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EFFECT OF ZrO_2 ADDITIONS ON FABRICATION OF ZrO_2/Mg COMPOSITES VIA FRICTION-STIR PROCESSING

VPLIV DODATKA ZrO_2 NA IZDELAVO ZrO_2/Mg KOMPOZITOV Z VRTILNO-TORNIM POSTOPKOM

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ZrO_2/Mg composites were prepared with friction-stir processing (FSP) using different ZrO_2 amounts. The effects of FSP on the microstructure, mechanical properties and damping capacities of ZrO_2/Mg composites are discussed in this paper. After friction-stir processing, the central region of the stir zone of a ZrO_2/Mg composite obtained fine, uniform, equiaxed crystal grains. An addition of ZrO_2 can obviously refine the grains and improve the microhardness and mechanical properties of ZrO_2/Mg composites. The ZrO_2/Mg composites prepared with friction-stir processing have better damping capacities. During a strain-damping test, the damping behavior of ZrO_2/Mg composites follows the Granato-Lücke dislocation damping theory (G-L theory). The ZrO_2/Mg composites with a groove width of 0.8 mm had the best damping capacities.

Keywords: ZrO_2/Mg composites, friction-stir processing, damping capacity, G-L theory

Avtorji so pripravili ZrO_2/Mg kompozite z vrtilno tornim postopkom (FSP, angl.: friction stir processing) z različno vsebnostjo ZrO_2. V članku opisujejo vpliv FSP postopka na mikrostrukturo, mehanske lastnosti in sposobnost za dušenje izdelanih ZrO_2/Mg kompozitov. Po izvedenem FSP postopku je v centralnem delu ZrO_2/Mg kompozitov (v coni mešanja oz. vrtenja) nastalo homogeno področje drobnih enakoosnih kristalnih zrn. Dodatek ZrO_2 je očitno udrobnil mikrostrukturo kompozita, zvišal mikrotrdoto in izboljšal ostale mehanske lastnosti ZrO_2/Mg kompozitov. Izdelani FSP ZrO_2/Mg kompoziti imajo tudi boljše sposobnost dušenja. Deformacijsko-dušilni preizkus je pokazal, da se karakteristika dušenja izdelanih ZrO_2/Mg kompozitov dobro ujema z Granato–Lücke dislokacijsko teorijo dušenja (G-L teorija). Kompoziti ZrO_2/Mg s širino brazde 0,8 mm so imeli najboljšo sposobnost dušenja.

Ključne besede: ZrO_2/Mg kompoziti, vrtilno-torni proces, sposobnost za mehansko dušenje, G-L teorija

1 INTRODUCTION

Magnesium and magnesium alloys are widely used because of the advantages of their low density, high specific strength, high damping capacity, noise reduction and so on.[1–6] Pure magnesium has the best damping capacity, but poor mechanical properties, such as low hardness, low yield strength and tensile strength, which limit the range of its application.[7–8] Several efforts to improve its mechanical properties through various severe-plastic-deformation (SPD) methods have been made since 1990s.[9] On the basis of experimental evidences, it is clearly believed that SPD could lead to a significant grain refinement as well as a profound influence on the precipitation processes.[10–11] Friction-stir processing (FSP) is a solid-state SPD technique, invented by The Welding Institute (TWI) in 1991.[12] The effects of FSP on the microstructural evolution and mechanical-property modification of various materials, in particular, magnesium alloys have been thoroughly investigated.[13–15]

The change of the grain size and grain orientation during friction-stir processing is an important factor affecting the damping performance of magnesium alloys. Therefore, friction-stir processing provides a great possibility to resolve the contradiction between the mechanical properties and damping performance of magnesium alloys.[16–20]

ZrO_2 particles have a high melting point and their doping to an alloy can inhibit the grain growth. When the alloy is plastically deformed, ZrO_2 particles are uniformly distributed along the grain boundaries, impeding the movement of dislocations and bending the dislocations deformed around them, promoting the formation of a concentrated deformation zone. So ZrO_2 particles can be used as a reinforcing phase to improve the hardness and elongation of a material. Liu et al.[21] added M-ZrO_2 and T-ZrO_2 particles into the AZ31 magnesium alloy to obtain ZrO_2/Mg composites with friction-stir processing, and the microstructure and mechanical properties of the composite was studied.

In this paper, ZrO_2/Mg composites were prepared with friction-stir processing. The effects of ZrO_2 amounts on the microstructure, mechanical properties

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and damping properties of ZrO_2/Mg composites were investigated.

2 MATERIALS AND METHODS

The material used in this study was the AZ31 magnesium alloy in the hot-rolled state with a composition of (in *w*/%): Al–2.54, Zn–0.943, Mn–0.332, Si–0.0165 and the Mg balance. The ZrO_2-reinforced particles exhibit monoclinic crystals with a particle size of 20–50 nm.

The base material was cut into a specimen of (300 × 160 × 4) mm. A groove with a depth of 2.5 mm and widths of (0.6, 0.8, and 1.0) mm was machined along its centerline. The shoulder diameter, pin diameter and pin height were (16.0, 4.0 and 3.8) mm, respectively. The FSP tool was rotated at 1500 min^{-1} in the clock-wise direction. The traverse speed was 50 mm/min. The tilted angle was 2.5°.

For an optical examination, samples were sectioned, cold mounted, polished with the silicon dioxide paste with a grain size of 1 μm and finally etched in a solution of oxalic acid (2 g), nitric acid (2 mL) and distilled water (98 mL), and then examined using a ZEISS optical microscope. The microhardness of the ZrO_2/Mg composites was measured with a Vickers hardness tester with a load of 100 gf for 10 s. Tensile testing was performed on a SANS CMT5205 material testing machine at a stretching rate of 2 mm/min at ambient conditions. Damping samples were machined to dimensions of (35 × 10 × 1) mm. The damping capacity was measured with a dynamic mechanical analyzer (NETZSCH DMA-242C) in the single cantilever deformation mode. For the measurements of the strain amplitude dependence of damping capacity, the range of the strain amplitude was from 1 μm to 200 μm, and the measurement frequency was 1 Hz.

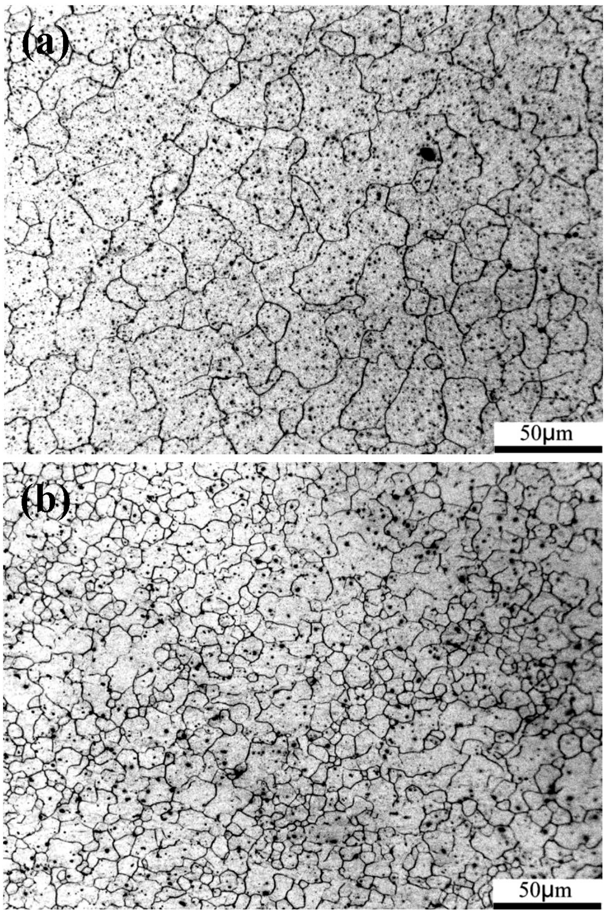

Figure 1: Metallographic microstructural comparison of the central regions of the stir zones: a) FSPed AZ31, b) FSPed ZrO_2/Mg composites

3 RESULTS AND DISCUSSIONS

3.1 Microstructures

Figure 1 presents the microstructure of the central region of the stir zone. **Figure 1a** shows the AZ31 magnesium alloy and **Figure 1b** shows the ZrO_2/Mg composite. The microstructure of the central region of the stir zone was subjected to vigorous mechanical stirring during the friction-stir process, which caused a severe plastic deformation. Coarse grains were broken, resulting in a noticeable refinement and fine, uniformly equiaxed crystal grains. The grain size of the stir zone of the AZ31 magnesium alloy was 16.91 μm and that of the ZrO_2/Mg composites was just 10.26 μm. It can be seen that an addition of ZrO_2 can effectively reduce the grain size. This is because after an addition of ZrO_2 reinforced phase particles, the alloy grain boundaries are pinned, thus greatly limiting the grain-boundary migration and playing a significant role in the refinement of grains.

3.2 Mechanical properties

Figure 2 shows the microhardness distribution along the cross-sections of a ZrO_2/Mg composite. **Figure 2a** presents the microhardness test position on the cross-section of the ZrO_2/Mg composite. The distance between the vertical and horizontal lines of a mesh unit is

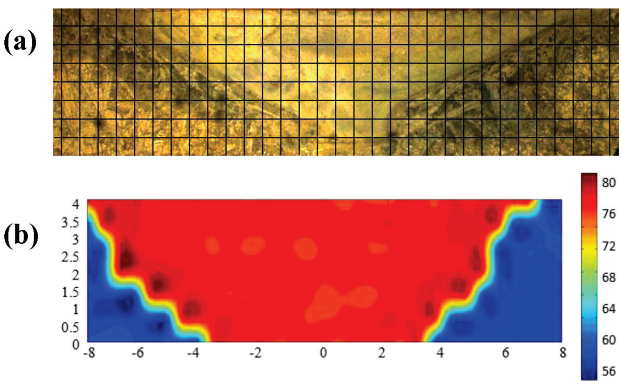

Figure 2: Microhardness distribution in the friction-stir zone of a ZrO_2/Mg composite a) microhardness test position (distance between the vertical and horizontal lines: 0.5 mm) and b) microhardness cloud map (unit of X- and Y-axes: mm)

0.5 mm. The intersection points are the test points for the microhardness measurement, forming a matrix of 31 × 15 points, which was plotted as the microhardness distribution in the Matlab software, as shown in **Figure 2b**.

The microhardness of the ZrO_2/Mg composites was related to the distribution of the ZrO_2 reinforced phase in the magnesium-alloy matrix. As can be seen from **Figure 2b**, the microhardness was in a range of 75–79 HV and the fluctuation was small. However, the microhardness values in the boundary region of the friction zone were relatively high, more than 80 HV, indicating a large fluctuation. It can be seen that after the friction-stir processing, the organization in the central region was very uniform, while there was still a certain degree of agglomeration in the boundary region. This is because during the friction-stir processing, the stirring force could not be involved in the boundary region, which was not the effective area of the composite material. Nevertheless, it showed that the FSPed ZrO_2/Mg composite material was uniform and dense.

The addition of ZrO_2 enhanced the tensile strength and yield strength of the ZrO_2/Mg composites. **Figure 3** shows the yield strength (YS), ultimate tensile strength (UTS) and elongation (EL) of the FSPed AZ31 magnesium alloy and ZrO_2/Mg composites with groove widths of (0.6, 0.8 and 1) mm, respectively. It can be seen that the addition of ZrO_2 can effectively improve the tensile properties, exhibiting a very good enhancement effect.

With an increase in the ZrO_2 amount, the tensile properties of the ZrO_2/Mg composites first increased inconspicuously and then decreased. This was because the distribution of the ZrO_2-rich phase was not uniform and there was a large degree of agglomeration after the friction-stir processing where the distribution of agglomerates in the AZ31 magnesium alloy matrix was also not homogeneous. When the ZrO_2/Mg composites were subjected to tensile loads, cracks were first generated around these agglomerates and they prematurely cracked. This indicated that an excessive addition of ZrO_2 cannot improve the tensile properties of ZrO_2/Mg composites because of the agglomerates.

3.3 Damping capacity

Figure 4 shows the damping-capacity dependence on the strain amplitude of the ZrO_2/Mg composites with groove widths of (0.6, 0.8 and 1) mm, showing a typical dislocation strain/damping spectrum. The damping values Q_0^{-1} (ε = 10^{-4}) of the ZrO_2/Mg composites with different ZrO_2 amounts at a low strain amplitude were 0.0111, 0.0117 and 0.0101, respectively. When the damping capacity is higher than 0.01, it is generally considered that the alloy exhibits a high damping capacity.[22] Therefore, the ZrO_2/Mg composites prepared with the friction-stir method showed a good damping performance.

According to the G-L theory:[23,24]

$$Q^{-1}(\varepsilon) = Q_0^{-1} + Q_H^{-1}(\varepsilon) \tag{1}$$

$$Q_0^{-1} = \frac{\rho B L_C^4 f}{36 G b^2} \tag{2}$$

$$Q_H^{-1} = \frac{C_1 \exp^{(-C_2/\varepsilon)}}{\varepsilon} \tag{3}$$

$$C_1 = \frac{\rho F_B L_N^3}{6 b E L_C^2} \tag{4}$$

$$C_2 = \frac{F_B}{b E L_C} \tag{5}$$

where C_1 and C_2 are the material constants; ρ is the dislocation density; F_B is the binding force between dislocation and weak pinning points; L_N and L_C are the average dislocation distances between strong pinning points and weak pinning points, respectively; b is the Burgers vector; E is the elastic modulus.

Taking the logarithmic transformation of Equation (2), we get Equation (6):

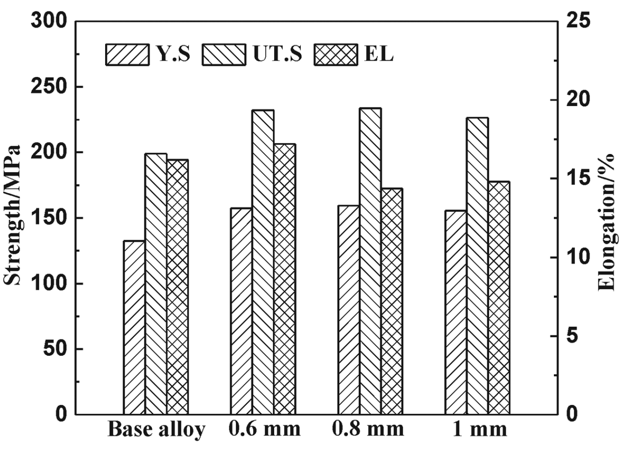

Figure 3: Yield strength (YS), ultimate tensile strength (UTS) and elongation (EL) of ZrO_2/Mg composites with different ZrO_2 amounts

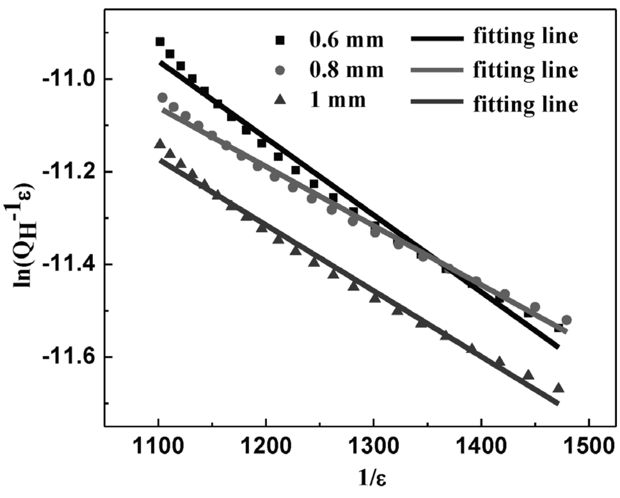

Figure 4: Damping-capacity dependence on the strain amplitude of ZrO_2/Mg composites with different ZrO_2 amounts

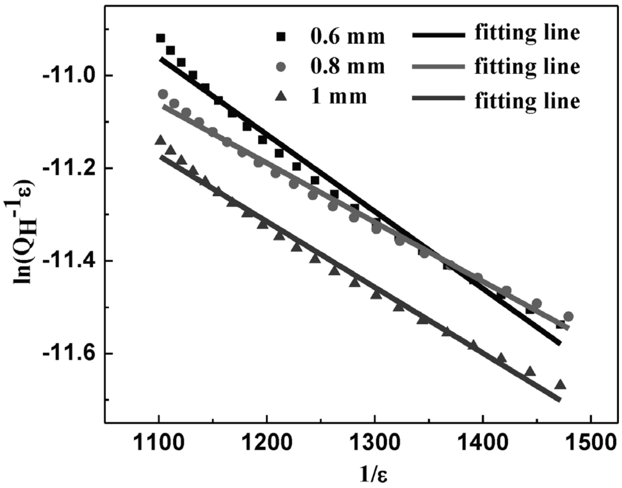

Figure 5: G-L plots for ZrO_2/Mg composites with different ZrO_2 amounts

$$\ln(Q_H^{-1}\varepsilon) = \ln C_1 - \frac{C_2}{\varepsilon} \quad (6)$$

The damping-capacity dependence on the strain amplitude of the stir zone at different ZrO_2 amounts is in good accordance with the G-L model, as shown in **Figure 5**. It can be seen that the G-L model curves for the ZrO_2/Mg composites with different ZrO_2 amounts follow a linear relationship. Therefore, the damping behavior of the ZrO_2/Mg composites with different ZrO_2 amounts under a high-strain condition was consistent with the G-L theory. For further analyses, the values of C_1 and C_2 calculated in accordance with the G-L plots[25] are shown in **Table 1**.

Table 1: Values of C_1 and C_2 according to G-L plots

Contents	0.6 mm	0.8 mm	1 mm
C_1×10^{-5}	10.820	6.403	6.705
C_2×10^{-3}	1.66	1.28	1.42
$(C_1/C_2)^{1/3}$	3.399	3.394	3.216

From Equations (4) and (5), the $(C_1/C_2)^{1/3}$ value is proportional to L_N. With an increase in the ZrO_2 amount, the $(C_1/C_2)^{1/3}$ value of the ZrO_2/Mg composites became smaller, showing that the distance from the strong pinning point L_N gradually became smaller, but the change was not obvious. This was because with the increase in the ZrO_2 amount, the density of the dislocations or other defects (such as grain boundary, phase boundary, etc.) in the ZrO_2/Mg composites and the concentration density of impurity atoms on the dislocation line were increased, reducing the average dislocation distance between strong pinning points (L_N). The increase of the distance of L_N was beneficial for the improvement of the high-strain damping capacity. It showed that the damping capacities of ZrO_2/Mg composites at the high-strain amplitude decreased with the increase of ZrO_2.

Combined with the damping values at the low-strain amplitude, the ZrO_2/Mg composites with groove widths of 0.8 mm had the best damping capacities.

4 CONCLUSIONS

(1) After friction-stir processing, fine uniform equiaxed crystal grains were obtained in the central region of the stir zone of ZrO_2/Mg composites. An addition of ZrO_2 refined the grains and improved the microhardness and mechanical properties of ZrO_2/Mg composite samples.

(2) The ZrO_2/Mg composite samples prepared with friction-stir processing had better damping capacities. In the strain-damping test, the damping behavior of ZrO_2/Mg composites followed the G-L theory. The ZrO_2/Mg composites with groove widths of 0.8 mm had the best damping capacities.

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