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MWCNTs/AZ31 surface composites fabricated by friction stir processing

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Abstract

Multi-walled carbon nanotubes (MWCNTs) were successfully dispersed into a magnesium alloy (AZ31) using friction stir processing (FSP). Distribution of the MWCNTs was changed on the basis of the travel speed of the FSP tool. The grain size of the MWCNTs/AZ31 surface composites was smaller than that of the FSPed AZ31 without the MWCNTs. The addition of the MWCNTs appears effective for fabricating the composites consisting of fine matrix grains. The maximum microhardness of these composites was ~78 Hv, which is almost double that of the AZ31 substrate (41 Hv). It is considered that both the grain refinement of the AZ31 matrix and the reinforcement by the MWCNTs increased the microhardness of the surface composites.

Keywords: MWCNTs; AZ31; Surface composites; Friction stir processing; Grain refinement; Microhardness

1. Introduction

Multi-walled carbon nanotubes (MWCNTs) have been tested for reinforcing various matrices [1–3] because they have many unique mechanical [4–9] and physical properties [10–14]. For example, such reinforcements can improve the strength of matrices due to the extremely high tensile strength of the MWCNTs. However, there are few reports about MWCNTs reinforced metal matrix composites because the uniform dispersion of MWCNTs in metal is exceedingly difficult. Additionally, MWCNTs easily react with the molten metal during the usual fabrication process, such as the vortex method [15]. These shortcomings limit the applications of the MWCNTs.

Recently, much attention has been paid to a new surface modification technique named friction stir processing (FSP) [16–19]. FSP is a solid state processing technique to obtain a fine-grained microstructure. This is carried out using the same approach as friction stir welding (FSW), in which a rotating tool is inserted into a substrate and produces a highly plastically deformed zone. It is well known that the frictioned zone consists of fine and equiaxed grains produced due to dynamic recrystallization [20]. Though FSP has been basically advanced as a grain refinement technique, it is a very attractive process for also fabricating composites. Mishra et al. [21] fabricated the SiC/Al surface composites by FSP, and indicated that SiC particles were well-distributed in the Al matrix, and good bonding with the Al matrix was generated.

In this study, the MWCNTs were dispersed in a magnesium alloy (AZ31) which is one of the best structural materials to decrease the weight of various vehicles. The MWCNTs/AZ31 surface composites were produced by FSP, and their metallographic examinations and hardness tests were carried out.

2. Experimental procedure

Commercially available MWCNTs (outer diameter: 20–50 nm, length: ~250 nm) synthesized from hydrocarbons, and an AZ31 rolled plate (thickness: 6 mm) were used in this study. The MWCNTs are typically entangled with each other and contain a few graphite granule inclusions (Fig. 1). The strong aggregation makes a uniform dispersion of MWCNTs in the matrix difficult.

The MWCNTs were filled into a groove (1 mm × 2 mm) on the AZ31 plate before the FSP was used, as shown in Fig. 2. The FSP tool made of SKD61 has a columnar shape (Ø 12 mm) with a probe (Ø 4 mm, length: 1.8 mm). The probe was inserted

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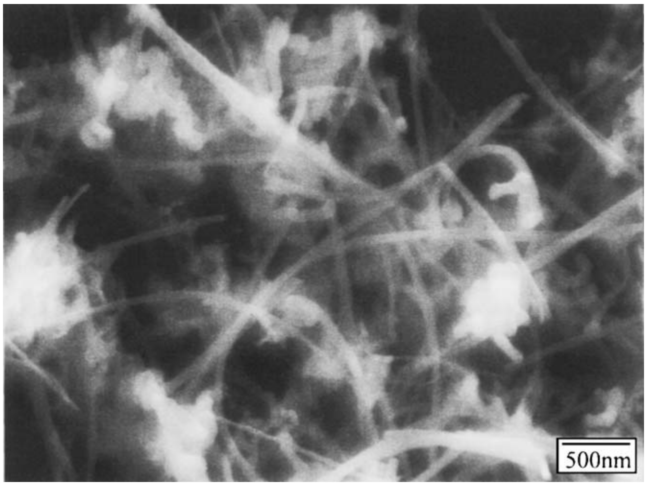

Fig. 1. SEM image of the as-received MWCNTs.

into the groove filled with the MWCNTs. A constant tool rotating rate of 1500 rpm was adopted and the travel speed was varied from 25 to 100 mm/min. The tool tilt angle of 3° was used.

Transverse sections of the as-received AZ31 and as-produced FSPed samples were mounted and then mechanically polished. The distribution of the MWCNTs was observed by SEM (JEOL JSM-6460LA), and the grain size of the etched sample was evaluated by optical microscopy. The grain size was estimated using the mean lineal intercept method. The microhardness was measured using a micro-vickers hardness tester (Akashi HM-124) with a load of 200 g.

3. Results and discussion

Figs. 3 and 4 show OM and SEM images obtained from the surface composites fabricated by the FSP, respectively. The dispersion of the MWCNTs in the AZ31 matrix was related to the travel speed of the rotating tool. Entangled MWCNTs, which were similar to the as-received MWCNTs, could be observed in the sample FSPed at 100 mm/min. Though the sample FSPed at 50 mm/min showed a better dispersion of the MWCNTs, there were some regions which included the aggregated MWCNTs. On the other hand, a good dispersion of the MWCNTs, which were separated from each other, could be observed for

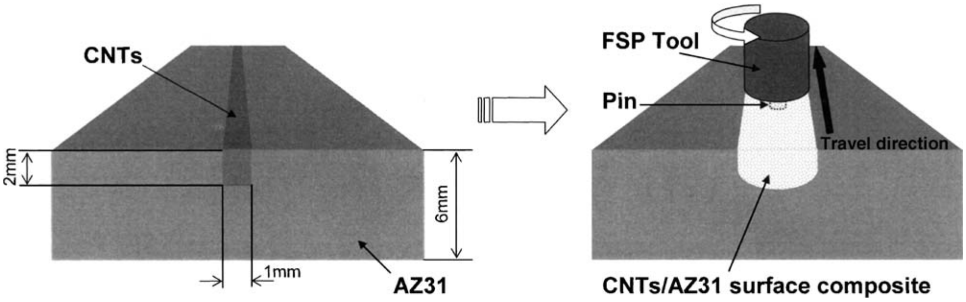

Fig. 2. Schematic of the friction stir processing.

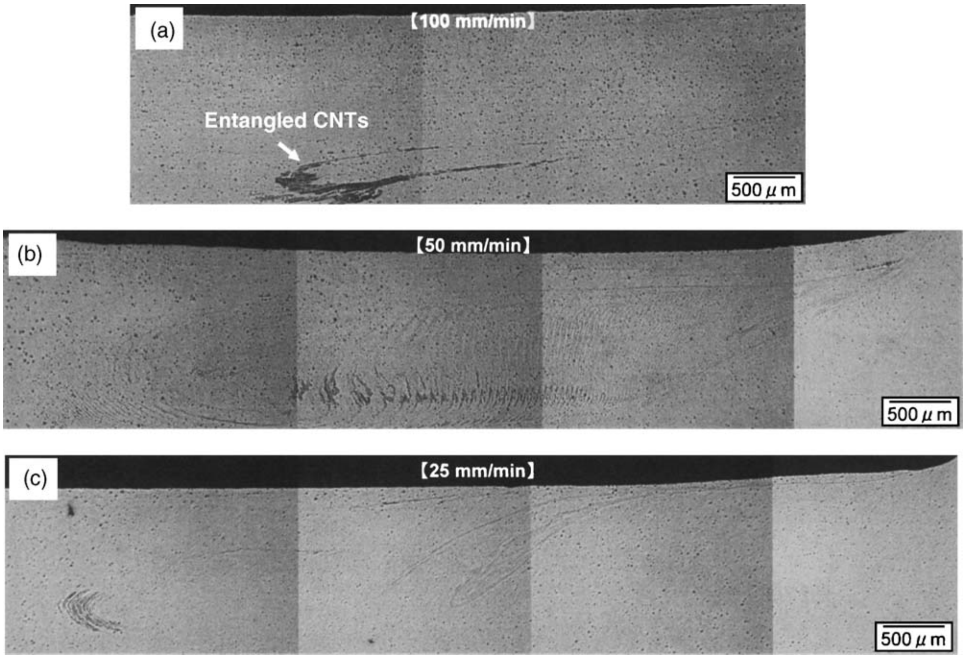

Fig. 3. OM images of the MWCNTs/AZ31 surface composites. The constant tool rotating rate was 1500 rpm. The travel speeds of (a), (b) and (c) were 100, 50 and 25 mm/min, respectively.

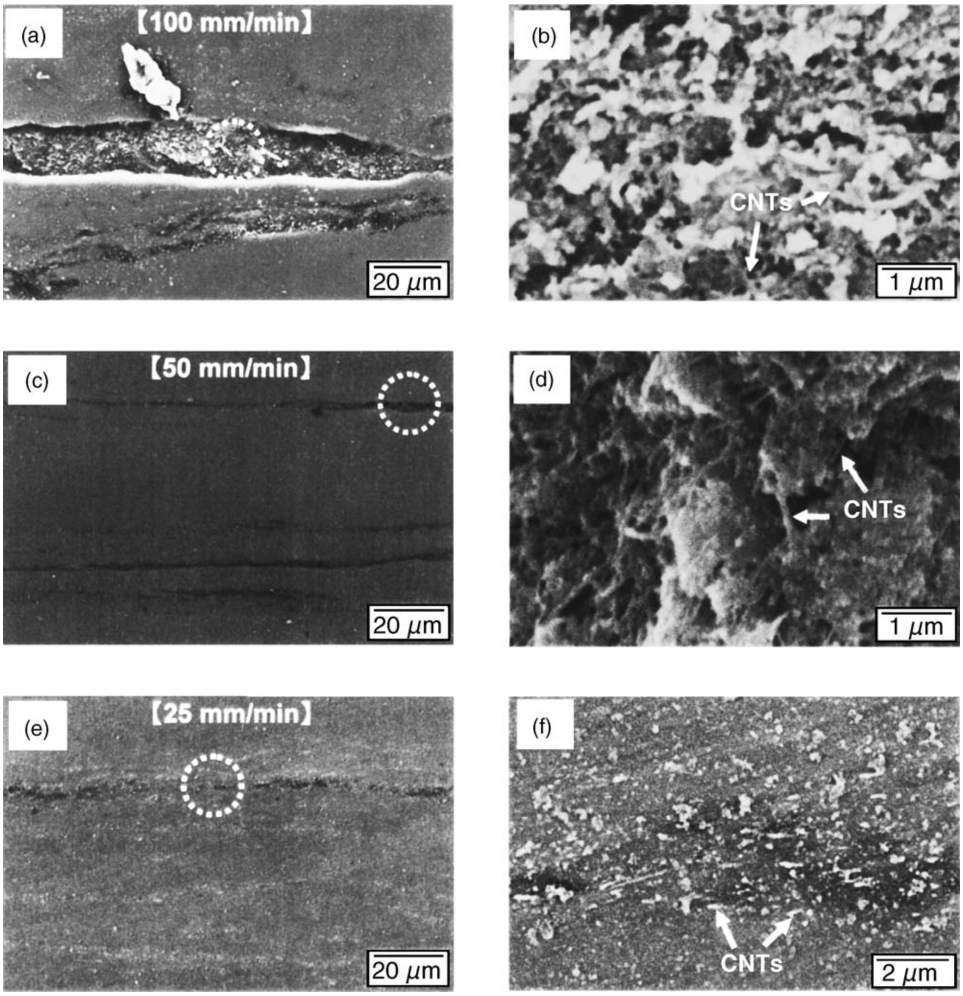

Fig. 4. SEM images of the MWCNTs/AZ31 surface composites. (b), (d) and (f) are enlargements of the microstructure inside a dotted circle for (a), (c) and (e), respectively. The constant tool rotating rate was 1500 rpm. The travel speeds of (a), (c) and (e) were 100, 50 and 25 mm/min, respectively.

the sample FSPed at 25 mm/min. Only the travel speed of the rotating tool determined the friction heats in the matrix because the tool rotating rate was constant (1500 rpm) in this study. It is considered that the travel speed of 100 mm/min was too fast to produce enough heat flow to produce a suitable viscosity in the AZ31 matrix for the dispersion of the MWCNTs.

Fig. 5 shows the indentation prints marked by the microhardness test under a 200 g load. The optical micrographs were obtained at the same magnification in order to compare the size of the indentation prints. The average microhardness of the as-received AZ31 was 41 Hv (Fig. 5(a)). On the other hand, the indentation print for the MWCNTs/AZ31 surface composite was obviously smaller than that of the as-received AZ31, as

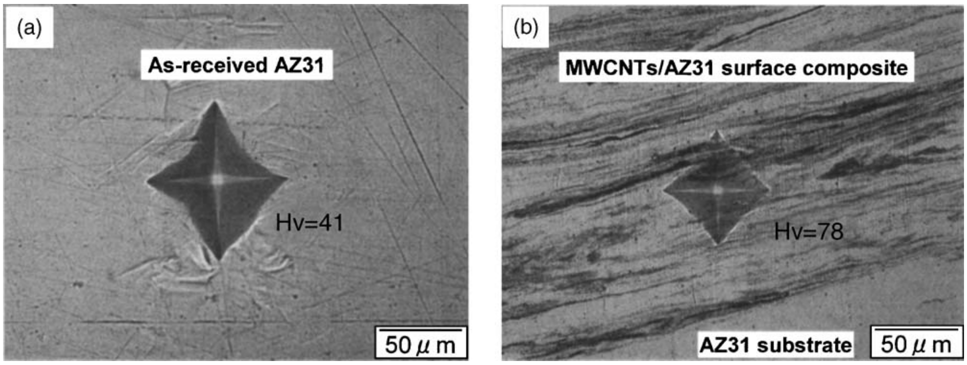

Fig. 5. The representative indentation prints for (a) as-received AZ31 and (b) the MWCNTs/AZ31 surface composites. The FSP condition was 1500 rpm and 50 mm/min.

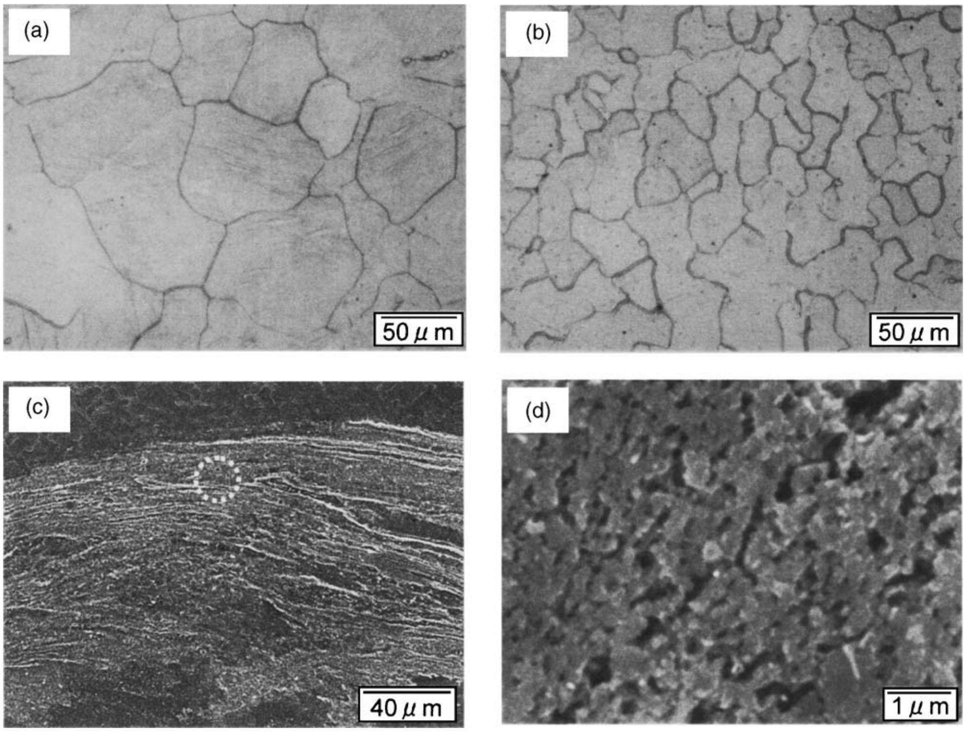

Fig. 6. Optical micrographs showing grain size of (a) as-received AZ31 and (b) FSPed AZ31. SEM image (c) and (d) showing the microstructure of the MWCNTs/AZ31 surface composite, (d) is enlargement inside a dotted circle for (c). The FSP condition was 1500 rpm and 50 mm/min for (b) and (c).

shown in Fig. 5(b). The value of 78 Hv could be measured as the maximum microhardness for the composites. In addition, the maximum microhardhess for the samples treated by the FSP without MWCNTs was 55 Hv. It is suggested that the grain refinement and the extremely high strength of the MWCNTs resulted in a significant increase in the microhardness for the MWCNTs/AZ31 surface composites.

The grain size of the AZ31 matrix was obviously refined by the FSP as shown in Fig. 6. As reported by other researchers [22–24], it seems that the grain refinement was caused due to dynamic recrystallization during the FSP. However, the FSP with the MWCNTs more effectively reduced the grain size of the AZ31 matrix in which some grains were less than 500 nm as shown in Fig. 6(c and d). It is considered that the pinning effect by the MWCNTs retarded the grain growth of the AZ31 matrix. The fine grain seems to be very useful for enhancing the mechanical properties, and also attractive for the low temperature superplasticity of the magnesium alloys [25].

4. Conclusions

The MWCNTs/AZ31 surface composites were successfully fabricated by the FSP. The microstructure and microhardness were evaluated by a view of observing the grain size and the dispersion of the MWCNTs. The obtained results can be summarized as follows:

(1) MWCNTs can be dispersed into the AZ31 matrix using the FSP.
(2) The dispersion of the MWCNTs is related to the travel speeds of the rotating tool. A good dispersion of the MWCNTs, which are separated from each other, is obtained for the sample FSPed at 25 and 1500 rpm.
(3) The FSP with MWCNTs obviously increases the microhardness of the substrates. The maximum microhardness for the composites is 78 Hv, while that of the sample treated by the FSP without MWCNTs and the as-received sample are 55 and 41 Hv, respectively.
(4) The addition of the MWCNTs promoted grain refinement by the FSP. Grains less than 500 nm are easily obtained.

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