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NANO-SiO_2 PARTICLES REINFORCED MAGNESIUM ALLOY PRODUCED BY FRICTION STIR PROCESSING

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Abstract. Friction stir processing (FSP) was employed to produce SiO_2/AZ31 composites for increase of the hardness of AZ31 matrix. SiO_2 particles with sizes of less than 0.2 μm were uniformly dispersed into the matrix processed by FSP. As a result, the addition of SiO_2 particles resulted in grain refinement of equi-axed ultrafine grains with sizes of less than 1 μm evolved in composite zone. Both the grain refinement and dispersion of SiO_2 particles caused hardness of the composites to 90 Hv, which was 1.83 times higher than that of the original alloy.

1. INTRODUCTION

Magnesium alloys attract great attention for their excellent properties such as light weight, high specific strength, high recyclability, etc. They have been wildly used as a light structural material in order to reduce CO_2 emissions, for example, through reducing the weight of automobile parts [1]. The hardness of magnesium alloys is not sufficient to general usage. This will limit their applications. It is well known that hardness of fine-grained materials is higher than that of coarse-grained ones due to a larger fraction of grain boundary. Fine grained structure, however, can be thermally unstable at elevated temperatures. The fraction of boundary decreases with grain growth at high temperature, resulting in any reduction of the hardness. Another useful way to strengthen the alloys is to fabricate metal matrix composites (MMCs) [2]. MMCs are produced by introducing any reinforced phases into metal matrix. Some addition of reinforcements into metallic matrix can improve the stiffness, wear, creep properties, etc., comparing with conventional engineering materials. Particle-reinforced composites have been paid great attention because they have generally rather isotropic properties and also are relatively inexpensive for manufacture. Several methods to fabricate particle-reinforced composites have been developed and studied, e.g. such as stir casting [3], squeeze casting [4], molten metal infiltration [5], powder metallurgy [6], etc. From the industrial point of view, most of these technologies include heating treatment and so consume good energy during the process and may be not economic. Furthermore, it is difficult for nano/sub-micro scale particles to disperse uniformly into metal matrix via these technologies. So a more advanced technology with low energy saving is strongly desired to produce MMCs.

Friction stir processing (FSP) derived from friction stir welding is a very useful surface modification technique [7]. FSP is a very convenient process without any special die and can be carried out at room temperature. During the process, a rotating tool is inserted into a monolithic workpiece to producing sever plastic deformation. Because of friction occurring at the tool-work piece interface,

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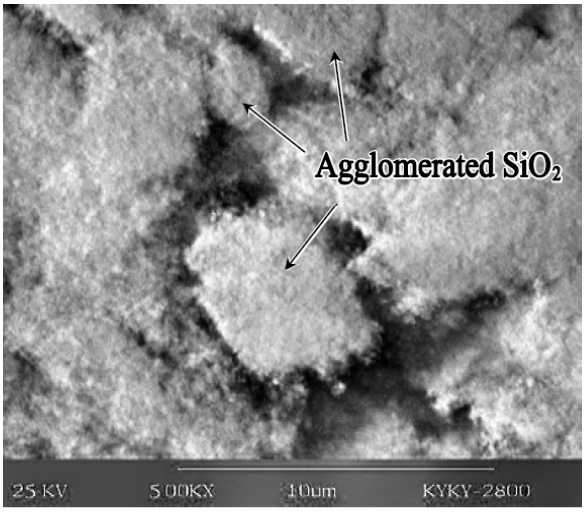

Fig. 1. SEM image of as-received nano-SiO_2 particles.

temperature is usually reached to a relatively high level and so dynamic recrystallization may take place during the process, leading to grain refinement in deformed zone [8]. FSP can be also used to introduce discontinuous reinforcement into metal matrix. It is reported in [9,10], for example, that Mg-based and Al-based composites are successfully fabricated by FSP and show excellent performance. This technology shows a great potential for producing MMCs. At the present work, FSP was employed to produce nano-composites in magnesium-based alloy. The microstructural development and hardness elevation accompanied by FSP were studied.

2. EXPERIMENTAL PROCEDURE

The metal matrix used was AZ31 magnesium alloy with the following chemical composition (wt.%): Al, 2.68; Zn, 0.75; Mn, 0.68; Cu, 0.001; Si, 0.03; Fe, 0.003; Mg, bal. Nano-SiO_2 particles with average sizes of ~20 nm were commercially available and used as reinforcement particles. The nano-SiO_2 particles used are agglomerated together with a clusters size of about 5~8 μm (see Fig. 1). A tool which has a columnar of Ø 20 mm and a probe of Ø 5 mm and length: 3.5 mm was used for FSP (Fig. 2a). AZ31 Mg alloy was machined to a plate and a groove with the dimensions of width 3 mm and depth 2.5 mm in center of a surface tested, as shown in Fig. 2b. First nano-SiO_2 particles were filled in the groove. Then the probe was inserted into the groove filled by the nano-SiO_2 particles and moved with a rotating rate of 1200 rpm and a travel speed of 50 mm/min.

A cross-section of the plate machined after FSP was prepared by standard metallographic techniques and etched in a solution of 10 ml acetic acid, 4.2 g picric acid, 10 ml water, and 70 ml ethanol. The microstructures were observed by optical microscopy (OM), and scanning electron microscopy (SEM). SEM was employed to examine the microstructures with particles dispersion. Vickers hardness tests were conducted using a 300 g load for 15 s on the surface of the plate before and after FSP.

3. RESULTS AND DISCUSSION

Fig. 3 shows a typical optical microstructure developed in stirred zone after FSP. The regions that appear gray in color are composed of new fine grains with nano-SiO_2 particles. It is clearly seen that ultrafine grains are developed in stirred zone comparing with original grains existing in lower hand side of Fig. 3. This may suggest that these fine and equiaxed grains are developed during FSP due to operation of dynamic recrystallization. Fine grained layers and coarse grained matrix are connected very soundly, because neither cracks nor exfoliations were observed on the interface. SiO_2 particles were relatively uniformly dispersed in stirred zone and

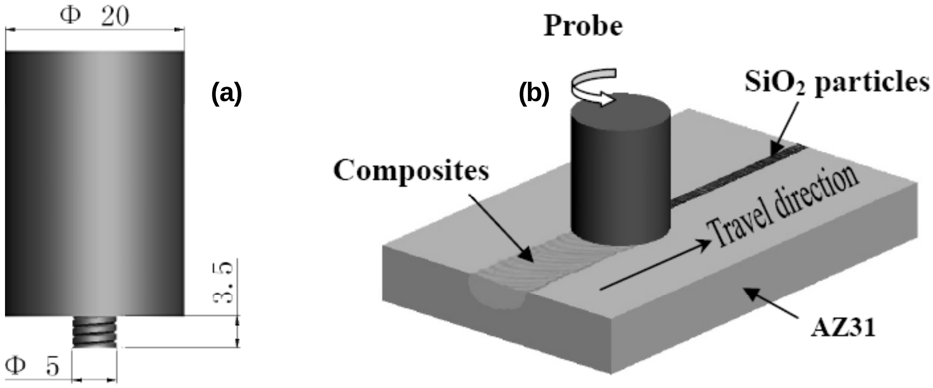

Fig. 2. Drawing of (a) probe and (b) friction stir processing.

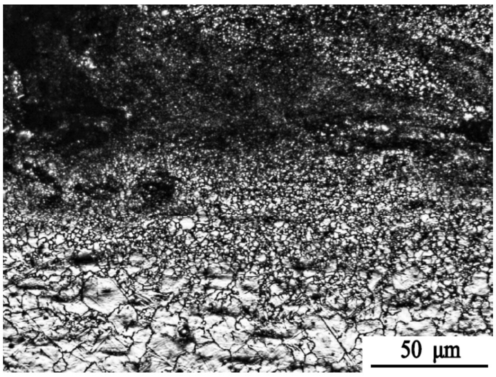

Fig. 3. A typical microstructure in composite zone of AZ31 Mg alloy with SiO_2 particles dispersed by FSP.

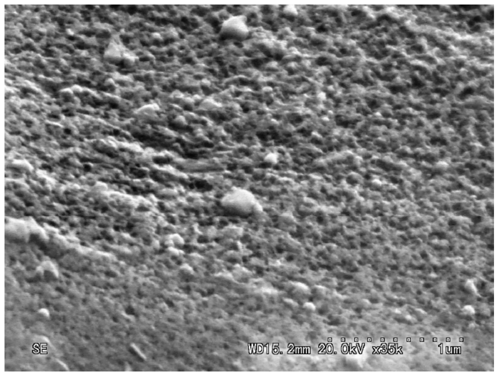

Fig. 4. SEM micrograph showing fine grains with dispersion particles developed by FSP.

ultrafine grained structure with sizes of less than 1 μm are evolved in composite zone. On the other hand, coarse grains of about 3~4 μm exist outside the composites zone. It is concluded that dispersion of SiO_2 particles can effectively promote grain refinement of the matrix.

SiO_2 particles dispersed in the composite zone were observed by SEM. A typical microstructure containing SiO_2 particles is shown in Fig. 4. It is seen that the particles are uniformly dispersed into the matrix of AZ31 after FSP. They were separated from each other with cluster sizes of less than 0.2 μm. New grains evolved in composite zone are much finer than those outside the regions with no particles (Fig. 3). It is concluded that dispersion of SiO_2 particles play a significant role in grain refinement. Some of SiO_2 particles locate on grain boundaries or triple junctions and can restrict grain boundary migration at elevated temperature. This may cause grain growth to operate more difficult and then the thermal stability of the matrix to increase. Y. Morisada et al. [11] also pointed out that pinning effect due to particles dispersed is one of critical reasons for more effective grain refinement taking place during FSP.

For particle-reinforced materials, one of the critical microstructure parameters is the particle interspacing L, which can be roughly estimated from Eq. (1) [12],

$$L = \frac{d}{2}\left(\frac{2\pi}{3V_f}\right)^{\frac{1}{2}}, \tag{1}$$

where d is the average particle diameter and V_f is the particle volume fraction. It is considered that the smaller the particle interspacing L is the finer grain structure could be obtained. According to Eq. (1), a smaller value of particle interspacing L can be achieve by reducing particle diameter d or increasing the particle volume fraction V_f. Ductility of composites generally decreases with increase in V_f, and so an usual way to decrease L is to reduce d. It is suggested in [13] that the addition of 3% nano-powders in metal could stabilize the grain size of less than 500 nm even at elevated temperatures and enhance the superplasticity.

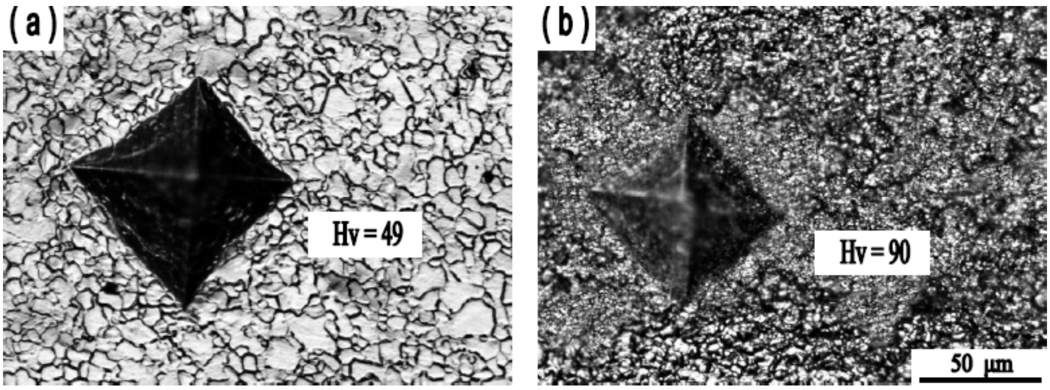

Fig. 5. Microstructures and indentations of Vickers hardness of the AZ31 plate (a) before and (b) after FSP.

Vickers hardness tests were conducted using a 300 g load on the etched surfaces of plate. Typical results for as-received AZ31 and FSPed plate are shown in Fig. 5a and Fig. 5b, respectively. It is clearly seen that the grain size in FSPed plate (Fig. 5b) is significantly refined from the initial one of ~25 μm (Fig. 5a), and in addition, a much finer microstructure is evolved at the region of around the SiO_2/AZ31 composites. The hardness of the composites was 90 Hv. This is about 1.83 times higher than that of the as-received AZ31, i.e. around 49Hv. It is concluded that a significant increase in hardness could be attributed to both the dispersion of high strength SiO_2 particles and grain refinement taking place in the region of SiO_2/AZ31 composites developed during FSP.

4. CONCLUSIONS

Nano-SiO_2 particles were successfully dispersed into AZ31 magnesium alloy via friction stir processing (FSP). The microstructures and microhardness changes before and after FSP were investigated by OM, SEM and Vickers hardness tests. The main results can be summarized as follows:

1. SiO_2 particles were uniformly dispersed into AZ31 matrix after FSP with sizes of less than 0.2 μm.
2. New grains size evolved in composite zone is less than 1 um and much finer than that in the regions outside the stirred zone.
3. Hardness of the SiO_2/AZ31 composites was 90 Hv and is about 1.83 times higher than that of the as-received AZ31.

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