Effect of ball-milling parameter on mechanical and damping properties of sintered Mg-Zr alloy

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Abstract: Effects of ball-milling parameter on structures and properties of sintered Mg-1.5Zr (mass fraction, %) alloy were researched by metallographic analysis, mechanical properties tests and DMA technology. The results indicate that with 310 r/min rotation speed, the microstructure of the sintered alloy is greatly refined, and Zr-phase distributes uniformly. The micro-hardness, bending strength and damping capacities are the greatest under 310 r/min rotation speed. The damping peak of sintered Mg-1.5Zr alloy increases with increasing frequency under the testing conditions. The relaxation time meets the Arrhenius relationship, and shows the characteristics of relaxation damping.
Key words: Mg-Zr alloy; ball-milling parameter; damping capacities

1 Introduction

Damping capacity is a critically important material property from the viewpoint of vibration suppression, noise control and instrument stability enhancement in automotive, architectural and aerospace industry. Thus, special interest is paid to search for new materials simultaneously exhibiting good mechanical properties and high damping capacity[1]. Among all commercial metallic materials, pure magnesium exhibits a high damping capacity, while its poor mechanical properties prevent its potential applications. During the past decades, considerable efforts for the development of high-damping Mg alloys have been devoted in improving mechanical properties by adding strengthening particles or alloying elements[2−6].

Since K1X1-F (containing 0.6%Zr, mass fraction) and its improved K1-A damping alloys appeared in the last century, they have been still the most widely used Mg-based damping alloys for their outstanding damping capacity and casting properties[6]. As an important alloying element, Zr addition not only refines the grain of magnesium alloy, but also improves damping capacities because of the strong interaction of Zr atom with the dislocation. Preparation of Mg-Zr damping alloys is currently focused on the casting method, and the addition of Zr in the magnesium alloy is limited because of its very low solubility in Mg, thus, the development of high-damping Mg-Zr alloys is mainly focused on adding other elements such as Y and Ca, and reports on further addition of more Zr contents to the alloys are rare[7−10]. Recently, powder metallurgy (P/M) is found to be a promising method to develop high performance Mg alloys[11]. In this work, Mg-1.5Zr alloys were fabricated by mechanical alloying (MA) and P/M method. The effect of ball-milling rotation speed on microstructure, mechanical and damping properties of the sintered Mg-1.5Zr (mass fraction, %) alloys obtained were evaluated.

2 Experimental

2.1 Sample preparation

In the experiment, magnesium and zirconium powder with a purity of 99.0% and the average particle size of 74 μm were mixed to obtain Mg-1.5Zr alloy

powder, and the ball milling processing parameters are as follows: ball-to-power mass ratio of 5:1, milling time of 10 h, rotation speed of 280, 310 and 350 r/min, respectively. The sample was pressed at the DY−30 pressing machine with self-made two-way suppression dies with the size of 6.5 mm × 6.5 mm × 39.0 mm. Samples were sintered in the crucible electrical resistance furnace of 7.5 kW under SF_6+CO_2 atmosphere protection, at sintering temperature of 550 °C for 5 h.

2.2 Testing method

Corroded by 4% nitric acid alcohol, sintered samples were used to observe and analyze the microstructure by an ordinary optical microscope. Hardness of the sample was tested using the HXS−1000A micro-vickers hardness tester (2 N, 15 s). Each sample was tested 5 times and then the average value was taken. Bending strength was tested by a universal material testing machine according to GB/T 6569−1986 test method with the specimen size of 6.5 mm × 6.5 mm × 39 mm. The density was measured using Archimedes method.

The damping capacity of the specimen was measured using a NETZSCH 242C DMA (dynamic mechanical analyzer) in single cantilever measurement mode, and evaluated by the loss tangent (tan φ). The size of the testing specimen was 35 mm × 5 mm × 1 mm, with the tolerance of ±0.05 mm. The testing conditions were: the heating rate of 5 °C/min, the temperature ranging from 27 to 275 °C, the frequency of 5 Hz and the amplitude of 6×10^{-5} m. The damping−temperature curves of sintered Mg-1.5Zr alloy were also measured under different frequencies (1, 5, 10 and 20 Hz).

3 Results and discussion

3.1 Microstructure observation

Under conditions of 10 h milling time and 5:1 ball-to-power mass ratio, the microstructures of sintered samples for different milling rotation speeds are shown in Fig.1. Light-colored parts are Mg phase, while dark-colored parts are Zr-phase or porosity, and the Zr phase distributes in the grain and at grain boundary.

The ball-milling rotation speed has an obvious effect on the size, shape and distribution of Zr particles. With the ball-milling rotation speed of 280 r/min, the alloy has a coarse microstructure with Zr-phase distributing unevenly and many porosity (black block)[12]. When the milling speed is increased to 310 r/min, the microstructure of the sintered alloy is greatly refined, and the Zr particles distribute uniformly with very little porosity. When the milling speed reaches 350 r/min, the microstructure of the alloy becomes coarse again, but it has a uniform grain size and little porosity.
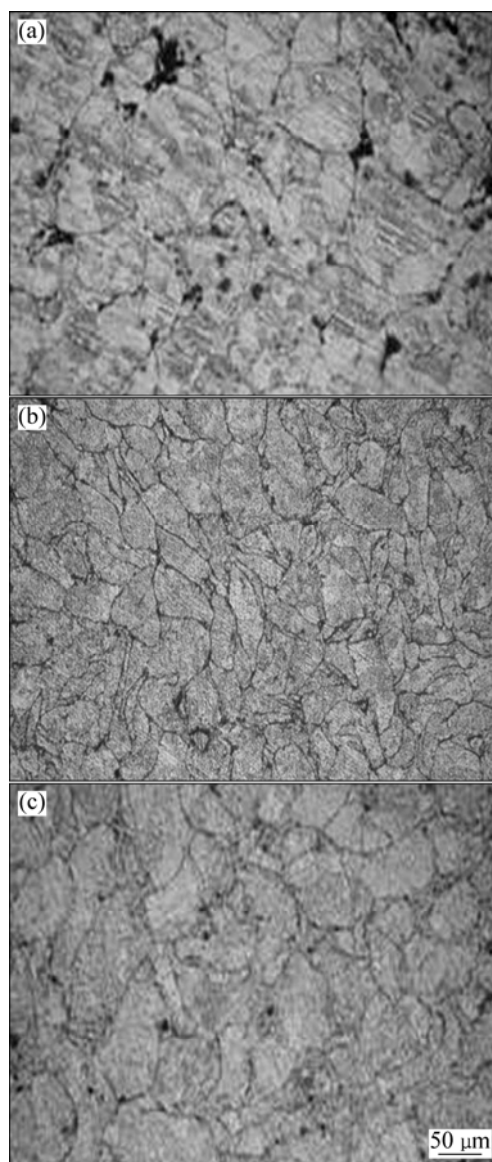

Fig.1 Microstructures of sintered Mg-1.5Zr alloys under different milling rotation speeds: (a) 280 r/min; (b) 310 r/min; (c) 350 r/min

3.2 Mechanical properties

Table 1 lists mechanical properties and density of sintered Mg-1.5Zr alloys with different rotation speeds. It can be seen that both microhardness and bending strength increase with the increase of ball-milling rotation speed. Under the same ball-to-power mass ratio and milling time, the number of collisions between ball and ball, ball and powder increases with increasing rotation speed, which is beneficial to refining powder, and finally to refining the microstructure of sintered alloys. The microhardness, bending strength and density of the sintered alloys under the speed of 310 r/min are higher than those of other alloys, indicating that the increase of strength and hardness of the sample is related to the density, grain refinement, solid solution strengthening and so on[13].

Table 1 Mechanical properties and densities of Mg-1.5Zr sintered alloy at different milling rotation speeds

Rotation speed/ (r·min^{-1})	Microhardness (HV)	Bending strength, σ_{bb}/MPa	Density/ (g·cm^{-3})
280	44.09	176.94	1.708 7
310	46.73	222.63	1.743 0
350	45.63	182.94	1.709 7

3.3 Damping capacities of alloys

Fig.2 shows the temperature dependence of damping capacity of the sintered alloys under conditions of frequency of 5 Hz and amplitude of 6×10^{-5} m. The damping capacity of the sintered alloy is the best with the rotation speed of 310 r/min. The grain is small and the size is uniform at the rotation speed of 310 r/min, and the area of grain boundary increases significantly, so the interface damping, which is caused by the interface viscous sliding, increases obviously. Moreover, it can also be seen that the damping of the three alloys all changes slightly with increasing temperature below 150 °C, and then increases rapidly with increasing temperature, and the damping peak appears at about 220 °C; afterwards, the damping capacity changes gently with the temperature. This is because temperature affects the average concentration of impurity atoms on the dislocation line, and the length of dislocation line increases with increasing temperature, resulting in increased damping capacity[3, 14]. In addition, the increase of temperature makes it easy for the dislocation line pin off from the pinning points, which is another reason for the increased damping capacity of the alloy.
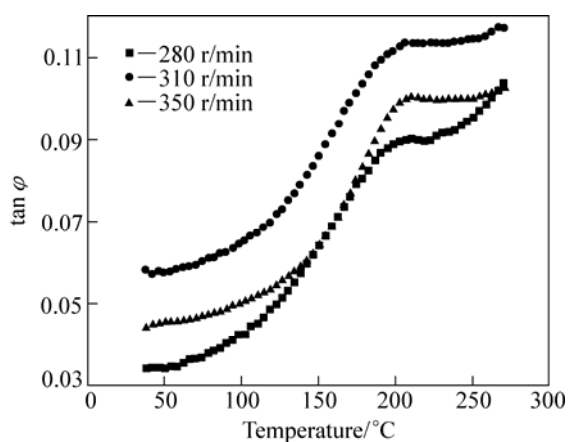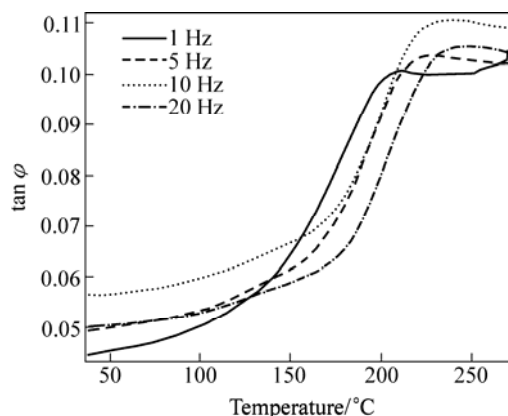

Fig.2 Evolution of damping capacity with temperature of sintered Mg-1.5Zr alloy at different rotation speeds

Fig.3 shows damping capacity versus temperature of sintered Mg-1.5Zr alloys under different frequencies. The sample was prepared under conditions of rotation speed of 310 r/min, milling time of 10 h and ball-to-power mass ratio of 5:1. With the increase of

frequency, the temperature of damping peak increases from 212.1 °C (1 Hz) to 252.2 °C (20 Hz), which means that the damping peak moves to high-temperature. According to the relationship between damping peak temperature and frequency, and the fact that the relaxation time meets Arrhenius relationship, the internal friction peak should be a relaxation peak. The internal friction peak is related to thermal activation process, which is different from resonant damping peak generated by G-L theory based on the model of string vibration. In addition, the damping−temperature curves of sintered Mg-1.5Zr alloy under other rotation speeds show the same characteristics of the relaxation damping.

Fig.3 Damping capacity versus temperature of sintered Mg-1.5Zr alloy under different frequencies

3.4 Damping mechanism

Internal friction is determined by the product of sliding distance along the grain boundary and sliding resistance. At low temperatures, the viscosity sliding distance along the grain boundary is small, so the internal friction is very little. At high temperatures, the sliding resistance along the grain boundary is small, so the internal friction is very little, too. Only in a middle temperature range, when both the sliding distance and sliding resistance are not too small, the internal friction can attain its maximum[15−16]. This would explain why the damping peak only appears at a middle temperature.

GE[15] proposed the sliding model on the mechanism of internal friction peak on grain boundary. It is considered that there are a lot of disordering groups on grain boundary, and the atoms slide under stress, but the atoms sliding will gradually slow down due to the locked effect of the grain boundary, which results in strain relaxation. At the same time, LEAK put forward the model of grain boundary movement. On grain boundaries, due to the existence of alloying atoms, the diffusion of atoms are affected by the contiguous alloying atoms, and the activation energy is changed, thus, the damping peak appears.

The internal friction of alloys is primarily due to the

following three aspects: the first is the dislocation-based damping, the second is the interface damping at high temperatures, and the third is the microstructure defects damping, which mainly refers to the internal friction caused by irreversible motion of defects (porosity, fine cracks) in alloys. For the thermal activation relaxation process, relaxation time meets Arrhenius relationship:

$$\tau = \tau_0 \exp[H/(rT)] \tag{1}$$

where τ_0 is the pre-factor, H is the activation energy and r is the Boltzmann constant.

Relaxation time of damping peak meets

$$\omega\tau_p = 1 \tag{2}$$

where ω ($\omega = 2\pi f$) is the angular frequency and τ_p is the relaxation time of damping peak.

According to damping capacity versus temperature of sintered Mg-1.5Zr alloy at different frequencies above, the fitting curve of $\ln(2\pi f)$ with T_p^{-1} is shown in Fig.4. The results show that the relaxation time τ_p meets Arrhenius relationship, which further validates that the damping peak is the relaxation process of thermal activation. It can be calculated from the slope of fitting curve in Fig.4 that $H=1.42$ eV, which is slightly higher than grain boundary activation energy of 1.38 eV of the pure Mg. This may be due to the segregation of Zr atoms in the Mg-Zr alloy grain boundary. Zr atoms segregation increases the sliding resistance of grain boundary and finally makes the relaxation activation energy of grain boundary increase.
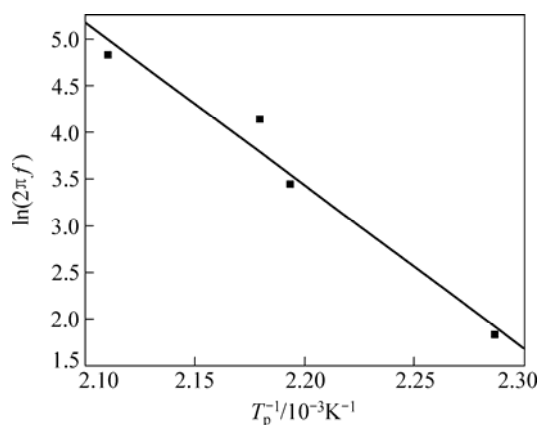

Fig.4 Fitting curve of $\ln(2\pi f)$ with T_p^{-1}

4 Conclusions

1) The damping Mg-1.5Zr alloys were prepared by mechanical alloying and powder sintering method. When the ball-milling rotation speed is 310 r/min, the mechanical properties and damping capacities are the best, which should be ascribed to higher dislocation density and refinement of grain size.

2) Internal friction of the alloy is mainly attributed to dislocation-based damping, interface damping at high temperature and micro-crystal structure defects damping. Damping peak of sintered Mg-1.5Zr alloy increases with increasing frequency under the testing conditions. The relaxation time meets Arrhenius relationship, and shows the characteristics of relaxation damping.

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