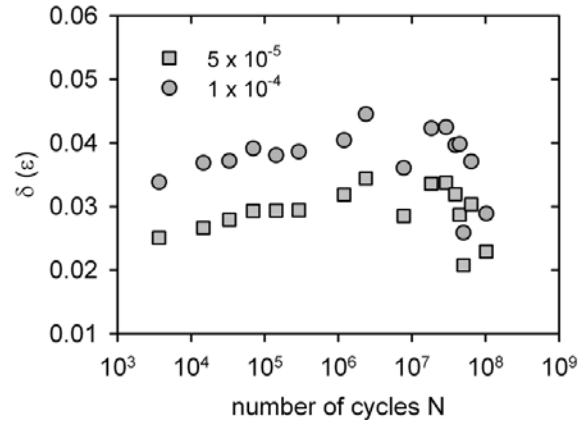

Fig. 3. Logarithmic decrement depending on number of bending cycles at room temperature estimated for two strain amplitudes.

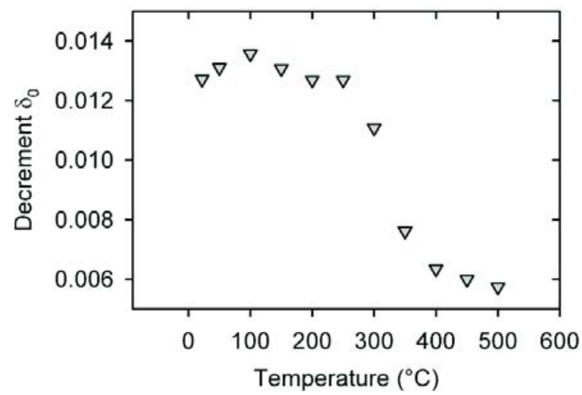

Fig. 4. Amplitude independent component depending on upper temperature of the thermal cycle.

4. Discussion

4.1. Thermal cycling

The amplitude independent material damping can be caused by different mechanisms (thermoelastic damping, grain boundary sliding, magnetic effects, and point defects). The significant contribution to the damping in magnesium is dislocation damping. Temperature dependence of the amplitude independent component of the logarithmic decrement δ_0 is introduced in Fig. 4. It is obvious that the values of δ_0 are very high. Similar damping measurements were realised on a magnesium nanocomposite with alumina particles prepared with the same method [4]. The damping in the amplitude independent region estimated at the as received state is substantially lower $\delta_0(\mathrm{Mg} + 3\mathrm{Al_2O_3}) = 0.00175$ while $\delta_0(\mathrm{ZrO_2}) = 0.0127$. The high damping in Mg + $3\mathrm{ZrO_2}$ nanocomposite is very probably due to a weak bonding between zirconia nanoparticles in comparison with alumina nanoparticles where the bonding is nearly perfect.

Very high damping in the composite with the zirconia nanoparticles is caused by an interfacial slip due to weak bonding between particles and matrix. In this case the frictional energy loss caused by the sliding at the interfaces may become a primary source of damping. The damping component due to interfacial slip under the applied stress amplitude σ_0 can be expressed as [5–7]:

$$\delta = \frac{3\pi^2}{2}\,\frac{\kappa\sigma_{\mathrm{r}}(\varepsilon_0 - \varepsilon_{\mathrm{cr}})}{\sigma_0^2/E_{\mathrm{C}}}\,V_{\mathrm{p}}, \tag{4}$$

where κ is the friction coefficient between both components of the composite, σ_{r} is the radial stress at the interface corresponding to stress amplitude σ_0 (strain amplitude ε_0). The critical interface strain $\varepsilon_{\mathrm{cr}}$ corresponding to the critical interface shear stress τ_{cr} is the strain at which the slip at the interfaces begins. E_{C} is the elastic modulus of the matrix. Assuming that $\varepsilon_{\mathrm{cr}}$

is much lower than ε_0 one obtains

$$\delta = \frac{3\pi^2}{2}\,\frac{\kappa\sigma_\mathrm{r}}{\sigma_0}\,V_\mathrm{p}. \tag{5}$$

The stress concentration factor $k = \sigma_\mathrm{r}/\sigma_0$ has been reported to be 1.1–1.3 [7]. The model does not take into account possible effects of temperature or frequency on damping, and therefore the predictions of the model may be taken only as the first approximation. The effect of an additional damping due to the influence of particles can be clearly seen (Fig. 5). The measured increase in δ_0 due to the addition of ZrO_2 particles to Mg is about 0.008–0.009. Taking for the friction coefficient a typical value of ~ 0.08, the relation (5) predicts a value for an additional damping owing to interfaces $\delta_{0\mathrm{i}} = 0.04$. The discrepancy between the predicted and measured value may be caused by a non-uniform strain state in specimens. They were subjected to bending and hence, strain reaches its critical value at which the interface sliding starts only in some sections of the specimen. High values of δ_0 obtained for magnesium nanocomposite are very probably caused by grain boundary sliding supported by diffusion processes.

Decrease of the damping after thermal cycling with the upper temperature higher than 250 ℃ is obvious from Fig. 4. This decrease indicates some changes in the interface between particles and the magnesium matrix. Nanoparticles were prepared by the evaporation with the pulsed radiation of a laser. Resulting powder was a mixture of crystalline and amorphous particles [8]. The observed decrease of damping is very probably due to partial crystallisation of ZrO_2 nanoparticles and consequently better bonding at the interface [9]. It restricts the interface sliding and energy dissipation. Thermal cycling of the nanocomposite generates thermal stresses at the magnesium/zirconia interface due to a big difference between the thermal expansion coefficient (CTE) of the magnesium matrix and zirconia nanoparticles. These thermal stresses can achieve the yield stress in the matrix and then new dislocations are generated in the matrix. An increase in the dislocation density near reinforcement can be calculated as [10]:

$$\Delta\rho = \frac{B\,f\,\Delta\alpha\,\Delta T}{b\,(1-f)}\,\frac{1}{r_\mathrm{f}}, \tag{6}$$

where b is the magnitude of the Burgers vector of dislocations, B is a geometrical constant, $\Delta\alpha$ is the absolute value of CTE difference, f is volume fraction of the reinforcing phase and r_f its minimum size. Newly created dislocation may increase the amplitude dependent component of decrement. This increase of $\delta_\mathrm{H} = \delta - \delta_0$ observed after thermal cycling at higher temperatures is obvious from Fig. 5 where the increase of damping is visible.

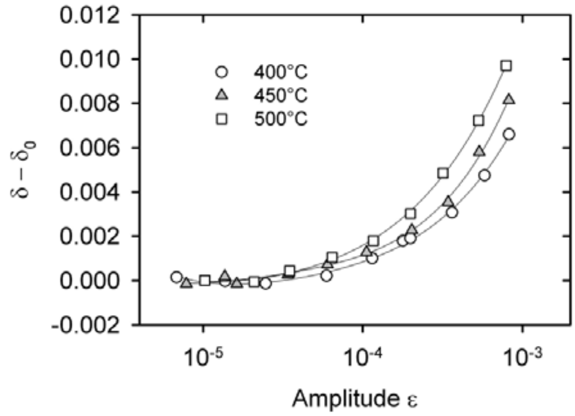

Fig. 5. Amplitude dependent component measured after thermal cycling at temperatures 400–500 ℃.

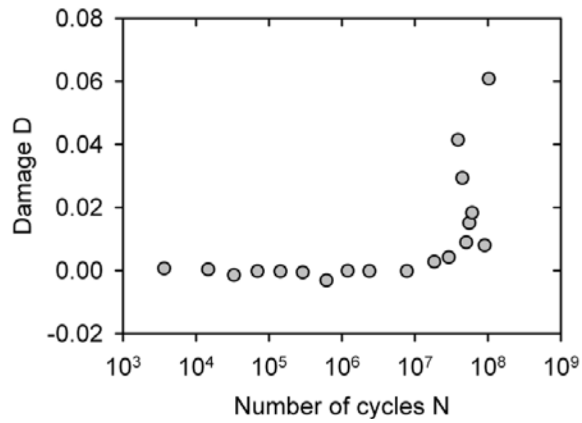

Fig. 6. Damage parameter plotted against number of cycles.

To summarise the discussion to this point, the high damping of the Mg + ZrO_2 is due to the interface friction caused by weak bonding at the interface between magnesium matrix and zirconia nanoparticles. Observed decrease of the decrement after thermal cycling at temperatures higher than 250 ℃ may be explained by the changes in the interface. The phase transformation in zirconia nanoparticles improves the bonding in the interface and restricts the interface sliding. Better bonding after thermal treatment at higher temperatures has a consequence in thermal stresses in the vicinity of the interface and newly created dislocations in plasticized zones.

4.2. Bending cycling

The logarithmic decrement in the strain amplitude independent region at low frequencies yields in the

Granato and Lücke theory [11–13]:

$$\delta_0 = \frac{\pi \omega B_\mathrm{d}}{36 G b^2} \rho \ell^4, \tag{7}$$

where G is the unrelaxed shear modulus, b is the Burgers vector and B_d is the damping force per unit length of dislocation per unit velocity, ρ is the dislocation density and ℓ is length of the shorter dislocation segments pinned at weak pinning points, which may be foreign atoms or small clusters of the point defects. Observed increase of the decrement with the number of cycles N_C (see Fig. 3) is caused by the increase of the dislocation density. Cycling in the region between 3×10^3 and 2.4×10^6 cycles leads to an increase of the decrement. The observed increase of the decrement indicates an increase of the dislocation density and also an increase in the distance between weak pinning points. Further cycling leads to the gentile decrease of the decrement up to 2.9×10^7 cycles. This decrease indicates a decrease of the dislocation density due to interactions between dislocations. Higher dislocation density restricted the slip length of vibrating dislocation segments. The decrement estimated for higher number of cycles than 2.9×10^7 decreases with increasing number of cycles. This decrease is very probably connected with the formation of cracks. This is in accordance with the development of the damage parameter with the number of cycles. The damage of the specimen after N cycles, $D(N)$, can be defined as [14]:

$$D(N) = 1 - \frac{E_\mathrm{N}}{E_0} = 1 - \frac{f_\mathrm{N}^2}{f_0^2}, \tag{8}$$

where E_N is an effective Young's modulus of the specimen after N cycles and E_0 is the Young's modulus for $N = 0$ and f_N and f_0 are the resonant frequencies of the specimen after N cycles and for $N = 0$, respectively. This definition is usual and reasonable [14] because $D(N) = 0$ for undamaged specimen, $D(N)$ increases when cracks start propagating in the specimen and $D(N) = 1$ for a broken specimen. Figure 6 shows the damage D plotted against the cycle number N. A rapid increase of D occurred for $N > 2 \times 10^7$. The measured decrease of the resonant frequency at the end of fatigue process indicates a stiffness loss due to the formation of cracks.

5. Conclusions

Thermal and mechanical cycling of the Mg/ZrO_2 nanocomposite invoked irreversible changes in the microstructure. These changes are detectable using non destructive damping measurements. Internal friction measurements are the useful tool for the study of fatigue of materials.

Acknowledgements

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