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THE INTERNAL FRICTION OF SINGLE CRYSTALS, BICRYSTALS AND POLYCRYSTALS OF PURE MAGNESIUM

TARCIE WEWNĘTRZNE MONOKRYSZTAŁÓW, BIKRYSZTAŁÓW I POLIKRYSZTAŁÓW CZYSTEGO MAGNEZU

The internal friction of magnesium single crystals, bicrystals and polycrystals has been studied between room temperature and 450°C. There is no internal friction peak in the single crystals, but a prominent relaxation peak appears at around 160°C in polycrystals. The activation energy of the peak is 1.0 eV, which is consistent with the grain boundary self-diffusion energy of Mg. Therefore, the peak in polycrystals can be attributed to grain boundary relaxation. For the three studied bicrystals, the grain boundary peak temperatures and activation energies are higher than that of polycrystals, while the peak heights are much lower. The difference between the internal friction peaks in bicrystals and polycrystals is possibly caused by the difference in the concentrations of segregated impurities in grain boundaries.

Keywords: Magnesium, single crystal, bicrystal, polycrystal, grain boundary, internal friction

Badania tarcia wewnętrznego w monokryształach, bikryształach i polikryształach magnezu przeprowadzono w zakresie temperatur między temperaturą pokojową a 450°C. W monokryształach magnezu nie występuje pik tarcia wewnętrznego, ale wyraźny pik relaksacyjny pojawia się przy około 160°C w polikryształach. Energia aktywacji piku wynosi 1,0 eV, co jest zgodne z energią autodyfuzji Mg przez granice ziaren. Z tego względu pik tarcia wewnętrznego występujący w polikryształach można przypisać relaksacji granic ziaren. W przypadku trzech badanych bikryształów temperatury pików pochodzących od granic ziaren i ich energie aktywacji są wyższe niż w przypadku polikryształów, ale wysokości tych pików są znacznie niższe. Różnica między pikami tarcia wewnętrznego w bikryształach i polikryształach jest prawdopodobnie spowodowana przez różnicę stężeń zanieczyszczeń segregujących na granicach ziaren.

1. Introduction

The grain boundary internal friction in fcc metals (such as aluminum [1-7]) has been extensively studied, while the magnesium as a hexagonal crystal was relatively less examined. One relaxation peak was first found in 99.97% polycrystalline magnesium at medium temperature (490K) and attributed to grain boundary sliding by Kê [1], similar results of Mg polycrystals were obtained by others [8-12]. On the other hand, an internal friction peak around 410K in 99.9999% Mg was considered to be induced by dislocation gliding [13]. Besides, two relaxation peaks (at 0.4 T_m and 0.67 T_m) were observed in nanocrystalline Mg [14], where the low and high temperature peaks were attributed to dislocation motion and grain boundary relaxation, respectively. In order to get more information about the grain boundary internal friction in pure Mg, internal friction spectra of Mg single crystals, bicrystals and polycrystals were investigated.

2. Experimental

The raw material used in this study was 99.99 wt.% pure Mg cast ingot, the main impurities (in wt.%) were as follows: Si, 0.0026; Zn, 0.0017; Al, 0.0008; Mn, 0.0007; Fe, 0.0005; Ni, 0.0002. Single crystals and bicrystals were grown by vertical Bridgman method. The misorientation angles of the bicrystals with [0001] symmetric tilt grain boundaries (STGB) were 9.5°, 25.0° and 38.0° ($\Sigma 7$). The polycrystalline samples were produced with a piece of bulk single crystal through hot rolling and annealing at 450°C for 1 h, and the mean grain size was about 160 μm. Internal friction specimens were cut to the same dimension of 60 mm ×4 mm ×2 mm. The grain boundary plane in the bicrystals was located at the middle of the width and aligned along the length of specimen. To prevent the possible influence of residual stresses remaining in the specimens during preparation and mounting, only the internal friction data of specimens after annealing in-situ at 450°C for 1 h were used.

The internal friction spectrum and relative dynamic modulus were measured by forced vibration method in an automatic inverted torsion pendulum under primary vacuum between room temperature and 450°C with heating/cooling rate 2°C/min. The strain amplitude was 10^{-5}, and the measured frequencies were 0.2-4.0 Hz.

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3. Results and discussion

Figure 1 shows the temperature dependence of internal friction and relative dynamic shear modulus of Mg single crystal and polycrystal. For the single crystal, there is no apparent peak in the measured temperature range and the relative modulus changes as a straight line. For the polycrystal however, there is a prominent relaxation peak around 160°C (at 1 Hz), the corresponding relative shear modulus drops dramatically around the peak temperature.

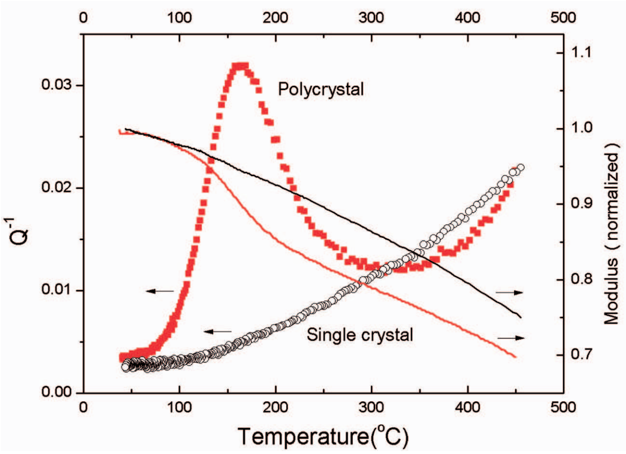

Fig. 1. Internal friction and normalized modulus of the Mg polycrystal and single crystal measured at 1.0 Hz

The peak in the polycrystals shifts to higher temperature at higher frequency, as shown in Fig. 2, indicating that the peak is a thermally activated relaxation peak [15]. After subtracting the background by a fitting program [2-7], the net internal friction peaks, and then the peak temperatures with different frequencies can be obtained. The Arrhenius plot for the polycrystal is shown in Fig. 3. The measured activation energy is 1.0 eV, and the pre-exponential factor of the relaxation time is 10^{-12} s, which are listed in Table 1.

The activation energy is consistent with that of grain boundary diffusion of Mg [16]. Therefore, the peak in the polycrystals originates from grain boundary relaxation. The present result of Mg polycrystals is consistent with that observed by Kê [1] and other authors in conventional Mg polycrystals [8,9] and fine-grained Mg polycrystals prepared by equal channel angular pressing [10,11].

TABLE 1

The peak temperature T_p (at 1 Hz), activation energy H, and pre-exponential factor of the relaxation time for the Mg polycrystal and three bicrystals

	Polycrystal	Bicrystal 9.5°	Bicrystal 25.0°	Bicrystal 38.0° (Σ7)
T_P (°C) at 1 Hz	163	254	190	210
H (eV)	1.00±0.05	2.3±0.1	1.7±0.1	2.3±0.1
lg τ_0 (s)	-12.1±1	-23.5±1	-20.6±1	-23.2±1

Figures 4, 5 and 6 show temperature dependence of internal friction and relative modulus of the three bicrystals (9.5°, 25.0° and 38.0° (Σ7)) after annealing at 450°C. Different from the polycrystal, the internal friction peak temperatures of three bicrystals are increased to the range of 190-250°C (1 Hz), and the peak heights are only at the level of 0.01, much lower than that in the polycrystal. The peaks are frequency-dependent, thus they are also thermally activated relaxation peaks. By comparing with the data of the single crystal, the peaks in the bicrystals should also be attributed to grain boundary relaxation.

The activation energies of the peaks for the three types of bicrystals were obtained from Arrhenius plots shown in Fig. 3. The relaxation parameters are listed in Table 1. The measured activation energies for the three bicrystals are about twice as that in polycrystals, while the pre-exponential factors are much smaller.

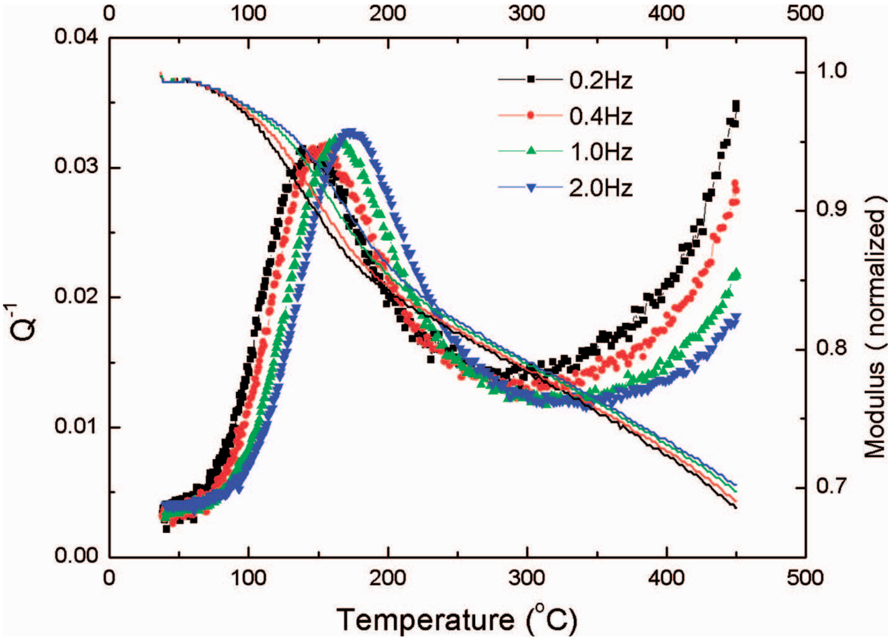

Fig. 2. Internal friction and normalized modulus of the Mg polycrystal measured at different frequencies

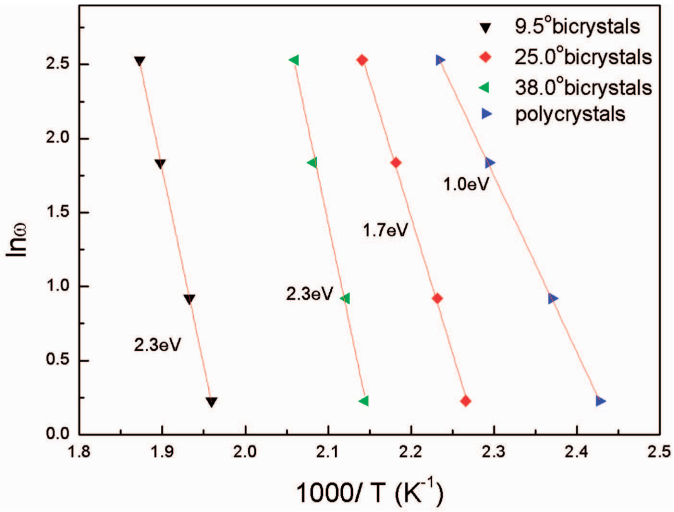

Fig. 3. Arrhenius plots of lnω versus 1000/T for the grain boundary peaks of the polycrystal, and 9.5°, 25.0°, 38.0° bicrystals, where $\omega = 2\pi\ f$ is the angular frequency

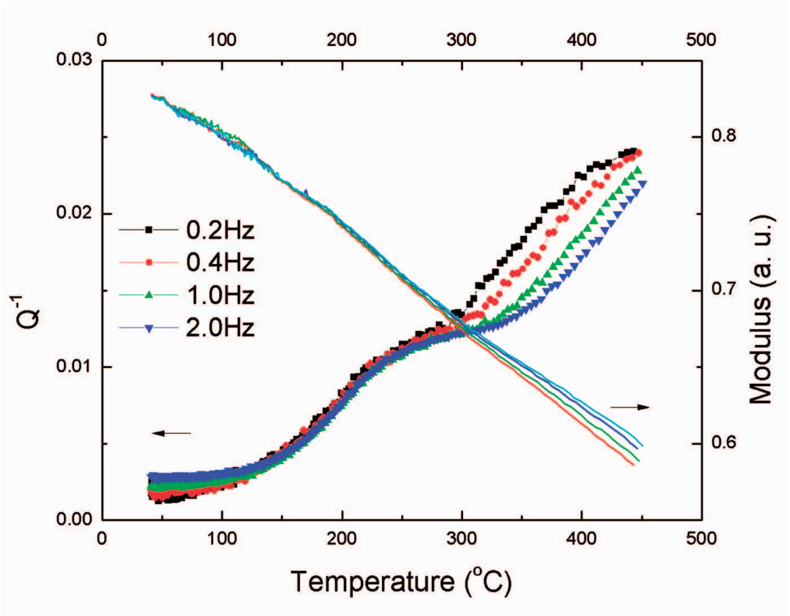

Fig. 4. Internal friction and relative modulus of pure Mg bicrystals with 9.5° [0001] symmetric tilt grain boundaries

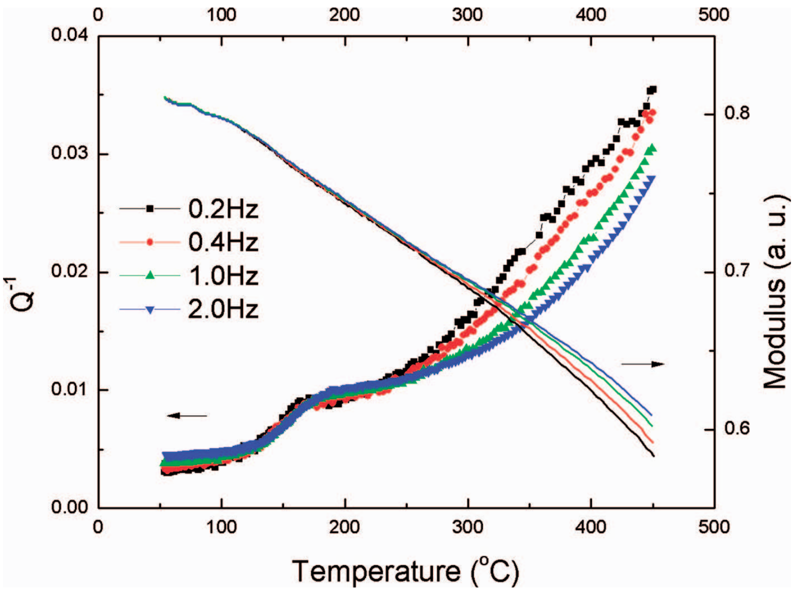

Fig. 5. Internal friction and relative modulus of pure Mg bicrystals with 25.0° [0001] symmetric tilt grain boundaries

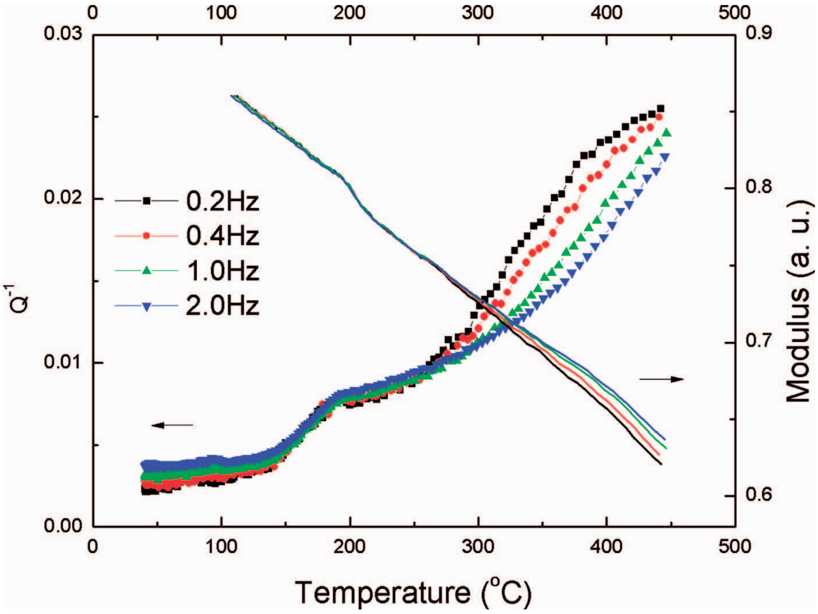

Fig. 6. Internal friction and relative modulus of pure Mg bicrystals with 38.0° [0001] symmetric tilt grain boundaries

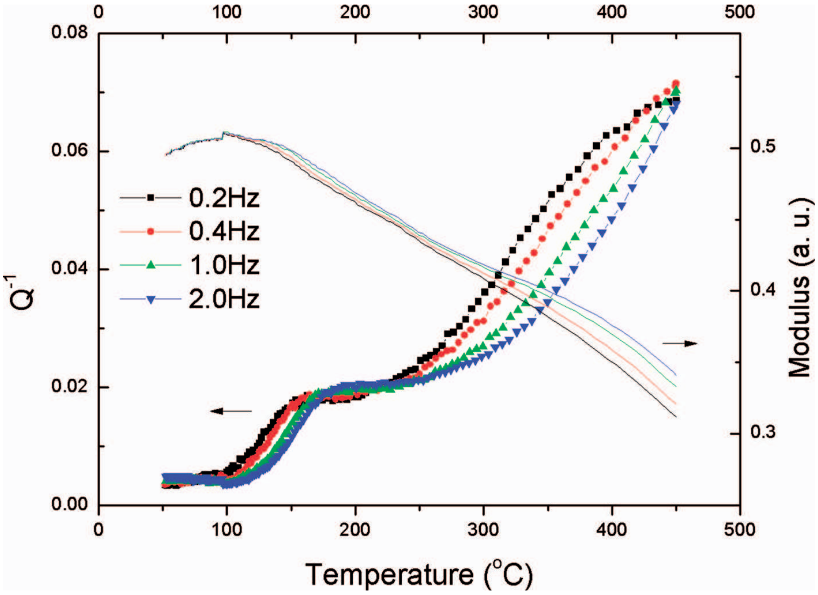

Fig. 7. Internal friction and relative modulus of pure Mg bicrystals with 25.0° [0001] grain boundaries. The specimen dimensions are 60 mm ×2 mm×2 mm, so the grain boundary density of the bicrystal is twice as that of the bicrystal in Fig. 5

Figure 7 shows the results of the 25° bicrystal with dimensions of 60 mm×2 mm×2 mm. In Figures 1-6, the dimensions of all the specimens are 60 mm×4 mm×2 mm, so the grain boundary density (i.e. grain boundary area in unit volume of specimen) of the bicrystal in Fig. 7 is twice as that of the bicrystal in Fig. 5. Comparing Fig. 7 and Fig. 5, we can see that the peak height of the former is about twice as that of the latter. Moreover, the peak shifting with frequency can be better distinguished in Fig. 7. The activation parameters obtained from the data of Fig. 7 and Fig. 5 are identical, indicating that the peak mechanism does not change with the variation of specimen dimension (or grain boundary density). These results are consistent with the work on Al bicrystals [6] and confirm that the observed peak in Mg bicrystals originates from grain boundary.

The difference between the peaks in the bicrystals and polycrystals may be attributed to the difference in the concentration of segregated impurities. Since the total grain boundary area in the polycrystals is several orders higher than that in the bicrystals, the concentration of segregated impurities at grain boundaries in polycrystals will be much less than that in the bicrystals. Hence, the peak in the polycrystals can be considered as a "pure" grain boundary peak, while the peaks in the bicrystals are an "impurity" grain boundary peak. The unexpected relaxation parameters (high activation energy H and small pre-exponential factor τ_0) in the bicrystals may be related to the segregated impurities and their coupling effect [3,4,17-20].

It should be noted that in the high temperature range of internal friction spectra of the three bicrystals, a very weak peak appears around 400°C superposed on the high temperature background. This peak seems to appear only at very low frequencies (or might be shifted out of the measuring temperature range for higher excitation frequencies). Consequently the relaxation parameters of the peak cannot be estimated. The origin of the peak superposed on the high temperature background should be further examined.

4. Summary

There is no internal friction peak in the Mg single crystals, but a prominent relaxation peak appears around 160°C (at 1 Hz) in the Mg polycrystals. The peak in the polycrystals originates from grain boundary relaxation. For the three bicrystals studied here, the grain boundary peak temperatures and activation energies are higher than that of polycrystals, while the peak heights are much lower. The difference between the internal friction peaks in bicrystals and polycrystals is possibly caused by the difference in the concentrations of segregated impurities in grain boundaries.

Acknowledgements

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