Mechanical damping in magnesium prepared by ball milling in medium temperature region

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Abstract

Internal friction in ultrafine-grained Mg was measured by forced vibration method over a wide temperature interval from room temperature up to 750 K and at several frequencies. The specimens were prepared by milling procedure in an inert atmosphere and subsequent compaction and hot extrusion. The linear grain size of specimens used was estimated by X-ray line profile analysis to be about 100–150 nm. Two peaks were obtained in the internal friction spectrum, one at a temperature of $\approx$ 375 K and one at $\approx$ 631 K (at the frequency of 1 Hz). The influence of prestraining and subsequent annealing was observed on the low temperature peak. The activation energy of the two peaks was estimated to be 1.16 eV and 1.77 eV, respectively. The activation volume has been estimated for low temperature peak. The low temperature peak has dislocation origin, while the high temperature peak is conditioned by the grain boundary sliding.

K e y w o r d s: ultrafine-grained material, internal friction, dislocation motion, grain boundary sliding

1. Introduction

Internal friction spectrum is the temperature or frequency dependence of damping. Internal friction in a material is the result of internal processes, which occur during alternating stress cycles imposed on it and these processes originate in the interactions between the structural components in the material. The presence of point, line, and planar defects within a stressed material often causes internal friction to occur because of the atomic movement, rearrangement, or realignment of the defects under application of the stress. These techniques can be used to characterise types of structure defects. Nanocrystalline materials are characterised by a small grain size, typically in the range 1–100 nm; materials with the grain size between 100 nm and 1 μ are usually called as the ultrafine-grained. Both have prominent properties owing to their high volume fraction of grain boundaries. There was a possibility to study the role of dislocations and grain boundaries in a material with the small grain size and to contribute to explanation processes occurring during plastic deformation of nanocrystalline magnesium. Internal friction of nanocrystalline materials was reported only in few papers [1–10]. Internal friction in microcrystalline magnesium reinforced by alumina and zirconia nanoparticles has been investigated in [11]. In the internal friction spectrum of high purity magnesium (99.9999 %) polycrystal, investigated in a medium temperature range, three peaks were observed at 1 Hz: P_0 ($\sim$ 260 K), P_D ($\sim$ 340 K) and P_1($\sim$ 430 K) [12, 13]. Seyed Reihani et al. [14] estimated in high purity magnesium a stable peak at 350 K (at a frequency of 1–2 Hz). Similarly Fantozzi et al. [15] found a pronounced relaxation peak at 420 K. A high temperature peak in 99.9 % Mg was found by Kê [16] at $\approx$ 470 K at 0.5 Hz.

The present paper reports the two internal fric-

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tion peaks observed in ultrafine-grained Mg (hereafter UFG-Mg) in the medium temperature interval. Based on the experimental results, the mechanisms related to both the peaks are discussed. Preliminary results of experiments were presented in [17].

2. Experimental procedure

UFG-Mg samples were prepared by milling procedure in an inert atmosphere and subsequently compacted and hot extruded. The grain size of specimens used was estimated by X-ray line profile analysis to be about 100–150 nm. Internal friction spectra were measured in a DMA 2980 (TA Instruments) apparatus in a single heating and cooling process. Measurements were performed at various frequencies from 0.1 up to 2 Hz. Throughout the measurements the strain amplitude was 1.2×10^{-4}. The heating as well as cooling rate was $1\,\mathrm{K\,min^{-1}}$. No grain growth during heating up to 750 K was observed. In our previous paper [17], we measured the internal friction spectrum from room temperature up to 720 K for three frequencies 0.5 Hz, 5 Hz and 50 Hz. Two developed peaks in the internal friction spectrum were obtained for 0.5 Hz at temperatures of ≈ 370 K and ≈ 620 K. The peaks shifted to higher temperatures with increasing frequency. The high temperature peak was shifted at 5 and 50 Hz to the temperatures out of the measured interval. Therefore, in this study, the temperature interval measured was extended to 750 K. Because the peaks described in the literature have their fixed denomination, we depicted in this study the low temperature peak for the relaxation maximum obtained in the interval 350–383 K and the high temperature maximum for the relaxation maximum found in the interval 586–640 K.

3. Results and discussion

Figure 1 shows the temperature dependence of the internal friction tan φ measured at 0.1 Hz during heating (heating rate $1\,\mathrm{K\,min^{-1}}$). Two peaks showing different intensities have been found: the weak low temperature peak and the more intense high temperature peak. Figure 2 shows the internal friction of nanocrystalline Mg at different frequencies during the first heating cycle measured in multi-frequency mode. It can be seen that both peaks shift to a higher temperature with increasing frequency, indicating that they may be relaxations peaks. The internal friction peaks are assumed to be imposed on top of a background IF_b expressed by

$$IF_\mathrm{b} = A + B\exp(-C_\mathrm{b}/kT), \qquad (1)$$

where k is the Boltzmann constant and T is the absolute temperature, A, 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 two relaxation peaks. Exact temperatures of both peaks estimated for various frequencies are introduced in Table 1. The peak widths are broader than those for a Debye peak, characterised by single relaxation time. The internal friction peak appears at the condition $\omega\tau = 1$ [18], with

$$\omega\tau = \omega\tau_0 \exp(\Delta H/kT), \qquad (2)$$

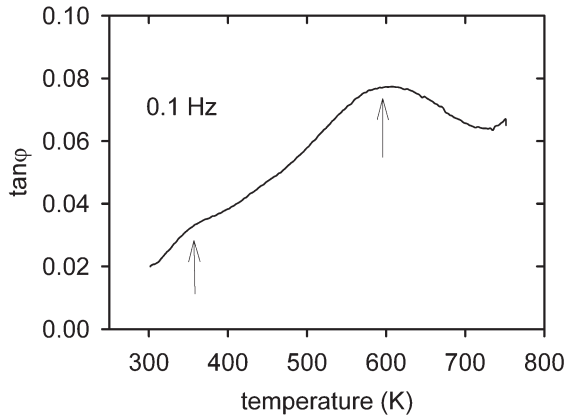

Fig. 1. Internal friction spectrum measured at 0.1 Hz with two peaks.

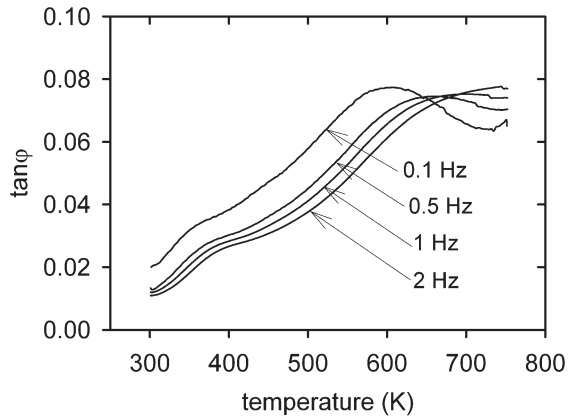

Fig. 2. Internal friction spectrum measured for various frequencies.

Table 1. Temperatures of the relaxation peaks estimated for various frequencies

Frequency (Hz)	T_peak (K)	T_peak (K)
0.1	352.8	586.4
0.5	367.9	617.7
1	375.2	631.1
2	382.7	640.3

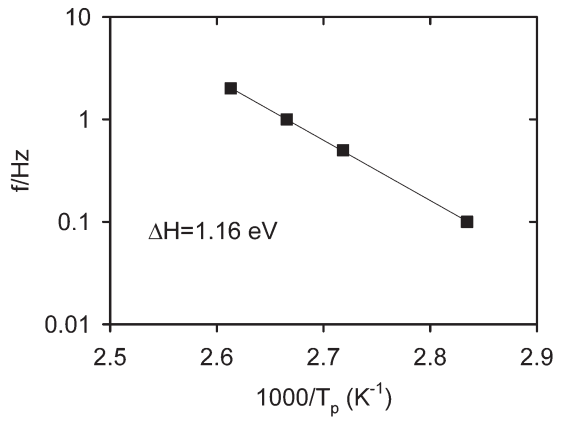

Fig. 3. Arrhenius plot and activation enthalpy obtained for the low temperature peak.

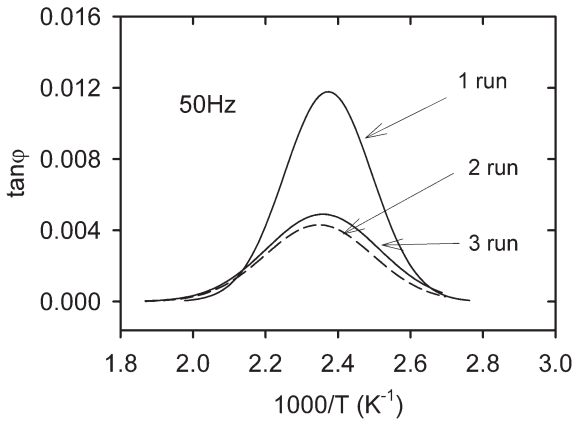

Fig. 4. Substructed low temperature peak after pre-straining of 2.55 %.

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 3 shows the semilogarithmic plot of the frequency versus the reciprocal value of the peak temperature T_P – so called Arrhenius plot.

3.1. Low temperature peak

From the slope of the straight line in Fig. 3, the mean activation enthalpy ΔH for the low temperature peak has been obtained and its value is 1.16 ± 0.05 eV. Internal friction in a high purity magnesium (99.9999 %) has been studied in [12] at a frequency of 1 Hz using torsion pendulum. The performed experiments showed in the temperature interval above the room temperature an internal friction peak depicted by authors P_D placed at ≈ 340 K and peak P_1 at ≈ 430 K. The position of the P_D peak is very close to our low temperature peak. Authors of [12, 13] described this peak to dislocation effects.

As far as our low temperature peak may have a dislocation nature, pre-straining of the sample at low temperature should influence the height of the peak. Deformation by rolling at room temperature ($\varepsilon \approx 2.55$ %) increases the strength of the low temperature peak, as it is obvious from Fig. 4 (the peak was extracted from the internal friction spectrum subtracting the background damping). The peak is stronger only in the first run after pre-straining, in the second and third run the height of the peak is approximately the same as in the as-prepared state. This result is different from an estimation presented in the paper [12], where probably the identical peak P_D vanished after annealing at 570 K. In our previous study, the amplitude dependence of decrement in nanocrystalline magnesium has been measured [19]. An existence of the amplitude dependent component of the logarithmic decrement indicated free dislocation loops in the material. The population of dislocations in the as-received sample is very stable. It is not influenced by any annealing up to 750 K. Beside this original dislocation population, the pre-straining of the sample produced new dislocations which were practically completely recovered in the measuring heating course.

In the case of relaxation processes linked to dislocation motion, the activation area $A = V/b$ (b is the Burgers vector) is the area that a dislocation has to move in order to activate the process, this means to cross over the barrier. Therefore, the product $V \cdot \sigma^*$ represents the work done by the applied stress to promote the motion over the local barrier. Neglecting the entropy term, the activation volume can be expressed as

$$V = -\frac{\partial \Delta H}{\partial \sigma} = -k \ln(\omega\tau_0) \frac{\partial T_\mathrm{p}}{\partial \sigma}, \qquad (3)$$

using Eq. (2) and the condition $\omega\tau = 1$ for the peak appearance. Figure 5 shows the internal friction spectrum measured at different strain amplitudes ε (maximum strain amplitude between 7.3×10^{-5} and 2×10^{-4}). As the strain amplitude increases the peak shifts towards lower temperatures and its height increases while the background increases moderately.

Using $\ln(\omega\tau_0) = -35.95$ ($\tau_0 = 3.9 \times 10^{-17}$ s, $\omega = 2\pi$ Hz), and for $\partial T_\mathrm{p}/\partial\sigma = (1/E)\, 4.8 \times 10^4$ (linear regression from Fig. 6, $\sigma = E\varepsilon$, E is Young's modulus), we obtain the value for the activation volume $V(\mathrm{exp}) = 5.7 \times 10^{-28}$ m$^3 \approx 17b^3$ ($E = 42$ GPa).

Considering geometrical interpretation of the activation volume

$$V = bd\ell = bA, \qquad (4)$$

where the energy barrier has a width of d and ℓ is the length of the dislocation segment between barriers. Parameters of the thermally activated process (activation enthalpy and activation volume) are typical

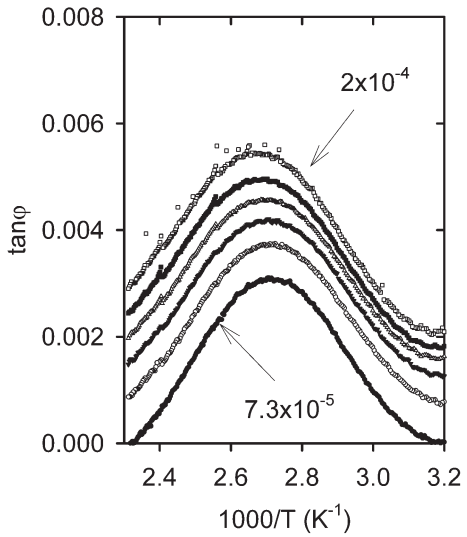

Fig. 5. Background subtracted low temperature peak obtained for various strain amplitudes.

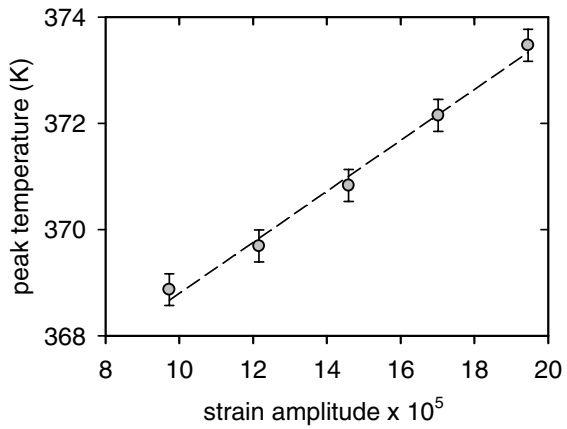

Fig. 6. Dependence of the peak temperature on the strain amplitude obtained for low temperature peak.

for certain barriers and their experimental estimation gives a possibility to identify the main barrier relevant for dislocation motion. Usually the thermally activated process is studied in plastic deformation as, for instance, investigated by Boček [20, 21]. On the other hand, the inelastic behaviour of metallic materials can be considered as the thermally activated dislocation motion. Hexagonal close packed magnesium deforms on many possible glide systems with dislocations of the Burgers vector $\langle a\rangle = 1/3\,[11\bar{2}0]$ in basal, prismatic and first-order pyramidal planes and with dislocations of the Burgers vector $\langle c+a\rangle = 1/3\,[11\bar{2}3]$ in first and second-order pyramidal planes. The most important dislocation relaxation is observed at temperatures of about $1/3\,T_{\mathrm{D}}$ (where T_{D} is Debye temperature). Dislocation glide due to the kink pair formation may give rise to a relaxation peak, so called Bordoni peak. Fantozzi et al. [15] found Bordoni peaks B_1 and B_2 at temperatures of 40 K (screw dislocation) and 80 K (edge and mixed dislocations), respectively. The main features of this relaxation were described in the literature several times, for example in [22].

The main deformation mode in magnesium is the basal slip of $\langle a\rangle$ dislocations. The secondary conservative slip may be realised by $\langle a\rangle$ dislocations on prismatic and pyramidal planes of the first-order. Couret and Caillard studied the prismatic glide in magnesium in a wide temperature range by TEM [23, 24]. They showed that the screw dislocations with the Burgers vector of $1/3\,[11\bar{2}0]$ are able to glide on prismatic planes and their mobility is much lower than the mobility of edge dislocations. The deformation is controlled by thermally activated glide of those screw dislocation segments. A single controlling mechanism has been identified as the Friedel-Escaig mechanism. This mechanism assumes a dissociated dislocation on a compact plane (0001) that joints together along a critical length L_{r} producing double kinks on non-compact plane. The activation energy for this process is

$$U = 2U_{\mathrm{K}} + 4U_{\mathrm{C}} + 2U_{\mathrm{R}}, \tag{5}$$

where $2\,U_{\mathrm{K}}$, $4\,U_{\mathrm{C}}$ and $2\,U_{\mathrm{R}}$ are the formation energies of the kink pair, of the four constrictions and of the two recombined segments, respectively. The activation area is proportional to the critical length between two kinks and it should be small:

$$A \propto L_{\mathrm{r}} b. \tag{6}$$

The theoretical prediction leads to a value of $2\,U_{\mathrm{K}}$ superior to 1.2 eV and to an activation area of about 15 b^2. This mechanism should give an internal friction peak similar to the Bordoni relaxation. The Friedel-Escaig mechanism is also sometimes called as pseudo-Peierls mechanism. Additionally in this model, the maximum of the internal friction peak must increase with increasing oscillating stress (strain amplitude) from a certain critical value.

As far as the experimental results are concerned, we can observe that:

(i) the activation energy is $\Delta H = (1.16 \pm 0.05)$ eV;

(ii) the activation volume is $V(\mathrm{exp}) = \sim 17b^3$;

(iii) the strength of the peak increases with the oscillation amplitude (Fig. 5).

Thus, the comparison between theoretical predictions and experimental results leads to the conclusions that the observed relaxation is very probably pseudo Bordoni relaxation based on glide of screw dislocations on non-compact planes controlled by the Friedel-Escaig mechanism. Dislocations within very small grains are pinned at the grain boundaries and

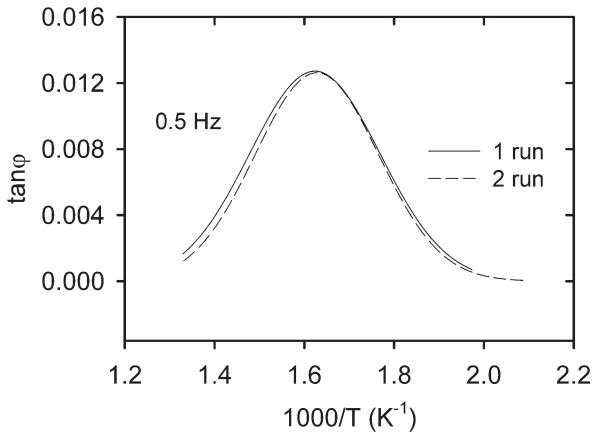

Fig. 7. Background subtracted high temperature peak after pre-straining of 2.55 %.

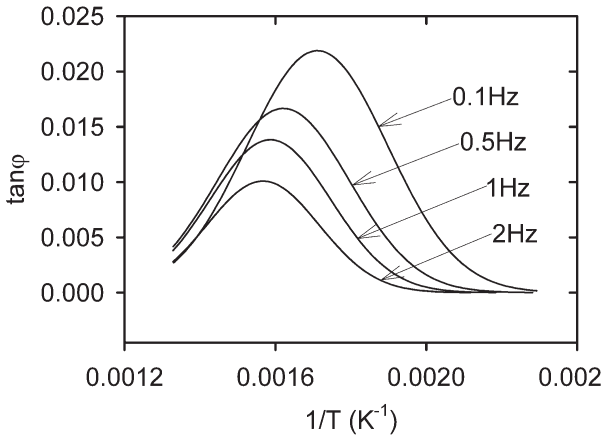

Fig. 9. Background subtracted high temperature peak obtained for various frequencies.

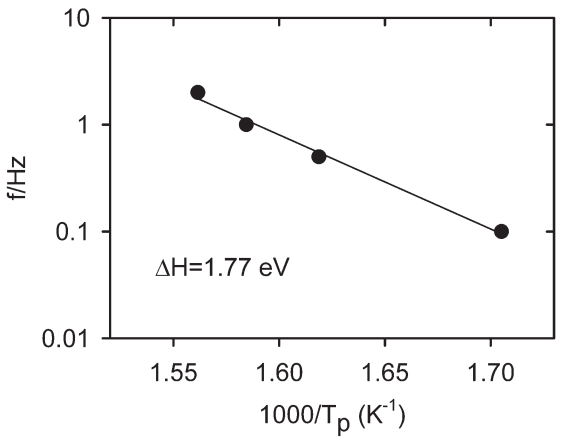

Fig. 8. Arrhenius plot obtained for the high temperature peak.

this is the reason why this dislocation population is so stable. On the other hand, newly created dislocations during cold rolling are only slightly pinned and they are recoverable during heating part of the measurement cycle.

3.2. High temperature peak

The high temperature peak has been observed at a temperature of $\sim$ 618 K (at the frequency 0.5 Hz). The cold pre-straining of the sample did not affect the strength of the high temperature peak, as it is seen from Fig. 7. The position and height of the peak are very stable during heating as well as cooling. The activation energy was obtained from the frequency dependence of the peak temperature (Arrhenius plot) to be 1.77 $\pm$ 0.05 eV (Fig. 8).

The existence of the high temperature peak in magnesium has been reported by Kê [16]. He described this peak as being related to the grain boundary relaxation. Similarly a high temperature peak has been found in Mg-4Li alloy [25].

Grain boundaries in the material with the small grain size contain a 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 [26], grain boundary dislocation segments vibrate under the 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 is not depending on the grain size:

$$(\tan\varphi)_{\max} = \frac{G\rho_{\mathrm{s}}b^2}{C}, \tag{7}$$

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. The peak height depends on frequency (Fig. 9). The relaxation time in this model is

$$\tau = \frac{kT}{b^2 C_{\mathrm{j}} D\, K} \exp\left(\frac{\Delta H_{\mathrm{j}} + \Delta H_{\mathrm{GB}}}{kT}\right) = \tau_0 \exp\left(\frac{\Delta H}{kT}\right), \tag{8}$$

where C_{j} is the linear density of jogs along the dislocation line, D is the diffusion coefficient, ΔH_{j} and 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 [27]. Comparing with the estimated activation enthalpy value of 1.77 eV, the possible jog formation energy should be approximately 0.8 eV, which seems to be a reasonable value. The high temperature peak in this study is observed at a higher temperature than that given in [16]. The surface of grains was passivated during the milling by 1 % of oxygen in protective argon atmosphere. MgO particles in the grain boundaries may influence grain boundary diffusion characteristics.

4. Conclusions

Nanocrystalline magnesium specimens were prepared by milling and hot extrusion. The grain size was estimated to be about 100–150 nm. Internal friction was measured as a function of temperature with four frequencies ranging from 0.1 to 2 Hz. Two relaxation peaks were observed: a peak at 375 K (at the frequency of 1 Hz) with activation energy of 1.16 eV, and a peak at 631 K (at the frequency of 1 Hz) with activation energy of 1.77 eV. The estimated small values of the activation volume and the peak sensitivity to the stress amplitude lead us to conclude that the low temperature peak is due to the screw dislocation motion on the prismatic plane controlled by the Friedel-Escaig mechanism. Pre-straining by the cold rolling did not influence the high-temperature peak. The grain boundary sliding is probably the reason for the appearing of this peak. A higher temperature at which the high temperature peak occurs may be caused by the existence of small MgO particles in the grain boundaries.

Acknowledgements

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