Microstructure and wear characterization of aluminum matrix composites reinforced with industrial waste fly ash particulates synthesized by friction stir processing

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Abstract

Fly ash (FA) is a waste product of coal combustion in thermal power plants which is available in massive quantities all over the world causing land pollution. This paper reports the characterization of AA6061 aluminum matrix composites (AMCs) reinforced with FA particles synthesized using friction stir processing (FSP). The volume fraction of FA particles was varied from 0 to 18 in steps of 6. The prepared AMCs were characterized using optical microscopy (OM), scanning electron microscopy (SEM) and electron backscattered diagram (EBSD). The wear rate was estimated using a pin-on-disc wear apparatus. FA particles were observed to be distributed homogeneously in the AMC irrespective of the location within the stir zone. The EBSD micrographs revealed remarkable grain refinement in the AMC. The
incorporation of FA particles enhanced the microhardness and wear resistance of the AMC. The strengthening mechanisms of the AMC were discussed and correlated to the observed microstructures. The wear mechanisms were identified by characterizing the wear debris and worn surfaces.

Key words: Aluminum Matrix Composites; Friction Stir Processing; Fly Ash; Wear.

1. Introduction

Aluminum alloys reinforced with various ceramic particles, universally known as aluminum matrix composites (AMCs) have become the focus of the current materials era due to their superior properties such as light weight, high wear resistance, low thermal expansion, high strength to weight ratio etc. AMCs are progressively phasing out aluminum alloys in a wide range of applications in automotive, aerospace, marine and nuclear industries [1–3]. Although industries gaze to exploit the benefits of AMCs, it still remains a daunting task to produce AMCs possessing all desirable properties at an economical price. The production cost of AMCs can be controlled if inexpensive reinforcements such as fly ash (FA) and natural minerals are used. FA is reasonably an economical reinforcement compared to conventional reinforcements such as silicon carbide (SiC), aluminum oxide (Al₂O₃), titanium carbide (TiC) and boron carbide (B₄C). The combustion of coal in thermal plants around the world releases FA as a byproduct in massive quantities which goes as a waste causing environmental impact. Incorporating FA to produce AMCs is a sensible way to reduce the cost of AMCs and land pollution [4–7].

Several researchers attempted to produce and characterize AMCs reinforced with FA particles using liquid metallurgy and solid state methods in the past decade [8–17]. Sobczak et al. [8] characterized the interfacial reaction products in Al/FA AMCs prepared using hot pressing. Rohatgi et al. [9] synthesized A356/FA AMCs using pressure infiltration and

The literature survey revealed that it is possible to produce AMCs reinforced with various types of FA particles using several techniques which include stir casting, compo casting, squeeze casting, powder metallurgy etc. But those production methods were always associated with multiple defects such as porosity [10,12,13], voids [9], particle clusters [15], inhomogeneous distribution [16] and brittle intermetallics owing to interfacial reaction [8,11]. Those defects diminish the mechanical and tribological properties and reduce the performance of Al/FA AMCs during service. Further, the wettability of FA particles with molten aluminum is poor which requires treatment of FA particles [12,15] or addition of wettability agents [11] leading to an increase in the cost of the AMC. The poor wettability weakens the interfacing bonding between the aluminum matrix and the FA particles limiting the load bearing capacity of the AMCs. Therefore, development in production method is crucial to exploit the advantages of low cost Al/FA AMCs.
Friction stir processing (FSP) is a promising method to produce surface and bulk AMCs and gained more attention in the last five years [18,19]. FSP is capable of overcoming the shortcomings of casting and powder metallurgy routes. Mishra el al. [20] conceived an idea to use FSP to create AMCs. The working principle of FSP was derived from friction stir welding (FSW) which was invented at The Welding Institute (TWI) in 1991. FSP induces severe plastic deformation and material flow due to the frictional heat and translation of material across the tool. The ceramic particles are packed along the FSP direction by applying any one of the methods which include straight groove, V groove, circular holes etc. The rotating action of the tool and plasticized material flow mix with the packed ceramic particles to form the composite. AMCs produced at optimized FSP parameters will be free of porosity, clusters and interfacial reaction. Moreover, FSP is not sensitive to the type of ceramic particles commonly employed in engineering applications. Hitherto, FSP has been successfully employed to produce AMCs reinforced with SiC [21], Al₂O₃ [22], TiC [23], Si₃N₄ [24], Ni [25], CNT [26], NiTi [27], TiN [28] and solid lubricants [29,30].

The structure and properties of Al/FA AMCs produced using FSP is not reported in literatures. Hence, the objective of the this research work is to synthesize AA6061/FA AMCs using FSP and characterize the evolution of microstructure, distribution, micro texture and sliding wear behavior. Aluminum alloy AA6061 is extensively used in industries and possesses good castability, weldability, reasonable strength and corrosion resistance [31].

2. Experimental procedure

Aluminum alloy AA6061 plates of size 100 mm x 50 mm x10 mm were used for this research work. The chemical composition of aluminum alloy AA6061 is presented in Table 1. A groove of 5.5 mm deep was made along the centre line of the plates using wire cut electrical discharge machining (WEDM) and compacted with FA particles. The average size
of FA particles used in this work was 2 μm. SEM micrograph of FA particles is shown in Figs. 1a and b. A pinless tool was initially used to cover the top of the groove after filling with FA particles to avoid the particles from scattering during FSP [32]. A tool made of HCHCr steel having threaded pin profile was used for the present study. The tool had a shoulder diameter of 18 mm, pin diameter of 6 mm and pin length of 5.8 mm. FSP was carried out on an indigenously built FSW machine. The process parameters employed were: tool rotational speed = 1600 rpm; traverse speed = 60 mm/min and axial force = 10 kN. The process parameters were adopted from Rejil et al. [33] which were optimized to yield desirable distribution of second phase particles without macroscopic defects in the FSP zone. The FSP procedure to produce the composite is available elsewhere [32]. Two passes were applied in opposite directions to achieve better distribution of FA particles. FSP was processed on three such plates by varying the width of the groove (0.4, 0.8, and 1.2 mm) to have four levels of volume fraction of FA particles (0, 6, 12, and 18 vol.%). Zero volume fraction refers to unprocessed aluminum alloy AA6061. The theoretical and actual volume fractions of FA particles were calculated as reported by Sathiskumar et al. [34].

Specimens were obtained by cutting the friction stir processed plates at its centre perpendicular to the processing direction. They were polished as per the standard metallographic procedure and etched with Keller’s reagent. The digital image of the macrostructure of the etched specimens was captured using a digital optical scanner. The microstructure was observed using a metallurgical microscope, scanning electron microscope (SEM) and electron backscattered diffraction (EBSD). Selected samples were electro polished using a mixture of perchloric acid and methanol to observe micro texture using EBSD. The microhardness was measured using a microhardness tester at 500 g load applied for 15 seconds at various locations in the composite.
The sliding wear behavior of AA6061/FA AMCs was evaluated using a pin-on-disc wear apparatus (DUCOM TR20-LE) at room temperature according to ASTM G99-04A standard. Pins of size 6 mm x 6 mm x 40 mm were prepared from the FSP zone by WEDM. The wear test was conducted at a sliding velocity of 1.0 m/s, normal force of 20 N and sliding distance of 3000 m. The polished surface of the pin was slid on a hardened chromium steel disc. A computer aided data acquisition system was used to monitor the loss of height. The volumetric loss was computed by multiplying the cross sectional area of the test pin with its loss of height. The wear rate was obtained by dividing volumetric loss to sliding distance. The worn surfaces of the test specimen were observed using SEM. The wear debris which were scattered on the face of the counterface were carefully collected and characterized using SEM.

3. Results and discussion

The representative crown appearance of the stir zone of friction stir processed AA6061 with FA particles is presented in Fig. 2. The surface of the crown is flat without any depressions, voids and discontinuities. There are no flaws on the crown surface. Semi circular striations which were formed due to the rubbing action of the rotating tool are seen on the crown. The spacing between the striations is equal to the ratio of traverse and tool rotational speeds i.e. weld pitch [35]. These striations do not affect the microstructure of the stir zone beneath the crown. But they indicate the material flow across the stir zone. A smooth crown appearance is essential as it leads to the formation of internal defects in the stir zone. The smooth crown appearance can be attributed to the optimized parameters used in this work.

3.1. Macrostructure of AA6061/FA AMCs
The macrostructures of AA6061/FA AMCs are depicted in Figs. 3a–c. The stir zone area which houses the AMC is obviously evident in all the figures. The periphery of the stir zone is marked using a black line. The identity of the groove on the aluminum plate before FSP is totally disappeared. It confirms that the formation of the composite is complete and plasticized material flow is continuous. The rubbing of the tool shoulder and the shearing of the pin develop ample frictional heat which turns the aluminum matrix around and below the tool into plastic state. The rotating and translation movement of the tool moves the plasticized aluminum from advancing side to retreading side. This movement of material flow forces the groove to yield and combines the packed ceramic particles with the plasticized aluminum alloy. The speed at which the tool rotates and translates determines the intensity of mixing resulting in the formation of the composite.

The width of the stir zone steadily decreases across the depth of the stir zone. The material flow characteristics during FSP influence the difference in the stir zone width. Two modes of material flow are present during the formation of stir zone as observed by Kumar and Kailas [36]. Both the shoulder and the pin of the FSP tool create two kinds of material flows i.e. “pin driven flow” and “shoulder driven flow”. The scale of resultant material flow changes along the depth of the stir zone. The shoulder driven flow of plasticized material is more dominant at the top and inferior at the bottom of the stir zone. Therefore, the width of the stir zone decreased along the depth of the stir zone. The area of the stir zone was computed using an image analyzing software. The area of the stir zone was estimated to be 50 mm² at 6 vol. % FA and 41 mm² at 18 vol. % FA. The area of the stir zone shrinks as the volume fraction of FA particles is raised from 6 vol. % to 18 vol. %. FA particles were initially compacted into the groove at the middle of the plate. The same tool was used without alteration in dimensions for FSP of all plates. The reason for reduction in stir zone area can be attributed to the following aspects. The increased volume fraction of FA particles elevates
the flow stress of the plasticized AMC. Because, the non deformable FA particles present resistance to the movement of plasticized aluminum. Secondly, FA particles behave as a thermal barrier and insulate the frictional heat from reaching the aluminum to certain extent. The consequence is to reduce the available friction heat for plasticization. The above mentioned two aspects magnify the flow stress value required to plasticize the AMCs. Hence, the area of the stir zone reduces as volume fraction is raised.

It is apparent from Figs. 3a–c that the stir zone is free from defects such as pin holes, tunnels, voids, cracks and kissing bonds. Those defects are often faced in FSW/FSP [37,38]. Defects minimize the area of the stir zone and cause the composite weaker to sliding wear and tensile loading. None of those defects occurred in the stir zone. Defects arise owing to numerous factors which are not limited to inadequate heat generation, material flow and consolidation. The process parameters control those factors appreciably. Absence of defects can be related to the chosen set of process parameters used in this work.

Fig. 4 shows a correlation between theoretical and actual volume fraction of FA particles in the AMC. The actual volume fraction is noted to be lower than that of the theoretical volume fraction under all experimental conditions. The theoretical volume fraction was estimated taking into consideration of the projected area of the pin. However, the frictional heat plasticizes additional aluminum higher than the swept volume of the pin. The compacted FA particles blend with more aluminum than that is determined. So, the volume fraction is reduced.

3.2. Microstructure of AA6061/FA AMCs

The selective SEM micrographs of the developed AA6061/FA AMCs are presented in Figs. 5a–f. The distribution of FA particles is seen all over the aluminum matrix. The distribution can be considered as reasonably homogeneous in the composite. The plasticized
aluminum matrix flows from advancing side to retreading side during FSP and cause the groove to cave in. The plasticized material is finally forged at the backside of the tool. The rotating tool exerts an intense stirring action which combines the compacted FA particles in the groove with the plasticized aluminum. The distribution of the particles in the aluminum matrix is a function of tool rotational speed [39]. The homogeneous distribution can be related to the tool rotational speed used in this research work. It is evident from the SEM micrographs that most of the FA particles are located within the grain boundaries. There is no segregation or arrangement of particles along the grain boundaries. The distribution is completely intragranular. Segregation deteriorates the mechanical and tribological properties of the composite. The ceramic particles naturally moves within the aluminum melt due to density gradient and cause segregation and clustering of particle. This is the limitation of liquid metallurgy routes. The free movement of the particles due to the density gradient is absent in FSP since the whole process is completed in solid state without melting of the aluminum matrix.

The severe plastic strain induced by the FSP on the aluminum matrix has the tendency to break and alter the shape and size of reinforcement particles [40]. The shape and size of FA particles are remarkably altered in comparison to the initial morphology as seen in Figs. 1a and b 1. Most of the FA particles lost its spherical shape. The vigorous stirring action of the tool broke spherical FA particles into irregular shapes. Some FA particles which were larger in size managed to withstand the severe plastic strain to retain its shape. The breaking of FA particles produces large number of fragment debris. It is interesting that there is no clustering or segregation of debris. This implies that the fragmented debris also merged with the plasticized aluminum and dispersed homogeneously in the composite. The size of debris is observed to be ranging in the order of nanometer to sub micron. The change in size of FA particles subsequent to FSP creates functionally graded local regions within the composite.
Figs. 6a and b reveal the SEM micrograph of AA6061/FA AMCs at higher magnification. The interface details of several FA particles are seen in this figure. Some researchers observed pores around reinforcement particles in AMCs developed using FSP [39]. No pores were observed around any of FA particles in Figs. 6a and b. Absence of pores can be attributed to sufficient material flow and plasticization of aluminum matrix at the chosen process parameters. The interfacial bonding between the reinforcement particle and the aluminum matrix contributes to a major role to transfer the load effectively during tensile loading and sliding wear. Excellent interfacial bonding is a prerequisite despite homogeneous distribution to enhance the properties. The temperature rise during the formation of the composite affects the interfacial bonding strength to a larger extent. The aluminum reacts with FA particle to form intermetallics at elevated processing temperature [8,11]. The reaction products deposit at the interface and weaken the interfacial bonding strength. No such intermetallics or reaction products were noticed around the FA particles. The temperature rise during FSP is insufficient to initiate any interfacial reaction.

Figs. 7a–h represent the optical photomicrographs of AA6061/18 vol.% FA AMCs snapped at various regions within the stir zone. The FA particles are distributed in every region inside the stir zone. There is no region which is free of FA particles. The micrographs reveal that the distribution of FA particles is independent of the region within the stir zone. The variation in the distribution of FA particles from the advancing side to the retreading side or from the top side to the bottom side is negligible. This result contradicts few researchers who observed significant change in the distribution of reinforcement particles across the stir zone [41–43]. The negligible variation can be correlated to sufficient plasticization of aluminum matrix and dispersion of FA particles to all regions of the stir zone at the chosen tool rotational speed. It is difficult to obtain constant distribution of reinforcement particles across the whole composite synthesized using casting methods. The velocity of the
solidification front will be varying across the mould which induces considerable variation in
the distribution of reinforcement particles across the composite castings. It is further observed
in Figs. 7g and h that there is no formation of onion rings at the bottom of the stir zone. This
indicates that the temperature variation from top to bottom of the stir is insufficient to mix the
flows of plasticized material to form onion rings.

EBSD images of AA6061/FA AMCs at various volume fractions and the effect of
volume fraction on average grain size are depicted in Figs. 8a–d and Fig. 8e respectively.
Coarse grains (Fig. 8a) which are perpendicular to rolling direction are seen in the matrix
alloy. The average grain size was measured to be 72 μm. The grains in AA6061/FA AMCs
are considerably finer in comparison to matrix alloy. The formation of fine equiaxed grains is
the result of dynamic recrystallization due to intense plastic deformation. The strain rate
during FSP can attain values up to 80s⁻¹ at the contact surface of the tool pin and the matrix
material which is enormous compared to other traditional severe plastic deformation
processes (0.1– 80s⁻¹) [20]. Such a huge strain rate leads to refinement of grains in the
aluminum matrix. The grain size of the composite is reduced with an increase in volume
fraction of FA particles. This leads to a conclusion that the FA particles act as grain refiners.
The movement of the grain boundaries is pinned by the FA particles which slow down the
rate of grain growth caused by dynamic recrystallization. This is known as pinning effect
which refines the grain size. The curve in Fig. 8e is not linear. The slope of the curve is very
abrupt from 0 to 6 vol. % and modest after 6 vol. %. This can be explained by taking into
account the mechanism of grain refinement in FSP to that of casting routes. The ceramic
particles serve as grain nucleating sites in casting routes and limit the freely growing
aluminum alloy. The intense plastic deformation during FSP is the additional mechanism
which offset the pinning effect of particles at low volume fraction.
3.3. Microhardness of AA6061/FA AMCs

Fig. 9a represents the effect of FA particles on the micro hardness of AA6061/FA AMCs. The reinforcement of FA particles remarkably improved the micro hardness of AA6061/FA AMCs. The microhardness was measured to be 62 HV at 0 vol.% and 125 HV at 18 vol.% FA particles. The microstructural changes caused by the reinforcement of FA particles are accountable for the improvement in micro hardness. The strengthening mechanisms are detailed as follows. FA particles are distributed all over the aluminum matrix which provides Orowan strengthening [44]. The fine distribution of FA particles resists the motion of dislocations and the path of dislocations is thwarted. The presence of FA particles multiplies the dislocation density of AA6061/FA AMCs compared to unreinforced AA6061. Secondary dislocations are formed due to the thermal mismatch and the differential deformation between the aluminum matrix and the FA particles. The dislocation motion is slowed down due to an increase in dislocation density across the AMC. Further, the difference in thermal contraction between the aluminum matrix and the FA particles results in quench hardening effect. According to Hall-Petch relationship, the grain size affects the mechanical properties of metallic materials. The grain size of AA6061/FA AMCs is smaller to that of aluminum matrix due to grain refinement of FA particles. The fine grains results in increased micro hardness. The effect of the aforementioned mechanisms multiplies as the volume fraction of FA particles is increased. Moreover, increasing the volume fraction of FA particles reduces the distance between them which causes an increase in the required stress for dislocation movement between the FA particles. Consequently, micro hardness is increased.

3.4. Sliding wear behavior of AA6061/FA AMCs
Fig. 9b depicts the wear rate of AA6061/FA AMCs as a function of vol. % of FA particles. It is obvious from the figure that the wear rate of AA6061/FA AMCs decreases as a function of FA content at constant wear test conditions. The wear rate was estimated to be 411 x 10^{-5} \text{mm}^3/\text{m} at 0 vol. % and 203 x 10^{-5} \text{mm}^3/\text{m} at 18 vol. %. FA particles enhanced the wear resistance of the AMCs extensively. The factors described in sec. 3.3 strengthen the AMCs and contribute to improvement in wear resistance of the AMCs. Archard’s law provides a relationship between hardness and wear rate of metallic materials. The volume loss of material during sliding wear is inversely proportional to the hardness of the AMC according to Archard’s expression. Higher the hardness of the AMC, lower will be the wear rate. Because the increase in hardness of the AMC increases the resistance to sliding wear. The aluminum matrix surrounded by the particles is easily worn away during sliding. Hence, the effective contact area between the AA6061/FA AMC specimen and the counter disc is reduced in comparison to unreinforced AA6061. FA particles bear the applied normal load. The superior interfacial bonding between the aluminum matrix and the FA particles retard the detachment of FA particles from the aluminum matrix during sliding. The good interfacial bonding and the homogeneous distribution of FA particles reduce the coefficient of friction [45]. The aforesaid factors lead to higher wear resistance of AA6061/FA AMCs. It is evident from Fig. 9b that the wear rate of AMCs with increasing content of FA particles is not linear. The non linear behavior is due to the occurrence of complex wear mechanisms of composites during sliding.

The worn surface of AA6061/FA AMCs at various volume fractions of FA particles is presented in Figs. 10a–d. The worn surface of aluminum alloy AA6061 in Fig. 10a displays large amount of plastic flow, fragmentation marks and deep craters. Groove pattern becomes to appear on this worn surface. Frictional heat develops during sliding wear which forces the
material to plasticize. The plasticized aluminum is primarily sticky to the counterface and consequently removed as sliding progresses. The plasticized aluminum alloy is furthermore exposed to the cutting action of the asperities of the counterface in the absence of FA particles. The cutting action and the removal of material in lumps produce a crater on the worn surface. The wear mode appears to be largely adhesion and abrasion to a smaller amount. The worn surfaces of AA6061/FA AMCs exhibit flat surface and are clearly different to that of aluminum alloy AA6061. Parallel groove like patterns are evident on the worn surface which are the marks of abrasive wear mechanism. The wear mode has transferred to abrasion from adhesion. This transfer of wear mode is prominent for achieving a significant improvement in the wear performance of AA6061/FA AMCs. The plastic flow of the matrix at the edges of wear tracks is too little due to the reinforcement of FA particles. The worn surfaces are covered with numerous loose and compacted wear debris. The wear debris do not adhere to the worn surface due to its hard nature. No craters are seen on the worn surfaces due to the homogeneous distribution of FA particles in the aluminum matrix. The grooves are uniformly distributed throughout the worn surface and in due course broke off to turn into debris.

The wear debris of AA6061/FA AMCs at various volume fractions of FA particles is presented in Figs. 11a–d. It is observed from Fig. 11a that the wear debris of AA6061 reveals a thin plate like morphology along with a minor amount of fine debris. The plasticized aluminum matrix is subjected to the cutting action of the counterface. The plate like morphology suggests that the material removal rate during sliding is high and confirms that the operating wear mechanism is adhesive. A minor amount of fine debris can be attributed to fragmentation of asperities during the initial stages of sliding wear. It is evident from Figs. 11a–d that the volume fraction of FA particles in the AMC considerably influences the
morphology and size of the wear debris. When the volume fraction of FA particles is increased, the wear debris morphology tends to be spherical and the size becomes finer (Figs. 11b–e). The spherical morphology suggests that the operating wear mechanism in the AMC is abrasive. The surface contact between the composite specimen and the counterface is small compared to unreinforced matrix alloy. FA particles project outside the surface and bear the load initially. As sliding proceeds the projected particles are either fragmented or detached from the specimen surface and trapped between the counterface and the sliding specimen. A new layer of projected particles will subsequently make contact with the counterface. The trapped particles alter two body abrasion wear into three body abrasion wear and creates fine spherical wear debris. The formation of spherical wear debris is similar to the conventional ball milling. The wear debris is ground to fine size till the specimen covers the set sliding distance. The spherical wear debris can be attributed to the reduction in wear rate of the AMC in comparison with matrix alloy. The spherical wear debris change sliding contact into rolling contact. The amount of friction is less in rolling contact compared to sliding contact. The material removal rate is decreased. The above discussed effects magnify as the volume fraction of FA particles is increased. The net result is formation of finer spherical wear debris and lower wear rate.

4. Conclusions

AA6061/(0,6,12 and 18 vol. %) FA AMCs were successfully synthesized using FSP. The microstructure, microhardness and sliding wear behavior were characterized. The following conclusions were derived from the present research work.

- The increase in volume fraction of FA particles caused a reduction in the area of the composite. The area of FSP zone was measured to be 50 mm$^2$ at 6 vol. % and 41 mm$^2$ at 18 vol. %.
The FA particles were distributed homogeneously in the composite irrespective of the volume fraction. The distribution was independent upon the location within the stir zone.

The FA particles experienced a change in size and shape due to high strain rate induced by FSP. The interface between the FA particle and the aluminum matrix was observed to be clean without the formation of any kind of intermetallics.

The grain size of the composite was refined remarkably by the combination of intense plastic deformation and the pinning effect of FA particles.

FA particles enhanced the microhardness of the composite. The microhardness was measured to be 62 HV at 0 vol. % and 125 HV at 18 vol. %.

FA particles improved the wear resistance of the composite. The wear rate reduced as the volume fraction of FA particles was increased. The wear rate was found to be $411 \times 10^{-5}$ mm$^3$/m at 0 vol. % and $203 \times 10^{-5}$ mm$^3$/m at 18 vol. %.

FA particles affected the wear mode as well as the morphology of the wear debris. The increased volume fraction of FA particles altered the wear mode from adhesion to abrasive. The wear debris changed from thin plate at 0 vol. % to spherical shape at 18 vol. %.

The enhancement of properties confirms that FA particles can be used as reinforcement for AMCs and will lead to reuse of FA particles avoiding land pollution.
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Table Captions

Table 1 Chemical composition of AA6061 aluminum alloy.