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Sliding wear behavior of AZ91/B₄C surface composites produced by friction stir processing

Hemendra Patle, B Ratna Sunil and Ravikumar Dumpala

1 Department of Mechanical Engineering, Visvesvaraya National Institute of Technology, Nagpur 440010, India
2 Department of Mechanical Engineering, Bapatla Engineering College, Bapatla 522101, India
E-mail: ravikumardumpala@mec.vnit.ac.in

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Abstract

In the present study, the surface of AZ91 Mg alloy was modified by incorporating boron carbide (B₄C) particles using friction stir processing (FSP). Sliding wear behavior of these developed AZ91/B₄C surface composites was investigated against AISI 52100 steel ball using linear reciprocating tribometer. Hardness tests reveal that the hardness of the fabricated surface composite (∼137.47 HV) is significantly increased compared to the base metal (∼95.5 HV) due to the presence of B₄C particles. Wear tests were conducted on the samples at two different sliding velocities; 0.06 m s⁻¹ and 0.12 m s⁻¹. It was observed that at higher sliding velocity of 0.12 m s⁻¹, AZ91/B₄C surface composite exhibited lower friction coefficient value in comparison to that of the base metal, whereas it is vice versa at the low sliding velocity of 0.06 m s⁻¹. However, surface composites exhibited superior wear resistance at both the sliding velocities, in comparison to that of the base metal. Scanning electron microscopy and energy-dispersive spectroscopy analysis of the wear tracks were carried out to understand the wear mechanisms. From the observations, a combination of abrasive, adhesive, and oxidative wear mechanisms were found to be prominent.

1. Introduction

Magnesium alloys are light weight metals and also exhibit high strength to weight ratio compared with aluminium alloys and steels. Therefore, components and structures made of magnesium alloys allow to replace heavy weight metallic components used in aerospace, transportation and electronics industry [1]. However, low hardness and less wear resistance of Mg alloys limits their wide applications [2]. To improve the wear resistance of magnesium alloys, hard reinforcement particles can be added into the surface. Such composites are named as surface metal matrix composite (SMMCs). Mechanical properties of SMMCs depend on the type of matrix material, reinforcing particles and the method used for the development of surface composites. Several techniques were adopted to produce magnesium based surface metal matrix composites (SMMCs) [3–5]. Friction stir processing (FSP) is one of such successful methods used for the development of surface composites [6–12]. FSP is a severe plastic deformation technique that allows the processing of materials within the solid state itself. A few investigations have shown that FSP of magnesium alloys improves the hardness and wear resistance of magnesium alloy [13–20].

Among all Mg alloys, AZ91 Mg alloy is most popular that is widely used for structural applications. AZ91 Mg alloy possesses excellent castability, suitable mechanical properties, moderate corrosion resistance and commercial availability with reasonable price [21]. Previous studies have explored the sliding wear behavior of AZ91 Mg alloy and its composites [8, 14–16, 22–26]. From these findings, it can be learned that the abrasion and adhesion were the common wear mechanisms in magnesium alloys during testing at lower sliding velocities and when testing at higher sliding velocities, more oxidation occurs due to increased frictional heating.

SiC, Al₂O₃, TiC, TiB₂, SiO₂, MWCNT, WC etc can be used as reinforcement particles for fabrication of Mg based composite [8, 14, 23, 24]. SiC and Al₂O₃ are the most commonly used reinforcement particles to fabricate AZ91Mg alloy based surface composites using FSP. B₄C particles possess lower density, higher hardness, good
thermal and chemical stability comparable to SiC and Al₂O₃ [27, 28]. Nanaksharan et al reported the preliminary wear characteristics of B₄C reinforced AZ91 matrix composite fabricated by FSP [29]. However, detailed analysis on the wear track characteristics of AZ91/B₄C surface composites is limited in the literature. In the present

Figure 1. (a) Photograph of grooved AZ91 Mg workpiece, (b) SEM micrograph of as received B₄C particles, (c) photograph of square taper pin tool (4.25 mm major dia. and 2 mm minor dia., 3 mm pin length, 15 mm shoulder dia.), (d) workpiece fixed on the worktable and (e) photograph of upper surface of fabricated AZ91/B₄C surface composite.

Figure 2. Macrograph of FSPed sample (AZ91/B₄C surface composite).

Figure 3. SEM micrograph showing the distribution of B₄C particles in AZ91/B₄C surface composite.
study, the effect of addition of B$_4$C on the sliding wear behavior of AZ91 Mg alloy was studied against AISI52100 steel ball and the detailed analysis is presented.

2. Experimental details

Commercially available cast billet (Exclusive Magnesium, India) of AZ91 magnesium alloy (weight%: 8.68% Al, 0.84% Zn, 0.03% Mn, 0.002% Fe and the rest Mg) was used for experimental studies. Plates were prepared in the size of 120 mm $\times$ 50 mm $\times$ 6 mm from the alloy billet to conduct experiments. To incorporate the secondary phase particles into the surface of the plate, 2 mm $\times$ 1 mm sized groove was machined at the top of the middle section on the plate (figure 1(a)). FSP experiments were performed using a dedicated NC friction stir welding machine (FSW-3T-NC, R.V. Machine tools, India). B$_4$C particles of size 10–15 $\mu$m were used as reinforcement particles as shown in figure 1(b). B$_4$C particles were filled in the groove and then a pin less tool was employed to
close the groove to prevent the particles scattering during FSP. Then, FSP was conducted using square taper pin tool as shown in figure 1(c) [30]. Based on preliminary experiments, the rotational and travel speeds of the FSP tool were kept at 1400 RPM and 25 mm min$^{-1}$, respectively, to obtain defect-free samples. Figure 1(e) shows the processed AZ91 Mg/B$_4$C surface composite plate. Theoretical volume fraction of B$_4$C powder that has been filled in the groove was calculated as $\approx 15\%$, using the following expression [31, 32]:

\[
V_t (\%) = \frac{\text{Area of groove}}{\text{Projected area of tool pin}} \times 100\%
\]

Where, Area of groove = Groove width $\times$ Groove depth
Projected area of tool pin = Pin diameter $\times$ Pin length.

Samples were extracted from the FSPed region to perform microstructure, microhardness and wear studies. For microstructure study, samples were polished and chemically etched using picric acid reagent. Microstructure observations were carried out using scanning electron microscope (SEM, JEOL JSM-6380A). Microhardness tests were conducted by vickers indentation method (Mitutoyo, HM-112) across the processed zone by applying 100 g load for a dwell time of 10 s.

Dry sliding wear tests were conducted on the base metal (BM AZ91 Mg) and the composite (AZ91/B$_4$C) samples using linear reciprocating tribometer (Ducom, TR-281). Wear tests were conducted against the counter body made of AISI52100 steel ball of 6 mm diameter and 62 HRC hardness, at sliding velocities of 0.06 m s$^{-1}$ and 0.12 m s$^{-1}$. Normal load and sliding distance were kept constant as 10 N and 500 m respectively. Tests were
conducted at room temperature and 40%−50% relative humidity. Mass loss of specimens was measured, using a digital weighing balance (CAS-84, Contech, India) with accuracy ±0.0001 g. Each test was repeated for three times in order to check the reproducibility. Worn surfaces of the tested specimens, collected debris and wear scar formed on the counter surface were examined and analyzed using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS).

3. Results and discussions

3.1. Microstructure

Figure 2 shows the macrograph of the FSPed AZ91/B4C sample in which the distribution of B4C particles can be seen within the stir zone. Any defects, such as warm-hole, void, cavity and tunnel defect were not observed in the processed zone. Basin shape stir zone was appeared after the completion of FSP pass, which is the general feature of FSP process [16]. The starting grain size was measured as 166.5 μm and after FSP, the grain size was observed as decreased to 12.581 μm. During FSP of AZ91, dynamic recrystallization refines the microstructure of the material and reduces the size of the grains and increases the hardness of the material [33].

Figure 3 shows SEM micrograph obtained from the stir zone of FSPed AZ91/B4C sample. Distribution of B4C particles in the stir zone can be clearly observed. The corresponding EDS analysis (figure 4) shows the elemental composition of the matrix and the dispersed B4C particles. SEM analysis revealed that B4C particles were dispersed uniformly in the stir zone. Distribution of particles plays an important role in the enhancement of hardness and wear resistance of fabricated surface composite [8, 14]. Due to the intense heat generation during FSP, phase transformation in Mg alloy is a known phenomenon. In our earlier study, dissolution of intermetallic phases (β phase, Mg17Al12) in the solid solution grains (α phase) was observed after friction stir processing in AZ91 Mg alloy [30]. Along with the grain refinement, decreased intermetallic phases and addition of secondary phase particles influence the mechanical behavior of the composites. It was observed that size of the few particles was decreased to less than 10 μm. However, major fraction of the size of the particles was observed as within the starting range (10−15 μm) after FSP.

Figure 8. SEM-BSE micrographs and EDS analysis of worn surface of (a) BM AZ91 Mg and (b) AZ91/B4C surface composite at sliding velocity of 0.06 m s⁻¹.
3.2. Hardness measurements

Figure 5 shows the variation of microhardness across the stir zone of FSPed AZ91/B4C surface composite. Hardness has been observed as increased for the composite compared with the base metal. Interestingly, hardness was observed as increased towards the retreating side within the stir zone compared with advancing side due to the higher fraction of B4C particles in the composite. The distribution of B4C was uniform as seen in the microscopic observations. However, macroscopically, higher amount of B4C was observed in the composite close to the retreating side of the nugget zone. This is due to the material flow pattern in the nugget zone as reported by Kondaiah et al [18]. Average microhardness of BM AZ91 Mg alloy was measured as 95.5 HV while for FSPed AZ91/B4C, microhardness was observed to be increased up to 137.47 HV. During FSP, dynamics recrystallization causes the refinement of microstructure. In the present study, grain size was observed as reduced from a starting size of 166.5 μm to 12.581 μm after FSP. During FSP of AZ91 Mg alloy, dissolution of hard brittle intermetallic phase (Mg17Al12) into the solid solution magnesium grains (α phase) reduces the hardness [34]. Furthermore, the addition of reinforcement particles (B4C) in the matrix increases the hardness of the composite. Additionally, FSP also leads to develop texture in Mg alloys [34, 35]. Therefore, the measured hardness values of the composite are the result of the combined effect from the grain refinement, dissolution of intermetallic phase, and distribution of reinforcement (B4C) particles in the matrix [27].

3.3. Wear studies

Figures 6(a) and (b) show the friction curves obtained for BM AZ91 Mg and AZ91/B4C surface composite samples from the wear tests conducted at sliding velocity of 0.06 m s⁻¹ and 0.12 m s⁻¹ at constant applied load of 10 N. At lower velocity (0.06 m s⁻¹), BM sample shows less friction coefficient than the composite sample. Distribution of B4C particles in composite sample works as barriers against sliding action of counter body (AISI52100 steel ball) causes the increase in friction between the testing and counter surface. At higher velocity (0.12 m s⁻¹), friction coefficient was observed as decreased for the composite sample as compared with BM sample which can be attributed to the hard surface and reduced adhesive contact because of higher sliding velocity (figure 12(d)) [36]. At higher velocity, friction curve was observed as more stable for composite sample compared with BM sample. In both test conditions, lower mass loss was observed for the composite sample, when compared with BM sample as shown in figure 7. Distribution of B4C particles reduces the direct contact of...
counter surface with the specimen surface and also bears the direct load applied from the counter surface [16]. The load-carrying capacity of B₄C particles reduces the surface wear rate by reducing the direct load encountered on the test surface; therefore, the composite has shown relatively lower mass loss.

Figures 8(a) and (b) show the worn surface morphology of wear track obtained for BM and composite samples at a sliding velocity of 0.06 m s⁻¹. The formation of the grooves was observed on the wear track parallel to sliding direction. Ploughing of material was also observed on the samples. Formation of grooves and ploughing are the indications of abrasive wear mechanism [37]. Dark spots represent the oxidation of the tested surface. Due to frictional heating, magnesium surface was oxidized during testing. Formation of oxide layer as observed in the present study gives the indication of oxidative wear mechanism [22].

Figures 9(a) and (b) show the worn surface morphology of wear track obtained for BM and composite sample at sliding velocity of 0.12 m s⁻¹. Grooves were observed as shallower on both the samples compared with deeper grooves formed on the samples tested at 0.06 m s⁻¹ sliding velocity. Series of cracks were appeared on the tested surfaces. These are the typical features of delamination wear and are in agreement with previous findings [38, 39]. Delamination wear is a fatigue related wear mechanism in which repeated sliding nucleates subsurface cracks and propagate [37, 40]. Linking of these subsurface cracks promote the shearing of surface and subsequently removes the material in the form of flakes and further form the craters on the worn surfaces.

Figures 10(a) and (b) show the wear debris morphology obtained for BM and composite sample at 0.06 m s⁻¹ sliding velocity. Due to abrasive action of hard asperities of steel ball, material from the sample has been removed in the form of fragments. In composite samples, some of the B₄C particles have come out of the testing surface and stuck between the sliding surfaces and acted as a third body. These particles dig into the test surface and remove the material into fragments and ribbon-like shapes [37]. EDS analysis of debris collected from the composite samples confirms the presence of iron along with other elements, reveals that the steel counter surface was also worn out due to trapped hard B₄C particles.

Figures 11(a) and (b) show the morphology of debris collected from tested samples at 0.12 m s⁻¹. Due to delamination, sheet-like flake shaped debris and tiny wear particulates were formed. However, debris of composite samples was noticed as finer than BM debris. Wear debris morphology of composite samples reveals...
Figure 11. SEM micrographs and EDS analysis of wear debris of (a) BM AZ91 Mg and (b) AZ91/B₄C surface composite at sliding velocity of 0.12 m s⁻¹.

Figure 12. Wear morphology of counter body against BM AZ91 Mg at sliding velocities of (a) 0.06 m s⁻¹ and (b) 0.12 m s⁻¹, and AZ91/B₄C surface composite at sliding velocities of (c) 0.06 m s⁻¹ and (d) 0.12 m s⁻¹, after 500 m sliding distance.
that the hard B₄C particles can act as hard barriers that moderate the intensity of plastic deformation and relieve delamination [23].

Figure 12 shows the wear morphology of counter bodies (AISI52100 steel ball) used for conducting the wear tests. Wear morphology shows the transfer of material from matrix surface to counter body surface in all the test conditions. Figure 13 shows the SEM image of wear scar formed on counter body used against the composite. Corresponding EDS analysis also confirms the presence of material transfer from the tested sample. Transfer of material from the tested sample to counter surface reveals the involvement of adhesive wear mechanism [41, 42].

As can be seen in figures 12(a) and (c), at lower sliding velocity (0.06 m s⁻¹), counter body used for composite testing shows less adherence of matrix material compared with BM. It reveals that the presence of B₄C particles in composite sample reduced the intensity of adhesive wear compared with BM sample. At lower sliding velocity, surface of counter body is adhered by more amount of testing material, compared with the counter body used for higher sliding velocity tests for both the samples, figures 12(b) and (d). At higher velocity, more frictional heat is generated during the sliding of mating surfaces, which in turn increases the oxidation of testing surface. These oxide layers fill the spaces in valley between asperities of tested surfaces. This oxide layer works as a protective layer during testing the surface and decreases the transfer of material from testing surface to the ball surface. At higher sliding velocities, oxidative wear tend to more predominant than the adhesive wear.

4. Conclusions

In present study, B₄C reinforced surface MMCs of AZ91 Mg alloy has been produced and the effect of B₄C particles reinforcement on the hardness and wear characteristics of the composite was investigated. The conclusions drawn from the results of this study are as follows.

(i) Hardness was observed to be increased after the addition of B₄C particles in AZ91 Mg alloy by FSP. Average microhardness for the surface composite AZ91/B₄C was measured as 137.47 HV, which is 44% more than the base metal microhardness (95.5 HV). The increased hardness for the surface composites is due to the dispersed B₄C and microstructure refinement in the stir zone.
(ii) Lower wear rate was observed for AZ91/B$_4$C surface composite compared with BM AZ91 Mg, under both sliding velocity conditions. It is attributed that the addition of B$_4$C particles increased the hardness and decreased the direct contact load during wear tests in the composites compared with base metal.

(iii) At 0.06 m s$^{-1}$ sliding velocity, abrasive and severe adhesive wear were observed as the predominant wear mechanisms along with participation of oxidative wear mechanism for BM AZ91 Mg and AZ91/B$_4$C surface composite samples. Whereas, at 0.12 m s$^{-1}$ sliding velocity, delamination and oxidative wear were the predominant wear mechanism along with participation of abrasive and mild adhesive wear. These observations strongly suggest that the sliding velocity significantly influences the wear mechanisms in AZ91/B$_4$C surface composites.

**ORCID iDs**

Hemendra Patle @ https://orcid.org/0000-0001-8635-9601
B Ratna Sunil @ https://orcid.org/0000-0001-9855-7808
Ravikumar Dumpala @ https://orcid.org/0000-0003-1500-8809

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