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Synthesis-microstructure-mechanical properties-wear and corrosion behavior of an Al-Si (12%)—Flyash metal matrix composite

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In the present investigation, Aluminium based metal matrix composite containing up to 15% weight percentage of flyash particulates were successfully synthesized using vortex method. The properties like density, hardness, microhardness, ductility and ultimate tensile strength were investigated. The MMC produced was also subjected to corrosion, dry sliding wear and slurry erosive wear test to investigate its behavior under different material wearing conditions. The results of microhardness revealed higher hardness of the matrix material in the immediate vicinity of flyash particle. The addition of flyash particles reduces the density of composite while increasing some of their mechanical properties. The results of wear studies have shown that the resistance to wear increases with increase in percentage of flyash. Corrosion resistance decreases with increase in flyash content. The macrostructural and microstructural characteristics of the MMC were investigated with particular emphasis on the distribution of flyash particles in the matrix. Macrostructural studies have shown near uniform distribution of flyash particles in matrix. Analysis of fractured surface of tensile test specimen is also made which revealed brittle fracture behavior of MMCs. © *2005 Springer Science + Business Media, Inc.*

1. Introduction

The flexibility associated with metal matrix composites (MMC) in tailoring their physical and mechanical properties as required by the end application have made them suitable candidate for a spectrum of applications related to automobile and aeronautical sectors [1, 2].

The emergence of novel processing techniques coupled with the need for lighter materials with high strength and stiffness has catalyzed considerable scientific and technological interest in the development of numerous high performance composite materials as serious competitors to the traditional engineering alloys. The majority of such materials are metallic matrixes reinforced with high strength, high modulus and often brittle second phase in the form of fiber, particulate, whiskers embedded in a ductile metal matrix. The reinforced metal matrix composites offer opportunities for sufficient improvement in efficiency, reliability and mechanical performance over traditional base metals. In particular, the particulate reinforced metal matrix composites are attractive because they exhibit near isotropic properties compared to continuously reinforced counterparts and are easier to process using standard metallurgical methods. Particulate reinforced MMC's provide additional advantage of being machinable and workable. The primary disadvantage of all MMC's however, is that they suffer from low ductility and inadequate fracture toughness compared to their constituent matrix material [3].

Nowadays the main focus is given to Aluminium as matrix material because of its unique combination of good corrosion resistance, low electrical resistance and excellent mechanical properties. Reinforcement material in MMC's may be carbide, nitrides and oxides. For the past few years new particulate composite containing flyash has been developed. Flyash is byproduct of coal combustion, which is used as filler or functional extenders in plastics, paints, resins and additive

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TABLE I Chemical composition of Al alloy used as matrix material in wt%

Si	Fe	Cu	Mn	Mg	Zn	Al
12.2	0.322	0.002	0.621	0.065	0.0215	Bal

to cement. Millions of tons of flyash powder are generated in coal based thermal power plants and only a small portion is being utilized [4]. Flyash is a waste by product and is being used as filler in aluminium matrices [5] and various components such as pistons, engine cover, connecting rod castings have been made out of cast aluminium alloy—flyash composite [6]. Flyash particles are very light materials with a density of 2.1–2.6 g/cm^3 for precipitator flyash and density as low as 0.4–0.6 g/cm^3 for cenosphere particles.

2. Materials

2.1. Matrix material

The matrix material used in the experimental investigation was an Aluminium alloy (Si-12.2%) whose chemical composition (in weight percent) is listed in Table I. This alloy has a composition very close to the Al-Si eutectic. It therefore has a low melting point (577°C). Aluminium and silicon have no solid solubility below the eutectic and the microstructure solidifies as silicon particles in an aluminium matrix. Aluminium-silicon alloy in its unmodified state is extensively used in sand casting and die-casting. The molten metal has high fluidity and solidifies at constant temperature. Aluminium-silicon castings have good corrosion resistance and good weldability. The microstructure can be refined by rapid cooling to increase the strength and ductility.

2.2. Reinforcement material

The reinforcement material used in the investigation was flyash particulates of assorted size with an average particle size of 10 μm. The flyash was collected from Mettur thermal power plant, Tamilnadu, India and micrograph of flyash is shown in Fig. 1. Particle size was estimated by using SEM (scanning electron microscope) and sieve analysis. The spheroidal flyash particles contain both solid spheres (precipitators) and hollow spheres (cenospheres). The major components of flyash as received from the source and used for reinforcement are listed in Table II in weight percentage. The flyash consist mainly Al_2O_3 (30.40 wt%), SiO_2 (58.41 wt%) and Fe_2O_3 (8.44 wt%). The loss on ignition was found to be 1.6 wt% and cenosphere content was found to be 61 wt%. Gravity separation method was used to find the cenosphere content. Cenospheres are hard, hollow, free flowing, microspheres found in flyash. Average particle density was 1.36 g/cm^3. By using froth floatation method carbon content has been determined and was found to be 1.9 wt%. This value was found to be nearer to the loss on ignition value.

TABLE II Composition of flyash used as reinforcement in wt%

Al_2O_3	SiO_2	Fe_2O_3	TiO_2	LOI	Carbon content
30.40%	58.41%	8.44%	2.75%	1.6%	1.9%

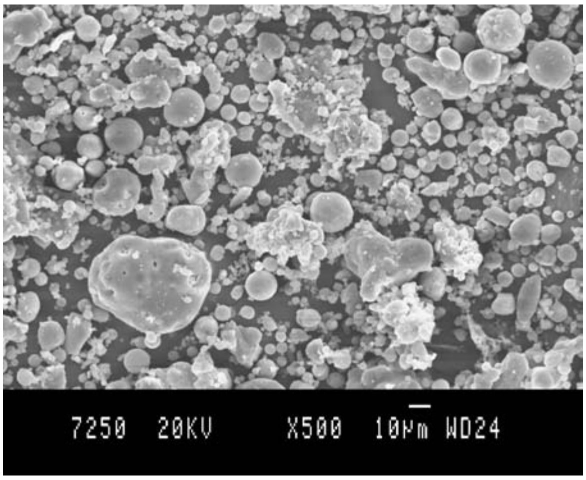

Figure 1 SEM micrograph of flyash used in the study.

3. Experimental procedure

3.1. Processing

The synthesis of the metal matrix composite used in the present study was carried out by using stir casting method. Al-12%Si alloy in the form of ingots were used for the trials. The cleaned metal ingots were melted to the desired super heating temperature of 800°C in graphite crucibles under a cover of flux in order to minimize the oxidation of molten metal. 3-phase electrical resistance furnace with temperature controlling device was used for melting. For each melting 3–4 kgs of alloy was used. The super heated molten metal was degassed at a temperature of 780°C. Flyash particulates, preheated to around 600°C were then added to the molten metal and stirred continuously by using mechanical stirrer at 720°C. The stirring time was maintained between 5–8 min at an impeller speed of 550 rpm. During stirring, Magnesium was added in small quantities to increase the wettability of flyash particles. The dispersion of the preheated flyash particulates was achieved in accordance with the vortex method [7, 8]. The melt with the reinforced particulates were poured into the dried, coated, cylindrical permanent metallic moulds of size 50 mm diameter and 175 mm height. The pouring temperature was maintained at 680°C. The melt was allowed to solidify in the moulds. For the purpose of comparison, the base alloy was cast under similar processing conditions as described.

In this study flyash was added in weight percentage of 5, 10, 12, and 15% in Al (12.2 wt%Si) metallic matrix using conventional casting method. The successful incorporation of flyash particulates in the limits exceeding 10 wt% using this technique can be attributed to the enhanced wettability of flyash particulates as a result of the preheating of flyash particulates to 600°C prior to the addition in the superheated liquid metallic melt and addition of magnesium during stirring of metallic melt and flyash particulates mixture. Preheating of flyash particulates seems to assist in (i) removal of surface impurities (ii) free flow of particulates (iii) desorption of gases and (iv) altering of surface composition owing to the formation of thin oxide layer.

3.2. Density

Density measurements were carried out on the base metal and reinforced samples using Archimedes's principle [9]. The buoyant force on a submerged object is equal to the weight of the fluid displaced. This principle is useful for determining the volume and therefore the density of an irregularly shaped object by measuring its mass in air and its effective mass when submerged in water (density = 1 gram/cc). This effective mass under water will be its actual mass minus the mass of the fluid displaced. The difference between the real and effective mass therefore gives the mass of water displaced and allows the calculation of the volume of the irregularly shaped object. The mass divided by the volume thus determined gives a measure of the average density of the object.

3.3. Hardness and microhardness

Bulk hardness measurements were carried out on the base metal and composite samples by using standard Brinnel hardness test. Brinell hardness measurements were carried out in order to investigate the influence of particulate weight fraction on the matrix hardness. Load applied was 500 kgs and indenter was a steel ball of 10 mm diameter.

Microhardness measurements were carried out in order to investigate the influence of flyash particle on the matrix hardness. Load applied was 50 gms and indenter used was Vickers indenter. Microhardness measurements were made on the particle and in the vicinity of the particle. Round specimens of diameter 20 mm were prepared and polished on different grit of emery paper. An average of 5 readings were taken for both bulk hardness and microhardness measurement

3.4. Macro and microstructural characterization

Macrostructural study was conducted on the as processed and machined composite castings in order to investigate distribution of flyash particles retained in the MMC. Castings were plain turned on lathe to remove 5 mm of material to reveal the particle distribution on macroscopic scale.

Microstructural characterization studies were conducted on unreinforced and reinforced samples. This is accomplished by using scanning electron microscope. The composite samples were metallographically polished prior to examination. Characterization is done in etched conditions. Etching was accomplished using Keller's reagent.

3.5. Mechanical behavior

Mechanical Strength measurements were made by conducting tensile test. The cylindrical specimens were prepared as per ASTM standards E8. To minimize the effect of surface irregularities and surface finish, the gauge sections were ground using 800 grit emery paper in order to remove all circumferential scratches and machine marks.

3.6. Fracture behavior

Fracture surface characterization studies were carried out on tensile fractured reinforced samples in order to provide insight into the fracture mechanisms operative during tensile loading of samples. Fracture surface examinations were accomplished using SEM.

3.7. Sliding wear behavior

Wear has been defined as the displacement of material caused by hard particles or hard proturberances where these hard particles are forced against and moving along a solid surface [10, 11]. Two body sliding wear tests were carried out on prepared composite specimens. A Ducom, Bangalore make computerized pin-on-disc wear test machine was used for these tests. The wear testing was carried out at a constant sliding velocity of 95 m/min with a normal load of 14.7 N. A cylindrical pin of size 5 mm diameter and 40 mm length prepared from composite casting was loaded through a vertical specimen holder against horizontal rotating disc. Before testing, the flat surface of the specimens was abraded by using 2000 grit paper. The rotating disc was made of carbon steel of diameter 50 mm and hardness of 64 HRC. Wear tests were carried out at room temperature without lubrication for 2 h and 20 min. The principal objective of investigation was to study the coefficient of friction and wear.

3.8. Fog corrosion

The oldest and most wildly used Salt Spray [Fog] corrosion testing method [12] was used in the investigation. A fog of NaCl solution was introduced in to a closed chamber where specimens were exposed at specific locations. The concentration of the NaCl solution used was 3.5%. Corrosive fog was created by bubbling compressed air through hot deionized water—Salt solution which was maintained at a temperature of 50°C.

The specimens for fog corrosion test were prepared by cutting specimens of size 10 × 20 × 5 mm from the composite castings. The surface of specimens were abraded by using 600 grit size emery paper and degreased. Before testing, the specimens were weighed to an accuracy of 0.001 gms and exposed to corrosive atmosphere for a period of 240 h (10 days). The specimens were suspended in corrosive chamber at regular intervals exposing the abraded surface to salt solution fog. After corrosion testing, the specimens were immersed in clark's solution for 5 min and gently cleaned with a soft brush to remove adhered particles. After drying thoroughly the specimens were re-weighed to determine the percentage weight loss. One set of specimens were tested and weighed for every 48 h for 10 days and another set of specimens was tested continuously for 10 days.

3.9. Slurry erosive wear

Erosive wear is defined as the loss of material from a solid surface due to relative motion in contact with a fluid that contains solid particles [11].

The experimental arrangement for slurry erosive wear consists of stirrer, which can hold 4 specimens at a time, and a water-cooled pot. All 4 specimens were dipped in slurry of distilled water—silica sand and stirred at a speed of 376 m/min. The slurry was prepared by mixing 80-micron size silica sand with distilled water in the ratio of 1:2 proportions. The pH value of slurry is found to be around 8.5. The slurry wear test was performed at ambient temperature and testing time was 14 h.

The specimens for the slurry erosive wear test were cut from composite ingots and plain turned to a diameter of 7 mm. Before testing, specimens were weighed to an accuracy of 0.001 gms. After testing specimens were dried and re-weighed to determine percentage weight loss.

4. Results and Discussion

4.1. Density measurement

The results of density measurement on the base metal and reinforced materials are shown in Fig. 2. The results reveal that an increase in the percentage of flyash particulates in MMC decreases the material density. Lower density results especially because of presence of particles like Cenospheres, which are hollow spheres with very low density of 0.4–0.6 gm/cm^3. Earlier studies [13] and the results mentioned above show similar density variations.

4.2. Hardness measurements

The results of bulk hardness measurements conducted on the monolithic and reinforced materials are as shown in Fig. 2. The results reveal that an increase in the flyash particulates weight percentage in MMC increases the material hardness. Higher hardness results because of inclusion of flyash particles like cenospheres and presipitators [13].

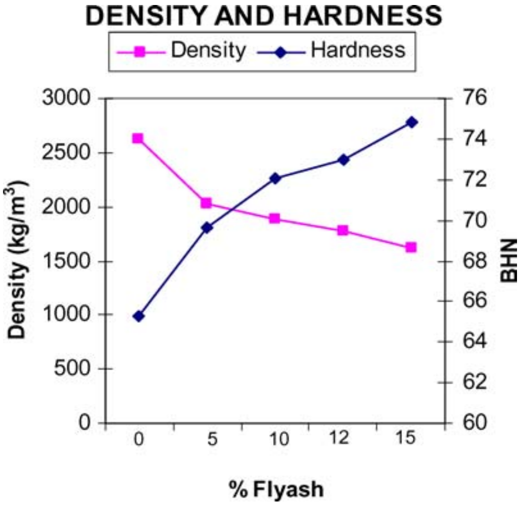

Figure 2 Variation of Density and Hardness with variation in percentage flyash.

TABLE III Microhardness of matrix material around flyash particle

Distance (μ)	Test1 (Hv)	Test2 (Hv)	Test3 (Hv)
0	533	463	489
10	316	379	396
20	234	307	257
30	134	167	138
40	117	248	210

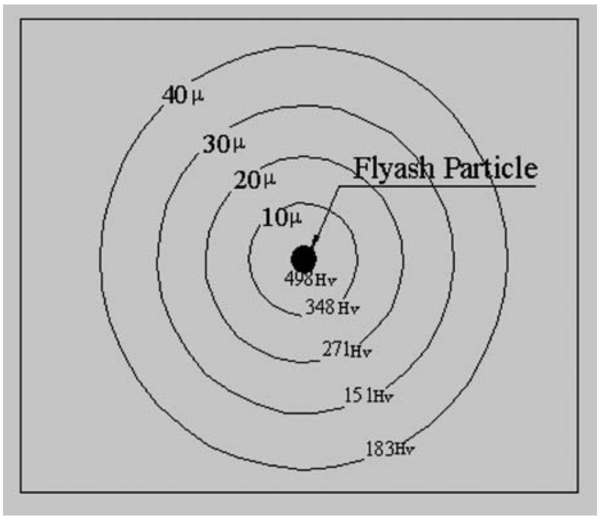

Figure 3 Average microhardness of matrix material around flyash particle.

The results of microhardness measurements conducted on the composite samples containing 12 wt% of flyash particles is shown in Table III. Measurements were made using 50 gms load. The results indicate that hardness vary in the vicinity of flyash particulate depending on distance from interface. But the variation does not show a clear trend. Near the particle-matrix interface the hardness value is higher compared to other regions. Fig. 3 shows average microhardness values at different distances from interface. Lack of clear trend in variation of microhardness values of matrix can be attributed to influence of neighboring particles which are present beneath and sides of the particle under test.

4.3. Macro and microstructural characterization

Macrostructural studies revealed reasonable uniform distribution of flyash particles and slight macrosegregation of particles. The distribution of flyash particles is influenced by the tendency of particles to float due to density differences and interactions with the solidifying metal. It is therefore a strong function of the solidification rate and geometry of castings [7]. Photo macrograph Fig. 4 shows the distribution of flyash particles (10% weight percentage) in permanent mold cast ingot. Higher concentration of flyash particles

Figure 4 Photo-macrograph of Al-10% flyash MMC.

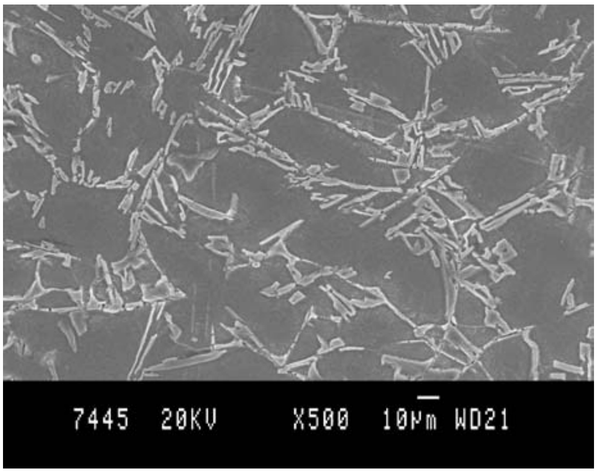

Figure 5 SEM micrograph of unreinforced matrix material.

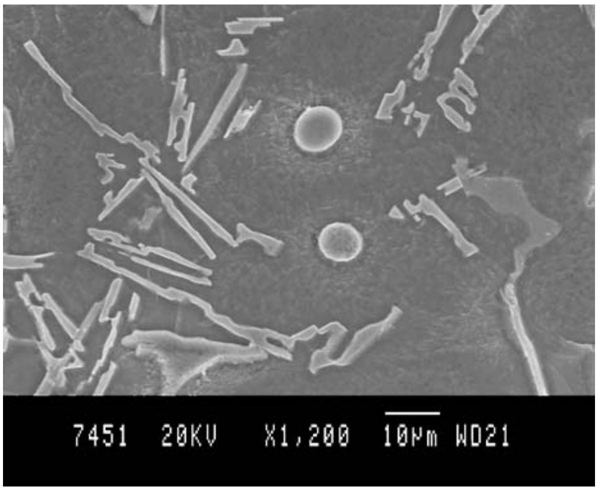

Figure 6 SEM micrograph of Al-15% flyash composite.

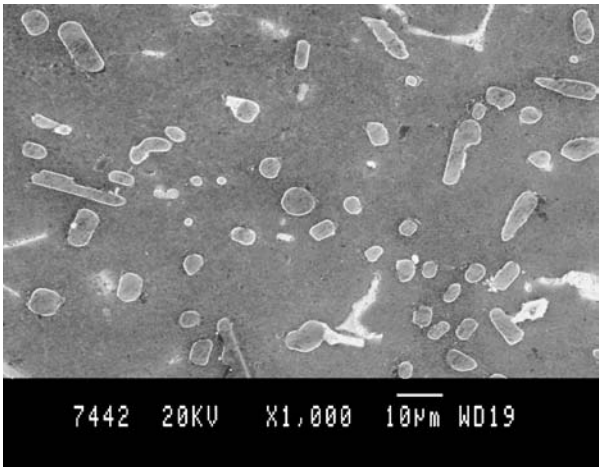

Figure 7 SEM micrograph of Al-15% flyash composite.

was obtained at the top and lower concentration at the bottom of the castings. Central 80% length of castings had near uniform distribution of flyash particles. A more uniform distribution of particles will be obtained by increasing the solidification rate.

Microstructure of matrix material is shown in Fig. 5. Microstructural studies of MMC with 10% flyash shows that there is no void or discontinuities (see Figs 6, and 7) and preferential presence of flyash particles away from silicon particles. At some places there was clustering of flyash particulates. The micrograph in Fig. 8 shows distribution of particulates with low magnification. Fig. 9 shows that there is good interfacial bonding between flyash particles and Al matrix. Good interfacial bonding can be obtained by heating of flyash particulates prior to dispersion and addition of magnesium in small quantities during stirring which improved wettability of flyash particles.

4.4. Mechanical behavior

The ultimate tensile strength and percentage elongation obtained during tensile test are shown in Fig. 10. It is found that the addition of flyash has significant effect on the tensile properties. The addition of flyash particles will increase the ultimate tensile strength of the material but ductility of reinforced samples is inferior compared to base metal. The addition of the flyash particles increases strength mainly by the load transfer from matrix to the reinforcement due to the differences in the elastic constants. There was significant decrease in % elongation, with 15% flyash composite showing lowest elongation of 2%.

The tensile fracture surface of flyash-reinforced samples is shown in Fig. 11. The fractographs taken in SEM revealed absence of dimples which are indicative of ductile fracture. The metal matrix composite specimens behaved as a typical brittle material. These results are consistent with mechanical properties results which show a reduction in elongation (see Fig. 10). As evident from the fractograph 12 there is no breaking or cracking of spheroidal flyash particles and because of destructive testing some particles with weak interface bonding were found dislodged. Fracture has taken place within the aluminium matrix and along matrix-reinforcement interface.

4.5. Fog corrosion

The results of salt solution fog corrosion test are shown in Fig. 13. The resistance to corrosion is good in

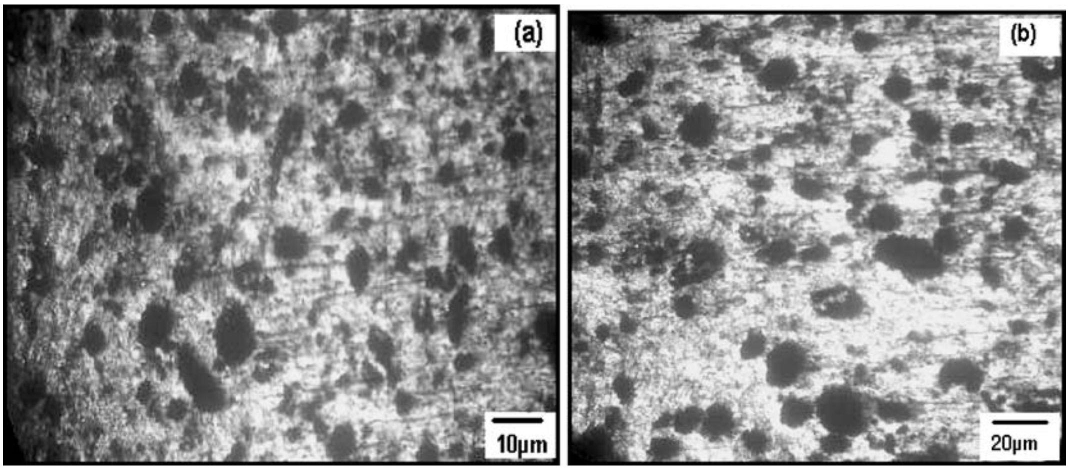

Figure 8 Optical micrograph of (a) 15% flyash composite (b) 10% flyash composite.

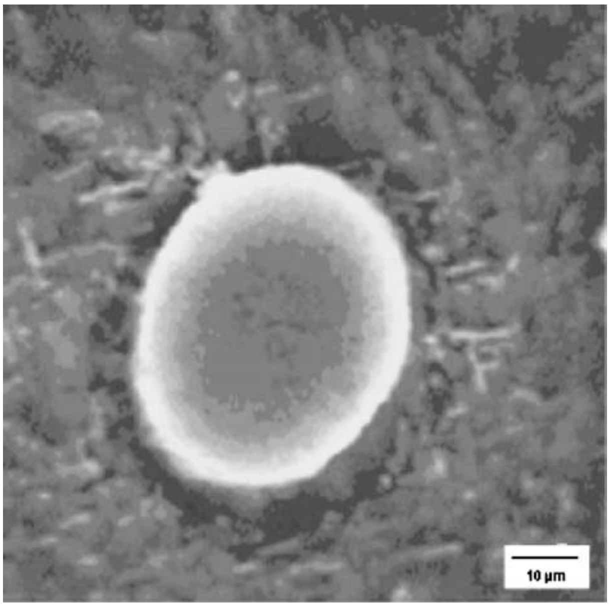

Figure 9 SEM micrograph of Al-flyash interphase.

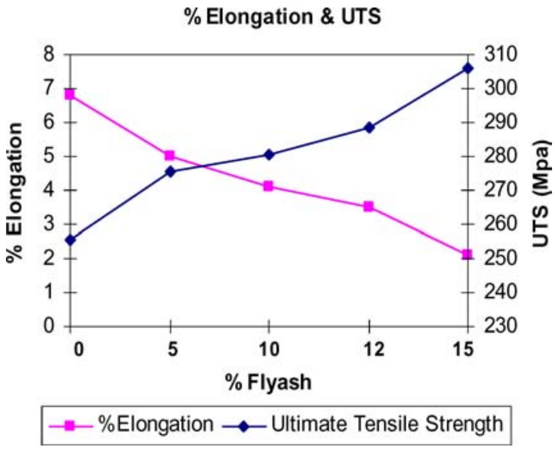

Figure 10 Variation of ductility and UTS with variation in percentage flyash.

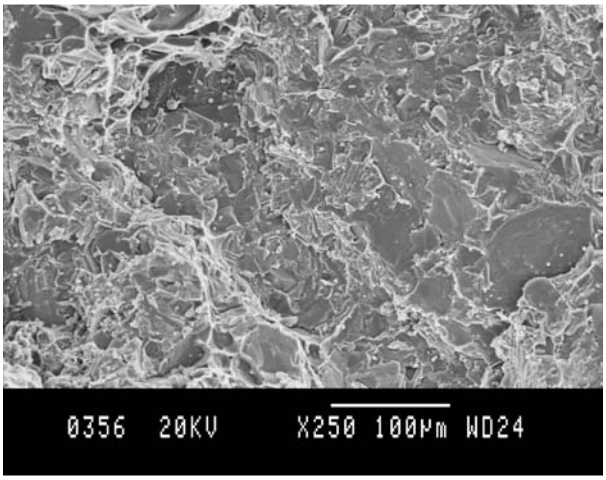

Figure 11 SEM fractograph of Al-10% flyash composite.

unreinforced specimen compared to reinforced composite specimens. Formation of oxide layer is visible within 6 h of commencement of test. The type of corrosion is pitting corrosion. After 24 h it is observed that the formation of pit is more rapid in reinforced samples than unreinforced samples. The presence of flyash particles will act as sites to initiate pits. There will be build up of corroded particle debris in the pits. Pits initiate at flaws within the surface film and at sites where the film is damaged mechanically under conditions in which self repair will not occur. Fig. 14 shows the weight loss of MMCs after 240 h of continuous test. Weight loss was found to be highest in metal matrix composite having 15% flyash.

4.6. Slurry erosive wear

The results of slurry erosive wear are shown in Fig. 15. The results show increase in slurry wear resistance with increase in flyash content. Compared to base metal, composite with 15% flyash showed less weight loss. The presence of flyash particles essentially improved wear resistance in the beginning 8–10 h. But after 8–10 h of testing weight loss was found to be almost nil in all cases. Decrease in weight loss is because of formation of oxide layer on the surface of the specimen which retards wear by acting as a protective layer. This is evident from the Fig. 16 where wear rate has decreased after 8–10 h of testing. After

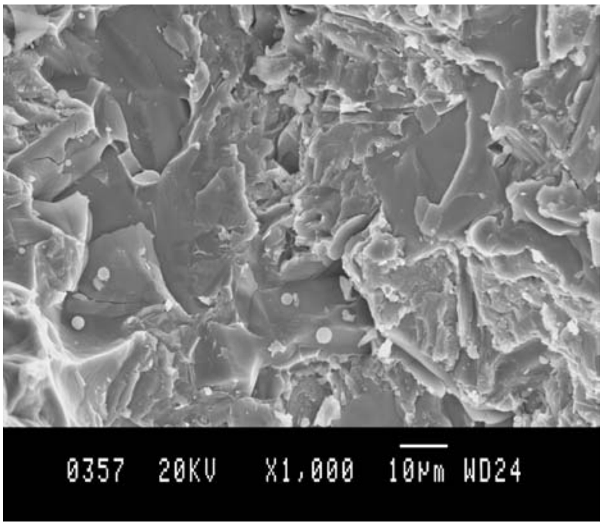

Figure 12 SEM fractograph of Al-10% flyash composite.

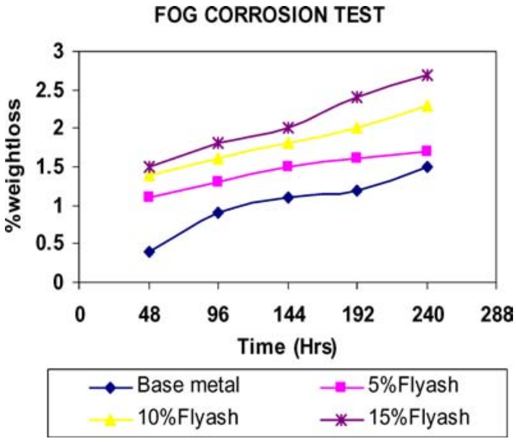

Figure 13 Percentage weight loss in fog corrosion test.

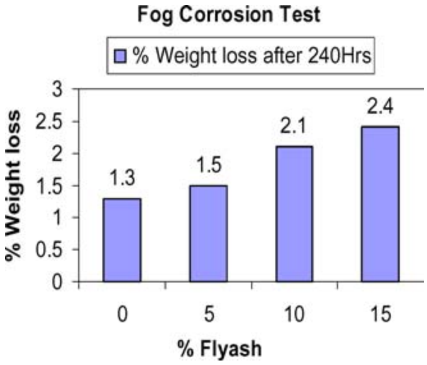

Figure 14 Percentage weight loss in fog corrosion test after continuous 240 h of testing.

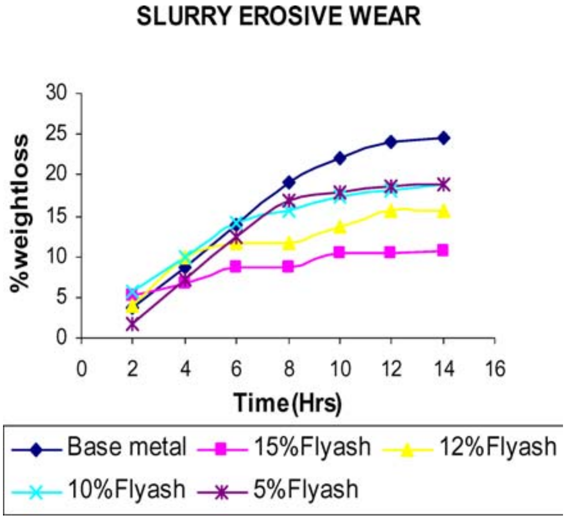

Figure 15 Percentage weight loss during slurry erosive wear.

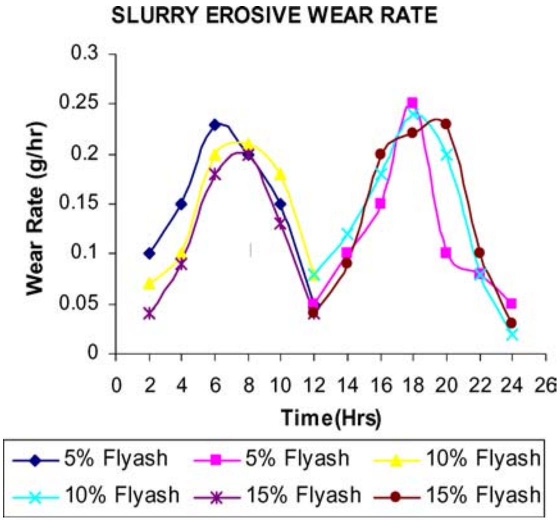

Figure 16 Variation of slurry erosive wear rate with time.

12 h of test, the specimens were reground with 1200 grade emery paper and slurry wear test was repeated with same experimental condition. The results of repeated test showed similar wear behavior as shown in Fig. 16.

4.7. Sliding wear behavior

Fig. 17 shows the results of dry sliding wear behavior of MMCs with different percentages of flyash content. From the graph it is evident that the resistance to wear has increased with increase in flyash content. Incorporation of flyash content significantly reduces wear. This is evident from the amount of wear observed for base metal and composite with 15% flyash content. This is because of the presence of hard flyash particles which will increase the overall bulk hardness of the material. The inclusion of flyash content will change the wear mode from mild to severe. This is evident from the comparison of wear behavior graph of base metal with that of composites. There is smooth, linear variation in wear for base metal compared to roughness observed in wear behavior curves of composite with flyash particles. The change in the coefficient of friction of aluminium matrix composites with time is shown in Fig. 18. It is observed that the coefficient of friction for composite with 15% flyash is lower. In composite with 15% of flyash, a steady state of nearly constant coefficient of friction was observed. In all other cases there is variation in coefficient of friction owing to the effect of material microstructural heterogeneity.

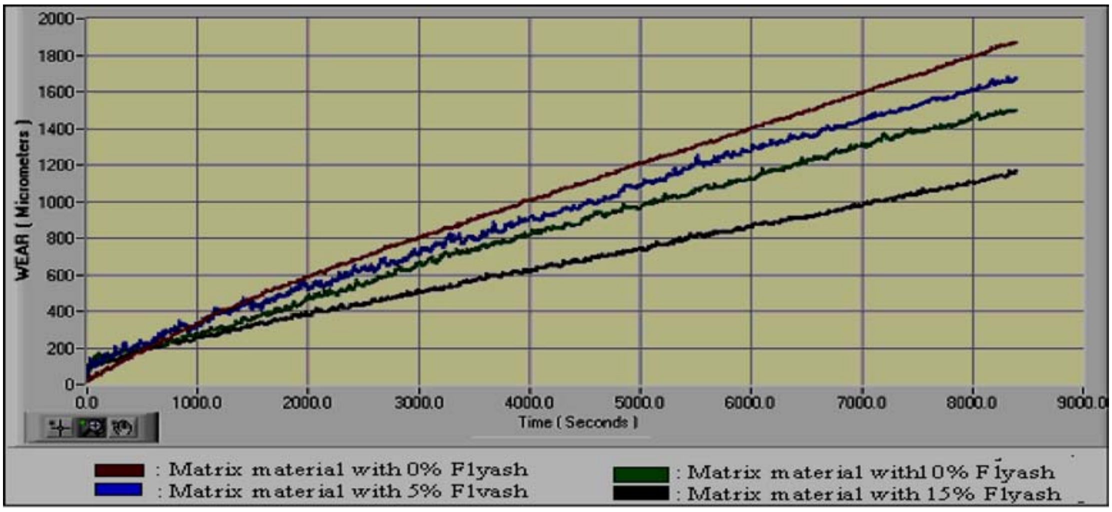

Figure 17 Sliding wear behavior of MMC with different percentages of flyash.

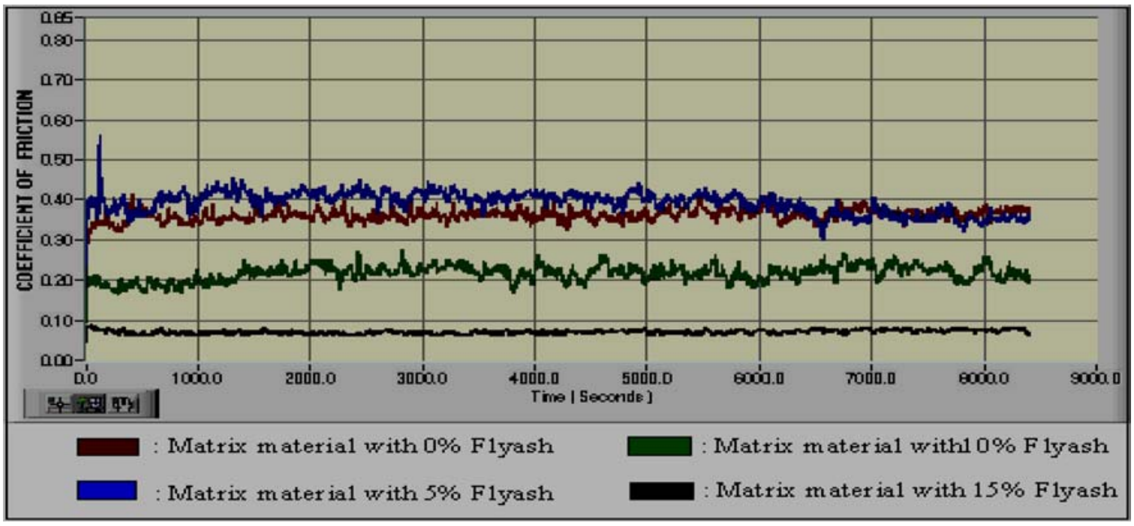

Figure 18 Variation of coefficient of friction of MMC with different percentages of flyash.

5. Conclusions

(1) Metal matrix composite up to 15% flyash was synthesized successfully by using vortex method.

(2) Macro and microstructure revealed near uniform distribution of flyash particles in the center portion of the casting. But there is slight agglomeration of flyash particles on macroscopic scale. The microstructure also revealed good interfacial bond between matrix and flyash particles.

(3) The density of MMC has decreased with increase in flyash content.

(4) The hardness of MMC increases with increase in flyash content and the microhardness was high near the vicinity of flyash particle.

(5) The Ultimate tensile strength increased with increase in flyash content. Where as ductility has decreased with increase in flyash content.

(6) The fractographs have shown that the fracture is of brittle in nature.

(7) The sliding wear resistance of MMC has increased with increase in flyash content. Similar trend is observed in slurry erosive wear.

(8) The corrosion resistance property decreases with increase in flyash content because of formation of pit around flyash particle. The area around flyash particle will be potential pit-initiating site.

(9) Incorporation of flyash particles in aluminium matrix can lead to the production of low cost aluminium composites with improved hardness, strength and wear resistance. These composites can find applications in automotive components like pistons, cylinder liners and connecting rods. These composites can also find applications where light weight materials are required with good stiffness and strength.

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