Slurry Erosive Wear Behavior of Hot Extruded Al6061-Si₃N₄ Composite

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Abstract. Silicon nitride possess excellent hot hardness and wear resistance coupled with very good corrosion resistance. Hence, in recent years, silicon nitride has been a serious contender as a reinforcement to develop light weight metal matrix composites for several technological applications. Al6061 is most popular matrix alloy as it possess excellent formability and in particular the quality of extrudates of Al6061 is quite high and are the most preferred in space and naval applications especially for support structures and torpedoes blades respectively. Improved corrosion and slurry erosion resistance on use of silicon nitride in nickel and aluminum alloy matrices have been reported by several researchers.

In the light of the above, this paper focuses on development of Al6061- 6wt% Si₃N₄ by stir casting the most economical and popular route followed by hot extrusion. Hot extrusion was carried out using 200T hydraulic press at extrusion ratio of 1:10 at a temperature of 550°C. Slurry erosion tests were carried out using 3.5% NaCl solution containing silica sand particles of size 312 µm at different rotational speeds varying between 300 rpm and 1200 rpm. The sand concentration was varied from 10 g/l to 40 g/l. Under identical test conditions, hot extruded Al6061- 6wt% Si₃N₄ composite do possess better slurry erosion resistance when compared with matrix alloy. The mechanism involved in the material removal process during slurry erosion process will also be discussed.

Introduction

Due to improved physical, mechanical and tribological properties coupled with ease of formability and light weight, aluminium alloy based composites are finding wide applications in the field of aerospace, automobile, defense, marine etc. Among them, Al6061 alloy based composites are more popular owing to its reasonable strength and ease of secondary processing such as extrusion and forging [1,2].

As regards the reinforcement in the matrix alloy, ceramics such as SiC, Al₂O₃, Si₃N₄, TiO₂, ZrO₂, have been quite popular. Many researchers have reported that the addition of Si₃N₄ to aluminium matrix do enhance its mechanical properties [3-6]. However, with the mandatory requirement of secondary processing of light weight MMCs, especially for automotive parts, there is a dire need to optimize the selection of the ceramic reinforcement. Si₃N₄ which possesses excellent hot hardness and tribological properties coupled with good strength at elevated temperature is currently the popular choice as a reinforcement in MMCs which needs secondary processing. Secondary processing in particular, hot extrusion of MMCs lead to several problems such as debonding due to large thermal mismatch between the reinforcement and the matrix alloy, inferior surface finish, interfacial reactions between the reinforcement and the matrix alloy and

lowering of the tool life of the die. Ramesh et.al have reported the elimination of interfacial reaction between SiC and aluminium alloy during hot extrusion by use of Ni-P coated SiC [7]. XIU Zi-Yang et.al have reported that extrusion has resulted in better homogeneity in the distribution of Si_3N_4 particles in composite leading to decrease in island clusters which are characteristic features of as cast composites [8]. Smagorinski et.al have reported that composites formed by extrusion do exhibit good combination of mechanical and physical properties such as CTE, elastic modulus and limit of relaxation [9]. Ramesh et.al have reported that hot extruded composites possess higher microhardness and lower adhesive wear rates under all studied loads and sliding velocities when compared with cast composites [10].

Improvement in thermal conductivity in $\text{Si}_3\text{N}_4/\text{Al}$ composites after annealing treatment has been reported by Yang Wen-Shu et.al [11]. Ramesh et.al have reported that hot extruded composites exhibit higher hardness and slurry erosive wear resistance when compared with as cast alloy and its composites [12]. Trzaska has reported that both Ni-P and composite of Ni-P/ Si_3N_4 layers have resulted in excellent corrosion resistance of aluminium substrate [13]. Ramesh et.al have reported the beneficial effect of Si_3N_4 as regards the drastic improvement of slurry erosion resistance of light weight cast composites [14]. Krishnaveni et.al have observed improved corrosion resistance for Ni-B- Si_3N_4 composite coatings compared to its plain counterpart [15].

The above discussed research works highlights the importance of investigating aluminium alloy-silicon nitride composites. Meager information is available as regards the slurry erosive behavior of light weight hot extruded aluminum alloy- Si_3N_4 composites. In the light of the above, this work focuses on use of Ni-P coated Si_3N_4 particulates as a reinforcement to develop high quality light weight hot extruded MMCs and characterizing its slurry erosive wear behavior in marine environment.

Experimental Details

Materials. Al6061 alloy was procured from Fenfee Metallurgicals, Bangalore, in the form of ingots and its chemical composition is reported in Table 1. The maximum constituent alloying elements present are Mg and Si. Silicon nitride of particle size ranging from 2 to 10 μm has been chosen as reinforcement (Supplied by ACE Rasayan, Bangalore). Prior to dispersing of Si_3N_4 in molten aluminum alloy, it has been surface treated with electroless nickel coating using a chemical bath whose constituent is reported in Table 2.

Si_3N_4 was subjected to immersion in toluene and propane bath followed by acetone wash. Further the surfaces of silicon nitride powder were activated using stannous chloride and hydrochloric acid solution which was followed by palladium sensitization of the surfaces. Completely dried Palladium activated Si_3N_4 powders were allowed to activate with the electroless nickel bath to achieve Ni-P coating. The temperature and pH of the bath was maintained at 85°C and 4.5 respectively. A duration of 30 minutes was adopted for coating. The coated Si_3N_4 powder was thoroughly cleaned with distilled water followed by heating in a oven to ensure its complete drying.

Table 1. Chemical Composition of Procured Al6061 Alloy

Si	Fe	Cu	Mn	Ni	Pb	Zn	Ti	Sn	Mg	Cr	Al
0.43	0.43	0.24	0.139	<0.05	0.024	0.006	0.022	0.001	0.802	0.184	Balance

Table 2. Bath composition used for electroless nickel coating

Sl. No.	Bath constituents	Quantity(g/l)
1	Nickel Chloride, hexahydrate $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	30
2	Sodium Succinate, hexahydrate $\text{Na}_2\text{C}_4\text{H}_6\text{O}_4 \cdot 6\text{H}_2\text{O}$	10
3	Hypophosphite, monohydrate $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$	20
4	Glycin $\text{H}_2\text{NCH}_2\text{COOH}$	10
5	Lead Nitrate $\text{Pb}(\text{NO}_3)_2$	0.02

Casting. Aluminum 6061 alloy ingots, 3Kg in quantity was melted using 6KW electrical resistance furnace. The melt was degassed by using commercially available chlorine based tablet (Hexachloroethane). The composite development set up is shown in Fig.1. The molten metal was agitated by use of ceramic coated mechanical stirrer rotating at a speed of 300rpm to create a vortex. Preheated Ni-P coated 6wt% Si_3N_4 (600°C , 1hr) was added slowly in to the vortex. The stirring duration was 10 min. The composite melt was then poured in to preheated metallic cylindrical moulds of diameter 80mm and height 110mm at a temperature of 700°C .

Fig.1 Electrical Resistance Furnace with Stirrer

Fig.2 Hydraulic Press

Hot Extrusion. The cast matrix alloy and the developed composites were machined to cylindrical billets of diameter 70 mm and height 100 mm. Graphite paste was applied to outside surfaces of the billets to minimize sticking of aluminum to the containers and the die. These billets were then heated in a furnace at a temperature of 550°C before inserting into a container and the extrusion die system which were preheated to 450°C using electrical heaters. The hydraulic press of capacity 200T used for extrusion is shown in Fig. 2. To minimize the friction at the interface of the die and billet, commercially available copper based lubricant was applied to the inner surfaces of the die and container.

The cast matrix alloy and its composites were hot extruded at a temperature of 550°C with an extrusion ratio of 1:10 using a 200T hydraulic press. The diameter of the extruded rod was 30 mm.

Slurry Erosion Test. Slurry erosion wear tests were conducted on polished and thoroughly cleaned rectangular samples of dimensions $8 \times 8 \times 40\text{mm}$. The samples were weighed before and after wear tests using an electronic balance of accuracy 0.1 mg. The details of this test setup are reported in our earlier works [2].

The slurry used was a mixture of silica sand and 3.5% of sodium chloride (NaCl) in distilled water. The sand concentration was varied from 10 to 40% in steps of 10% with its particle size maintained at $312\text{ }\mu\text{m}$. The rotational speed was varied from 300 to 1200 rpm in steps of 300 rpm. The total duration was 5 hrs for all the tests conducted. The worn surfaces were subjected to SEM studies to understand the mechanism of material removal.

Results and Discussion

Microstructural Studies. Alignment of the reinforced Si_3N_4 particles along the extrusion direction is observed as evidenced in Fig. 3(a). However, the extent of uniformity of distribution of Si_3N_4 particles as observed at high magnification is very good. Further, there is a drastic reduction in the particle size after extrusion as shown in Fig. 3(b). This can be attributed mainly to the high state of compressive stresses during the extrusion process which leads to fragmentation of the Si_3N_4 particles. The original particle size used was 2 to $10\text{ }\mu\text{m}$, while on extrusion majority of the Si_3N_4

particles have disintegrated in to particle size less than 2 microns. Smaller the particle size with high degree of homogeneity in the distribution of the reinforcement within the metallic matrix alloy leads to improved mechanical properties coupled with excellent wear and corrosion resistance.

(a) Optical Micrograph

(b) SEM

Fig.3 Microstructure of Hot Extruded Al6061-6wt% Si_3N_4

Slurry erosive wear results

Effect of slurry concentration. The effect of slurry concentration on erosive wear of hot extruded Al6061 and Al6061-6wt% Si_3N_4 composite are shown in Fig.4.

It is observed that mass loss increases with increase in slurry concentration in both hot extruded Al6061 alloy and its composites. An increase of three folds in the mass loss of the matrix alloy when compared with the composite is being observed at 40% silica sand concentration. This superior slurry erosion wear resistance can be attributed to the excellent corrosion resistance of silicon nitride coupled with its high hardness. Further, it is reported that silicon nitride reacts with moisture and form thin siliceous film which is highly protective in reducing the mass loss of the composites [14]. Fig.5 shows SEM photographs of worn erosive wear surfaces of hot extruded Al6061-6wt% Si_3N_4 composites for lowest and highest slurry concentration studied. It is observed that the extent of surface degradation of the composite is quite extensive at high slurry concentration. Severe damage to the matrix in the form of deep cracks and fragmentation of the reinforced silicon nitride have been observed at high slurry concentration which are indicative of larger material loss.

Fig.4 Variation of weight loss of hot extruded Al6061 and Al6061-6wt% Si₃N₄ composites at different slurry concentration

Effect of rotational speed. The effect of rotational speed on erosive wear of hot extruded Al6061 and Al6061-6wt% Si₃N₄ composite are shown in Fig.6.

It is observed that mass loss increases with increase in rotational speed for both hot extruded Al6061 alloy and its composites. An increase of 6.2% in the mass loss of the matrix alloy when compared with the composite is being observed at 1200 rpm. The increase in mass loss with increase in rotational speed can be attributed to the increased extent of plastic deformation due to increase in impact velocity. The extent of surface degradation of the composites at high slurry rotational speed of 1200 rpm is quite appreciable when compared at lower rotational speed of 300 rpm as shown in Fig. 7. Heavy grooving and fragmentation of the surfaces are observed at high speed, while extensive micro pits are the characteristics feature at low rotational speed leading to low material loss.

(a) 10% Slurry Concentration

(b) 40% Slurry Concentration

Fig.5 SEM photographs of worn erosive wear of hot extruded Al6061- 6wt% Si₃N₄ composites at different slurry concentration

Fig.6 Variation of weight loss of hot extruded Al6061 and 6wt% Si₃N₄ composites at different slurry rotational speed

(a) 300 rpm for 30% Slurry Concentration

(b) 1200 rpm for 30% Slurry Concentration

Fig.7. SEM photographs of worn erosive wear of hot extruded Al6061- 6wt% Si₃N₄ composites at different rotational speed

Conclusion

Hot extruded Al6061-6wt% Si₃N₄ composites exhibit excellent slurry erosion wear resistance in 3.5% NaCl sand slurry.

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