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PREPARATION OF 5083 AI-SIC SURFACE COMPOSITE BY FRICTION STIR PROCESSING AND ITS MECHANICAL CHARACTERIZATION

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Abstract. In this study, surface composite based on 5083Al matrix reinforced with nano-sized silicon carbide particles has been fabricated by Friction stir processing (FSP). Microstructure, hardness and wear behavior of the surface composite have been investigated and compared with those of 5083Al alloy. It is observed that the friction stir processed (FSPed) sample possess higher hardness, especially in the nugget zone, as compared to 5083Al. The tribological studies showed that wear resistance of the surface composite is observed to be significantly inferior to that observed for 5083Al, as a result of high coefficient of friction and higher friction force developed during the course of sliding wear. This leads to detachment of hard SiC particles from the surface of FSPed sample. Microstructural analyses of worn track and wear debris reveal that the primary wear mode in FSPed is abrasive whereas in 5083Al both abrasive and adhesive wear modes are operative.

Key words: Friction Stir Processing, Microhardness, SiC, Wear, SEM

1 Introduction

Aluminum alloys are very promising for structural applications in aerospace, military and transportation industries due to their light weight, high strength-to-weight ratio and excellent resistance to corrosion. However, the low hardness and low strength of aluminium alloys limit their use, especially for tribological applications. In comparison to unreinforced aluminum alloys, aluminum/aluminum alloy matrix composites reinforced with ceramic phases exhibit higher strength and hardness, improved tribological characteristics, and increased resistance to creep and fatigue [1]. For applications involving aluminum and its alloys where surface contact is involved, the useful life of the components is mainly determined by their surface properties such as wear resistance and hardness. Therefore, it is highly desirable that surface layer of the component is reinforced by hard ceramic particles to achieve the desired hardness while the substrate still maintains the original structure with good ductility and thermal conductivity [2, 3-5].

Frictions stir processing (FSP), based on the principle of friction stir welding, is an emerging solid state metal working process and has proved to be a successful technique for fabrication of hybrid Surface Metal Matrix Composites (SMMCs) in aluminum/aluminum alloy plates [6-11]. This technique causes intense plastic deformation and high strain rates in the processed material resulting in precise control of the microstructure through material mixing and densification [12, 13]. Miracle [14] observed that FSP densifies the microstructure, refines the grain size, results in closure of porosities and provides a convenient method to improve the surface properties of aluminum alloy by forming surface composites. Ma et al. [15] investigated the effect of multipass FSP on the microstructure and tensile properties of a cast aluminium-silicon alloy. They concluded that Si particles were uniformly distributed in the entire processed zones created by multiple-pass FSP and both the strength and ductility of the transitional zones were lower than those of the nugget zones. Darras et al. [16] observed that FSP refines the microstructure from an average grain size of about 6 µm to about 3-4 µm and showed that the hardness of the processed sheets was extremely sensitive to the processing parameters. Rao et al. [17] investigated the effect of single and double pass FSP on micro structural refinement of hypereutectic Al-30% Si alloy. Chen et al. [18] investigated the effect of processing parameters on microstructure and mechanical properties of Al-Al11Ce₃-Al₂O₃ in-situ composite produced by Ce₃-Al₂O₃ FSP. The study indicates a significant difference between the compressive and tensile strength of the composite produced under different processing conditions. Mahmoud et al. [13] studied wear characteristics of surface-hybrid-MMCs layer fabricated on aluminum plate by FSP. It is found that the addition of reinforcement ceramic powder (SiC, Al₂O₃ or mixture) to an aluminum matrix is beneficial in reducing the wear loss especially at relatively low loads. Dolatkhah et al. [19] produced metal matrix composite (MMC) through FSP on the surface of 5052 aluminum sheets by using SiC of 5 µm and 50 nm sizes. The results showed that the reversal of tool rotation direction between FSP passes,

increase in number of passes and decrease of SiC particles size enhance hardness and wear properties of the composites.

The literature survey reveals that FSP can be successfully employed to enhance the surface properties (viz. micro-hardness, wear resistance etc.) of metals and their alloys. However, the work pertaining to enhancement of surface properties of aluminum/aluminum alloys through friction stir processing (FSP) is rather scant. The present study deals with FSP of 5083Al alloy. The alloy is highly resistant to attack by both seawater and industrial chemical environments, apart from retaining exceptional strength after welding. But the tribological applications of this alloy is limited due to its poor hardness. The 5083Al alloy is typically used in shipbuilding, rail cars, vehicle bodies, mine skips and cages, pressure vessels, TV towers and drilling rigs [9]. The present study investigates the effect of doping the surface of 5083 Al with nano-sized SiC particles through friction stir processing on its mechanical behavior viz. microhardness and wear resistance.

2 Experimental Procedure

In this study 5083-Al alloy in plate form (Length: 150 mm, Width: 100 mm, Thickness: 6 mm) has been selected for the purpose of carrying out FSP. The chemical composition of the alloy, as estimated through spectroscopic technique, is Si (0.027%), Mg (4.4%), Cu (0.02%), Fe (0.19%), Mn (1.19%), Zn (0.026%), Cr (0.06%) and Ti (0.05%). The surface of 5083Al plate is doped through FSP with nano-sized SiC powder (size \cong 60-100 nm) to produce 5083Al-SiC surface composite.

The set up used for carrying out friction stir processing consists of a CNC vertical milling machine and a specially designed rotating FSP tool. The fixture for holding the base plate, while carrying out FSP, was designed and fabricated in house (Fig. 1). The fixture consists of a rectangular base plate of dimensions 400 mm x 200 mm x 20 mm. Three rods, having square X-section (25 mm x 25 mm) and length 300 mm were machined to achieve surface finish of the order of 5 μ m. Out of these square rods, two rods were drilled with counter sunk holes (4 Nos.) to adjust allen bolts of size M10. These two rods were fixed at the ends of rectangular base plate (Fig. 1). The square rod-2 consists of three-additional drilled holes with M12 internal threads to accommodate hexagonal bolts of size M12. The third square rod was placed between rods 1 and 2 and was movable with the help of hexagonal bolts tightened to square rod 2. The base plate, to be friction stir processed, was held tight between the square rods 1 and 3. The base plate was also tightened to the rectangular base plate with the help of two MS strips (S1 and S2) which were screwed to rectangular base plate with the help of hexagonal bolts (M8). The whole of the fixture (Fig. 1) was fitted to the bed of CNC milling machine with the help of sliding screw arrangements.



Fig. 1 Fixture for holding specimen during FSP

A specially designed tool with shoulder and pin was used for the purpose of FSP. The tool was made of HSS with hardness ranging around 61-62 HRc, achieved through hardening and tempering treatment. The specifications of FSP tool used are given as below.

Pin and Shoulder Material:

High Speed Steel (Heat treated-M2)

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| Shoulder Dimension: | Dia. $= 12 \text{ mm}$, Length $= 60 \text{ mm}$ |
|---------------------------------|---|
| Pin Dimension: | Dia. = 4 mm, Length = 3.5 mm |
| Hardness of Tool: | 61 to 62 HRc |
| The symbol M2 refers to a speci | al type of HSS in tungsten-molybdenum series having |

The symbol M2 refers to a special type of HSS in tungsten-molybdenum series having high wear resistance. The carbides in it are small sized and evenly distributed.

The fixture containing duly gripped base plate was placed and tightened to the bed of CNC vertical milling machine. Subsequent to this, a number of holes of dia. 2 mm and 2 mm deep were drilled on the surface of base plate by keeping the centre distance between two consecutive holes as 4 mm (Fig. 2).



Fig. 2 Drilling holes on the top surface of base plate

Followed by drilling, the nano-sized alumina powder paste, prepared by mixing alumina powder in kerosene oil, was poured inside the drilled holes. The kerosene oil helps in retaining the alumina powder in the base plate during the course of subsequent FSP. Thereafter, the FSP tool was mounted in the spindle of CNC milling machine and the spindle was set to rotate at 1200 RPM and the machine bed was also given a feed of 40 mm/min. The FSP tool was plunged into the drilled hole, provided in the base plate, to a predetermined depth of 3.8 mm. As the tool progressed along the centre line of the drilled holes, a layer of 5083Al- SiC nano-composites was produced on the surface of base plate (Fig. 3).



Fig. 3 Image showing tunneling effect in friction stir processed 5083Al Plate

2.1 Microhardness Measurement

Microhardness of the friction stir processed samples was measured at various locations, ranging from the processed zone to base metal, with the help of Vicker's microhardness tester. In order to perform microhardness tests, the specimens were mirror polished to view the indentation marks. Firstly the FSPed samples were cut with hand hacksaw into small sizes along the longitudinal as well as transverse sections. The cut pieces were cold mounted by using a freshly prepared cold setting acrylic mixture of self cure polymer (powder) and self cure monomer (hardener). The mounting of the samples was performed inside a hollow MS pipe, enclosing

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the cut specimen, placed over a metallic-greased surface. The epoxy mixture was allowed to cold set for 5 min. The mounted samples (Fig. 4) were first ground on a flat belt grinder, followed by manual grinding with successive grades of emery papers of grit size ranging from 250 to 2000. The final polishing of the sample was done on a buffing machine by using diamond paste and continuously running water.

The hardness measurements were taken on the surface of samples at an interval of 2 mm, on both sides of centre line of FSP zone. The microhardness of the base alloy was also measured in a similar fashion. The size of indentation was measured with the help of a micrometer and an eyepiece having magnification of the order 10X and 40X, fitted on the hardness tester. The indentation marks were created with the help of a pyramid shape diamond indentor at a load of 20 gm and dwell time of around 10 sec.



Fig. 4 Mounted specimens

2.2 Wear Test

The wear behavior of the specimens, both unprocessed and FSPed, was evaluated on a pin-on-disk type wear testing machine (DUCOM make). The flat specimen having square X-section (4 mm x 4 mm) and thickness 6 mm were cut from the nugget zone of the FSPed sample and from the base alloy plate. These flat specimens were tight fitted in a pin shaped holder having diameter 10 mm and 30 mm long and provided with a circular slot to adjust the cut specimen. The pin holding the flat specimen was tightened into the grips provided with the wear testing machine, which was secured inside the loading arm, carrying a normal load of 5 kg. The sample was held against a rotating disc made of EN 32 steel (HRc 62) at a constant sliding speed of 1.56 m/sec. The wear test was conducted for a total sliding distance of 3000 m with the corresponding test duration of 32 min. The coefficient of friction during the wear test was monitored on a PC directly attached to the wear testing machine. The weight loss of the sample was measured by using an electron weighing balance, having an accuracy of 0.001 mg, during the course of wear test by stopping the machine after every 8 minutes of sliding. For the purpose of measuring weight loss of the pin, it was removed from the holder. After measuring the weight loss of the sample, the pin was replaced in the holder by keeping the same orientation for carrying out the wear test.

3 Results and Discussion

In order to investigate the effect of doping SiC particles on 5083Al plate through FSP, the specimens were characterized for microhardness and wear test, as described in previous section. The results obtained for friction stir processed samples are compared with those obtained for unprocessed 5083 Al.

3.1 Microhardness Profile

Figure 5 shows microhardness profile of the samples measured across top surface of the FSPed and unprocessed 5083Al samples. It is observed that the hardness of FSPed zone is significantly higher than that observed for the base alloy. The hardness is maximum (155 HV) in the centre of FSPed zone (nugget/stir zone) and decreases on either side to reach around 84 HV at a distance of around 2 mm from the centre of FSPed zone. Unlike FSPed sample, the microhardness of the base alloy remains almost uniform, ranging between 37 to 44 HV.

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Microhardness of the FSPed samples was also measured across the transverse section at different locations as shown in Fig. 6(a). The indentation marks 1, 2, 3, 4 and 5 are spaced at an interval of 2 mm whereas the indentation marks 5, 6, 7, 8, 9 and 10 are equispaced at a depth of 1 mm. As one moves from mark-5 towards mark-1, the micro hardness decreases from 210 HV to 91 HV (Fig. 6b), implying again that the subsurface hardness is also maximum in the nugget/stir zone and decreases significantly on moving away from the centre of FSPed zone. The comparison of Fig. 5 and Fig. 6(b) indicates that the subsurface hardness of the FSPed sample, at a given depth, is significantly higher as compared to the hardness measured at the corresponding location on the top surface. As one moves downward from mark-5 towards mark-10, the microhardness decreases from 210 HV to 49 HV (slightly higher than the hardness of 5083Al).



Fig. 5 Hardness profile measured across top surface of the FSPed and unprocessed 5083Al samples





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Fig. 6 (a) Location of indentation marks (b) Hardness profile across transverse section of FSPed sample

3.2 Wear Behavior

It is observed in Fig. 7 that for both the samples (FSPed and unprocessed 5083Al), the weight loss increase with sliding distance. The weight loss observed in FSPed sample is initially low up to a sliding distance of around 750 m and thereafter it increases significantly. However, the wear rate noticed for the 5083 alloy sample is significantly lower than that noticed for FSPed alloy sample. Unlike FSPed sample, the wear rate for 5083Al remains almost uniform over the entire sliding distance.



Fig. 7. Variation of weight loss with sliding distance



Base alloy



(a)

(b)

Fig. 8. Comparison of (a) coefficient of friction and (b) frictional force for FSPed and unprocessed 5083 Al

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High wear resistance exhibited by the base alloy is also evident from the lower coefficient of friction (avg. COF = 0.27) noticed for the base alloy than the FSPed alloy (avg. COF = 0.50) as depicted in Fig. 8 (a). The higher friction force observed for FSPed sample is responsible for the removal of hard SiC particles from the surface of FSPed sample thereby giving rise to more wear. On the other hand, due to masking of 5083Al surface by the soft Al phase, during the course of wear, relatively low wear rate is observed for 5083Al (Fig. 7).

Further, the presence of SiC particles on the surface of FSPed sample causes increase in hardness as well as coefficient of friction. The high value of friction coefficient and frictional force noticed during the wear testing of FSPed sample could be attributed to the fact that the hard reinforcing SiC particles damage the outer layer of rotating steel counter-face, thus increasing the roughness of steel disc. This in turn results in damage and destruction of the reinforced SiC particles. The damaged or crushed SiC particles get trapped between the sliding surfaces and additionally damage the 5083Al-SiC composite surface layer of FSPed sample through abrasion. The detachment of more and harder reinforcing SiC particles from the surface of FSPed sample leads to increase the friction coefficient and causes more weight loss of the FSPed sample.

In comparison to the unprocessed 5083 Al sample, the friction coefficient observed for FSPed sample fluctuated more (Fig. 8a). This may be due to the detachment of hard SiC particles from the surface of FSPed sample that act as a barrier against the sliding track and consequently increases the friction coefficient noticed for FSPed sample as compared to 5083 Al sample. In 5083 Al sample, the layered morphology of base particles has lubricating effect and helps in lowering the friction coefficient.

3.3 SEM of Worn Tracks

Scanning Electron Microscopy (SEM) of the worn track was carried out to identify the operating wear mechanisms in the sample. The SEM of the worn tracks for both 5083Al and FSPed alloy samples are shown in Fig. 9.



(a) $\int_{U_{2}} S_{3700} 15.0 kV 67.3 mm x100 SE} for the formula for the$

Fig. 9 Scanning electron micrographs of worn track: (a) 5083Al and (b) FSPed alloy specimens

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Fig. 9a (i) shows the worn track of 5083Al sample. During the initial period of wear, small pieces of materials are ploughed out from the surface of 5083Al sample which get adhered to the steel disc. These adhered particles abrade more and more material from the surface of 5083Al sample and lead to relatively higher wear. The presence of few grooves on the worn track, as showed in Fig. 9b (ii), results due to detachment of adhered material from the surface of 5083Al as a result of sliding action that causes plastic deformation on both sides of the groove. Hence it is revealed from the above discussion that the mode of wear in 5083Al is a combination of adhesive and abrasive.

Fig. 9(b) shows the worn surface of FSPed sample. The comparison of Figs. 9a(i) and 9b(i) reveals that the width of wear track decreased in FSPed sample as compared to 5083Al sample. The extent of wear in FSPed sample is relatively uniform than the 5083Al sample. The magnified view of the worn track of FSPed sample [Fig. 9b(ii)] reveals the presence of small pits, predominantly because of local removal of ceramic (SiC) particles from the surface of sample during the course of wear test. Though a few fine micro-scratch marks are observed on the worn track at lower magnification [Fig. 9b(i)] but no deep scratch marks are revealed at higher magnification [Fig. 9b(ii)]. Hence, it is revealed that the extent of wear in surface composite layer of FSPed sample is relatively uniform as compared to those observed in 5083Al sample. The mode of wear in FSPed sample is observed to be primarily abrasive.

3.4 EDS of Worn Track

Energy dispersive X-ray spectroscopy (EDS) analysis of the wear debris of FSPed and unprocessed 5083Al samples was conducted to evaluate the morphological features of wear debris. EDS of the worn debris obtained during the wear test of 5083Al sample was recorded as four different locations marked as a, b, c, and d in Fig. 10.



Fig. 10 SEM of worn track of 5083 Al sample



Fig. 11 EDS of wear debris of 5083Al at locations marked in Fig. 10

The EDS pattern, shown in Fig. 11(a), reveals that the wear debris comprises of carbon along with other elements like Al, Mg, and Fe. The presence of these elements indicates that the debris consist of elements present in 5083Al (e.g. Al, Mg) as well as in the steel disc (e.g. Fe, C), thereby confirming the abrasive as well as adhesive wear modes in 5083Al sample. The EDS pattern shown in Figs. 11(b) to 11(d) reveals that the extent of elements like Al, Mg and Fe is higher in the wear debris, which implies that the worn out particles/debris mainly result from the base alloy sample.

The EDS pattern of wear debris, at two different locations marked as (a) and (b) in Fig. 12, generated during the wear test of FSPed sample is shown in Fig. 13.



Fig. 12 SEM of worn track of FSPed sample

The EDS pattern shown in Figs. 13 (a) and (b) reveals the presence of mainly Al and O in the debris,

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apart from the traces of some other constituent materials of 5083Al as well as of the steel disc. This again confirms that the primary wear mechanism in FSPed sample is abrasive. The detachment of hard SiC particles from the surface of FSPed sample acts as a barrier to sliding and consequently leads to high friction coefficient and high wear rate of FSPed sample than that observed for 5083Al, in spite of higher surface hardness of FSPed sample.



Fig. 13 EDS of wear debris of FSPed sample at locations marked in (Fig. 11)

4 Conclusions

The present study reveals that the doping of 5083Al with hard SiC particles through FSP leads to significant increase in hardness of the surface composite produced on FSPed sample layer. The wear resistance of FSPed sample is inferior to that observed for 5083Al in spite of its higher hardness. It may be attributed to high coefficient of friction and higher friction force observed during the wear testing of FSPed sample, owing to detachment of hard SiC particles from the surface of FSPed sample during the course of wear. The operative wear mechanism in FSPed alloy is abrasive whereas in 5083Al sample both abrasive and adhesive wear mechanisms are operative.

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