

Fabrication of Surface-Hybrid-MMCs Layer on Aluminum Plate by Friction Stir Processing and Its Wear Characteristics

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Aluminum-base hybrid composites reinforced with mixtures of SiC and Al₂O₃ particles 1.25 μm in average size have been fabricated on an A 1050-H24 aluminum plate by friction stir processing (FSP) and their wear resistance has been investigated as a function of the relative weight ratios of the particles. A mixture of SiC and Al₂O₃ powders of different weight ratios was packed into a groove of 3 mm width and 1.5 mm depth cut on the aluminum plate, and covered with an aluminum sheet 2 mm thick. A FSP tool of square probe shape, rotated at a speed of 1500 rpm, was plunged into the plate through the cover sheet and the groove, and moved along the groove at a travelling speed of 1.66 mm/s. After the hybrid composite was fabricated on the Al plate, the homogeneity of the particles distribution inside the Al matrix has been evaluated from the macro/microstructure and hardness distribution. Moreover, the wear characteristics of the resulted hybrid composites were evaluated using a ball-on-disc wear tester at room temperatures at normal loads of 2 and 5 N. As a result, it was found that the reinforcement particles were distributed homogeneously inside the nugget zone without any defects except some voids that appeared around the Al₂O₃ particles. The average hardness decreased with increasing the relative content of Al₂O₃ particles. Regarding the wear characteristics, the wear volume losses of the hybrid composites depended on the applied load and the relative ratio of SiC and Al₂O₃ particles. The hybrid composite of 80% SiC + 20% Al₂O₃ showed superior wear resistance to 100% SiC and Al₂O₃ or any other hybrid ratios at a normal load of 5 N.
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1. Introduction

Surface metal matrix composites (SMMCs) exhibit a unified combination of high tribological properties of the surface and high toughness of the interior bulk metal when compared with both metal matrix composites (MMCs) and monolithic materials.¹⁻⁵ Selecting reinforcement remains one of the most critical factors in realizing the best properties from the resultant SMMCs.⁶⁻⁹ The widely used particles for reinforcing Al alloys are silicon carbide (SiC) and alumina (Al₂O₃).¹⁰⁻¹⁵ Besides their high hardness, they show low density and low cost comparing with other reinforcements. In the past two decades, the wear resistance of the aluminum alloys reinforced with SiC and Al₂O₃ in many forms (particles, whiskers and fibers) and sizes has been described by a huge body of publications.⁶⁻²⁰ Most studies⁶⁻¹⁶ proved that SiC was more effective than Al₂O₃ in the improvement of wear resistance of the resultant aluminum composite due to its higher hardness.⁶⁻¹⁶ On the other hand, some investigations¹⁷⁻²⁰ suggested that Al/Al₂O₃ composites were more stable and inert, and had better corrosion and high temperature behavior than Al/SiC.¹⁷⁻²⁰ In this sense, it may become practical to use both SiC particles and Al₂O₃ particles (hybrid) as reinforcements to gain both of their benefits for improving the composite properties.

Hybrid MMCs are engineering materials reinforced by a combination of two or more different type and/or form of substances in order to achieve the combined advantages of both of them. This gives a rather high degree of freedom in material design.¹⁶ It was reported in many works^{10-12,21-25} that the hybrid composites of SiC and Al₂O₃ exhibited better wear resistance than those reinforced only with the SiC or Al₂O₃.^{10-12,21-25}

It is generally known that the fabrication of SMMCs by traditional fusion surfacing techniques such as laser beam and thermal spraying may lead to the deterioration of MMCs properties.²⁶⁻³⁰ In the case of Al₂O₃, the Al₂O₃ may decompose to Al and oxygen gas (forming porosities) on contact with liquid aluminum in addition to the tendency of the Al₂O₃ particle to clustering.³⁰ Similarly, the SiC may react with molten aluminum to form brittle Al₄C₃ carbide.^{21,29} Moreover, one of the severe problems in preparation of hybrid composites by normal fusion process is the agglomeration of each reinforcement, which results in inhomogeneous reinforcement distribution in the matrix.³¹ Considering these problems, friction stir processing (FSP) seems to be a good candidate for successful fabrication of hybrid SMMCs on the Al plate.²⁹

Friction stir processing (FSP) is a microstructure modification technique developed by Mishra³² based on the concept of friction stir welding, but its goal is different.^{29,32,33} A non-consumable rotating tool consisting of a shoulder and a probe is plunged into a single piece of material, and provides frictional heating and mechanical mixing in the area covered by the tool.^{29,32,33} It is considered that SMMCs are one of the latest applications of FSP.³⁴⁻⁴⁰ The stirring action and frictional heat generated by the FSP tool can be used to distribute ceramic particles as reinforcement on the surface of light metals like aluminum and magnesium.³⁴⁻⁴⁰

The present authors^{39,40} succeeded in uniformly distributing SiC particles of 1.25 μm size in a surface layer of a few mm thickness on Al 1050-H24 plates to form surface composites by FSP.

In the present work, aiming to obtain the hybrid composites by FSP of excellent tribological properties, we plan to extend our study of surfacing the 1050-H24 Al to include the possibility to distribute two types of reinforcements: SiC and Al₂O₃ particles.

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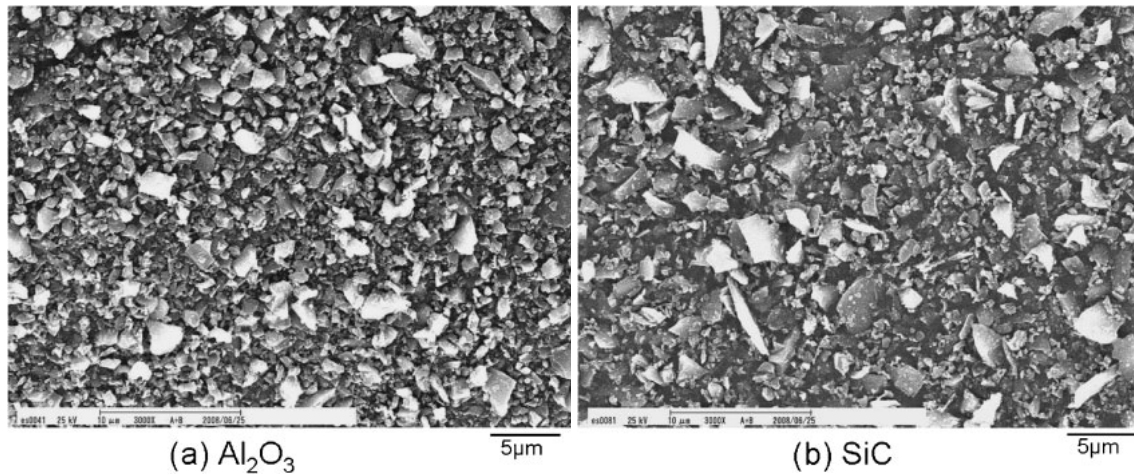


Fig. 1 SEM images of the as-received Al_2O_3 and SiC powders.

2. Experimental Setup

Commercially pure aluminum Al-1050-H24 plates of 5 mm thickness were used as the base material. Mixtures of SiC and Al_2O_3 particles at different ratios were used as the reinforcements. They had similar average particle diameters of $1.25\ \mu\text{m}$ as shown in Fig. 1.

A tool of steel SKD61 with a shoulder of 14 mm diameter and a square probe of 5 mm diagonal length and 3.3 mm height was used to perform the FSP. The reinforcement powder was packed in a groove of 3 mm width and 1.5 mm depth cut on the Al plate. An aluminum sheet of 2 mm thickness was used to cover the groove filled with the reinforcement powders to prevent them from going out before they were incorporated into the aluminum matrix during the FSP. The tool was rotated at a rotation speed of 1500 rpm, and travelled at a speed of 1.66 mm/s with a tilt angle of 3° . These conditions produced the best results in terms of homogeneity of the particles inside the nugget zone for producing the SMMCs of the pure Al reinforced with SiC particles.^{39,40} Double and triple passes were applied in order to improve the homogeneity of the reinforcement particle distribution. In the second pass, the tool was travelled along the same line as the first and third ones but in the opposite direction. This means that the advancing side of the first and third passes became the retreating side in the second pass (refer to Refs. 39) and 40)). Macro- and microstructure observations were performed on the cross-section of the FSP specimens. Scanning electron microscope (SEM, ELIONIX, ERA-8800FE) was used to study the distribution of reinforcement particles. The microhardness was measured with AKASHI Model Vickers hardness tester at 200 g load applied for 15s through the mid-plane of the cross-section of the nugget of each condition. The hardness distribution was measured along three parallel lines that were 1–2 mm deep from the upper surface and separated by a distance of 0.5 mm. The interval between the indentations on the same line was 0.75 mm. All measured data along the three lines were collected and averaged for each condition. The nugget zones produced by FSP triple passes were analyzed by X-ray diffractometer, (XRD, D 8 Discover with GADDS system, 35 kv, 80 mA, $\text{MoK}\alpha$ radiation) to identify experimentally

the phases that were present inside the FSP nugget zone after 3 passes.

The wear behavior of the SMMCs was evaluated by using a ball-on-disk (FPR-2100, RHESCA CO., LTD) tester in air at room temperatures. Stainless steel balls (SUS 304) with a diameter of 4.5 mm were used as the counterpart material. Before the test, all the specimens were ground on emery paper up to 600 grade until the end of the cover sheet, which was consider almost the FSP nugget zone center. The tests were carried out at normal loads of 2 and 5 N applied to the SUS 304 balls. The radius of the ball travelling was 6 mm, the rotation speed was 100 rpm (linear velocity of 31.4 mm/s) and the total rotation time was 1 ks. The friction force was recorded automatically against time by the tester software. The friction coefficient of the stainless steel ball against the SMMCs was calculated from the ratio of the friction force to the normal load. The wear volume loss was measured with a surface profilometer (SURFOM 1400D) at 8 different locations (every 45°) along the wear track, and the average of the measured values represented the mean wear volume losses of the specimen. Examples of the surface profilometer charts of the cross-section area of the wear tracks are shown in Fig. 2. The wear track was examined with a scanning electron microscope (SEM, ELIONIX, ERA-8800FE) at low magnifications to discuss the wear mechanism. For comparison, the wear behaviors of the nugget zone of a pure aluminum produced by FSP without reinforcements under the same processing conditions were also investigated.

3. Experimental Results and Discussion

3.1 Composites homogeneity after first pass

Macroscopic appearances of the nugget zone in which SiC, SiC + Al_2O_3 and Al_2O_3 powders were dispersed by the first FSP pass are shown in Fig. 3. In general, the macroscopic particle distribution inside the area under the shoulder that subjected to intense plastic deformation during FSP or so called “nugget zone”²⁹) was almost similar regardless the reinforcement type. This is due to that all the samples were fabricated at the same rotation speed of 1500 rpm and with a tool of the same design, which were the main parameters that control the material flow inside the nugget zone as mentioned

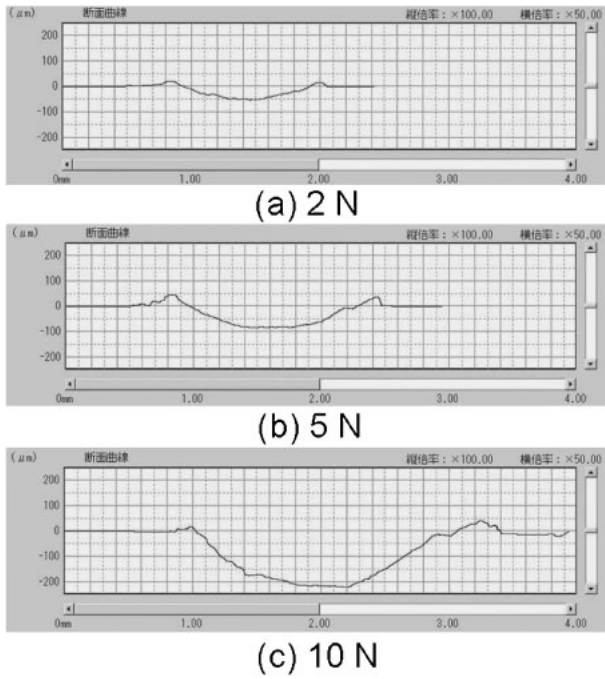


Fig. 2 Examples of surface profilometer charts of wear tracks produced at different loads.

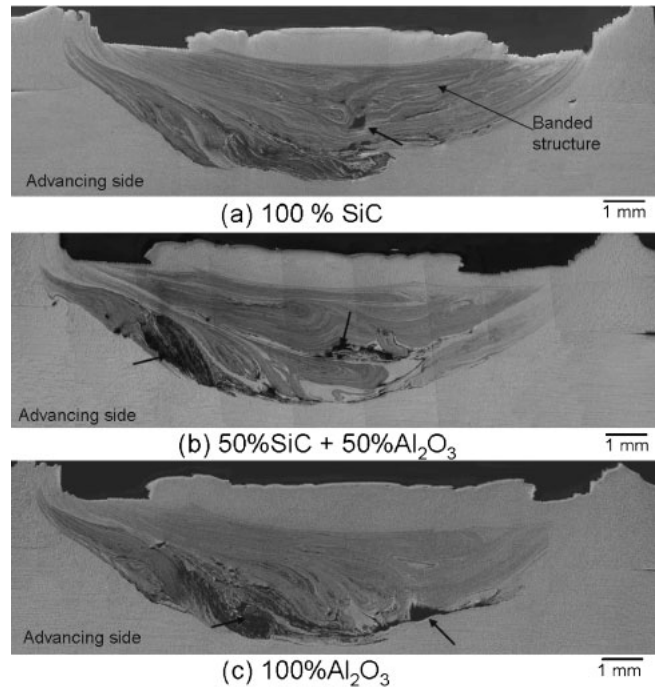


Fig. 3 Micrographs of the nugget zone cross-section after the first FSP pass.

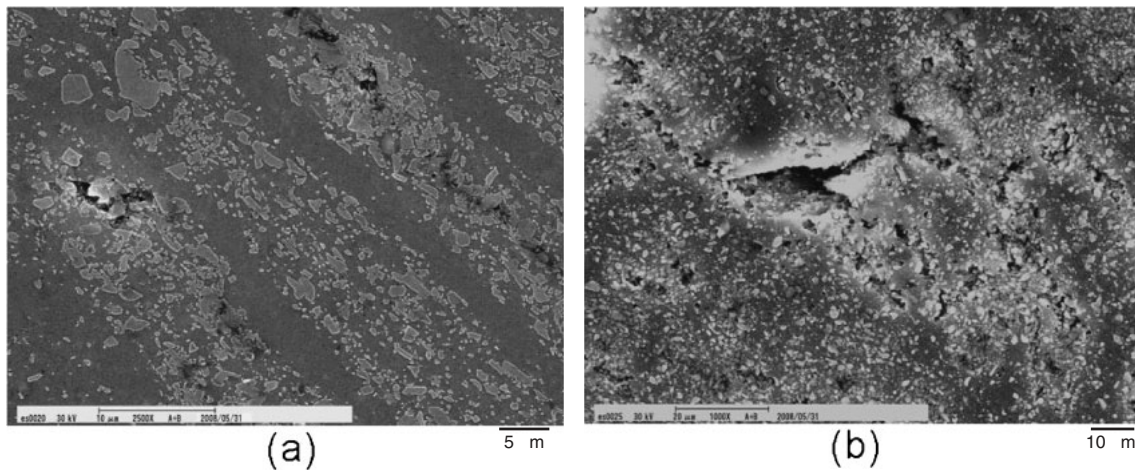


Fig. 4 SEM images of some local areas in Fig. 3 after the first pass: (a) banded structure in Fig. 3(a) and (b) reinforcement particles clusters in Fig. 3(b).

in our previous works.^{39,40} The reinforcement particles were distributed in a banded structure towards the top of the advancing side, leaving the retreating side and the central top portions with low density of reinforcement particles. The banded structure was clearer when the reinforcement was SiC particles as shown in Fig. 4(a). Some reinforcement particles clustering were observed inside the nugget zone as indicate by arrows in Fig. 3 and as shown in Fig. 4(b). These clustering areas were increased with the content of Al₂O₃ particles (compare Fig. 3(a) with Fig. 3(c)).

3.2 Composites properties after triple passes

Macroscopic appearances of the nuggets cross-sections produced by triple FSP passes with different relative contents of SiC and Al₂O₃ powders are shown in Fig. 5. As expected, the reinforcement particles were distributed more widely and

uniformly in the nugget zone for all weight ratios of SiC and Al₂O₃ powders by applying three FSP passes. This was due to the stirring action generated in every pass by the rotated tool. In the same time the reinforcement density on the advancing side became almost similar to that on the retreating side. This was due to the opposite travelling direction of the second pass to the first and third passes. The macroscopic behavior of the powder mixtures of SiC and Al₂O₃ inside the nugget zone was quite similar regardless their relative content except for small variations in the nugget zone homogeneity; i.e., small reinforcement clustering was detected at the lower part of the nugget zone when the ratio of Al₂O₃ was not less than 80% (see Figs. 5(h) and (i)). On the other hand, some small elongated onion ring structures were appeared inside the nugget zone when the SiC ratio was high (see Figs. 5(a), (b), and (c)).

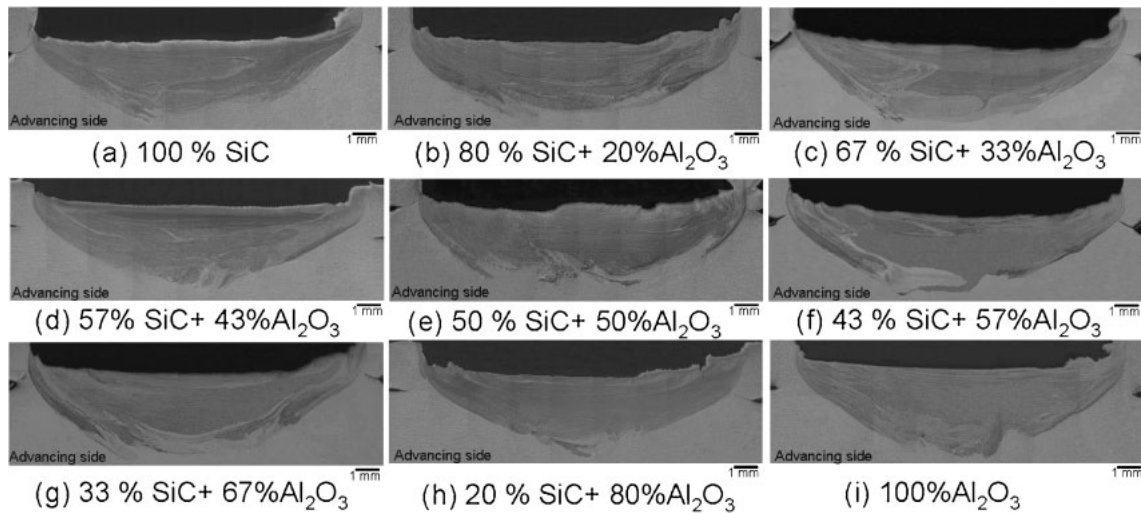


Fig. 5 Micrographs of the nugget zone cross-section of different hybrid composites.

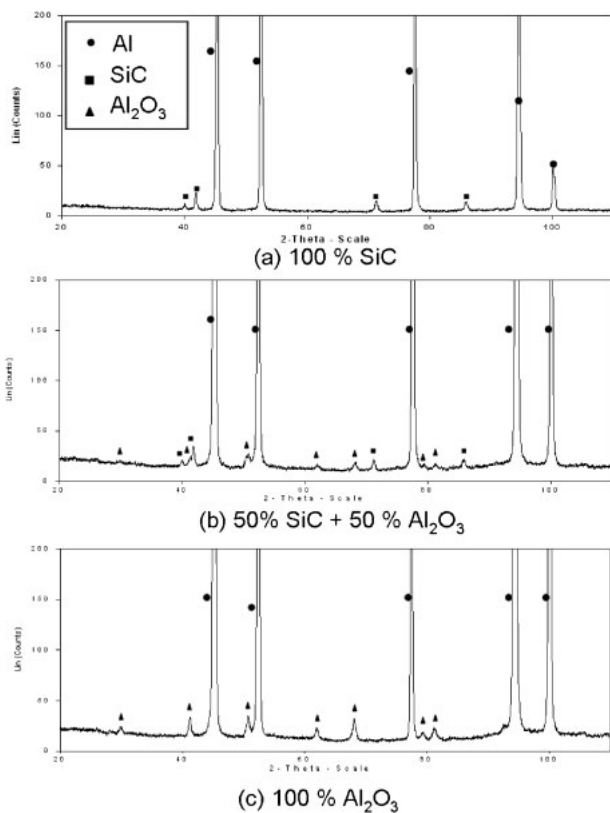


Fig. 6 XRD patterns of nugget zone cross-section after triple passes.

On the other hand, as stated in our previous paper,⁴⁰⁾ the multi-pass FSP has almost no effect on the size of the nugget zone. This is due to that the following FSP pass was done after the sample was cooled down to room temperatures.⁴⁰⁾ Moreover, the FSP tool was eroded by the hard ceramic particles (SiC and Al₂O₃) and the worn material (mainly iron) reacted with Al matrix to form iron aluminides as described in details in our previous work.⁴⁰⁾

The XRD patterns of the nugget zone cross-sections that had different reinforcement ratios are shown in Fig. 6. It was clear that there were no new phases except the added

reinforcement powders (SiC, Al₂O₃) and the aluminum matrix. This suggests that no reaction occurred between the SiC and Al₂O₃ powders or between the aluminum matrix and the powders during three FSP passes, which can be considered to be a merit of the FSP as a surfacing technique over other traditional fusion surfacing techniques such as laser beam and thermal spraying.²⁹⁾ It was reported by many authors^{14,21,30)} that there were undesirable interfacial reactions between the matrix and the reinforcement during fabrication by liquid surfacing techniques. For example, Majumdar *et al.*,¹⁴⁾ detected large Al-carbide plates at the SiC/Al of SiC particles introduced onto Al-substrate by laser injection. This reaction product Al₄C₃ is very brittle, which degrades the composite material especially in humid environments.¹⁴⁾

Microscopically, the SEM images of the centers of the nugget cross-section, as shown in Fig. 7, suggest that the particle distribution in the matrix was almost uniform under all the processed conditions. These areas represent the most important area in the nugget zone practically, this is due to that surfacing is done by applying many adjacent parallel passes to cover the area of interest. The adjacent pass will overlap the advancing or retreating side of the previous one which share in more particle distribution. The central portion of the nugget tends to remain as it is without changes. However, presence of small voids around some particles or at the particle/matrix interface was observed in the nugget zones of higher Al₂O₃ contents. By EDS analyses, it was found that these micro voids were mainly located around the Al₂O₃ particles while it was less noticeable around SiC particles as shown in Fig. 8. This may be related to the better wettability of the SiC with aluminum matrix than that of Al₂O₃.^{6,12)}

The EDS mapping of Si and O in the center of the nugget zone of different powder ratios were carried out to observe the SiC and Al₂O₃ particles distribution. The reliability of oxygen by EDS is relatively weak, but we use it only to detect the Al₂O₃ particles not to measure its amounts. As shown in Fig. 9, it is clear from the mapping that both SiC and Al₂O₃ particles were mixed together in the nugget zone; i.e., they

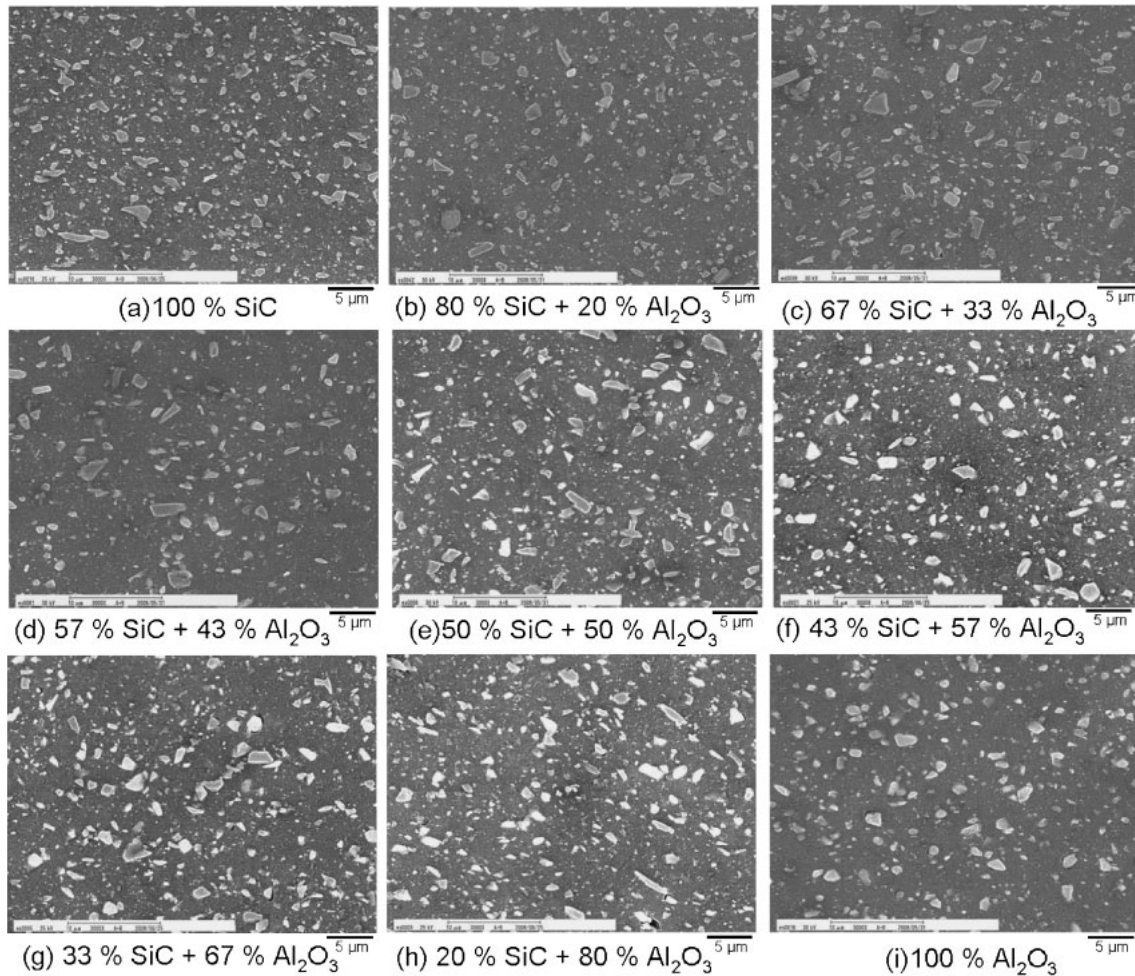


Fig. 7 SEM micrographs of the nugget center with different hybrid composites.

exhibited no separate clustering or banding of one type of reinforcement compared with the other, or in other words, they behaved as one type of reinforcement. This may be due to the strong stirring action which was considered to be one of the most prominent advantages of FSP over all welding and surfacing processes.^{29,33–40} It was reported by some literatures^{22,31} that one of the problems in the fabrication of the hybrid-MMCs by fusion processes or even by other ordinary solid-state processes such as power metallurgy (PM) was the agglomeration of the different reinforcements, especially the finer particles.²²

In contrast to the reinforcement particles distribution over the most areas of the nugget zone, there were some inhomogeneity in the peripheral areas as shown in Fig. 10. The reinforcement particles tended to be dispersed in banded structure as shown in Fig. 10(a). In the retreating and advancing sides, reinforcement particles of relatively high density or clustering were detected as shown in Figs. 10(b) and 10(c). In the lower part of the nugget zone, when the ratio of Al_2O_3 was higher than 80%, some dense reinforcement areas were observed as shown in Fig. 10(d). These inhomogeneous distributions appeared only in the peripheral areas of the nugget zone where the effect of the tool stirring action became weak.

The average microhardness of the nugget zone is plotted against the relative content ratio of Al_2O_3 in the reinforce-

ment in Fig. 11. As compared with average hardness ~ 23 HV of the FSP nugget zone without reinforcement, the hardness values of the reinforced composites were increased by twice (~ 47 HV) for 100% Al_2O_3 and almost three times (~ 60 HV) for 100% SiC. It was revealed from the diagram that the hardness tended to decrease almost linearly with an increase in the relative amount of Al_2O_3 content in the composite. This is partially because of the higher hardness of the SiC particles than Al_2O_3 particles. The existence of micro voids at the Al_2O_3 particle/matrix interface also seems to share in lowering the hardness of the composite material. According to the previous works, the obtained hardness values are quite similar to other MMCs fabricated by FSP and also other methods if we take into consideration that our substrate was pure Al. For example, Morisada *et al.*³⁵ distributed SiC particles in AZ31 ($\text{HV} \approx 40$) with FSP, and the hardness of the resulted MMCs was about 70 HV. Ünü²⁰ fabricated Al/ Al_2O_3 MMCs with powder metallurgy and the resulted hardness was 55 HV. Reihani¹⁰ fabricated Al6061/SiC with squeeze casting and the hardness was about 85 HV. On the other hand Anandkumar¹¹ surfaced the Al-Si (90 HV) alloy with SiC particles by laser cladding and the hardness reached 120 HV.

3.3 Wear characteristics

In order to investigate the effect of Al_2O_3 /SiC ratio on the

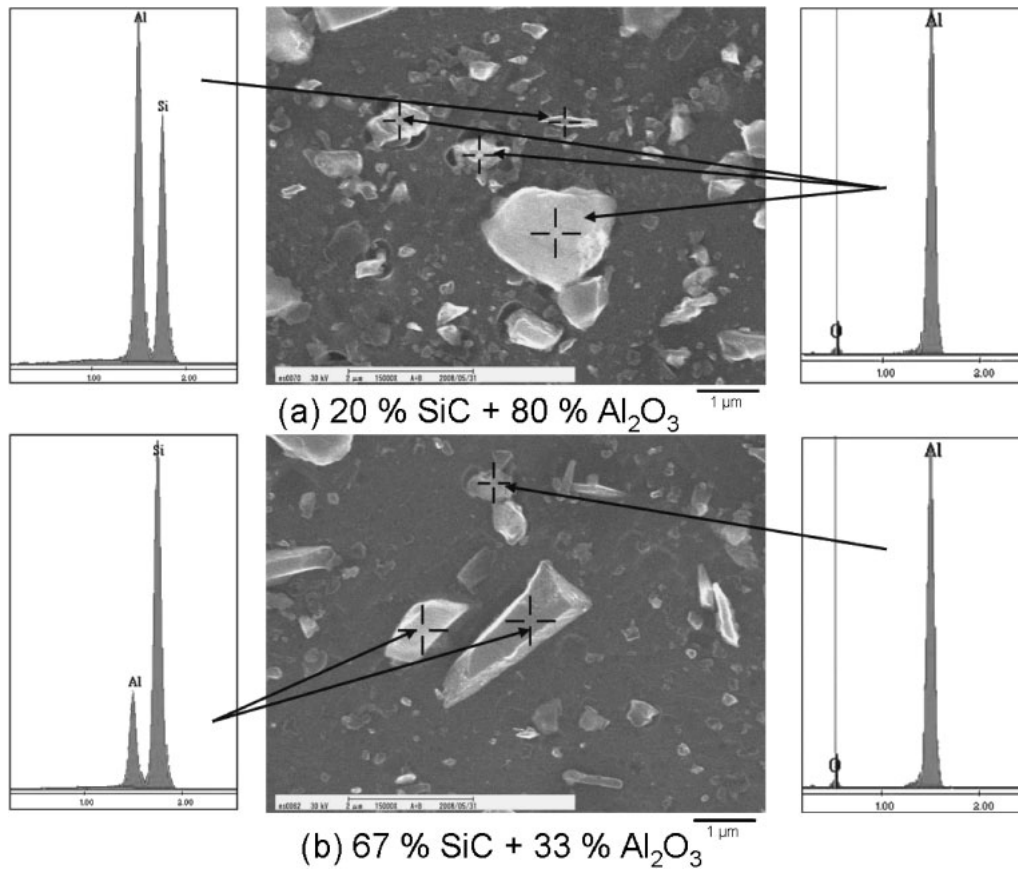


Fig. 8 SEM images of particles of SiC and Al₂O₃ incorporated in the nugget zone and EDS spectra for some of them: (a) 80% Al₂O₃ and (b) 33% Al₂O₃.

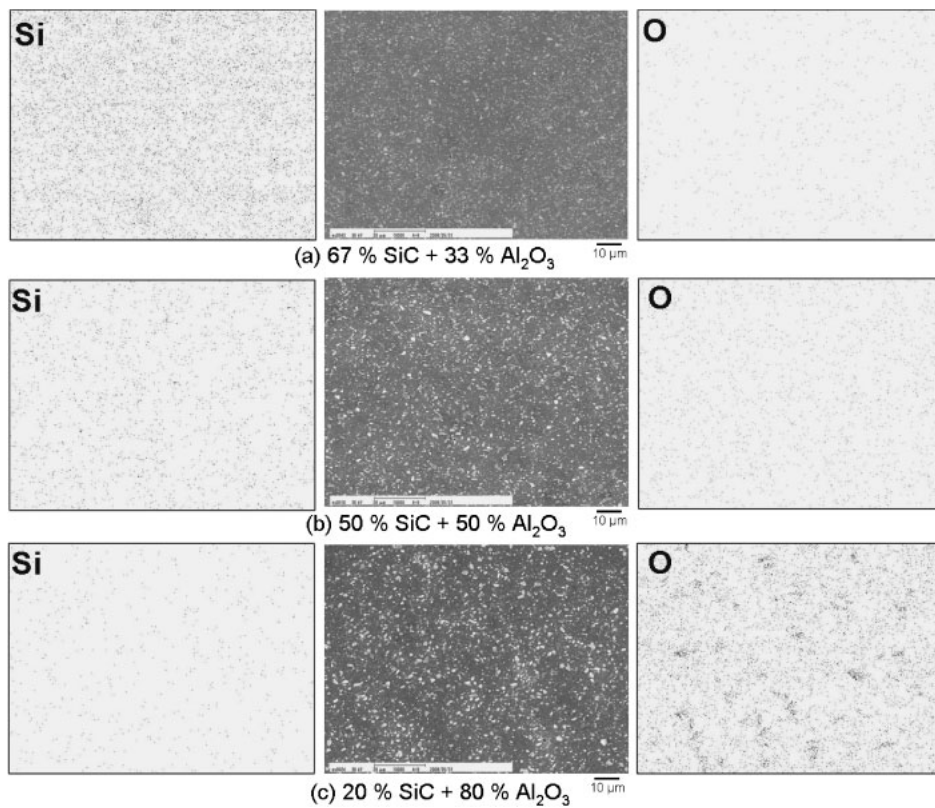


Fig. 9 EDS image mapping of Si (for SiC) and O (for Al₂O₃) at the center of nugget zone containing hybrid composite of: (a) 67% SiC + 33% Al₂O₃, (b) 50% SiC + 50% Al₂O₃, and (c) 20% SiC + 80% Al₂O₃. (the central figures are SEM images of the nugget zone center while the right and left ones are their EDS mapping)

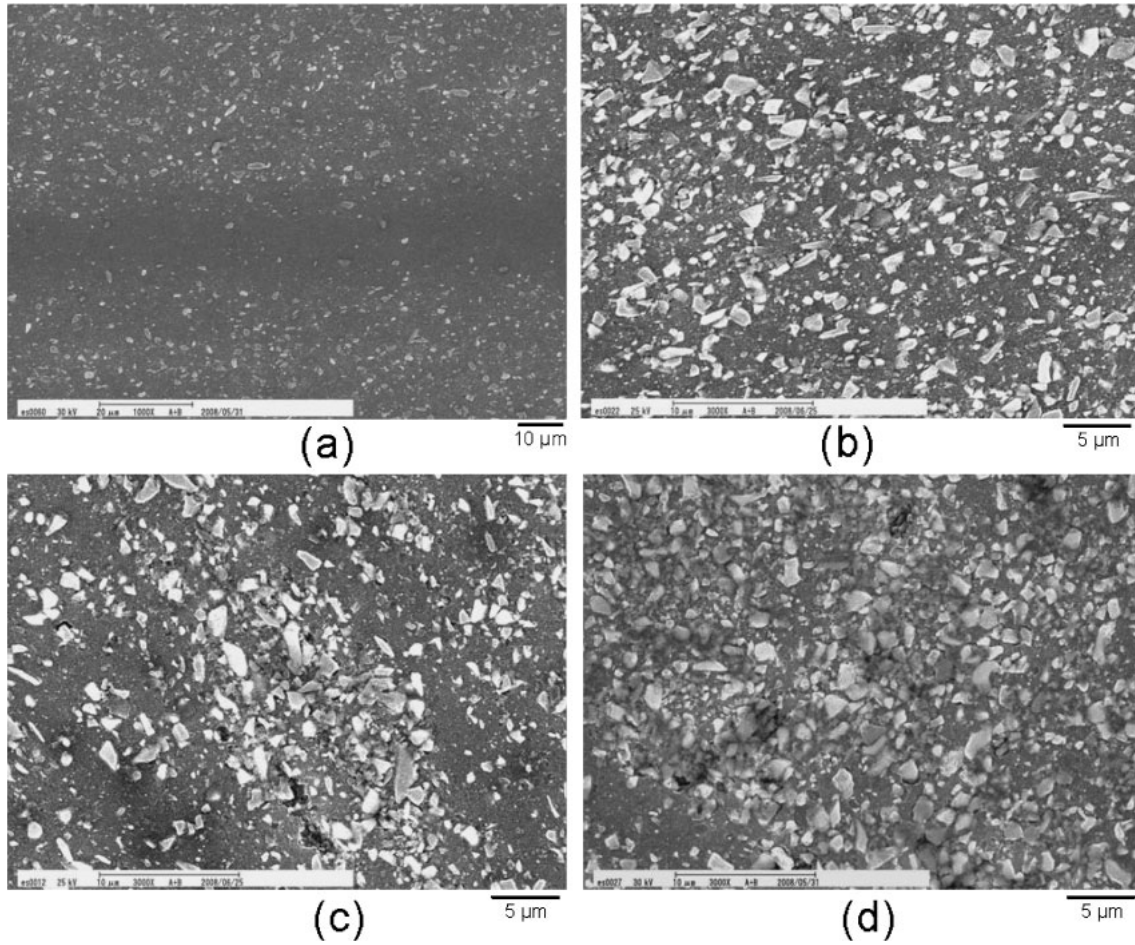


Fig. 10 SEM images of some local areas inside the nugget zones: (a) Banded structure in sample a, (b) high reinforcement density in retreating side of sample e, (c) Elongated clustering in advancing side of sample g, and (d) Al_2O_3 particles clusters in bottom side of sample i.

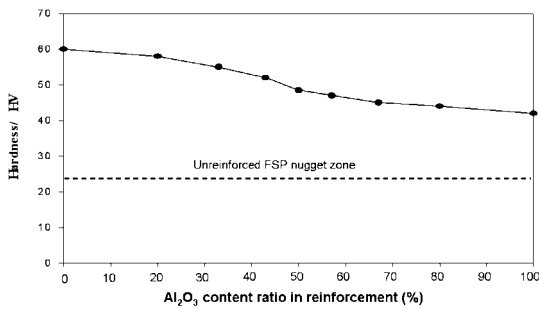


Fig. 11 Effect of the Al_2O_3 content ratio in the reinforcement on the average hardness of the resulted FSP nugget zone measured at mid-cross-section.

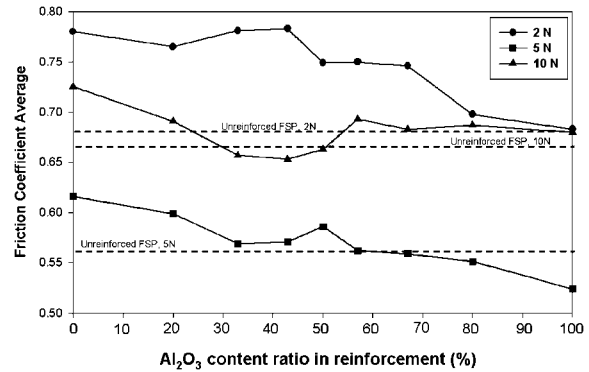


Fig. 12 Effect of hybrid ratio of reinforcements on the average friction coefficient at normal applied loads of 2, 5, 10 N.

wear resistance of the FSP nugget zones, the average friction coefficients and wear volume losses were plotted against the Al_2O_3 relative ratio in reinforcements in Figs. 12 and 13, respectively. As shown in Fig. 12, the average friction coefficient values exhibited general tendency to decrease with increasing the relative content of Al_2O_3 at normal loads of 2 and 5 N. It was reported that the Al_2O_3 powder had a lower friction coefficient 0.4⁴¹⁾ than that of SiC 0.7⁴²⁾ when they were tested under the same conditions. Therefore, it may be expected that the composites that contained higher ratios of Al_2O_3 particles will have lower friction coefficients.

Furthermore, it is clear that the average friction coefficient decreased with increasing the normal applied load from 2 to 5 N at all composite ratios. The decrease in the average friction coefficient with normal applied load was in agreement with many works.^{43–46)}

Figure 13 shows that the addition of reinforcement powder (SiC, Al_2O_3 or mixture) to an aluminum matrix was beneficial in reducing the wear volume losses at normal loads of 2 and 5 N. The wear volume values, at normal loads of 2 N, increased with the Al_2O_3 particles relative ratio in the

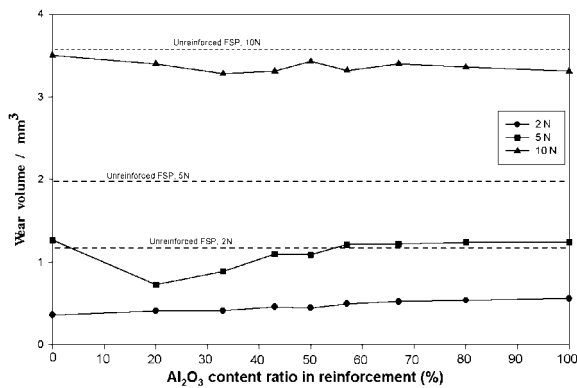


Fig. 13 Effect of hybrid ratio of reinforcements on wear volume losses at normal applied loads of 2, 5, 10 N.

hybrid composites. On the other hand, the hybrid composites containing Al_2O_3 particles relative ratio of 20% were superior in wear resistance to other composites at a normal load of 5 N suggesting that the hybrid composites exhibited better wear resistance than those reinforced with only one type of reinforcement.

4. Conclusion

In this work, we presented a detailed study on fabrication and wear properties of hybrid composites that was produced in a surface layer of pure Al plate by dispersing a mixture of SiC and Al_2O_3 particles through friction stir processing. The following conclusions can be drawn from the results obtained:

- (1) The reinforcement particles (SiC, Al_2O_3 or their mixture) were distributed almost homogeneously over the nugget zone by FSP without any defects except small some voids forming around the Al_2O_3 particles.
- (2) The average hardness of the resulted composites increased to about 60 HV at 100% SiC (almost 3 times that of the nugget zone without reinforcement) and it decreased with increasing the relative ratio of Al_2O_3 particles.
- (3) The average friction coefficient decreased with increasing the relative ratio of Al_2O_3 particles in the reinforcement.
- (4) Wear characteristics of the composites were varied depending on applied load and the relative ratio of SiC and Al_2O_3 . At a normal load of 2 N, the wear resistance decreased with increasing the Al_2O_3 particles ratio in the reinforcement. However, at a normal load of 5 N, the hybrid composites containing 20% Al_2O_3 + 80% SiC exhibited superior wear resistance to other relative ratios of the Al_2O_3 and SiC particles.

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