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Effect of friction stir processing with SiC particles on microstructure and hardness of AZ31

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Abstract

SiC particles were uniformly dispersed into an AZ31 matrix by friction stir processing (FSP). The SiC particles promoted the grain refinement of the AZ31 matrix by FSP. The mean grain size of the stir zone with the SiC particles was obviously smaller than that of the stir zone without the SiC particles. The microhardness of the stir zone with the SiC particles was reached about 80 Hv due to the grain refinement and the distribution of the SiC particles. Additionally, the SiC particle/AZ31 region showed fine grains even at elevated temperatures (∼400 °C) resulting in the pinning effect by the SiC particles. In contrast, the microhardness was significantly decreased attributed to the abnormal grain growth of the FSPed AZ31 without the SiC particles.

Keywords: Friction stir processing; AZ31; SiC particle; Grain refinement; Pinning effect; Microhardness

1. Introduction

Magnesium alloys are very attractive materials due to their low specific gravity, high specific strength, and high recyclability. They have been used as a structural material in order to reduce CO_2 emissions and increase power performance by reducing the weight of automobile parts. However, the mechanical properties, such as the hardness of the magnesium alloys, are not sufficient to enhance their applications. Though some processes to fabricate ceramics particle/magnesium alloys composites have been studied to improve the mechanical properties [1–4], the nonuniform dispersion of the particles is still a serious problem.

Recently, much attention has been paid to a new surface modification technique called friction stir processing (FSP) which is the same approach as friction stir welding (FSW) [5–9]. A rotating tool is inserted into a substrate and produces a highly plastically deformed zone. It is well known that the stir zone consists of fine and equiaxed grains produced due to dynamic recrystallization [10]. Recently, the authors' group reported MWCNTs as known a representative reinforcement agent, which has a poor dispersion property, was successfully distributed in the magnesium alloy by the FSP, and indicated that the MWCNTs addition was very effective for the grain refinement of the matrix [11].

In this study, the SiC particles were dispersed into AZ31 in order to reveal the effect of the FSP with the SiC particles on the microstructure and hardness of the magnesium alloy. The pinning effect of the SiC particles on the grain growth of the AZ31 matrix was also evaluated with respect to the change in the grain size after the heat treatment.

2. Experimental procedure

Commercially available SiC powder (mean diameter: 1 μm, 99% pure) was used (Fig. 1). The SiC powder was filled into a groove (1 mm × 2 mm) on the AZ31 plate before the FSP was carried out. This process was explained in detail elsewhere [11]. The FSP tool made of SKD61 has a columnar shape (Ø12 mm) with a probe (Ø4 mm, length: 1.8 mm). The probe was inserted into the groove filled with the SiC powder. A constant tool rotating rate of 1500 rpm was adopted and the constant travel speed was changed from 25 to 200 mm/min. The tool tilt angle of 3° was used. The FSPed samples were heated in an electric furnace to evaluate the microstructural change in connection with the pinning effect of the SiC particles (heat treatment condition: 200–400 °C, 1 h, in air).

Transverse sections of the as-received AZ31, as-produced FSPed samples, and heat-treated FSPed samples were mounted

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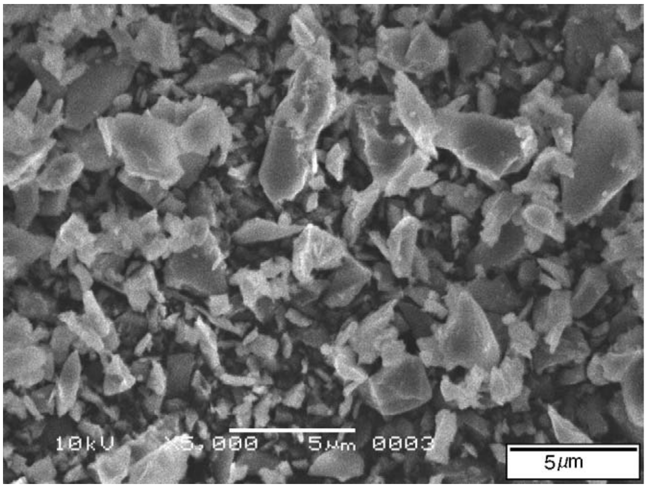

Fig. 1. SEM image of the as-received SiC particles.

and then mechanically polished. The distribution of the SiC particles was observed by SEM (JEOL JSM-6460LA) and TEM (JEOL JEM-1200EX), and the grain size of the etched sample was evaluated by optical microscopy. The grain size was estimated using the mean linear intercept method ($d = 1.74L$; L is the linear intercept size). The microhardness was measured using a micro-vickers hardness tester (Akashi HM-124) with a load of 200 g.

3. Results and discussion

3.1. Microstructure and microhardness

The SiC particles were well dispersed in the stir zone as shown in Fig. 2. No discernible defects and porosities could be observed. The interface between the AZ31 matrix and the SiC particle/AZ31 composite layer was sound without exfoliations. Fig. 2(b) shows that the SiC particle led the grain to be refined by the FSP through a recrystallization process. The grain size in the SiC particle/AZ31 region was clearly fine compared with that of the region without the SiC particles.

Fig. 3 shows representative photomicrographs of each sample. (b) and (c) correspond to the recrystallized stir zone of the FSPed samples. The mean grain size was 79.1, 12.9, and 6.0 μm for the as-received AZ31, the FSPed AZ31, and the FSPed AZ31 with SiC particles, respectively. The microhardness of the AZ31 could be estimated using the following equation [12]:

$$\mathrm{Hv} = 40 + 72d^{-1/2}$$

where d is the mean grain size of the AZ31. Based on this equation, the microhardness was calculated to be 48.1, 60.0, and 69.3 Hv for the as-received AZ31, the FSPed AZ31, and the FSPed AZ31 with SiC particles, respectively. These values showed a good agreement with the experimental results as shown in Fig. 4. However, the maximum microhardness of the SiC particle/AZ31 region was 76.2 Hv. It is considered that this vale was reflected by not only the grain refinement using the SiC particles, but also the high hardness of the SiC particles.

Fig. 5 shows the TEM microstructure of the stir zone with the SiC particles. Some fine SiC particles were observed on the grain boundary of the AZ31 matrix. It seems very effective to restrain the grain growth. It is considered that the SiC particles were smashed by the rotating tool during the FSP.

3.2. Effect of SiC particles on grain size

The grain size was related to the FSP conditions and the presence of the SiC particles are shown in Fig. 6. It is widely

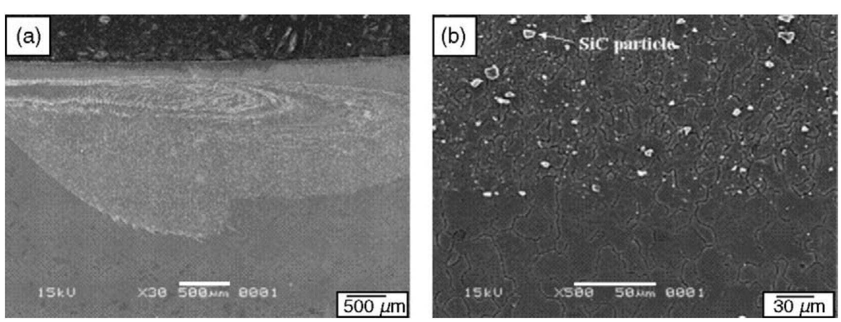

Fig. 2. SEM images of (a) stir zone with the SiC particle and (b) interface zone between the SiC particle/AZ31 and the AZ31.

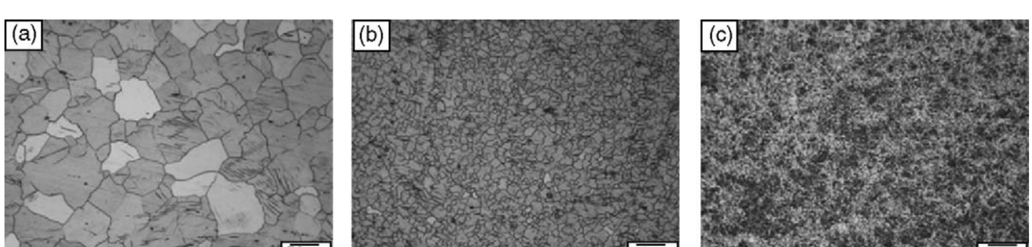

Fig. 3. Representative grain structures of (a) as-received AZ31, (b) FSPed AZ31, and (c) FSPed AZ31 with the SiC particles. The travel speed of (b) and (c) was 50 mm/min.

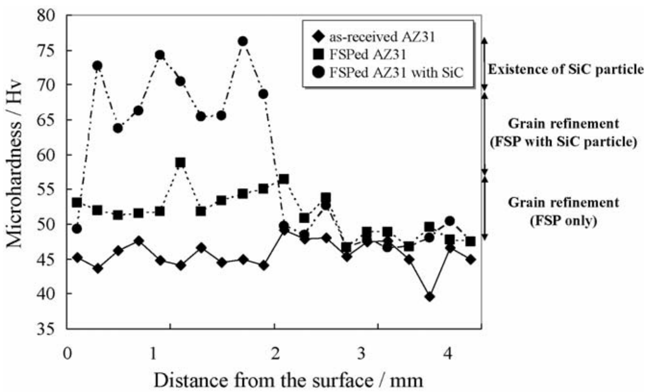

Fig. 4. Microhardness profile of the cross-section in as-received AZ31, FSPed AZ31, and FSPed AZ31 with the SiC particles. The travel speed of (b) and (c) was 50 mm/min.

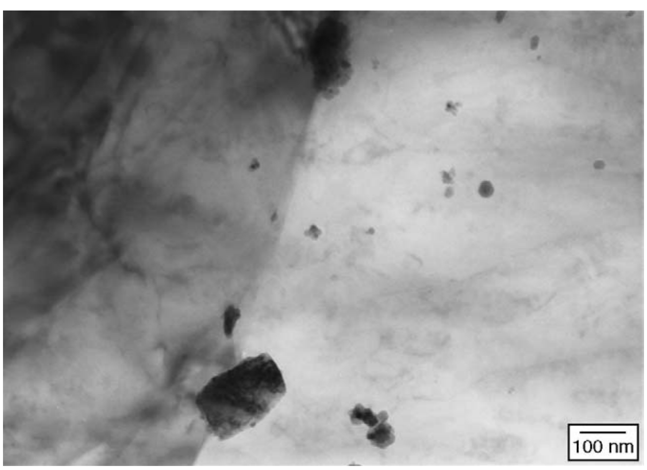

Fig. 5. TEM image of the SiC particles on the grain boundary of the AZ31.

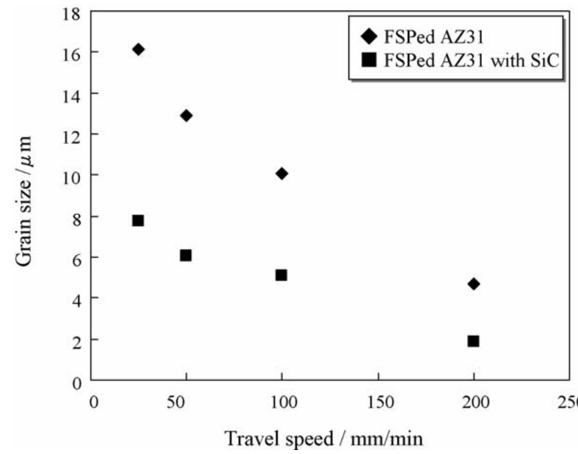

Fig. 7. Relationship between the grain size and the travel speed of the rotating tool.

reported that the grain size increases following the decrease in the travel speed of the rotating tool [13–16]. However, the grain size of the FSPed sample with the SiC particles at the travel speed of 25 mm/min is smaller than that of the sample FSPed without the SiC particles at the travel speed of 200 mm/min. The FSP with the SiC particles is considered to make fine grains more effectively due to the enhancement of the induced strain and the pinning effect by the SiC particles.

Fig. 7 shows the relationship between the grain size and the travel speed of the rotating tool. Though a slight increase in the grain size was confirmed for the FSPed sample with the SiC particle following the decrease in the travel speed, the grain size was less than 8 μm for all samples. The differences in the grain size between the samples with and without the SiC particle were smaller when the travel speed increased. This suggests that the particle size of the SiC used in this study cannot

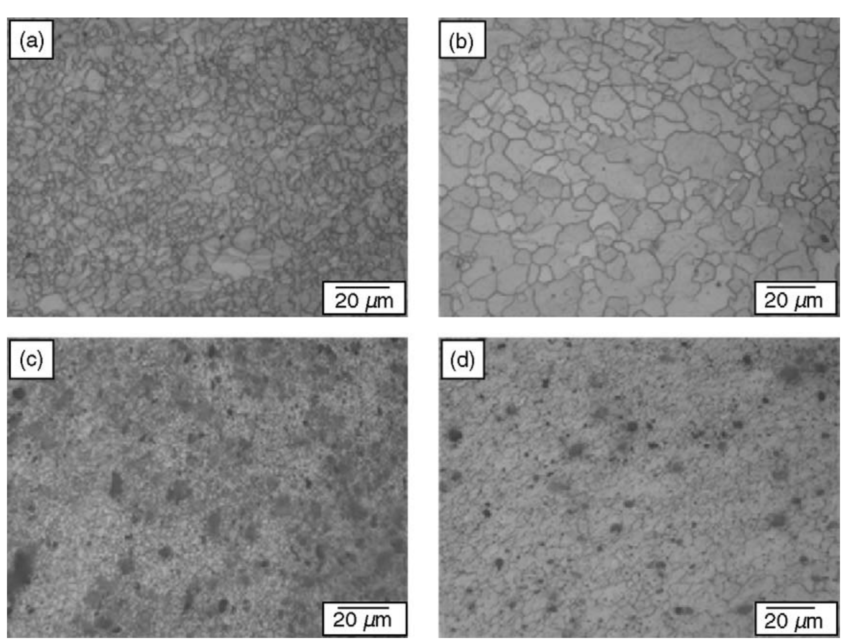

Fig. 6. OM images of the stir zone for the FSPed AZ31 (a and b) and the FSPed AZ31 with the SiC particles (c and d). Travel speed of the rotating tool was 200 mm/min for (a) and (c), and 25 mm/min for (b) and (d).

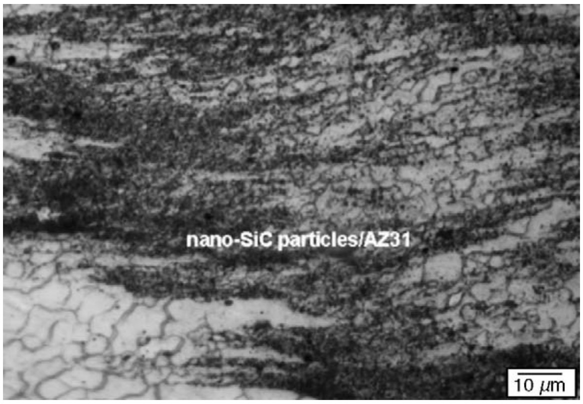

Fig. 8. OM image of the stir zone for the FSPed AZ31 with the nano-sized SiC particles. The constant tool rotating rate and the travel speed of the rotating tool were 1500 rpm and 50 mm/min, respectively.

effectively restrain the grain growth for the ultra-fine grain (less than 1 μm).

The optical microstructure of the stir zone with the nano-sized SiC particles (Sumitomo Osaka Cement Co., Ltd., mean individual particle size: 30 nm) is shown in Fig. 8. Though the dispersion of the nano-SiC particles is not uniform, the grain size on the nano-sized SiC particles/AZ31 region (∼1 μm) is clearly smaller than that of the SiC particles/AZ31 (6 μm) under the same FSP condition (1500 rpm, 50 mm/min). Lee et al. reported that the grain size of AZ61 with 5% and 10% nano-sized SiO_2 particles (individual particle size: ∼20 nm) in volume fraction after four FSP passes were 1.8 and 0.8 μm, respectively [17]. It is considered that a finer grain structure could be formed by the FSP with the uniform dispersion of smaller SiC particles.

3.3. Grain growth at elevated temperatures

Fig. 9 shows the change in the grain size by the heat treatment. The abnormal grain growth was confirmed above 300 °C for the FSPed sample without the SiC particle. The small grain size and the residual strain inducted by the FSP resulted in a serious grain growth. On the contrary, the grain size was nearly the same for the FSPed sample with the SiC particle even after the heat treatment at 400 °C.

As a consequence of the differences in the grain size, the microhardness was changed as shown in Fig. 10. The microhardness of the stir zone for the FSPed sample without the SiC particle decreased to ∼40 Hv by the heat treatment above 300 °C. This microhardness was lower than that of the as-received AZ31. This result revealed that the FSPed sample has an unavoidable problem related to the degradation of the mechanical properties attributed to the grain growth. The SiC particle addition overcame this problem because of its excellent pinning effect. For the FSPed sample with the SiC particle, the microhardness was not decreased below 300 °C whereas the value was scattered at 400 °C. It is considered that the region with the relatively low

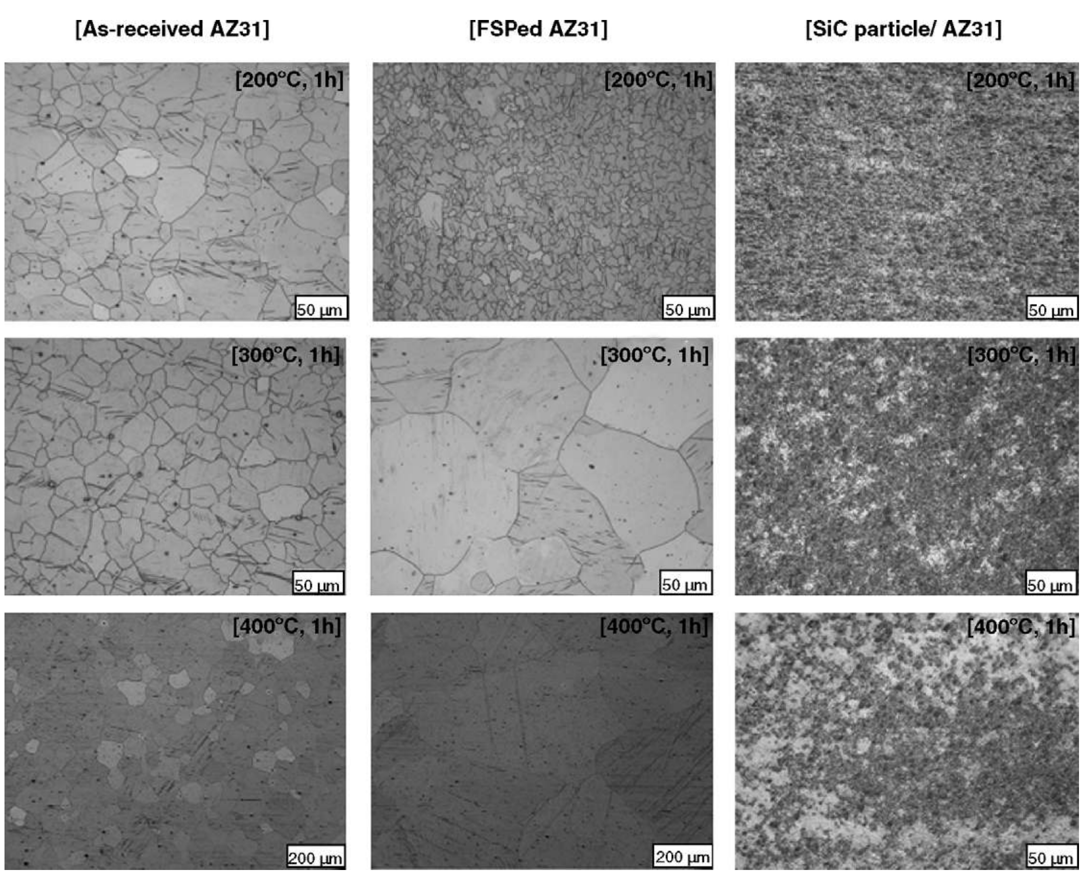

Fig. 9. Representative grain structures of the stir zone after the heat treatment.

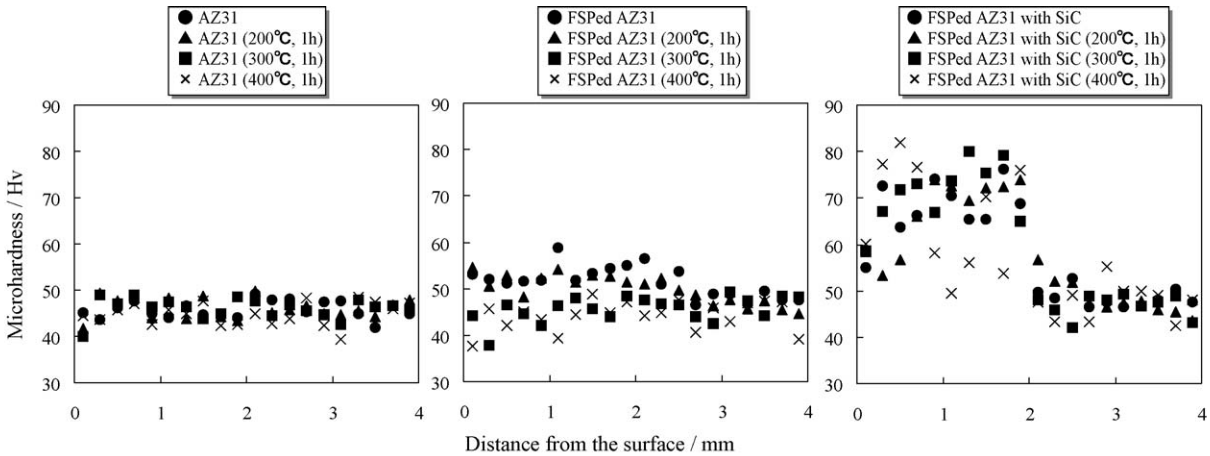

Fig. 10. Microhardness profiles of the cross-section in as-received AZ31, FSPed AZ31, and FSPed AZ31 with the SiC particles before and after the heat treatment. The travel speed of the FSPed samples was 50 mm/min.

microhardness in the stir zone contained a small amount of the SiC particles.

4. Conclusions

The SiC particle dispersed AZ31 was successfully fabricated by the FSP. The microstructure, microhardness and thermal stability were evaluated by the observation of the grain size and the dispersion of the SiC particles. The obtained results can be summarized as follows:

(1) The SiC particles lead the grain to be refined by the FSP.
(2) The microhardness of the stir zone with the SiC particles increases to about 80 Hv.
(3) The fine grain structure of the AZ31 fabricated by the FSP is unstable above 300 °C.
(4) The fine grain fabricated by the FSP with the SiC particles is maintained at the elevated temperatures (∼400 °C).

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