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PROBING OF POLYCRYSTALLINE MAGNESIUM AT ULTRASONIC FREQUENCIES BY MECHANICAL SPECTROSCOPY

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Abstract. In the present work, using the method of a composite piezoelectric oscillator at a frequency of 99-102 kHz, polycrystalline magnesium was studied under two types of treatment: deformation and subsequent annealing. The influence of these types of treatments on changes in the dislocation density and such an important structurally sensitive parameter as the dynamic Young's modulus has been established. The values of micro-yield stress are determined. With a longitudinal deformation of 1.7%, at the strain amplitude of 5×10^{-6} , its value decreased from 2.9 MPa to 1.7 MPa; after annealing at 400 °C, it increased to 5.8 MPa.
Keywords: Magnesium, mechanical spectroscopy, dislocation density, Young's modulus, internal friction, microplastic deformation

1. Introduction

Magnesium (Mg) and alloys based on it are widely used in the aerospace industry, shipbuilding and mechanical engineering due to its low weight, specific strength and environmental friendliness [1,2]. The low density of magnesium alloys has motivated the development of structural Mg alloys for automobiles and aerial vehicle, where weight reduction is needed to achieve high fuel efficiency [3]. In recent years, there has been a surge of interest in magnesium and its alloys as revolutionary materials for biomedical applications [4]. However, the poor plastic properties of Mg-based materials limit their applications [5]. At present, the development of new and improvement of existing Mg-based alloys does not stop [6,7,8]. For example, in 2001, rapidly solidified Mg–Zn–Y alloys were found to have excellent mechanical properties, including maximum tensile yield strength of ~600 MPa and elongation of ~5% at room temperature [7,8].

One of significant aspects of the properties of structural alloys is the study of the influence of microdeformations. Multiple microdeformations during the operation of the material lead to fatigue, wear, the formation of microcracks, and finally the fracture of samples or constructions [9]. The formation of new dislocations in the structure of the material lead to increase of internal stresses, which are the main reason for the degradation of materials strength properties. Knowledge of changes in the density of dislocations can be very useful in the practical applications due to the direct connection of this change with the fracture and wear of materials [10]. In this paper, we present the results of investigation of elastic and anelastic properties of pure polycrystalline magnesium using mechanical spectroscopy. The aim of our work is to study the changes in such a structurally sensitive parameter as the dynamic Young's modulus (YM) and mechanical damping or internal friction (IF) under deformation and annealing of magnesium.

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2. Method and samples

Polycrystalline magnesium samples with purity 99.99% in the shape of rectangular bars with characteristic dimensions of 2.5x2.5x24.5 mm were fabricated by casting. To study the microplastic behavior, the samples were passed through three processing steps: i) 1.7% longitudinal deformation carried out using a press ii) annealing at 200°C in vacuum iii) annealing at 400°C in vacuum. The measurements were taken before these processing steps and after each of them. The grain size in the experimental samples remained in the range from 10 to 100 μm.

All measurements were conducted using the composite piezoelectric oscillator technique at a oscillatory frequency about 100 kHz [11]. The size of the samples was specially selected to ensure resonance of longitudinal oscillation. The temperature range used in the experiments was from 80 to 300 K. The low temperature range is optimal for studying dislocation motions since the contribution of thermal vibrations of the lattice decreases. In this range, the temperature dependencies of oscillation frequency and the IF were measured in the sample at a fixed oscillatory strain amplitude 10^{-5}. Along with this, the amplitude dependence of IF was recorded at different temperatures and oscillation frequencies at constant temperature. Young’s modulus was determined using the resonant frequency of the sample:

$$E = 4\rho l^2 f^2, \quad (1)$$

where f is oscillatory frequency, ρ is density of magnesium, l is length of the sample.

The frequency change in these experiments was approximately in 99-102 kHz range. Internal friction was determined by the change in voltage on the quartz transducer [11].

3. Results

Figure 1 shows the temperature dependencies of YM and IF as a result of heating; in general, there is a tendency for YM and IF to decrease with increasing temperature. The dependencies of internal friction at low temperatures after deformation and after annealing at 200°C show the formation of a Bordoni relaxation peak close to 80 K [10], Fig. 1b. The value of Young's modulus over the entire range decreases by about 1.5%, as it is shown in Fig. 1a. To verify the adequacy of the obtained Young's modulus values, we used the traditional method of estimation of the elastic constants for polycrystals by Voigt – Reuss – Hill averaging [13]. Room temperature Young's modulus found with such averaging for magnesium is 43.2 GPa, which is close to our values determined before deformation and after annealing. Therefore, this indicates on the isotropy of the investigated Mg polycrystalline samples, which also means that the effects of texturing can be neglected [13].

Figure 2 demonstrates temperature spectra of IF on cooling and subsequent heating before deformation. The amplitude dependencies were measured during cooling at fixed temperatures, Fig. 3, and during heating the continuous temperature spectra were obtained (Fig. 1b). Places where the data for amplitude dependencies were taken can be seen from the curve discontinuities during the cooling processes, see Fig. 2. It also important to note, that after each event of measuring amplitude dependence (Fig. 3) the level of IF sharply increases (Fig. 2), which may be associated with an increase in the density of dislocations after each act of loading. The heating process does not lead to stress relaxation, and the heating curve goes higher than spectrum for cooling.

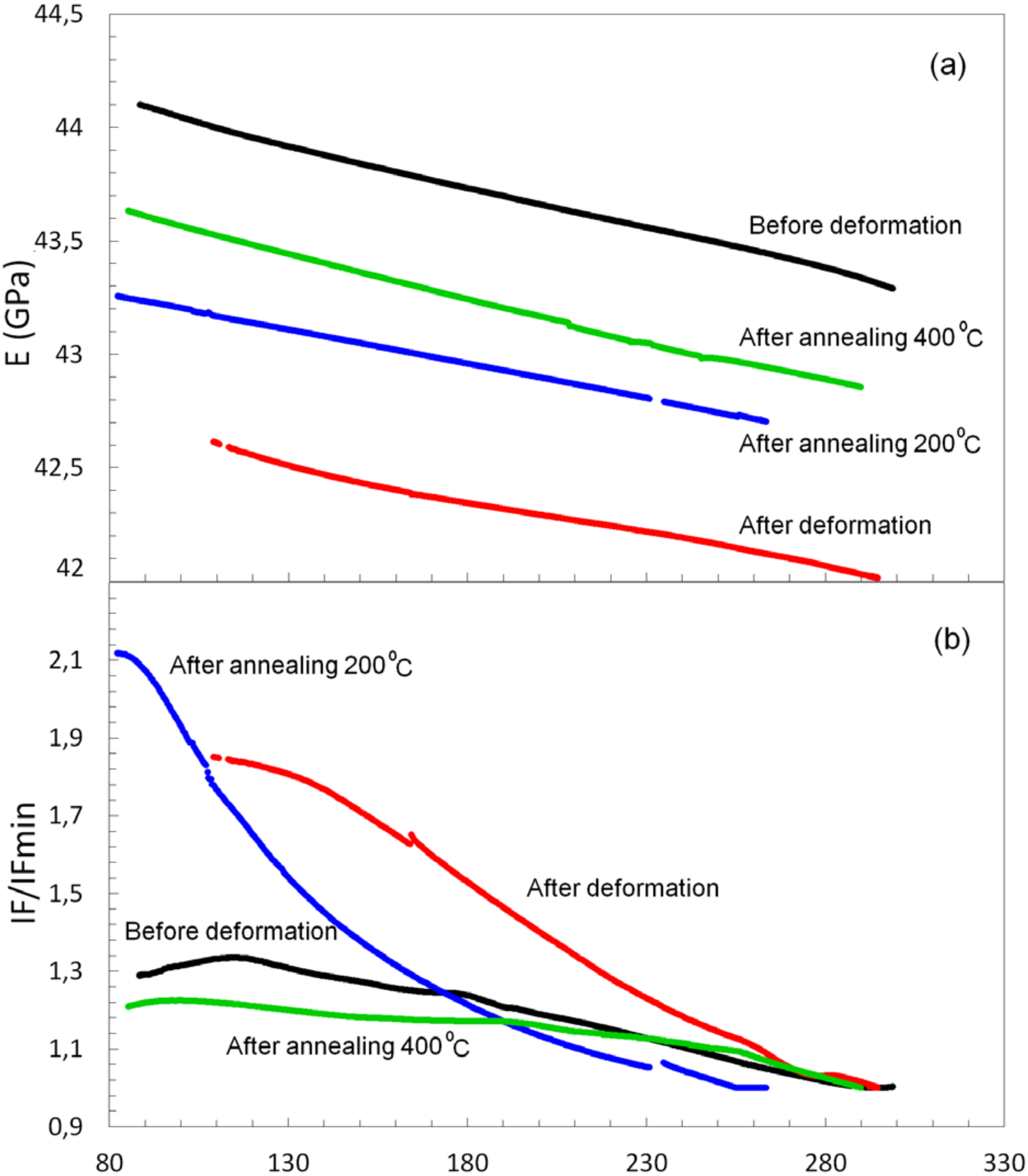

Fig. 1. Temperature dependencies of the dynamic Young's modulus (a) and normalized internal friction (b) for differently treated samples: — before deformation, — after deformation, — after annealing 200ºC, — after annealing 400ºC; oscillatory strain amplitude is 10^{-5}

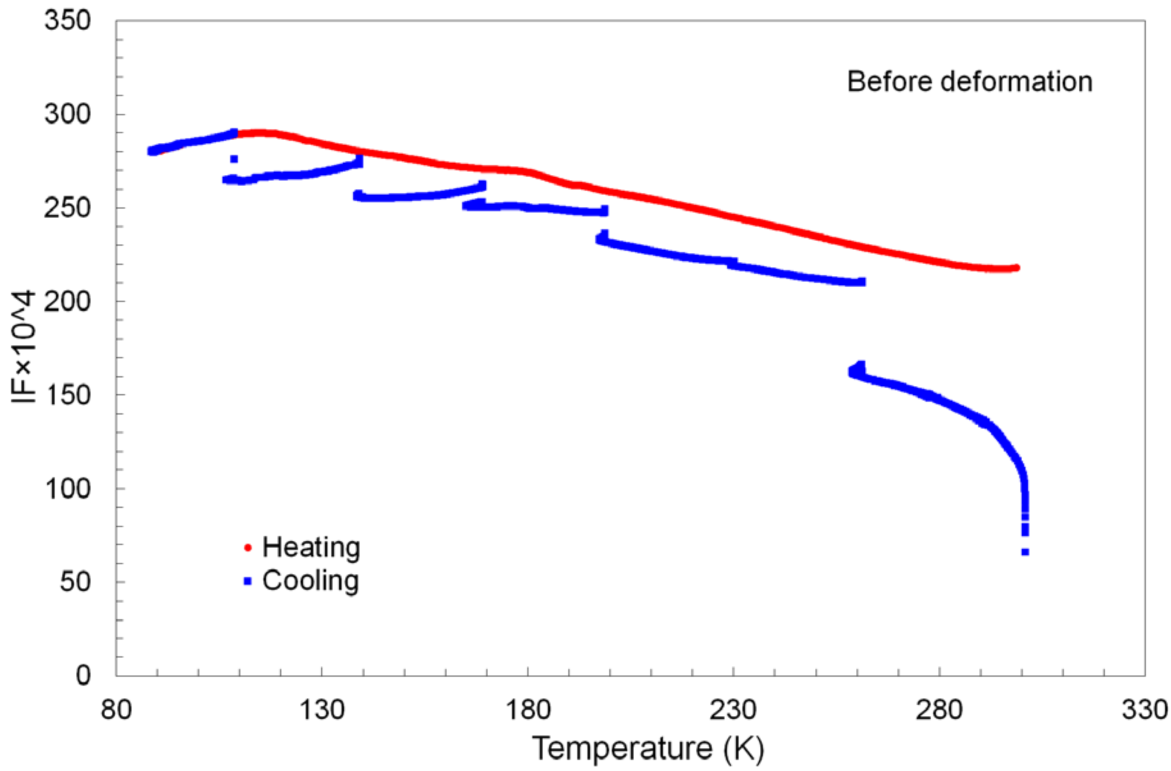

Fig. 2. Temperature dependence of internal friction in the cooling-heating cycle before deformation: — heating; — cooling; oscillatory strain amplitude is 10^{-5}

Figure 3 shows the amplitude dependences of the internal friction at various temperatures divided into 3 groups. For the first group the measurements were conducted before deformation of the sample, for the second – after deformation, and for the third – after deformation and deep annealing. Each individual curve consists of two parts: amplitude-independent internal friction (AIIF), where a change of the oscillatory strain amplitude is not accompanied by a change in the IF, (up to about 10^{-7}) and amplitude-dependent internal friction (ADIF), where a change in the strain amplitude is accompanied by a sharp change in internal friction (after about 10^{-7}) [14]. The amplitude dependences before deformation and after deep annealing are characterized by a pronounced increase in ADIF. At the same time, one can notice an increase in the hysteresis value (the difference between the forward and backward running) for these curves.

Our measurements also allowed us to evaluate the mechanical (or microplastic) properties of magnesium using stress – strain diagram. We explored the algorithm to calculate microplastic deformation as it was proposed in [15]. To estimate the contribution of microplastic deformation, we plotted the dependence of microplastic deformation versus anelastic strain amplitude for three cases: before deformation; after deformation and after deep annealing, Fig. 4.

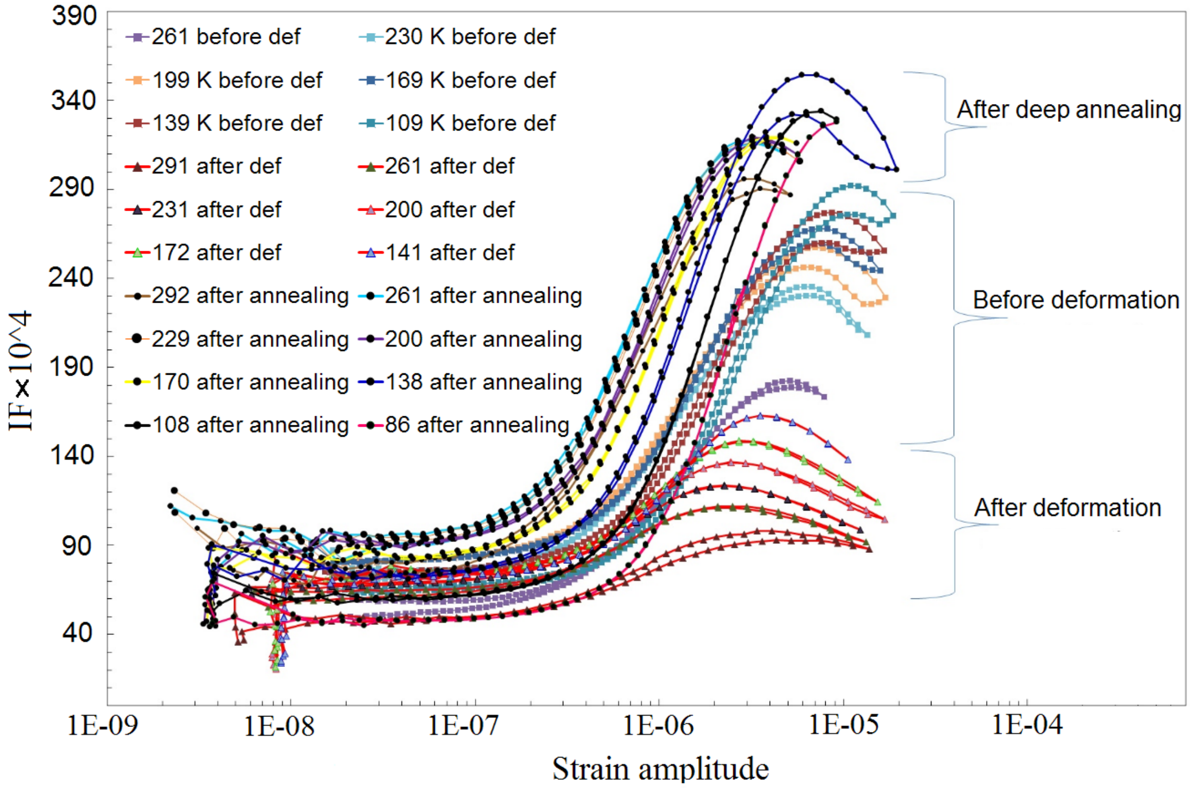

Fig. 3. Internal friction as function of the oscillatory strain amplitude at different fixed temperatures under different treatments of polycrystalline magnesium: before deformation; after deformation; after deep annealing

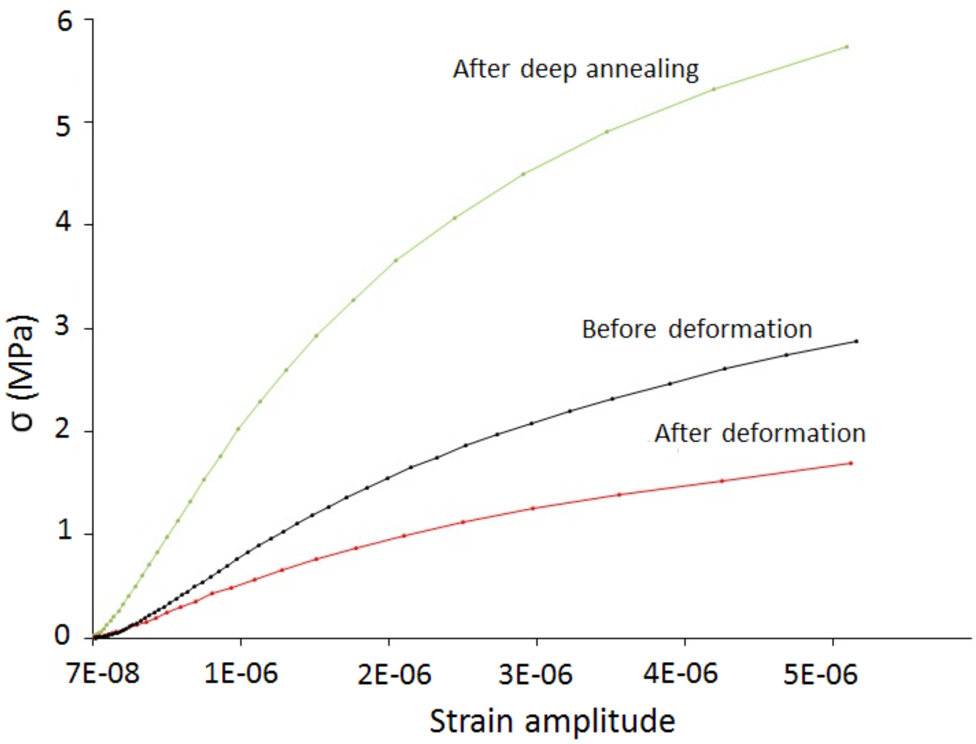

Fig. 4. Microplastic deformation diagram for different treatments of magnesium polycrystal: — before deformation; — after deformation; — after deep annealing

4. Discussion

A change in the dislocation density of the material directly affects such important structurally sensitive parameters as the dynamic Young's modulus and mechanical damping: plastic deformation produces additional (fresh) dislocations and results in YM decreasing and the increasing of IF. This effect was previously observed on numerous crystalline materials [12,13,16]. In this paper we show that the reduction of Young's modulus after deformation of 1.7% is accompanied by a thermally activated Bordoni peak, as it follows from Fig. 1. The basis of this relaxation process is the formation and movement of thermal or geometric kinks at edge, screw and mixed dislocations [17]. The changes in the elastic modulus under different treatment are clearly seen in Fig. 1a. A drop in YM during deformation is caused by a raise of the dislocation density in the material. After annealing the structure relaxes and this process is accompanied by a decrease in the dislocation density and in the height of the Bordoni peak until its complete disappearance, and at the same time in a raise in the Young's modulus, see Fig. 1. The dynamic elastic modulus clearly raises with the annealing temperature increase; this can be explained by the diffusion of point defects to dislocations hindering their movement and annealing of the dislocations themselves [18,19]. It is also worth noting the recovery of the curvature of the elastic modulus graph, Fig. 1a, after annealing at 400°C; this indirectly reflects the state of equilibrium of the structure [11]. Investigation of amplitude dependences of IF after different treatments of magnesium is a clear evidence of a change in the dislocation structure, Fig. 3. These ADIF dependences relate on the formation of the microplastic regions, in which the damping raises as a consequence of dislocation multiplication [13]. The motion of dislocations in some cases is controlled by point defects [18,19]. Figure 3 shows that the deformation of the sample is accompanied by a sharp drop of the amplitude-dependent part of IF and appearance of strain hysteresis, which is connected with partial blocking of the dislocation movement. The magnitude of microplastic deformation at various amplitudes can be seen from Fig. 4. An increase in the dislocation density leads to blocking the propagation of dislocations, thereby reducing the magnitude of microplastic deformation. With a strain amplitude about 5×10^{-6} the values of micro-yield stress drop from 2.9 MPa to 1.7 MPa, and after annealing it increases by more than 3 times reaching the level of 5.8 MPa.

5. Conclusions

We studied the evolution of dislocation density of a polycrystalline magnesium under plastic deformation and annealing by mechanical spectroscopy at a frequency about 100 kHz. It was found that micro-yield stress caused by the movement of dislocations is reduced by 1.2 MPa at a strain amplitude of 5×10^{-6} after longitudinal sample deformation of 1.7% due to the increased dislocation density inside the polycrystal. After annealing the value of micro-yield stress increased by more than 3 times, which indicates a decrease in stresses inside the structure due to both the diffusion of point defects to dislocations and the annealing of the dislocations themselves. At the same time these processes are confirmed by a pronounced changes in the dynamic elastic modulus.

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