Characterisation of floor tiles reinforced aluminium surface composite synthesized by friction stir processing

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Abstract. The present work employed Friction Stir Processing route for fabricating Aluminium Surface Composite. The fine ceramic particles obtained from floor tiles were selected as the reinforcement while Al6061 alloy as the matrix material. The fabricated surface composite was further investigated for microstructure and wear characterisation. Microstructural features depicted the successful incorporation and homogeneous dispersion of floor tile particles in the aluminium matrix. The reinforced particles enhanced the microhardness value to 92 HV which was about three times more than base metal. Higher wear resistance and lower friction coefficient exhibited on addition of floor tile particles in the matrix. The least wear rate of 4.9×10⁻³ mg/m and friction coefficient value of 0.30 was obtained in aluminium surface composite compared to 36.2×10⁻³ mg/m and 0.43 respectively.

Keywords: friction stir processing; aluminium surface composite; microstructure; wear; friction

1. Introduction

The alarming situation of global warming and environmental pollution has made the manufacturing industries to introspect and finds greener solutions to their energy requirements. The industries are conducting extensive research in the area of aluminium alloys which are reinforced with hard particles to form Aluminium Metal Matrix Composites (AMMCs). AMMCs have become the major part of current era materials and are considered to be an excellent light-weight alternative in the automobile, aerospace and marine components. The high specific strength, excellent machinability, high stiffness and superior surface properties are some of the factors responsible for the widespread use of aluminium composites [1-2]. However, the economical fabrication of aluminium composite with the desired properties has always been a major challenge for the industrial sector. The application of low-cost reinforcement such as floor tile, porcelain, marble dust, fly ash, rice husk etc has the potential of reducing the manufacturing cost of the composite. Ceramic particles obtained from waste floor tiles is the cheap alternative compared to costly conventional reinforcement such as SiC, B₄C, TiC etc. Floor tiles being non-biodegradable accumulate in the environment and can only be used as landfills, where they reduce the fertility of the soil. Hence application of ceramic tiles as reinforcement is a rational way to minimise the fabrication cost of AMMCs and its harmful effect on the environment [3-5].
AMMCs fabrication can be achieved through various fabrication techniques, popularly such as squeeze casting, stir casting, liquid infiltration and powder metallurgy. However, these methods involve certain limitations like segregation and non-uniform dispersion of reinforcement, reinforcement and matrix reaction to form undesirable second phase, presence of voids and complex process parameters involved. Moreover, these methods are only useful for bulk AMMCs fabrication [6-8]. There are certain applications where it is required the material surface to be strong and hard while its core to be soft. The following desired surface behaviour of the material can be fulfilled by the fabrication of Surface Composite or Surface Metal Matrix Composites (SMMCs) [9]. The Friction Stir Processing (FSP), working principle derived from Friction Stir Welding (FSW), is the newly emerging technology dedicated particularly for surface modification [10, 11]. FSP was initially developed for altering the microstructure of material through grain refinement leading to improved mechanical properties; imparting superplasticity and so on [12-14]. With advancement in technology, FSP was found to be unique methodology for producing the surface composites. The solid-state phenomena in FSP assisted in overcoming the limitations of conventional methods. The procedure for FSP aided surface composite fabrication can be referred from the literature [15-17].

The literature survey of FSP produced Aluminium Surface Composite revealed numerous investigations on the use of conventional reinforcement such as B₄C [18], SiC [19], SiC+Gr [20], B₄C+MoS₂ [21], ZrSiO₄ [22], CaCO₃ [23], TiN [24], TiB₂+BN [25], Al₃Fe [26] etc. The current trend towards green technology encouraged the development of low-cost surface metal matrix composites. The latest research performed by the researchers utilised alternative cheap reinforcements such as rice husk and fly ash to fabricate the surface composites. Dinaharan et al. employed different % of Fly Ash (FA) particles in Al6061 matrix through FSP technique and obtained Al6061/FA surface composite. The microstructural characterisation revealed homogeneous dispersion of FA particles and grain structure refinement in the Al6061 matrix. Further with increase in % of FA particles, there was significant enhancement in the microhardness and wear resistance of the surface composite [27]. Moreover, Dinaharan et al. reported similar microstructural, mechanical and wear behaviour when FA and Rice Husk particles were reinforced in AZ31 [28] and Copper [29] matrix respectively through FSP. The literature study revealed the research potential in the area of economical surface composites fabrication through FSP. The application of Floor Tile (FT) particle as a reinforcement has not been reported yet. In the present investigation, different % of FT particles was reinforced in the Al6061 matrix through Friction Stir Processing (FSP) technology to obtain Al6061/FT surface composite. The surface composite was investigated for the morphology of FT particles in 6061 matrix. The Al6061/FT surface composite was further characterised for microstructural and wear behaviours.

2. Material and methods
In the present study of surface composite fabrication, aluminium alloy of 6061 series was selected as the matrix material in the form of plates having 200×80×10 dimension. The chemical composition of Al6061 analysed through spectroscopy is depicted in table 1. The fine ceramic particles obtained from the ball milling of waste floor tiles were selected as the reinforcement. The size of Floor Tiles (FT) particles was determined with the help of sieve shaker equipment. The particles size was found to be ranging from 53 µm to 75 µm. After the material selection, the surface composite was prepared using Friction Stir Processing (FSP) technique. The working principle of the FSP procedure is depicted in figure 1.

Table 1. Chemical composition of Al6061 alloy

| Chemical element | Mn | Fe | Mg | Si | Cu | Zn | Ti | Cr | Others | Al |
|------------------|----|----|----|----|----|----|----|----|--------|----|
| % present        | 0.15 | 0.70 | 0.8-1.2 | 0.4-0.8 | 0.15-0.4 | 0.25 | 0.15 | 0.04-0.35 | 0.15 | Rest |
Figure 1. Schematic sketch of Friction Stir Processing (FSP) principle.

Figure 2. Equipments employed for performing FSP operation: (a) FSW set up; (b) FSP tool.
As per the FSP procedure, firstly the rectangular groove was prepared on the Al6061 plate surface with the help of milling cutter. The groove was properly cleaned with acetone for further processing. Three different grooves of width (1 mm) and depth (3, 4, 5 mm) were prepared on the plate to obtain the volume fraction of 12, 16 and 20 % of FT particles. The expression used for determining the volume fraction of reinforcement [30] is

$$\text{Volume fraction} = \frac{\text{Area of groove}}{\text{Projected area of probe}} \times 100$$

The FT particles were packed into the groove manually and then capping pass using pinless tool was done to close the groove. Finally, FSP tool performed the processing operation to obtain the surface composite layer. The indigenously developed Friction Stir Welding (FSW) machine “RV machine tools, FSW-4T-HYD” set up was employed for FSP operation. Fig. 2 exhibits the FSW set up and schematic sketch of FSP tool. The cylindrical FSP tool features were: 20 mm shoulder diameter, 5 mm pin diameter and length, H13 tool steel hardened to 50-60 HRC. The parameters employed during entire FSP experiment were: constant rotational speed of 1000 rpm, traverse speed of 30 mm/min, tilt angle of 0° and 2 FSP passes. The selection of parameters was made based on the literature and previous experience.

The surface composite layer obtained was further analysed for microstructure and wear characterisation. Samples for the same were delved from the processed region through CNC wire cut. Polishing, Optical microscopy (OM) images and X-ray Diffraction (XRD) techniques were employed for microstructural investigation. Polishing of samples was done with different grades of emery from coarse to fine for dry polishing and alumina powder for wet polishing. Finally the samples were etched for 15 seconds using Keller’s reagent etchant to reveal the grain boundaries. The used etchant is a chemical solution of distilled water (190 ml), HNO₃ (5 ml), HCl (3 ml) and HF (2 ml). Finally samples were observed under OM to acquire the microstructure images. The XRD (BRUKER D8 ADVANCE) was done to know the reinforcement existence in the Al6061 matrix. XRD spectrums were obtained from powder diffractometer with Cu-Kα radiation having wavelength of 1.5418. The microhardness testing machine (FISCHERSCOPE HM2000S) was utilised for determining the hardness of the samples at 300 mN load and 5 seconds dwell time. The pin-on-disc tribometer (TR-20L-PHM800-DHM850) was used for the dry wear analysis of the samples at ambient temperature conditions. The pin samples of 9 mm diameter were made to slide against the 100 mm EN 24 steel counter disc having hardness 58 HRC and surface roughness (Ra) value of 0.2 µm. The parameters selected for the tribometer test were: sliding speed of 1.5 m/s, sliding distance of 2000 m and normal load of 30 N. Wear samples were weighed using electronic weighing balance before and after test to determine the weight loss and finally the wear rate.

3. Result and discussions

3.1. Microstructural characterisation

The OM images shown in figure 3 depict the microstructural characteristics of Aluminium surface composites samples. It can be observed that the Floor Tile (FT) particles were properly embedded in the aluminium matrix through FSP procedure. The vigorous stirring action developed during FSP operation assisted in the fragmentation of FT particles, thus creating their homogeneous dispersion in the Al6061 matrix. The brittle nature of particles resulted in their uneven and non-uniform fragmentation. The morphology of surface composite also exhibited the formation of proper interfacial bonding between FT particle and matrix. Further, absence of defects in the processed zone resulted in the successful fabrication of Al6061/FT surface composite. Similar microstructural observations were reported in the FSP aided fabrication of Al1060/SiC [31] and Mg/SiC [32]. The morphology shown in figure 3 (d) depicts the presence of FT particles at the Grain Boundaries (GBs) of the Al6061 grain structure. It shows that the particles will provide the pinning effect thus restricting the grain growth of the base alloy. Navazani and Dehghani et al. [33] also reported the similar pinning effect of TiC particles when reinforced in the AZ31 matrix through FSP.
Figure 3. Optical Microscopy (OM) images of Al6061/FT surface composite at different magnifications.

Figure 4. XRD pattern of Al6061/FT surface composite.
The major components of floor tile particles consist of clay, silica and feldspar (K(AlSi₃O₈)) [34]. The XRD results of the FSP produced Aluminium surface composite sample was analysed through XRD spectrum shown in figure 4. The spectrum depicts the distinct major peaks of α-Al while marginal peaks of feldspar compound. Moreover, peaks of other compounds were not observed in the spectrum. The reason may be due to the insufficient temperature developed during FSP which restricted the interfacial reaction of matrix and reinforcement thus avoiding the formation of other compounds.

3.2. Microhardness analysis

The microhardness value (HV) of Aluminium surface composite samples reinforced at various % of FT particles is shown in table 2. The addition of FT particles increased the HV value of Al6061 alloy from 36 to a maximum of 92 HV (at 20 % FT). The appreciable amount of hardness present in ceramic FT particles resisted the penetration of composite samples resulting in enhancement of overall hardness. Moreover, homogeneous dispersion and proper bonding of FT particles in the Al6061 matrix, assisted in bearing the load developed during testing, thus increasing the microhardness of Al surface composites. The microhardness enhancement can also be attributed to the presence of dislocations produced due to unlike coefficient of thermal expansion values of matrix and reinforcement. The excessive dislocation density created hindrance in the deformation thus enhancing the hardness of surface composite samples. Additionally, aluminium surface composite fine-grain morphology obtained due to refinement of grain structure (provided by FSP) and pinning effect (provided by FT particles) also contributed to the hardness enhancement which can be justified accordingly by Hall-Petch relationship [35].

Table 2. Microhardness and Wear properties of the studied samples

|                              | Base Metal (BM) | 12 % FT | 16 % FT | 20 % FT |
|------------------------------|----------------|---------|---------|---------|
| Average Microhardness Value (HV) | 36             | 69      | 80      | 92      |
| Wear Rate (mg/m)             | 0.0362         | 0.0102  | 0.0075  | 0.0049  |
| Average Coefficient of Friction (COF) | 0.43           | 0.38    | 0.31    | 0.30    |

3.3. Wear characterisation

The wear behaviour investigated through pin-on-disc Tribometer equipment is depicted in table 2. The Aluminium surface composite samples exhibited significant reduction in wear rate compared to the base metal. The wear loss in surface composite sample reinforced at 20 % FT was 71 % lower than the Al6061 matrix. Moreover, wear rate follows decreasing trend with increase in volume fraction (%) of FT particles. The wear rate of sample having 20 % FT was approximately 52 % lower than 12 % FT composite sample. During wear testing, the weight loss (mg) of samples was measured after every 500 m revolution of the disc as shown by the plot in fig. 5. According to Archard relation [36], weight loss in the material and its hardness has notable relation. The significant amount of hardness obtained in surface composite samples contributed to less wear rate compared to the parent metal. Further, FSP aided homogeneous dispersion of FT particles in the 6061 alloy matrix, assisted in significant reduction of wear loss. It can be concluded that significant amount of hardness in surface composite samples contributed to their high wear resistance. The plot shown in fig. 6 depicted the inverse relation of microhardness and wear rate. Ahmadkaniha et al. [37] also reported less wear rate in FSP produced AZ91D/Al₂O₃ surface composite compared to AZ91 matrix.

The Coefficient of Friction (COF) relation with the sliding distance for all studied samples is shown in figure 7. The average COF value of Aluminium surface composite was 30 % lower (for 20 % FT) compared to the base metal. Additionally, fluctuation in COF value in case of composite was less as compared to the base metal. The load-bearing ability of FT particles contributed to lower COF value in aluminium surface composite. FT particles can absorb the direct normal load (30 N) during wear test thus exhibited lesser friction coefficient. The similar observation of friction coefficient behaviour was reported in FSP produced Al6061/B₄C [38] and ZM21/B₄C [39] composite. Moreover, strong load-
bearing effect in case of 20% FT composite sample due to more no. of FT particles contributed to least COF value compared to 12 and 16% FT samples.

**Figure 5.** Weight loss relation with the sliding distance.

**Figure 6.** Plot showing inverse relation of microhardness and wear rate.
Figure 7. COF relation with Sliding Distance for (a) Base Material (BM); (b) 12% FT; (c) 16% FT and (d) 20% FT.

4. Conclusions
The present experiment successfully reinforced the Floor Tiles (FT) particles (vol. fraction of 12, 16 and 20) in Al6061 matrix through Friction Stir Processing technique thus obtaining Aluminium Surface Composite. The major findings observed after the characterisation of the surface composite were:

1. The OM and XRD results confirmed the FT particles presence in the soft matrix of aluminium alloy. Moreover, FSP assisted in the fragmentation and uniform distribution of FT particles in the Al6061 matrix. FT particles behaved as pinning agents in the matrix, thus restricting the grain growth of the matrix.
2. The addition of FT particles increased the Microhardness value from 36 HV of base metal to a maximum of 92 HV.
3. The FT particles were found to be responsible for reduced wear rate and coefficient of friction value compared to the base metal. Further with increase in % of FT particles there was significant enhancement in the microhardness and wear resistance of surface composite.

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