Original Article

Fabrication of Al5083/B₄C surface composite by friction stir processing and its tribological characterization

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1. Introduction

The aluminum based metal matrix composites provide excellent specific strength, stiffness, high hardness, wear resistance, stability at high elevated temperature [1] with additional advantages of three dimensional isotropy and affordability [2]. The Al–B₄C composites are used in the bicycle frame, bullet proof vests, armor tanks, containment of nuclear waste, neutron absorber in nuclear power plant, transportation applications, etc. owing to their high hardness, low density and excellent thermal and chemical stability [2–4]. Al–B₄C composites are fabricated by various researchers in the past few years by stir casting [5–7], casting [8–10], squeeze casting [11] and mechanical alloying [12].

However, a surface property of the material is very important to achieve longer life of mechanical components. Dispersion of the nano reinforcements in the surface to have
a uniform layer is a difficult task. Existing processing techniques for processing of surface composites are based on liquid phase processing at high temperature such as laser melt treatment, electron beam irradiation and plasma spraying [13–15]. In these cases, there will be an interfacial reaction between the matrix and reinforcement, which leads to the formation of detrimental phases. The above problems can be eliminated by processing it below the melting point of substrate. The recent new surface modifying technique named Friction Stir Processing (FSP) [16] is developed by utilizing the principle of the friction stir welding (FSW). FSW was invented by the welding Institute UK in 1991 [17]. FSP is a solid state processing technique for producing a thick composite layer; the layer thickness can range from hundreds of micrometers to several millimeters. FSP is also useful to obtain a fine grained microstructure and to remove the cast defects with improved mechanical properties and wear resistance [18–20].

The successful fabrication of aluminum based metal matrix composites reinforced with particulates such as SiC...
Table 1 – Chemical composition of base material.

<table>
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<tr>
<th>Elements</th>
<th>Mg</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Zn</th>
<th>Si</th>
<th>Ti</th>
<th>Cu</th>
<th>Ni</th>
<th>Al</th>
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<tr>
<td>%wt</td>
<td>4.162</td>
<td>0.064</td>
<td>0.554</td>
<td>0.191</td>
<td>0.029</td>
<td>0.230</td>
<td>0.049</td>
<td>0.051</td>
<td>0.023</td>
<td>Bal</td>
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Fig. 5 – The configuration of the tensile specimen.

Fig. 6 – Optical micrographs of transverse cross-sectional of the FSP zone specimens (a) base material, (b) without particle one pass stir zone, (c) B₄C micro sized particle one pass, (d) B₄C micro sized particle three pass, (e) B₄C nano sized particle one pass, (f) B₄C nano sized particle three pass.

[21–24], Al₂O₃ [25–27], Si₃N₄ [28], B₄C [29,30], ZrO₂ [31] and TiO₂ [32] were reported. However, no literature is available on FSP of 5083 aluminum alloy with B₄C nano composites. The present work aims to fabricate 5083 aluminum alloy surface composite by FSP with the reinforcement of B₄C micro and nano particles. The effects of number of passes on the microstructure, micro hardness, tensile strength and wear behavior were investigated.
2. Materials and methods

Al alloy of 5083-O rolled plate of 8 mm thickness was used. The chemical composition is presented in Table 1. The micron sized B₄C particles with a purity of 99% having an average particle size of 20 μm and nano size B₄C particle of 99.5% purity with the mean particle size of 30–60 nm were used as reinforcement materials. The SEM micrographs of the particles are shown in Fig. 1. These micrographs reveal the size and morphology of micro and nano level particles. The tool made of H-13 tool steel hardened to 55 HRC with a shoulder of 18 mm diameter with threaded pin diameter of M6 × 1.0 of pin length of 5 mm was used for FSP. A groove (1 mm width and 3 mm depth) was made in the middle of the workpiece and the groove was then filled with the reinforcements. A probe-less FSP tool with shoulder only was initially used to prevent the escape of the particles from the FSP zone. Then the samples were subjected to various numbers of passes from one to three.
An indigenously developed FSW machine (11 kW, 40 kN) was used to fabricate the surface composite layer. The specimens were clamped on the hydraulic fixture with mild steel backing plate. The constant rotational speed and transverse speed were fixed at 1000 rpm and 25 mm/min respectively after number of trials. The advancing and retreating sides of the parent metals were changed after each FSP pass. For every pass, the specimen was allowed to be cooled to the room temperature and all the experiments were carried out at room temperature. Fig. 2(a) shows the photograph of the threaded tool used for the study and Fig. 2(b) shows its dimensions. Fig. 3 shows the schematic of FSP experiment. After FSP, the samples were cut in the transverse direction and polished with different grades of emery sheets and the samples were analyzed through optical microscope. A modified Poulton’s reagent (30 ml HCl, 40 ml HNO₃, 2.5 ml HF, 12 g CrO₃, and 42.5 ml H₂O) was used to study microstructure of the base metal and the stir zone of the surface composite layer (SCL). Scanning electron microscope (SEM) (Hitachi S3700) was used to examine the distribution of the reinforcement particles. XRD (BRUKER D8 ADVANCE) was used to identify the presence of phases in the Stir zone of SCL. The Vickers micro hardness (Future Tech Japan) values of the FSPed regions was measured along and perpendicular (cross section) to the processing direction using a load of 100 g with dwell time of 10 s. The tensile properties of the FSPed specimens were examined using computer controlled universal testing machine (Tinius Olsen H50KS) at a constant cross head speed of 1 mm/min in room temperature. The longitudinal tensile specimens prepared on the middle of the FSP stir zone by wire EDM as per ASTM: E8/E8M-011 standard. Figs. 4 and 5 show the size and configuration of the specimens for the tests. The wear behavior of the SCL was studied through pin on disk tribometer (make DUCOM) in room temperature condition. Wear test specimens of 10 mm diameter were cut from the middle of stirred zone of FSPed surface by wire EDM. Tribo tests were conducted as per ASTM G99-04 standard. The counterpart discs were made of EN-24 steel hardened to 58 HRC and the surface roughness (Rq) of 0.2 μm. A constant track diameter 100 mm was used in all the tests. Before the test, the surface of each pin was polished on 1200 grit emery paper. The wear test was conducted

Fig. 10 – Micro hardness profile of FSPed specimens with and without particles of the cross section region (as a function of depth).

![Micro hardness profile](image)

![Stress-strain curves](image)

Fig. 12 – Stress-strain curves for base metal and specimens FSPed with and without particles with different passes.

Fig. 11 – (a) Tensile specimens before tensile testing, (b) after testing.
at a sliding velocity of 2 m/s, normal force of 30 N and sliding distance of 3000 m. At every 500 m interval the samples were cleaned with acetone and weighed to an accuracy of 0.001 mg by electronic weighing balance. The applied load as well as the sliding speed were fixed in order to compare the active wear mechanisms in similar conditions. The friction coefficient between the pin specimen and disc was determined by measuring the frictional force with a stress sensor at the distance of 3000 m without stopping of the disc rotation at the same sliding velocity and normal force.

To understand the wear mechanisms the worn surfaces of the SCL and base metal were examined by SEM. The wear behavior of surface composite layer processed at single and multi (three) passes with micro and nano particles was compared with the wear behavior of base metal. FSPed surfaces without micro/nano reinforcement were also studied to have better understanding.

3. Results and discussion

3.1. Microstructural analysis

Microscopic observation of the SCL is shown in Fig. 6. The B₄C particles were found to be clustered in the stir zone after each FSP pass. The reinforcement particles are strengthening precipitates of Al5083 alloy that dispersed in the Al matrix. The finer grains were found in the SCL compared to the as received Al substrate, due to severe plastic deformation and dynamic recrystallization. Earlier reports also stated that the refinement of grains and uniform distribution of particles could be obtained by FSP [33]. With the increase in the number of FSP passes powder distribution becomes more homogeneous and reduced clustering of reinforcement particles is observed. Good dispersion of reinforcement occurs in the matrix and the size of the grain is also reducing. Typical XRD profile of nano
B₄C reinforced aluminum alloy specimen subjected to three stir passes is shown in Fig. 7. The clear peaks of aluminum and marginal peaks of B₄C are observed in the figure. Fig. 8 shows the SEM micrograph of cross section of three pass nano FSPed specimen with elemental composition.

3.2. **Hardness**

The hardness of base material was 82 HV. For experimental purpose, the average of three hardness values was taken in all the samples. Fig. 9 show the hardness profile of specimens FSPed without particle one pass, with micro and nano sized B₄C particles one pass and three passes. The obtained data is compared with the base material. Asadi et al. [34] have reported that hardness of the FSPed AZ91/SiC nano composite is observed to be increased due to uniform dispersion of SiC particles and refinement of the grain size of the base material. Shamsipur et al. [35] have reported that the hardness value could be increased by 6.4 times for FSPed Ti/TiN composite surface layer in compare with base material due to the uniform dispersion of reinforcements. Morisada et al. [36] have revealed that FSPed nanostructured tool steel exhibited much higher hardness in compared to base material. Faraji and Asadi [37] reported that the increased number of passes FSP in AZ31/Alumina nano composite the hardness is observed to be higher than that of single pass. The second pass homogenizes the particle distribution, decreases the grain size and produces the uniform hardness profile. According to the Hall–Petch relationship, decrease in grain size increases the yield strength, which in turn increases the hardness value also. Dolatkhah et al. [22] have achieved maximum of 116 HV hardened in FSP of 5052 Al with 50 nm SiC particles due to severe grain reduction. Shamsipur et al. [38] reported that microhardness value of Ti/SiC nano surface composite layer is 3.3 times greater than that of as received titanium substrate due to uniform dispersion of nano sized reinforcement particles after four FSP passes. Zahmatkesh and Enayati [39] have achieved higher hardness value in the stir zone of FSP in Al 2024 with Al₂O₃.
Fig. 15 – SEM micrograph of the worn out track of (a) base material, (b) specimen FSPed without particle one pass, (c) specimen B₄C micro particle one pass, and (g), (h), (i) are high magnification view of (a), (b) and (c) respectively.

nano particles than that of as received material due to uniform dispersion of Al₂O₃ particles in Al matrix. Raft et al. [19] have reported that microhardness value of A390/Al₂O₃ and A390/graphite surface composites exhibited higher hardness than the hardness of as received material. The hardness is increased due to grain refinement of the particles and higher hardnesh of reinforcement particles. Shafiei-Zarghani et al. [26] reported that there is a threefold increase in hardness with passes of FSP 6082/Al₂O₃ nano reinforcements and the dispersion of nano-size Al₂O₃ in uniform manner in the Al matrix. Increase in the number of passes of many composite systems [36–40] has proved the uniformity in distribution in hardness.

The surface composite layer with nano particle three pass exhibited higher hardness compared to nano particle single pass. The hardness of the SCL depends upon fine grain size of Al matrix, fine dispersion of reinforcing particles (B₄C) in aluminum matrix and quench hardening effect due to difference in thermal contraction coefficient of aluminum matrix and B₄C reinforcing particles [40]. The above mentioned literature supports the finding of increased hardness of FSPed surfaces. The Al5083/B₄C nano FSP surface composites exhibited higher hardness when compared with the FSP of micro reinforcement particles. For example, the Al5083/B₄C nano FSP three pass and Al5083/B₄C micro three pass surface composites exhibited hardness of 134.8 Hv and 116.4 Hv respectively. The detailed hardness values are presented in Table 2.

Fig. 10 shows the cross section (depth profile) of the microhardness from processed region to the unprocessed region. Base material hardness is given in the figure. The FSP processed with nano particle three pass surface exhibited 60% increase in hardness. This increase in hardness is attributed to defects closure by friction stir process and refinement of grains. Micro particle distribution further increases its hardness and nano particle reinforcement improves the hardness. Further improvement in hardness by number of passes is certainly attributed to severe deformation of the parent material, finer grain structure and uniformity in particle distribution.
3.3.  Effect on the number of passes

The number of FSP passes reduces the clustering effect, increasing the uniformity of distribution of particles, which in turn results in improvement of mechanical and wear resistance properties. This observation in micro and nano B$_4$C reinforcement is supported by literature in various other material systems. Mahmoud [41] studied that FSP of Al390 Al alloy with three passes and found that the hardness and wear resistance of the processed surface layer was improved and the coefficient of friction was observed to be slightly reduced when compared with the un-processed material. Shafei-Zarghani et al. [42] have reported that nano size alumina particles are more uniformly distributed in the aluminum matrix due to increasing the number of FSP passes. Alumina clusters were rarely reported. Increasing the FSP passes produces the better distribution of SiC and Al$_2$O$_3$ nano particles in the AZ91 matrix [43]. The hardness, strength, elongation and wear resistance of the composite layer are reported to be improved. After two passes, the nano sized Al$_2$O$_3$ particles are dispersed in a uniform manner in the Al matrix. Orowan strengthening can be very significant in the composites, the nano particles are well distributed in intra granularly and inter granularly. The cluster size is reduced and the integrity in the particle/matrix interface can be improved by increasing the number of passes.

3.4. Mechanical properties

Fig. 11(a) and (b) shows the tensile specimens before and after testing and Fig. 12 shows the result for the base material, and the processed specimens of micro and nano sized B$_4$C particles with one and three passes. The maximum tensile strength was achieved in the nano particles with three passes. Mechanical properties mainly depend on the fine grain size and dislocation strengthening. Restriction of movement
of grain boundaries is also influencing the tensile properties of the stirred zone material. Grain refinement is the important parameter to decide the super plastic behavior of metal [44]. FSP Al alloys forms equiaxed fine grains due to stirring the material and they exhibit excellent grain refinement. Fine grain size of nano sized FSP composites leads to a better tensile properties after the number of passes. Zohoor et al. [45] have reported that there is an increase in the strength of the Al alloy composite with the number of passes with the FSP effects on grain refinement and particle reinforcement on the Al alloy composite. FSPed Al–TiC in situ composites with two passes specimen exhibited the significant improvement of tensile properties due to homogenization, refinement of grain size and elimination of casting defects [46].

Fig. 13 shows the tensile fracture features of the specimens. Ductile fracture observed in all samples. However, a distinct difference of fracture morphologies can be observed between the as received material and FSPed composites with number of passes. The FSPed nano particles composite with three passes shows finer dimples, which are associated with finer reinforcement of nano particles in the matrix alloy. The nano sized composites with one pass FSP specimen improves the tensile properties and elongation, in three pass nano particle FSP specimen observed improved tensile properties and slightly decrease in elongation when compared with one pass nano particle FSP. The increase in dislocation density causes the reduction of elongation. The micro particle FSPed composites shows lower tensile strength compared to the nano reinforced composites and fracture morphologies show the larger dimple size with reduced ductility.

3.5. Wear properties

The wear behavior of the as received Al 5083 alloy, FSPed without particle one pass and FSPed with reinforcement particle (micro and nano sized B4C particle one pass and three pass) surface composite layer (SCL) were evaluated by a pin on disc tribometer. Fig. 14(a) shows the variation of weight loss with sliding distance of the SCL samples. The weight loss of the as received material is higher than the SCL samples. The hard B4C nano reinforcement particles exhibited the least wear amongst the tested specimens.

Fig. 14(b) shows the wear rate results. The wear rate of the as received Al5083 alloy sample is higher the SCL samples. The wear rate of the B4C nano particle with three pass sample has less wear rate as compared to the one pass nano particle and micro particle specimen.

Reasons for better wear resistance of the SCL as compared with unprocessed matrix alloy.

1. The dispersion of hard B4C reinforcement particles in the SCL in uniform manner, which increases hardness due to decrease in grain size in the Al matrix. These results are supported by Anwari et al. [47].
2. The fine grain size of matrix alloy due to FSP with number of passes.

Similar results were reported by Shafei-Zarghani et al. [42] for Al-Al2O3 and Dolatkah et al. [22] Al-SiC nano particles with four passes. The wear resistance of the SCL was superior to the as received Al substrate due to increase in hardness and presence of hard ceramic particles and matrix refinement. Fig. 14(c) shows the change in wear rate vs microhardness. According to the Archard’s equation, the wear rate is inversely proportional to the hardness of the material. Owing to the high hardness of nano particles and their bonding nature, the nano particles resist the penetration and cutting into the surface by the counter material [48-50].

Figs. 15 and 16 show the SEM micrograph of worn surface of the samples. The deep grooves occur in the base metal due to intensive material removal and plastic deformation. In nano composite with three pass specimen’s shallow grooves are seen on the surface in comparison with single pass nano composite layer. Presence of ceramic particle in the FSPed composite layer prevents the wear and drastic material removal of material. Fig. 17(a) and (b) shows the EDAX of worn surface of Al–B4C nano particle three pass FSPed specimen and Al–B4C micro particle three pass FSPed specimen.

Fig. 18 shows the variations of friction coefficient with sliding distance of the base metal and FSPed samples. From Fig. 18(f), it is clear that the average coefficient in the three pass nano particle has the lowest value of 0.4. The friction coefficient in nano composite layer is low due to decrease in adhesive and lower abrasive wear because of higher hardness. Furthermore the presence of worn out B4C particles acting as
Fig. 18 – Variations of friction coefficient with sliding distance in (a) base material, (b) FSPed without particle one pass, (c) FSPed with B₄C micro particle one pass, (d) FSPed with B₄C micro particle three pass, (e) FSPed with B₄C micro particle one pass, (f) FSPed with B₄C nano particle three pass.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Base material</th>
<th>Without particle one pass</th>
<th>Micro sized B₄C particle one pass</th>
<th>Micro sized B₄C particle three pass</th>
<th>Nano sized B₄C particle one pass</th>
<th>Nano sized B₄C particle three pass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wear rate (mg/m)</td>
<td>0.0057</td>
<td>0.00497</td>
<td>0.0046</td>
<td>0.00416</td>
<td>0.00363</td>
<td>0.00327</td>
</tr>
<tr>
<td>Friction coefficient</td>
<td>0.6</td>
<td>0.55</td>
<td>0.5</td>
<td>0.45</td>
<td>0.45</td>
<td>0.4</td>
</tr>
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</table>
lubricant is also evident from the low value of friction coefficient of nano composite layer with three passes. However, the maximum (average value) friction coefficient value of is 0.6 observed in base material. Presence of hard reinforcing material in FSPed with B4C particles prevents adhesive wearing and material removal. Table 3 describes the wear rate of the micro and nano particle specimens with one and three passes.

4. Conclusions

Al/B4C composites were fabricated successfully by one and three pass FSP. Effect of the reinforcing particle type and FSP passes on the microstructure, micro hardness, tensile strength and wear properties of the fabricated surface composite layer were investigated.

1. The microstructure in the FSPed specimens with nano B4C particles three passes exhibits fine grain size, higher hardness (124.8 Hv), ultimate strength (360 Mpa) and wear rate (0.00327 mg/m) in comparison to the base material hardness (82 Hv), ultimate strength (310 Mpa) and wear rate (0.0057 mg/m).

2. By increasing the FSP passes, the distribution of nano particles in the Al matrix becomes uniform which has resulted in increased hardness, in comparison with one pass FSP nano composite.

3. The microhardness of the Al/B4C surface nano composites is higher in comparison with B4C micro particles. The presence of nano size B4C particles contributes to produce ultra fine grain size.

4. The FSPed nano sized tensile specimen on the stir zone exhibited better mechanical properties than the as received Al 5083 alloy.

5. The wear properties of the Al5083 alloy were improved by addition of B4C nano particles in comparison with B4C micro particle, and the wear resistance of the nano SCL was higher than that of the unreinforced Al 5083 alloy.

Conflicts of interest

The authors declare no conflicts of interest.

REFERENCES


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