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Fullerene/A5083 composites fabricated by material flow during friction stir processing

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Abstract

Fullerene was successfully dispersed into A5083 by friction stir processing (FSP). Dispersion of the fullerene enhanced the grain refinement by recrystallization during the FSP and the grain size reached ∼200 nm. The hardness was also remarkably increased by both the grain refinement and the dispersion of the fullerene molecules.

In this study, material flow during the FSP was investigated with respect to the dispersion of the fullerene. It was revealed that the formation mechanism of the "onion ring" was closely related to the convectional flow induced by the shoulder of the rotating tool. This material flow by the shoulder is very important when fabricating the surface composites by the FSP in order to provide a uniform dispersion of the reinforcement.

Keywords: A. Metal-matrix composites; A. Nano-structures; A. Particle-reinforcement; A. Discontinuous reinforcement

1. Introduction

Much attention has been paid to friction stir processing (FSP) which is well known as a surface modification technique based on friction stir welding (FSW) [1–5]. It has been basically developed for deleting various defects in cast products or improving the mechanical properties of light metals by the grain refinement related to the Hall–Petch relation. Recently, the FSP has been studied as a new process for fabricating surface composites. In this case, the surface of metal plates was modified by the dispersion of various ceramic particles with a low energy consumption [6]. The author's group reported that magnesium alloy based composites reinforced by SiC particles, MWCNTs, or fullerene could be fabricated by the FSP [7–9]. The dispersion of the reinforcement enhanced the mechanical properties of the processed area. Specifically, the fullerene dispersed AZ31 showed three times higher hardness compared to the as-received AZ31. This indicated that the FSP seems to be the best way to use the fullerene molecule, which is the hardest and smallest particle, as a nanometer-sized reinforcement. The fullerene molecule is unstable in the matrix at high temperature during the fabrication process such as a sintering [10]. The FSP which is well known as a solid-state process should be appropriate process to disperse the fullerene into the metallic materials, because of its low process temperature. However, the dispersion mechanism by the FSP is still unclear. Investigation of the basic dispersion mechanism is indispensable for further development of the FSP. The aim of this study is the establishment of a fabricating process of the surface composites by the FSP using the fullerene dispersion.

Understanding of the material flow in the stir zone is inevitable for the FSW to obtain an excellent joining [11–13]. Although many researchers have mentioned the "onion ring" formed in the stir zone [14–16], its formation mechanism has not been explained in detail. Therefore, the

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other aim of this study is to reveal the formation mechanism of the onion ring using the visualized material flow attained by the fullerene dispersion.

2. Experimental procedure

Commercially available fullerene powder (95% pure, C_{60}: 80 vol%, C_{70}: 15 vol%, Honjo Chem. Co., Japan) was used. The fullerene powder was filled into a groove (1 × 2 mm) on an A5083 plate (mean grain size: 25.4 μm) before the FSP was carried out. This process was explained in detail elsewhere [7]. The FSP tool made of SKD61 has a columnar shape (φ12 mm) with a probe (φ4 mm, length: 1.8 mm). The probe was inserted into the groove filled with the fullerene powder. The constant travel speed was 50 mm/min and the constant tool rotating rate was changed from 500 to 2000 rpm. The tool tilt angle of 3° was used.

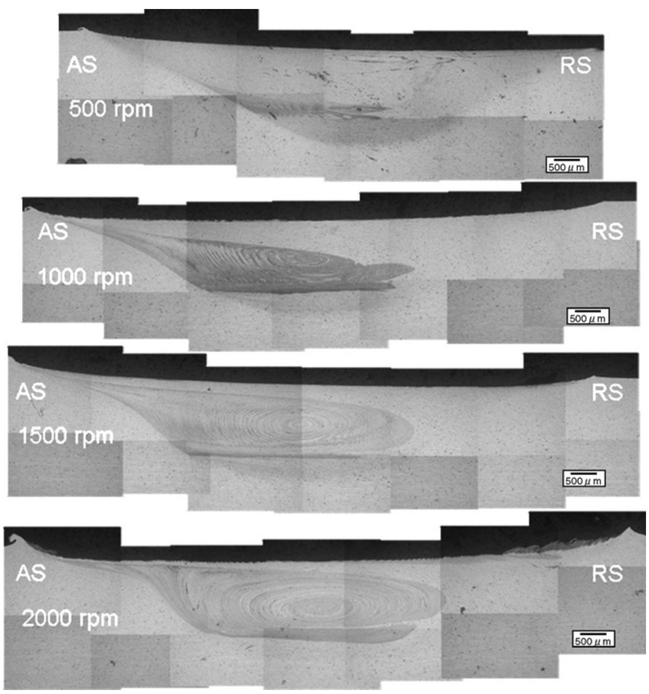

Fig. 1. OM images of the cross sections for the FSPed samples with the fullerene at various rotating speeds.

Cross and lateral sections of the FSPed samples were mounted and then mechanically polished. The material flow was observed by optical microscopy. The grain size of the matrix and the fullerene molecules in the matrix were observed by TEM (JEOL JEM-1200EX). 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. Material flow in stir zone

Cross sections of the FSPed samples are shown in Fig. 1. The material flow could be easily observed by the fullerene dispersion. For the sample FSPed at 500 rpm, fullerene was gathered in a specific area of the stir zone. On the other side, the fullerene was dispersed so as to form the onion ring in the stir zone for the sample FSPed at 1000–2000 rpm. The shape of the stir zone was changed from triangular to trapezoidal by the increase in the rotating speed. The onion ring could be clearly observed in the trapezoidal stir zone. It seems that the formation of the onion ring and the heat input were closely related. Fig. 2a shows a schematic of the material flow proposed by many researchers [17–19]. Though the material flow near the probe should be correct, it is difficult to form the onion ring using only

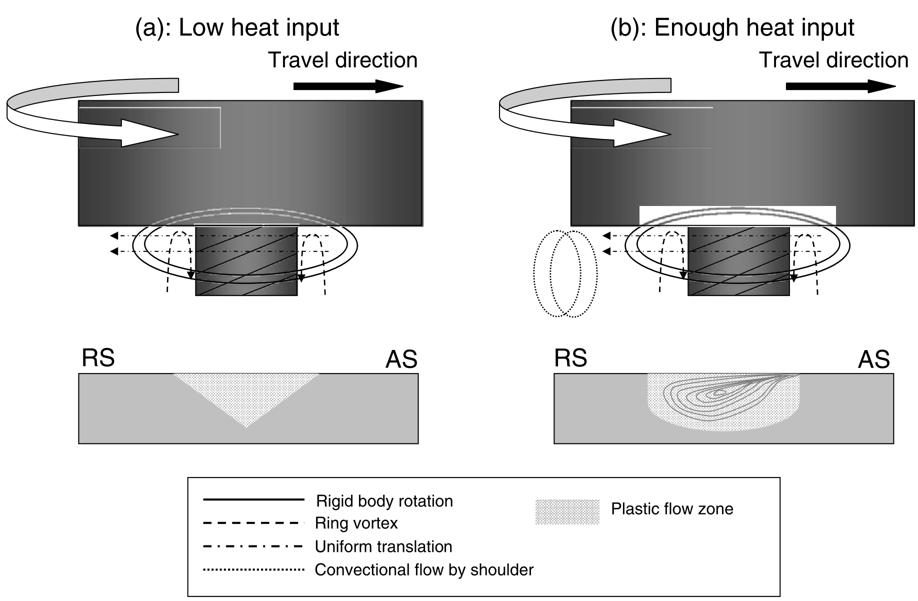

Fig. 2. Schematic of the relationship between material flow and heat input during FSP.

the proposed flows. Fig. 1 suggests that the convectional flow caused immediately before the consolidation of the stirred material should be indispensable for forming the onion ring. When the heat input is not sufficient, the flow does not occur, because of the high flow resistance. The onion ring could not be observed for the sample FSPed at 500 rpm. On the other hand, when the heat input is sufficient for the flow, a clear onion ring could be observed. Therefore, it is proposed that the onion ring is formed by the flow induced by the shoulder as illustrated in Fig. 2b.

Fig. 3 shows a lateral section of the fullerene/A5083 fabricated under various FSP conditions. The gap between the gray lines drawn by the fullerene dispersion corresponds to the revolutional pitch (travel speed of the rotating tool/rotating speed). In addition, the shape of the line is changed to an arc followed by the change in the rotating speed. The onion ring was observed in the samples with the clear arc lines. It is considered that the onion ring is the cross section of the overlapped cap shape formed by the convectional flow as shown in Fig. 4. It is difficult to form the cap shape under the small heat input condition, because the area of the plastic flow zone is not enough for generating the convectional flow. In this situation, a ditch should be momentarily formed at the rear of the probe during the FSP. The ditch was continuously filled by the stirred material due to the rotating tool. The stirred material in the ditch should be influenced by the rotating shoulder that generates the convectional flow as shown in Fig. 5.

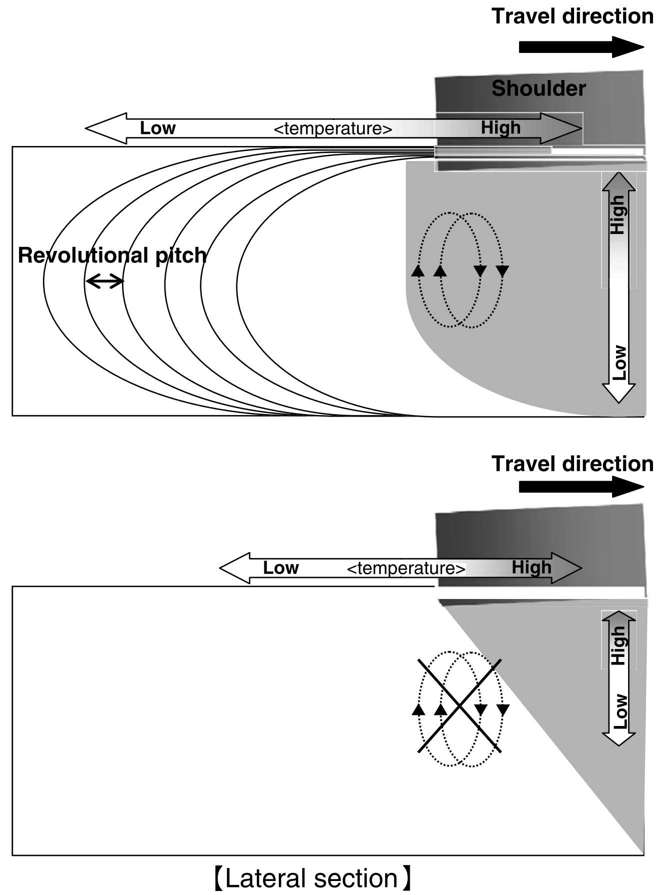

Fig. 4. Schematic of the relationship between the convectional flow by the shoulder and heat input.

3.2. Fullerene/A5083 nanocomposite

The gap between the lines in the onion ring is closely related to the revolutional pitch. The FSP with a smaller revolutional pitch can form a dense onion ring. If the fullerene is fully dispersed between the gaps, uniform composites could be obtained. Fig. 6 shows an optical microphotograph of the cross section for the FSPed sample with the fullerene paste at 1500 rpm. The fullerene paste was used to fill the much amount of fullerene into the groove. The volume fraction of the fullerene within the paste is 85%. The fullerene is uniformly dispersed in the center of the stir zone, because a significant amount of the fullerene could be filled in the groove using the fullerene paste. Based on these results, it is revealed that the

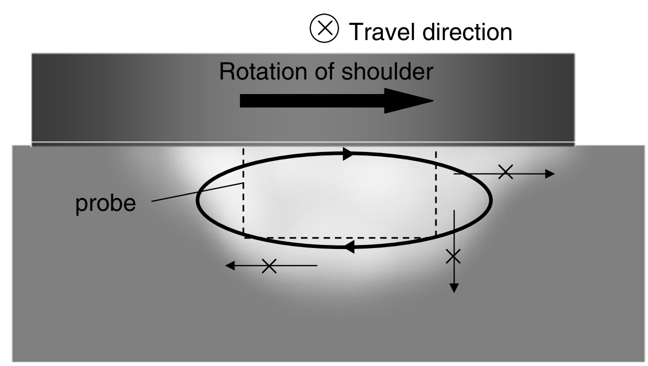

Fig. 5. Schematic of the convectional flow by the shoulder.

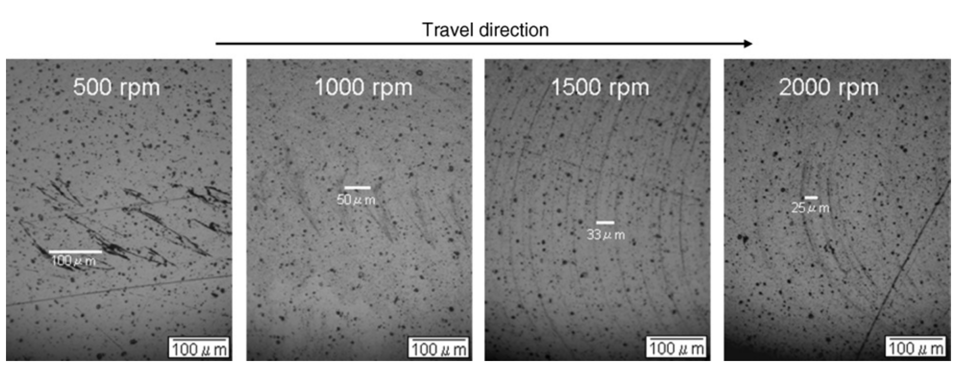

Fig. 3. OM images of the lateral sections for the FSPed samples with the fullerene at various rotating speeds.

dispersion condition could be controlled by the revolutional pitch and the amount of the reinforcement in the groove.

The grain size of the matrix is drastically decreased by the FSP with the fullerene dispersion as shown in Fig. 7.

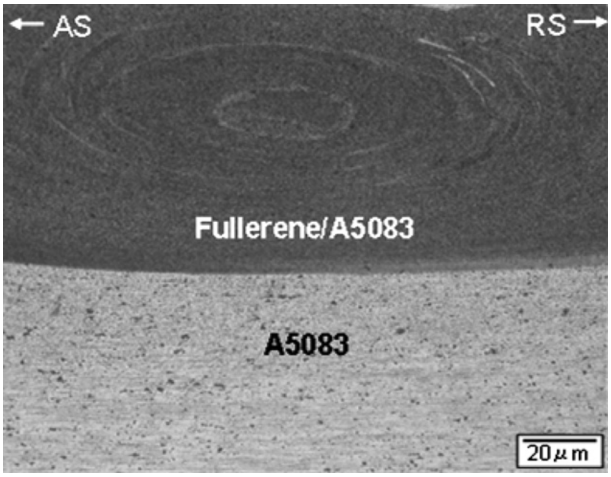

Fig. 6. OM image of the cross section for the FSPed sample with the fullerene paste.

There are single and somewhat aggregated fullerene molecules in the matrix. The grain of the matrix was refined to ~200 nm even under the FSP conditions of 50 mm/min and 1500 rpm. This FSP condition generated a relatively high heat input which leads to grain growth after the dynamic recrystallization. The recrystallizaiton during the FSP has been wildly studied from every aspect, because it is a very important phenomenon for the metallurgy and applications [20,21]. The grain size of about 1.8–9.8 μm were reported for the FSPed A5083 plate which was processed at various conditions [22]. It is considered that the grain growth was restrained by the pinning effect of the fullerene molecules dispersed in the grain boundary of the matrix.

Fig. 8 shows the depth profile of the microhardness. The hardness increased for the FSPed sample containing the fullerene dispersion. For the sample FSPed at 1000 rpm, some areas show a higher hardness due to the dense dispersion of the fullerene. Although the values are scattered, the FSPed sample with the fullerene paste shows a higher hardness which is the almost double that of the matrix from the surface to about 1.5 mm in depth. The indentation print for

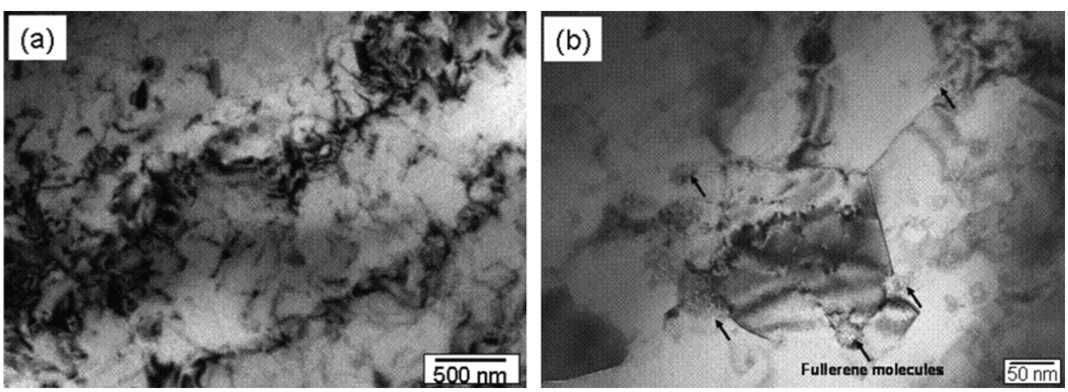

Fig. 7. TEM images of the FSPed sample with the fullerene paste: (a) low magnification and (b) high magnification.

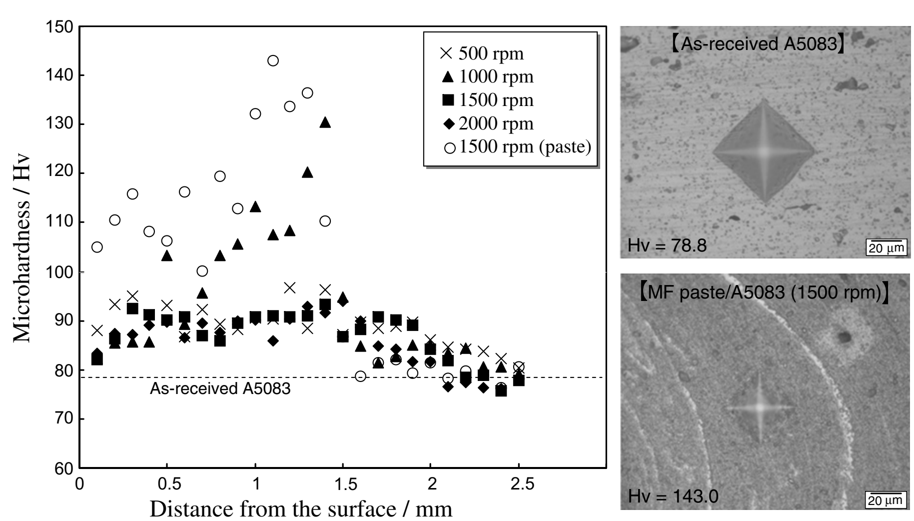

Fig. 8. Microhardness profile of cross sections in the FSPed samples and the representative indentation prints.

the region with fullerene was clearly smaller compared to that for the as-received A5083. The FSP with the fullerene obviously increases the microhardness of the substrates by the promotion of the grain refinement due to the pinning effect of the fullerene and its extremely high hardness. Though the fine grains were certainly produced by the recrystallization, the microhardness might be some influenced by the dislocation.

4. Conclusions

The fullerene/A5083 composites were successfully fabricated by the FSP. The material flow in the stir zone during the FSP was studied by visualization using the fullerene dispersion. Additionally, the microstructure and microhardness were evaluated by observing the grain size and the dispersion of the fullerene. The obtained results can be summarized as follows.

(1) The fullerene molecules can be dispersed into the A5083 matrix using the FSP.
(2) The onion ring is formed by the convectional flow due to the shoulder.
(3) The FSP with the fullerene obviously increases the microhardness of the substrates by the promotion of the grain refinement due to the pinning effect of the fullerene and its extremely high hardness.
(4) A5083 with a grain size of less than 200 nm is easily obtained using the fullerene dispersion.

Acknowledgements

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