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Synthesis and study of tribological properties of WC particulates reinforced Al Nanocomposites.

[1] Amarnath.G, [2] K.V.Sharma, [3] H.N.Girish, [4] Arulmani.L

[1][2][3][4] Department of Mechanical Engineering

[1][4] RR Institute of Technology, VTU, Bangalore -560090,India.

[2] Visvesvaraya College of Engineering, Bangalore University, Bangalore-560001, India

[3] Government College of Engineering Ramanagaram, VTU, Ramanagaram, India

Abstract: -- The tribological property of Al metal matrix composites, reinforced with WC Nano- particles is presented. Sliding tests were performed on a pin-on-disk apparatus under different contact loads. It was found that the reinforced Nano-WC particles could effectively reduce the frictional coefficient and wear rate, especially under higher normal loading conditions. In order to further understand the wear mechanisms, the worn surfaces were examined under the scanning electron microscope. A positive rolling effect of the nanoparticles between the material pairs was proposed which contributes to the remarkable improvement of the load carrying capacity of metal matrix nanocomposites.

Keywords: Metal matrix nanocomposite, tribological property, positive rolling effect, frictional coefficient, wear rate.

1. INTRODUCTION

Over the last three decades, many different types of composites have come into use. They consist of plastics, ceramics, and metals reinforced with fibers, laminates, whiskers, or particulates. Of particular interest are metal matrix reinforced composites; with one of the most popular families being represented by Al reinforced metal matrix composites [1]. The demand for improved mechanical performance and weight savings has focussed attention on a number of new materials, including Al metal matrix composites. These materials are formed by the addition of second phase, generally a ceramic material, to an Al matrix [2].

The reinforcement of Al alloys with either ceramic fibers or particulates is being investigated as a means of improving their stiffness and strength [3]. It is well known that apart from the changes made in the mechanical properties, the addition of ceramic phases in Al alloys in the form of fibers or particles may also alter their corrosion behavior. The reinforcement particles themselves were commercially pure ceramic materials that were insulators. However, in MMC, an interface forms between the reinforcement particles and the Al matrix during manufacturing [4-6]. Synthesis of monolithic and reinforced Al composites containing different amounts of WC particulates composites with varying amounts of WC was carried out using a disintegrated melt deposition technique. The reinforcement particles which are smaller than the matrix particles situate among the matrix particles, filling pores. This causes an increase in the density of the composites compared with the pure aluminum. However, the increase in agglomeration of the particles with increasing the nano-sized WC particulates leads to the increase of the density. It would be worth mentioning that nanoparticles are prone to agglomerate and pores retained in the agglomerated zones, which causes to decrease the density. On similar trend can be observed in micro hardness values. Agglomeration of sharp edged WC particulates in the matrix can be easily observed. Large dimples can be seen in the Al matrix due to the ductility of the matrix material and the relatively large powder particle size.

It has been demonstrated by previous studies on particulate reinforced materials exhibiting some degree of ductility that void nucleation and the final ductility may be sensitive to both the size and distribution of the particulates. In particular, it as been suggested that the local volume fraction as opposed to the average volume fraction may be an important parameter in determining the fracture of materials containing dispersions of particulates at high volume percentages.

Nano particulate reinforced metal matrix composites (MMCs) have been studied widely in recent years, essentially due to their promising advanced properties. Specific attention has been focused to aluminum matrices which are widely used in MMCs [7]. The advantages of aluminum and its alloys used as the composite's matrix among others are high specific strength and stiffness, good damping capacities, dimensional stability [8].The mechanical and tribological behavior of the MMC's has been studied extensively. Some information concerning to the wear behavior of Mg-based MMCs reveals that tribological properties of Mg alloys can be improved by the addition of hard ceramic fiber or particulate reinforcement [9].

2. EXPERIMENTAL

2.1 Composite preparation furnace

Three-phase resistance type 12 KW capacity furnace is used. The temperature range of the furnace is 1200°C with a control accuracy of ± 10° C fitted with seven segmented light emitting diode read out and partially integrated differential digital temperature controller. The shooting capacity of the furnace is 500°C per hour. It is fitted with an alumina crucible at its center and it can be tilted by 90 degrees on its horizontal axis enabling pouring of the melt.

2 .2 Preheating of reinforcement

Muffle furnace was used to preheat the particulate to a temperature of 500°C. It was maintained at that temperature till it was introduced into the Al alloy melt. The preheating of the reinforcement is necessary in order to reduce the temperature gradient and to improve wetting between the molten metal and the particulate reinforcement.

2.3 Melting of the matrix alloy

The melting range of Al alloy is 650 - 700°C. A known quantity of the Al alloy ingots were pickled in 10% NaOH solution at room temperature for ten minutes. Pickling was done to remove the surface impurities. The smut formed was removed by immersing the ingots for one minute in a mixture of 1 part Nitric acid and 1 part water followed by washing in methanol. The cleaned ingots after drying in air were loaded into the alumina crucible of the furnace for melting. The melt was super heated to a temperature of 540°C and maintained at that temperature. A Chromel-alumel thermocouple was used to record temperatures. The molten metal was then degassed using purified nitrogen gas. Purification process with commercially pure nitrogen was carried out by passing the gas through an assembly of chemicals arranged in a row (concentrated sulphuric acid and anhydrous calcium chloride, etc.) at the rate of 1000 cc/ minute for about 8 minutes.

2.4Mixing and stirring

Alumina coated stainless steel impeller was used to stir the molten metal to create a vortex. The impeller was of centrifugal type with three blades welded at 45° inclination and 120° apart. The stirrer was rotated at a speed of 500 rpm and a vortex was created in the melt. The depth of immersion of the impeller was approximately one third the height of the molten metal from the bottom of the crucible. The pre-heated reinforcing particles were introduced into the vortex at the rate of 120 gm/min.

Stirring was continued until interface interactions between the particles and the matrix promoted wetting. The melt was degassed using pure nitrogen for about 3-4 minutes and after reheating to super heated temperature (700°C), it was poured into the pre heated lower half die of the hydraulic press. The top die was brought down by applying a pressure of 100 kg/cm2 to solidify the composite. Both the lower and the upper half dies were preheated to 280°C, before the melt was poured into it. The applied pressure enables uniform distribution of the particulate in the developed composite.

2.5 Casting of composite melt under pressure

The distribution of particles in Al melt was uniform and without any noticeable agglomeration when the composite melt was cast in permanent dies after applying an optimum pressure in a hydraulic press. The castings were allowed to remain at room temperature for some time before subjecting to any heat treatment. A commercially available Al 6061 was considered as matrix material in this study. WC nanoparticulate was selected as reinforcements. The average diameter of nanoWC particle of 300 nm was used as reinforcement. Nano MMCs were fabricated in a special reaction chamber under a vacuum atmosphere. Melting of Al alloy reaching the temperature between 650 and 700 °C, the mixture of WC powder of wt.% of 5, 10 and 15%, was then introduced into the melt by carrier Argon gas. The Al melt was agitated by the propeller, and the WC was introduced into the melt. The task was carried out for an appropriate length of time to ensure the complete mixing of WC in the melt then pour into the die.

2.6 Wear test

The wear test was performed on a Duecom pin-on-disc apparatus according to ASTM standard. The specimen pins were rotated against a polished steel disc with the initial surface roughness of approximately 220 nm. The tests were conducted for 60 min each in a wide range of wear conditions, i.e., the normal load in a range from 10 to 40N and the sliding velocity from 1 to 3 m/s. During the test, the temperature of the disc was monitored by an Iron-constantan thermocouple contacted to the edge of the disc. The frictional coefficient was recorded and calculated by a ratio between the tangential force and normal load. The mass loss of the specimen was measured in order to calculate the specific wear rate by the following equation.

$$w_s = \frac{\Delta m}{\rho F_n L} \quad mm^3 Nm^{-1} \quad \text{------ (1)}$$

in which FN is the normal load applied on the specimen during sliding, □m is the specimen's mass loss, □ is the density of the specimen, and L is the total sliding distance. All the test results were summarized in Table 2. After wear tests, worn surfaces of specimens were examined by a scanning electron microscope (SEM).

3. RESULTS AND DISCUSSION

The worn surface of matrix Al is given in Fig. 1. Grooves paralleled to the sliding direction are clearly observed which suggest the wear process governed by abrasive wear mechanism. In this case, aluminium matrix material generally obtains a high wear rate depending on the original roughness of harder counterpart and the contact pressure. In a magnified view, viscous flow of materials is observed in a micron scale due to the high flash temperature occurring in real contact area. Owing to the low load carrying capacity, the softened Al was rapidly removed by the hard asperities of the metallic counterpart surface.

Figs2,3 and 4 present the worn surfaces of three composites without and with nano-WC, respectively. It is clear that the surfaces of both composites were quite smooth resulting from adhesive wear. In a magnified view, local matrix micro-cracks may occur, which were probably caused by the ''fatigue wear'' of adhesive contact . A metal layer may transfer onto the metallic counterpart surface during running-in stage, which then results in this adhesive wear mechanism. For the nanocomposite, slight nano-grooves were observed which are parallel to the sliding direction. The worn surface of the nanocomposite, because of the reduction of friction and contact temperature, the particles could effectively bear the load and were then gradually removed within a normal process. The particle thinning, particle fracture, and finally particle peeling shows the patterns of the particle fracture of nanocomposite with good interfacial bonding even under high pressure. In order to understand the role of nanoparticles for improving the wear performance of MMC composites.

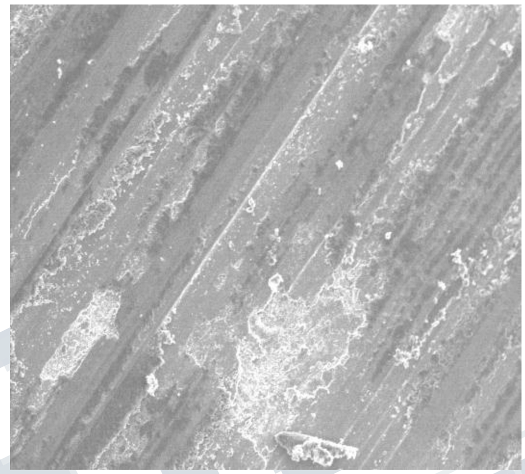

Fig. 1. SEM micrographs of the worn surfaces of matrix metal (Al) measured at 1MPa and 1 m/s

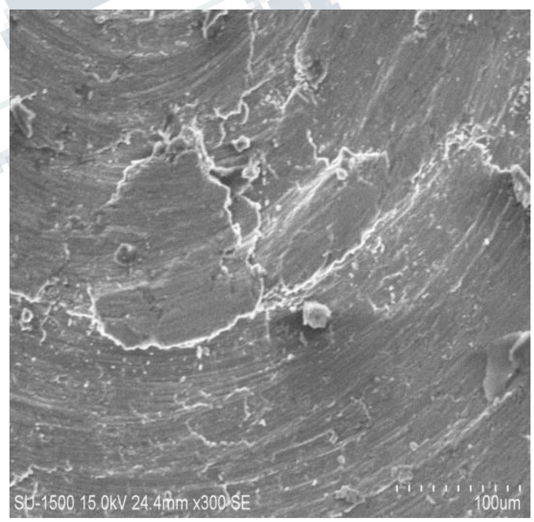

Fig. 2. SEM micrographs of the worn surfaces of Al/5% of WC MMCs measured at 1MPa and 1 m/s

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It can be seen that the particle surface is relatively smooth, but apparently tilted to the worn surface. Therefore, during friction process, the exposed particle underwent most of load and was impacted by the asperities of counterpart. In comparison with that of Al- based composite, the interfacial damage between particle and matrix did not occur due to the better interfacial bonding of Al matrix and WC nano particle.

The selected experimental design parameters are as given in Table.1which shows four factors and four levels used in the experiment. The orthogonal array of L16 type was used and is represented in Table 2. This design requires sixteen experiments with four parameters at each of these four levels. The interactions were neglected. The 5th column was assigned as error (E), and was considered randomly.

Table1: Selected parameters and their assigned levels

Symbol	Factors	Unit	Levels			
			1	2	3	4
A	WC	Wt. %	0	5	10	15
B	Load	N	25	50	75	100
C	Speed	m/s	0.4	0.8	1.2	1.6
D	Sliding distance	M	1000	2000	3000	4000

Table2 :Experimental data and sample statistics

Ex. No.	Main factors					Observed response (Wear rate x10^{-5} gm/m)			Average wear rate	Standard Deviation	S/N ratio (db)
	A	B	C	D	E	1	2	3			
1	1	1	1	1	1	1.6399	1.5625	1.7173	1.6399	0.07743	-26.5184
2	1	2	2	2	2	3.2381	3.3486	3.6879	3.4249	0.234394	-23.294
3	1	3	3	3	3	3.9837	4.6057	6.7929	5.1274	1.475493	-10.8194
4	1	4	4	4	4	9.4247	6.9869	4.8876	7.0997	2.270628	-9.90198
5	2	1	2	3	4	1.5129	1.1631	1.0788	1.2516	0.230184	-14.7081
6	2	2	1	4	3	2.8675	2.9406	3.3938	3.0673	0.285143	-20.634
7	2	3	4	1	2	3.1659	6.0795	4.1873	4.4776	1.478347	-9.62538
8	2	4	3	2	1	8.3024	5.2452	7.5881	7.0452	1.599258	-12.8796
9	3	1	3	4	2	1.1362	0.8021	1.1640	1.0341	0.2014	-14.2104
10	3	2	4	3	1	2.5543	2.8161	2.7335	2.7013	0.13387	-26.098
11	3	3	1	2	4	4.8224	4.8850	4.8972	4.8682	0.040146	-41.6746
12	3	4	2	1	3	6.2518	5.0851	7.1131	6.1500	1.01781	-15.6242
13	4	1	4	2	3	0.1992	0.2514	0.2227	0.2244	0.026143	-18.6755
14	4	2	3	1	4	1.4189	1.3624	1.4294	1.4036	0.036043	-31.8085
15	4	3	2	4	1	3.0667	2.8666	3.4356	3.1230	0.288632	-20.6846
16	4	4	1	3	2	5.6106	4.1768	4.5979	4.7951	0.736942	-16.2674

The S/N ratios were computed for wear rate in each of the 16 trial conditions and their values are as given in Table2. In order to estimate the effect of factor A (wt. % WC) on the average value of response variable, three observed responses were summed together at level 1 of factor A then the sum was divided by 4 to obtain the average response at that level of factor A. The average responses at level 2, 3 and 4 were obtained in the similar manner. The estimated individual factors effect on average wear rate of AL6061/WC composites is shown and presented graphically in Fig.5.The estimated standard deviation of individual factors effects on wear rate of AL6061 /WC composites from Table 2 are plotted in Fig.6. Both factor A and B again affect the wear rate variability. In order to minimize variability: the WC wt. % -level 4 (15 wt. %) and normal load – level 1 (25 N) should be used. In this work, the smaller wear rate is the indication of better performance. Therefore, the smaller-is-better for the wear rate was selected for obtaining optimum performance characteristics. The following S/N ratios for the larger-is-better case could be calculated with following equation:

$$\frac{S}{N} = -10\, X log\left(\frac{\bar{X}^2}{\sigma^2}\right)$$

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Table 3 Summary of Taguchi analyses of factors for optimized levels

Factors	Average response	Standard deviation	S/N ratio
A	A4	A4	A2
B	B1	B1	B4
C	C2	C1	C4
D	D3	D2	D3

In order to maximize the S/N ratio, the following assignments were done: factor A (wt. % WC) - level 2 (5 wt. %), factor B (load)- level 4(100N), factor C (speed) - level 4 (1.6 m/s), factor D (sliding distance) - level 3 (3000m).

In Fig. 5, Fig.6 and Fig. 7 key factors which are optimizing response are plotted and some significant levels are shown. The objective is to minimize the response average, reduce standard deviation and maximize the S/N ratio.

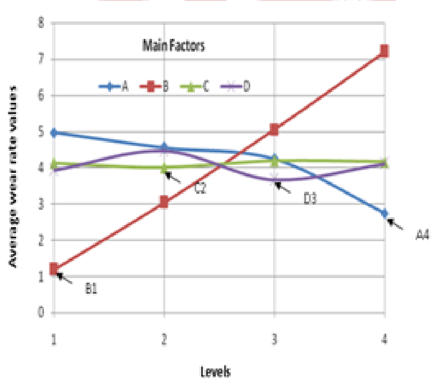

Fig.5 Estimated individual factors on average wear rate of 6061/ WC composites

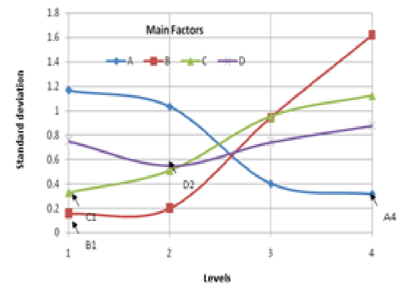

Fig.6 Estimated individual factors effect on standard deviation of Al6061/WC composites

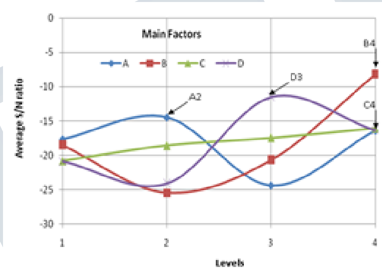

Fig.7 Estimated individual factors effect on average S/N ratio Al6061/WC composites

CONCLUSIONS

Table3 suggests that the optimum condition for the minimum wear rate is the combination of A4B1C2D3 levels of the respective control factors. This implies that in order to reduce the wear rate, the sliding velocity, load and sliding distance should be lowered, while increasing the reinforcement ratio. It is evident from Fig. 1 that applied load (factor B) has the greatest increasing effect on the wear rate. It has been observed that applied load is the most dominating factor controlling the wear behaviour of aluminium matrix alloy and composites.

Wear resistance of the composites were superior to the matrix alloy at all loads tested. The wt. % of reinforcement is the second most effective factor on the wear rate of the composite samples. It has been observed that the wear rate of the composites decreases with increasing content of WC reinforcement in the matrix alloy.

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