

The effect of friction stir processing with SiC particles on microstructure and hardness of Fe-7Al alloy

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Abstract

The surface of Fe-7Al (mass%) ferritic alloy plate was successfully modified by friction stir processing (FSP) with SiC particles. The stir zone with SiC addition had an average grain size of 4.3 μm , smaller than that of 7.0 μm in the stir zone without SiC addition. SiC particles introduced by FSP were broken into pieces by interaction with the rotating tool and were converted to Fe_3AlC_x

particles by reaction with the ferrite matrix. The hardness near the surface of the stir zone was significantly increased to 270 HV by introduction of particles, compared to the hardness of 200 HV in the stir zone without particle additions.

1. Introduction

Much attention has been paid to friction stir processing (FSP), which has the same principle as friction stir welding (FSW) [1-4]. A rotating tool with a probe and a shoulder is inserted into a substrate and produces a highly deformed zone. Due to dynamic recrystallisation, fine equiaxed grains can be obtained in the stir zone. Recently, some researchers have reported that SiC particles were successfully dispersed as a reinforcement in aluminum alloy, magnesium alloy, and pure copper by FSP [5-8]. They showed SiC addition was very effective in reinforcement and grain refinement of the matrix.

In this study, FSP with SiC particles was carried out on Fe-7Al (mass%) alloy. This alloy has lower density and higher damping capacity than plain carbon steel [9]. It has also room temperature ductility of 20 % or more owing to its disordered bcc structure [10,11]. These properties can contribute to reduced emissions and less noise for automobiles. However, Fe-7Al alloy has low strength because it is single phase of ferrite below its solidus temperature [12]. FSP with SiC particles could be expected to strengthen the surface of the alloy plate. The microstructure and

hardness in the stir zone were evaluated in order to clarify the effect of FSP with SiC particles.

2. Experimental procedures

Commercially available SiC powder with mean size of 2 μm and purity of 99% was used in this study. Fig. 1 shows the SiC powder particles. Fe-7Al (mass%) alloy plates in hot rolled condition were used. These plates were 80 mm in length, 80 mm in width and 6 mm in thickness. Fig. 2 presents the schematic illustration showing the friction stir processing in this study. A groove with 1 mm in width and 1 mm in depth was made on the surface of a plate. The SiC powder was filled into the groove and pressed with a spatula before FSP with SiC addition. A WC-Co alloy tool, which had a shoulder with 12 mm in diameter and an unthreaded probe 7 mm in diameter and 0.7 mm in length, was used. FSP was load-controlled at 1500 kgf. A tool rotating rate of 400 rpm and a traveling speed of 400 mm/min were adopted. The tool was tilted by 3° about the vertical axis. The FSP was carried out along the center line of the groove with and without SiC particles.

Microstructures of the stir zone were observed by optical microscopy. The FSPed samples were cross-sectioned perpendicular to the traveling direction, mechanically polished and etched with a 5 % nitric acid and ethanol solution. The grain size of the etched sample was estimated by using the mean linear intercept method. Crystal structures near the surface of the plates before and after FSP were identified by X-ray diffraction (Rigaku RINT 2500V). The diffractometer was operated at 40

kV and 40 mA. To prepare TEM samples, thin plates were cut near the surface of the stir zone and then mechanically polished to a thickness of 100 μm . The polished thin plates were finally twin-jet electropolished to make thin films using a solution of 5 % perchloric acid, 15 % glycerol and 80 % methanol at 20.5 V and $-30\text{ }^{\circ}\text{C}$. The thin films were observed with a TEM (JEOL 200CX) at 200 kV. The Vickers hardness profiles were measured along the centerline of the cross-section in the stir zone by using a micro-Vickers hardness tester (Akashi HM-124) with a load of 300 gf.

3. Results and discussion

An optical microscope image of the cross-section of Fe-7Al alloy after FSP with SiC particles is shown in Fig. 3. No obvious defects or superfluous flash could be observed. The distribution of particles appeared in a large area in the stir zone (SZ). The horizontal flow of the material is apparent. It is considered that the maelstrom current of the material hardly occurs owing to the use of an unthreaded rotating tool [13, 14]. In addition, the particles were distributed more in the advancing side (AS) than in the retreating side (RS). This result is consistent with those of other materials FSPed with particle additions [8, 15].

Fig. 4 shows XRD patterns of as-received and FSPed Fe-7Al alloy plates. The main microstructural constituent was ferrite before and after FSP. These results are consistent with Fe-Al binary phase diagram, showing that Fe-7Al alloy is single phase ferrite below its solidus temperature

[12]. Peaks of $\text{Fe}_3\text{AlC}_{0.69}$ (κ -carbide) with perovskite structure, and SiC were observed in the SZ with SiC addition. This implies that some of the SiC particles reacted with the matrix during FSP.

The dispersion of particles influenced the grain size of the matrix. Fig. 5 shows cross-section near the surface of the SZ after FSP with and without SiC addition. Fine equiaxed ferrite grains were observed in both stir zones. Besides, banded structures were observed in the SZ with SiC addition, which consisted of particle-rich regions and particle-depleted regions. The mean grain size of the ferrite in the SZ with or without SiC addition was $2.7 \pm 0.35 \mu\text{m}$ or $4.7 \pm 1.0 \mu\text{m}$, respectively. The FSP with SiC particles is supposed to reduce grain sizes efficiently due to increases of stored energy of the matrix and pinning effects of dispersed particles.

Microstructures in the SZ of Fe-7Al alloy after FSP were evaluated by transmission electron microscopy, as shown in Fig. 6. Ferrite grains and dispersed particles were observed in the stir zone after FSP with SiC particles. Few dislocations were found in the ferrite grains. It indicates that dynamic recovery and/or recrystallisation occurred during FSP. Additionally, dispersed particles were observed, which were much smaller than SiC particles before FSP, because they are fractured into finer pieces by the interactions with the rotating tool. This behavior agrees with the results of FSP with SiC particles on the other alloys such as aluminum alloys, magnesium alloys and pure copper [6-8].

Fig. 7 shows high magnification images of dispersed particles and the selected area diffraction

pattern of the particle indicated by an arrow. From this pattern, the particle is identified to be κ -carbide with perovskite structure. The result also corresponded to the carbide on the XRD pattern after FSP with SiC addition, as shown in Fig. 4. According to the reports on Fe-Al-C alloys, Fe_3AlC_x with perovskite structure should have $L1_2$ structure consisting of Fe and Al atoms with C atoms in the octahedral interstices [16, 17]. The carbide is known to occur in the disordered bcc phase in Fe-7Al-0.5C (mass%) alloy [18]. Consequently, it is considered that SiC particles were broken into fine pieces and converted to Fe_3AlC_x due to the reaction with the matrix during FSP.

Hardness depth profiles along the center line of the cross-section in the SZ are shown in Fig. 8. The hardness near the surface of the SZ reached 270 HV by introduction of SiC particles, which is greater than that of the base metal (200 HV). In contrast, the hardness in the SZ without SiC particles was not increased by FSP. This confirms that the additional hardening is associated with the addition of SiC particles is attributed to the dispersed Fe_3AlC_x particles and refined ferrite grains.

4. Conclusions

FSP with SiC successfully introduced a dispersion of particles in the affected surface of an Fe-7Al ferritic alloy plate. The microstructure and hardness were evaluated by comparison against a stir zone without the introduction of SiC particles. The results can be summarized as follows:

(1) The microstructure of the stir zone is found not to be severely deformed, indicating dynamic

recovery and recrystallisation during friction stir processing. A fine dispersion of particles was created, that led in turn to a fine distribution of ferrite grains in the stir zone.

- (2) SiC particles introduced by FSP are refined by interaction with the rotating tool and are converted into Fe_3AlC_x particles by reaction with the ferrite matrix.
- (3) The hardness near the surface of the stir zone can be significantly increased to 270 HV by the introduction of particles, compared to the hardness of 200 HV in the stir zone without particle additions.

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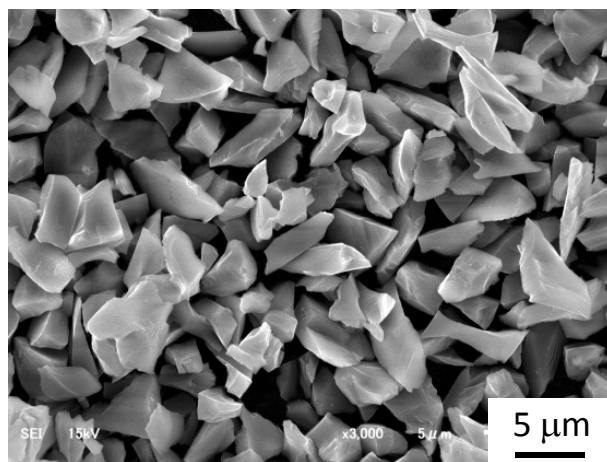


Fig.1 As-received SiC particles.

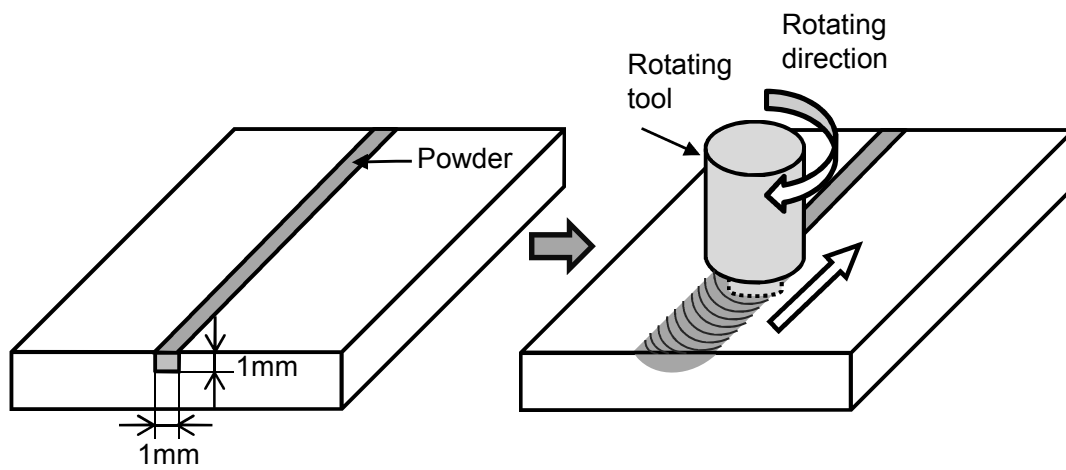


Fig. 2 Schematic illustration of friction stir processing with particle addition.



Fig. 3 Cross-section of Fe-7Al alloy after FSP with SiC addition.

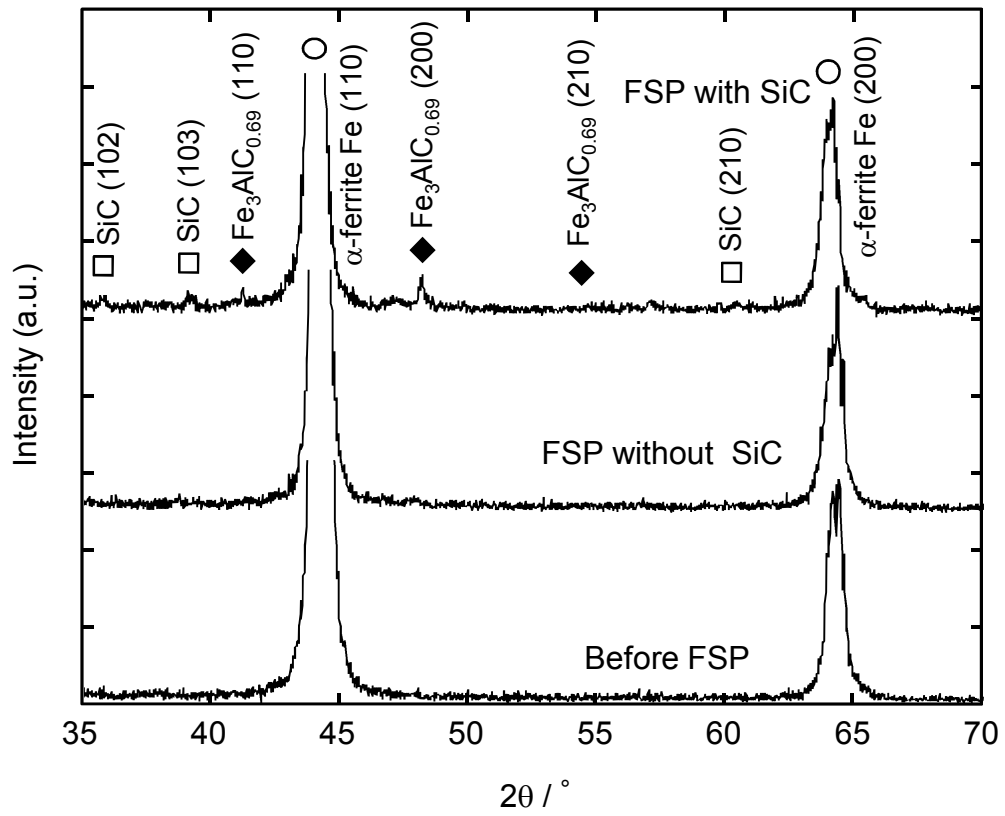


Fig. 4 XRD patterns of Fe-7Al alloy before and after FSP.

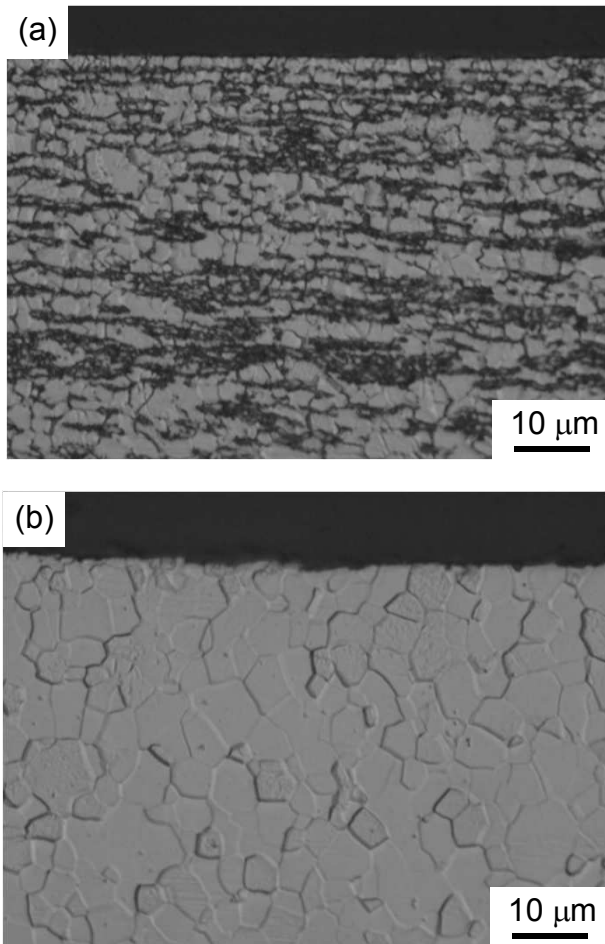


Fig. 5 Cross-section near the surface of the SZ after FSP: (a) with SiC addition and (b) without SiC addition.

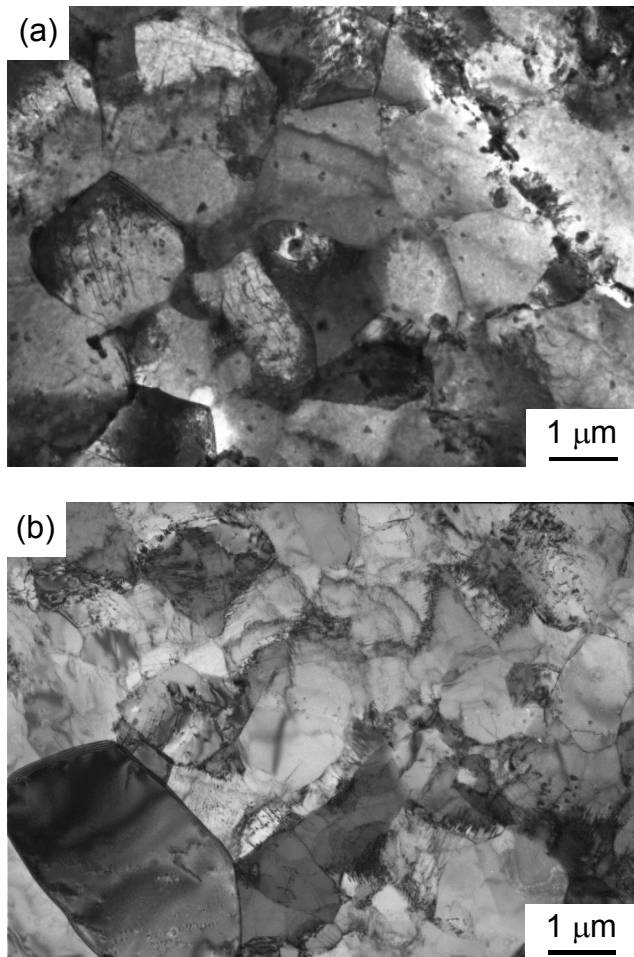


Fig. 6 Microstructures in the stir zone of Fe-7Al alloy after FSP: (a) with SiC addition and (b) without SiC addition.

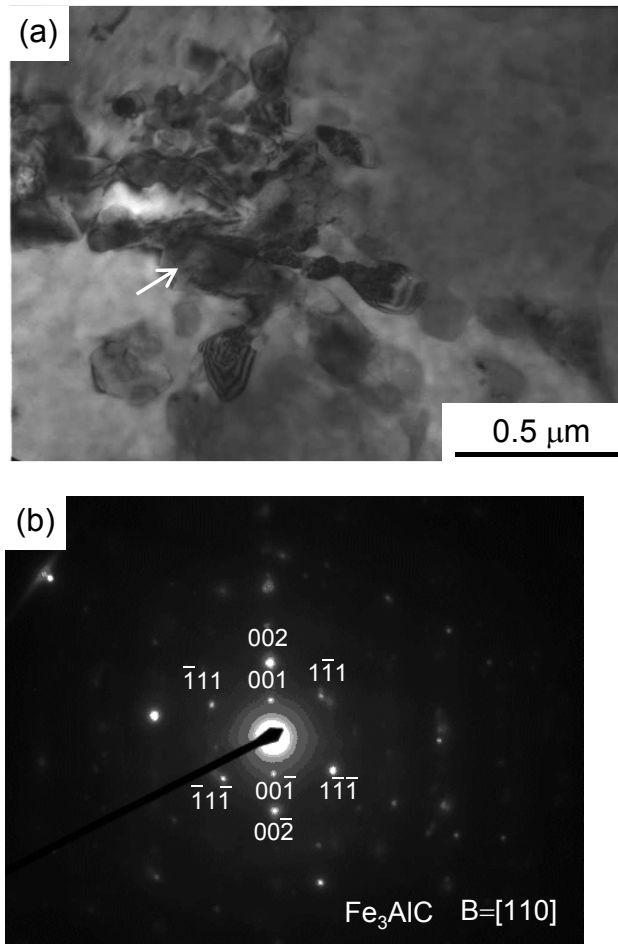


Fig. 7 Particles in the stir zone of Fe-7Al alloy after FSP with SiC addition: (a) bright field image and (b) selected area diffraction pattern.

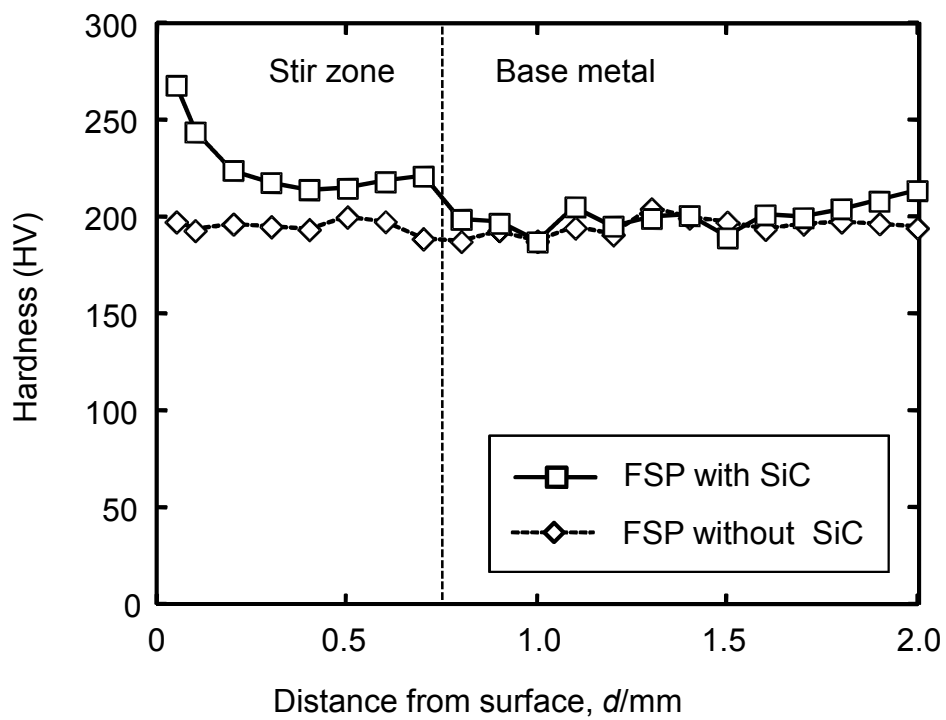


Fig. 8 Hardness depth profile along the centerline of the cross-section of the FSPed Fe-7Al alloy with and without SiC addition.