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A STUDY OF THE LOW STRESS SLIDING ABRASION WEAR BEHAVIOR OF MULLITE COATINGS

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ABSTRACT

Mullite ($3Al_2O_3$–$2SiO_2$), a ceramic with good thermal barrier and wear properties, was plasma sprayed on cast Aluminum A 356.0 alloy pins, to study the wear resistance of Aluminum parts coated with ceramics for use in aerospace, automobile, and industrial applications. The bond strength at the interface was improved by spraying Nickel-Chrome as a bond coat. This paper pertains to the study of low stress 3 body sliding abrasion wear loss of Mullite coatings. A specially designed pin on disc tribometer with features to apply lower pressures of 20 to 100 KPa was fabricated and tests conducted to measure the wear mass loss of all the specimens. Mullite was sprayed on cast Aluminum 356.0 T6 treated, with and without a bond coat and the wear performance was compared with that of bare wrought Aluminum 6063 and bare cast Aluminum A 356.0 in the soft and T6 treated conditions. The effect of heat treatment on cast alloy and the coating (with and without bond coat) on the wear performance has been studied. The wear scars were observed through a metallurgical microscope. The coating was found to be amorphous in nature and showed good wear resistance. The counter face was pasted with SiC emery of particle size 75 µm and with higher hardness and compressive strength than the coating. The results revealed that severe wear occurred, the wear factor falling in the range 10^{-2} to 10^{-5} mg/Nm, the regime of severe wear, in spite of the low stress applied. The wear behavior was correlated to the test conditions and the properties of the materials used. Mullite coatings on cast Aluminum with bond coat and the substrate T 6 treated performed better than other materials.

***Keywords**:* Low stress abrasion, Mullite, Mechanically Mixed Layer, Amorphous layer, Functionally graded duplex coating.

1. INTRODUCTION

1.1 Literature survey of wear of Aluminum alloys

Wear may be defined as the surface damage or removal of material from one or both of two solid surfaces in a sliding, rolling or impact motion to one another as a result of mechanical action. The wear phenomenon can occur due to adhesion, abrasion, surface fatigue and tribochemical reaction. In addition, many studies reveal that the wear situation comprising of the materials under consideration, its shape, weight, applied load, test duration and environment conditions, affect the wear rate and the wear mechanisms (Budinski and Budinski, 2005). Wear is a serious problem in many engineering applications, especially moving parts like bearings and engine parts. There is also an increasing demand for light weight materials with good wear resistance in the automotive and aerospace sectors (Gui, Kang, and Lee, 2000).

It is stated that Aluminum alloys exhibit mild and severe wear. A transition from mild wear to the severe wear depends on the applied load and the sliding speed during dry sliding wear (Alpas and Zhang, 1997). When the wear is severe, ductile materials such as Aluminum alloys experience substantial surface plastic deformation at the surface (Alpas and Singh, 1996; Dautzenberg and Zaat, 1973; Perrin and Rainforth, 1997).

One investigation (Horn and Zeigler, 1983) showed that pure Aluminum and non-heat treatable alloys together with cast Al-Si alloys have poor dry abrasive wear resistance and that cold work does not cause a significant improvement. Heat treatable Aluminum wrought alloys and cast alloys age hardened to optimum hardness showed improved performance. Another work (Rao and Sekhar, 1986), concluded that the wear rate of a range of Aluminum alloys was not inversely proportional to their Vickers's hardness. It is still not clear despite many studies which Aluminum alloy would offer the best wear performance and whether precipitation hardening would be optimum, or whether work hardening would be better (Ghazali, Jones and Rainforth, 2007). This applies to Aluminum MMC's (Metal Matrix Composites) also. Dry sliding wear behavior of Al-4Si-4Mg alloys with one to five % addition of Cerium was studied in one investigation (Anasyida, et.al., 2009). It was found that the wear resistance of the as-cast alloy improved. In another study, 2017 aerospace Aluminum alloy was characterized through metallographic investigations to develop proper precipitation strengthening and age-hardening heat treatment process parameters (Zainul Huda, 2008).

Both the alloys 6063 and 356.0 considered in this study are Magnesium Silicon alloys, the former being a popular aerospace material and the latter used for applications such as machine tool parts, aircraft wheels, pump parts, valve bodies, cylinder heads and engine blocks.

Wear studies have been conducted earlier in the meso, micro and nano scales for MEM'S (Micro Electro Mechanical Systems), with applied loads ranging from a few nN to 100 mN (Le, Luo, and Williams, 2008). Adhesive wear studies involved higher applied loads up to 50 N.Very little work has been conducted on low stress abrasion and sliding and fretting wear, which generally pertains to coatings (Kwang and Emmanuelle, 2000, Miyoshi et.al, 1999, Prehlik et.al., 2001, Yakovlev, 2004). Little or no work has been carried out in the load range 1 N to 3 N for bulk materials and coatings. Aluminum alloys coated with ceramics is expected to have a better wear performance and enough study has not been done.

Ceramic coatings on Aluminum can play a major role in enhancing the wear resistance of Aluminum under elevated temperature conditions in applications such as auto engines, jet engines, aerospace engines, brake shoes/drums and sleeve bearings. Aluminum has the advantage of low density, but performs poorly in wear resistant applications. The problem is aggravated when components are exposed to elevated temperatures. Hence appropriate ceramic coating plasma sprayed on Aluminum would improve the elevated temperature wear resistance of Al cast or wrought.

1.2 Low stress abrasion

Low stress abrasion involves the separation of small particles of the material of the superficial layer in conditions of friction usually sliding, caused by the presence of abrasives harder than the material of the rubbing surface. It is also called scratching abrasion and this is a primary mechanism of damage in 80 to 90% of all tribological wear. Surfaces subjected to low stress abrasion show that material has been removed by hard sharp particles plowing out material in furrows. Abrasion rates increase with the sharpness of the abradant, decrease as the hardness of the surface subjected to abrasion increases, decrease as the size of the abradant decreases and below a particle size of about 3 μm, scratching abrasion ceases; polishing wear commences and microchip formation no longer takes place. Abrasion rate is directly proportional to the sliding distance and the load on the particles or protuberances. Abrasion rates increase when the hardness of the abradant is more than twice the hardness of the surface subjected to the abrasion. In metals, microstructure (carbon content, carbides, hard phases, etc.) affects abrasion rates. The presence of hard micro constituents reduces wear. Fixed abrasives produce more abrasion than the same abrasive used in a three body, lapping mode.

Ceramics and Cermets can have effective resistance to low stress abrasion if the ceramic is harder than the abrasive and if cermets have a significant volume fraction of a phase that is harder than the abrasive (Kenneth G.Budinski, 1988).

The process of abrasive wear is dominant in conditions of dry friction. It is assumed that with a rise in hardness, there is a rise in the abrasive wear resistance of metals and alloys. There are exclusions from this rule:

In some instances, the softer metal wears less than the harder metal. This happens when hard abrasive particles embed themselves in the soft metal in a permanent way and act as abrasives with respect to the harder metal.

Also if the surface is excessively hard, it may also be very brittle. Consequently the material may crack around points of contact with abrasive grains and relatively large portions of the material may become detached from the surface. In such cases, wear and damage of the surface may be very significant (Tadeusz Burakowski and Tadeusz Wierzchon, 1999).

Many wear studies have been conducted for studying adhesive wear with higher test pressures up to 1000 KPa. Few studies have been conducted for lower pressures of 20 to 100 KPa to address the phenomenon of low stress abrasion, applicable for many automobile, aerospace and industrial applications where only lower stresses act on components.

2. EXPERIMENTAL WORK

2.1 Apparatus

The experimental test fixture is shown in Figure 1, which is a pin on disc tribometer. Here the flat pin of size 8 mm x 50 mm length is loaded precisely with weights of 1 N, 2 N and 3 N. The wear mass loss was measured for various test conditions and wear factor calculated and plotted. The wear mechanisms were identified and the wear scars viewed through an optical microscope and inference drawn. The test conditions such as load applied, sliding speed and distance simulate the real life situations of many practical wear applications. The set-up has been specifically designed and fabricated to test with low applied load.

2.2 Test conditions

Wrought Aluminum alloy 6063-O fingers of size 8 mm diameter and 50 mm length were machined from 12 mm diameter rods and cast Aluminum alloy A 356.0 rods of 12 mm diameter were melted and cast in permanent cast iron moulds and later machined to 8 mm diameter and 50 mm length fingers. Two types of cast samples were prepared namely the as-cast and T6 tempered conditions.T6 tempered cast specimens were coated with Mullite with and without bond coat. Dry sliding 3 body abrasive wear behavior of these materials under low stress conditions with applied load from 1 N to 3 N has been investigated using a pin on disc wear tester at a fixed sliding speed of 1.3 m/s and sliding distances of 82 m, to 574 m, in steps of 82 m.

SiC emery paper of grit 220 (75 µm and with irregular particle shape) was pasted on the EN 8 steel counter face and made to run against the fixed pin made of the Aluminum alloy. The emery sheets were replaced after every test duration of 7 minutes. The diameter of the disc is 60 mm and thickness 15 mm. Prior to testing, test samples were polished with emery paper and cleaned in acetone, dried and then weighed using an electronic balance having a resolution of 0.1 milligrams. After each test, the specimens were removed, cleaned in acetone and weighed in a similar fashion. At each load the mass loss from the surface of the specimens were determined as a function of the applied load and the sliding distance. Confirmation tests were also conducted to establish the values. Additional information pertaining to the wear situation is detailed below (Raymond, 2002) in Table-1.

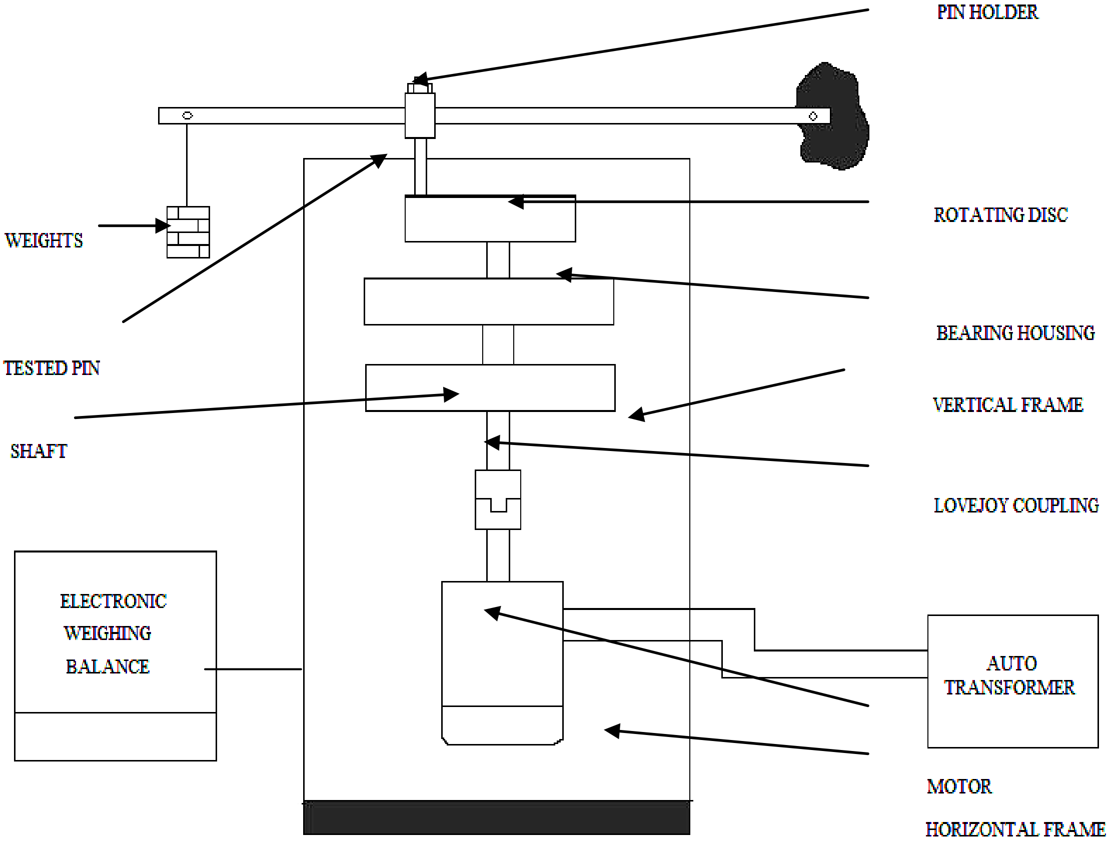


Figure 1: Pin on disc wear test apparatus

Table 1: The wear situation

Parameter	Description
No. of bodies	3 body (the abrasive particles are constrained by a counter face and trapped between the two independent bodies)
Stress level	Low stress upto 0.019 MPa to 0.059 MPa
Presence of fluid	Dry abrasion
Relative hardness of particles to surface	Surface softer than particles
Motion	Sliding, unidirectional and high speed (1300 rpm)
Contact geometry	Circular area in surface contact
Test environment	25° C, 50 % RH
Materials	Dissimilar

As stated in a previous section, the wear situation plays a major role in the wear behavior of any material. The wear situation in this study was expected to be more aggressive than the adhesive wear situation, used for the study by many authors. The wear factor (K) in mg/Nm is calculated as per the following formula:

K=$W \div LN$, where W is the wear mass loss in 'mg', L is the sliding distance in 'm', and N is the applied load in 'N'.

2.3 Metallographic observation

Specimens for the metallographic observations were prepared by standard polishing techniques. The microstructure of the specimens was investigated by means of an optical microscope model Censico with a magnification of 200 X. Keller's reagent with composition, 194 ml distilled water, 5 ml Nitric acid, 3 ml HCl and 2 ml hydrofluoric acid was used as the etching reagent.

The wear scars of the worn surfaces were similarly prepared and observed. The specimens were run for duration of three minutes for the various applied loads before polishing.

3. MATERIALS

3.1 Substrate

Wrought Aluminum 6063-O and cast Aluminum A 356.0 are the substrates used with composition as per ASM standards (Elwin, 1990) and properties as per Table 2.

Table 2: Properties of wrought Al 6063-O, cast Al A 356.0 and Mullite

Sl.No.	Property description	Al 6063	Al A 356.0	Mullite
1	Melting point , °C	600	650	1840
2	Density, g/cc	2.68	2.67	3.16
3	Thermal Expansion Coefficient ,10^{-6} °C	23.4	21.4	5.4
4	Thermal Conductivity at 20 °C, W/mK	200	167	9
5	Specific Heat KJ/kgK	0.91	0.963	1.1
6	Elastic modulus GPa	70	72.4	151
7	Hardness HB	25	42 (soft), 63 (T6)	1070 (Kg/mm^2)
8	UTS MPa @ R.T.	131	234	103.5
9	Poisson's ratio, 'υ'	0.33	0.33	0.25

3.2 Top coat

The top coat faces the hot combustion gases and hence should be a thermal barrier. Mullite ceramic of 100 micron thickness is plasma sprayed on the bond coat. The properties of Mullite are shown in Table 2. Powder particles of 40 to 60 µm were used for spraying.

3.3 Bond coat

It is most important to plasma spray a layer of bond coat of Nickel Chrome on the Aluminum substrate to a thickness of 50 microns before applying the top coat of Mullite to improve the adhesion of the coating to the substrate. This is due to the wide mismatch in the Young's modulus and the thermal expansion coefficients of the substrate material and the Mullite ceramic. The bond coat will reduce this mismatch and hence reduce the residual stresses in the coating and enhance the adhesive strength of the coating. The Nickel Chrome powder is from Powder Alloy Corporation, USA with the details given below.

Alloy: PAC98F1 REV C NICKEL CHROMIUM
Specification: PWA1319F
Particle size: 45 to 75 microns
Particle chemistry is given below in Table 3

Table 3: Particle chemistry

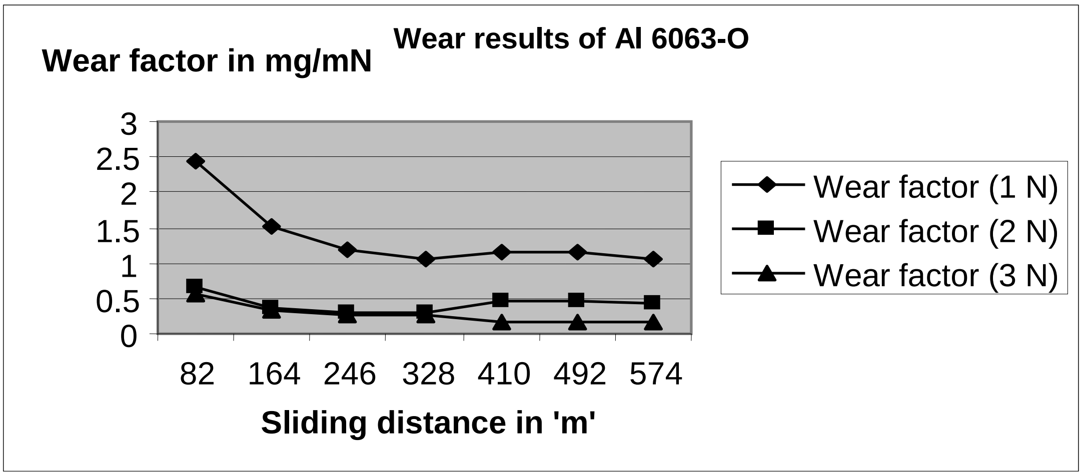
Element		C	Cr	Fe	Mn	Ni	Si
Results		0.015	19.58	0.35	0.004	78.60	1.29
Spec	Min		18.00				
	Max	0.25	21.00	1.00	2.50	80.00	1.50

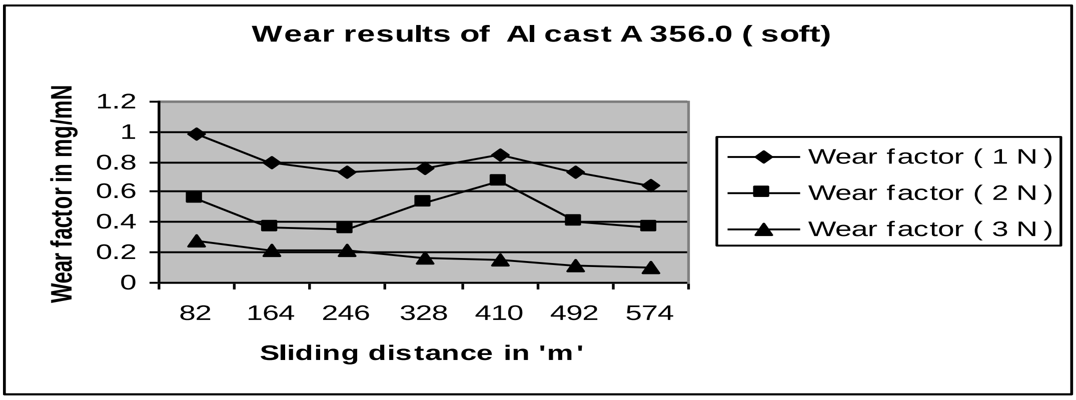
3.4 Counter face:
SiC fine emery sheet of grit 220, particle size 75 microns, irregular shape. SiC has a hardness of 2800 kg/mm^2 and compressive strength of 3900 MPa.

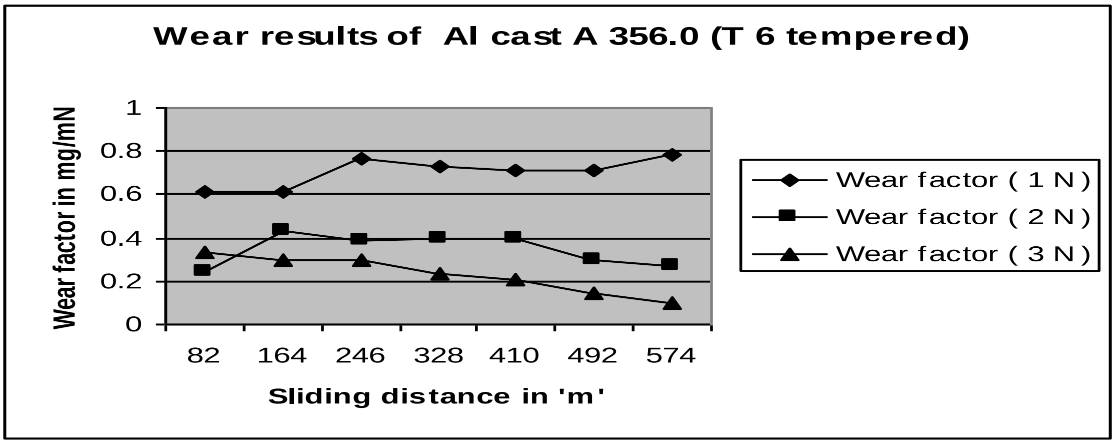
3.5 EDS/XRD/SEM study
Surface analysis by EDS and XRD confirmed the presence of all coating elements, such as Al, Si, Ni, Cr in the coating with bond coat and lesser % of Nickel Chrome in the coating without bond coat. The analysis also indicated the presence of Al_2O_3 and SiO_2 in the coating, due to the presence of Oxygen. The SEM image of the cross section of the coated sample showed the amorphous layer of Mullite well bonded with the bond coat, and the bond coat in turn blended well with the substrate (Viswanath and Vijayarangan, 2009).

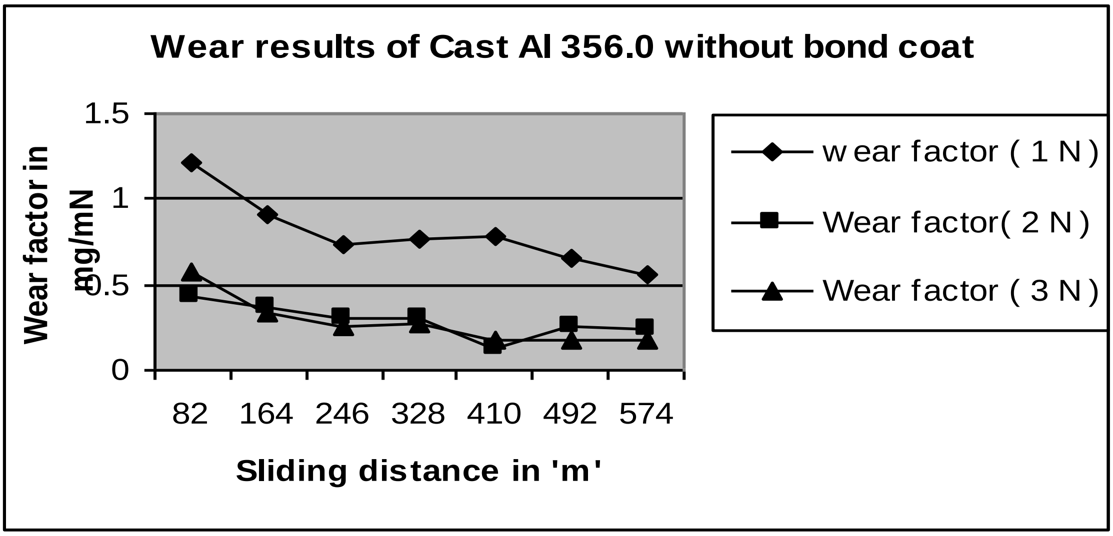
4. RESULTS AND DISCUSSION

The wear loss measured was used to plot the wear factor against sliding distance curves at the applied load range of 1 to 3 N as shown in Figure 2. Figures 3 to 6 show the wear scars of the materials.









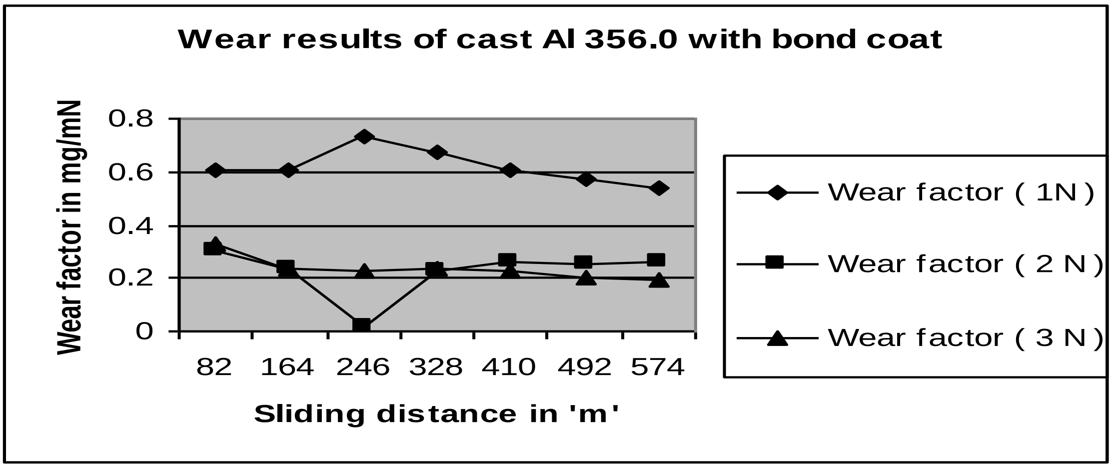


Figure 2 : Wear factor in 10^{-3} mg/Nm as a function of sliding distance and applied load

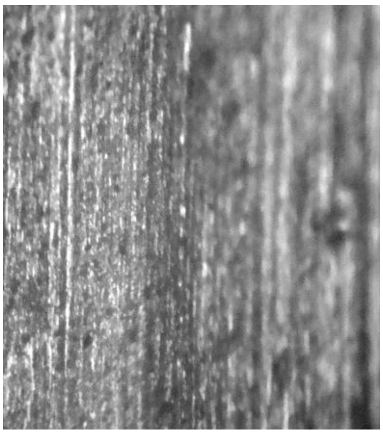


Figure 3: Wear scar of Al 6063-O for 3 N applied load

Notes: The wear scars show a plowing and grooving action due to low hardness and flow stress of the material. The wear rate is high though the MML is present. The dark patches are that of SiC.

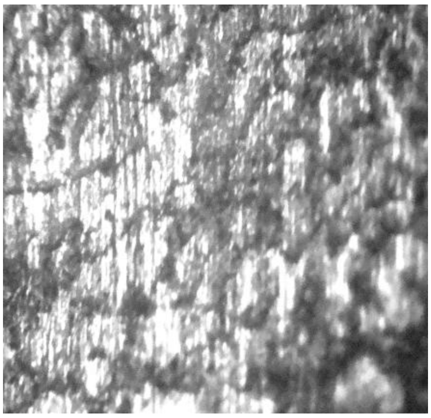


Figure 4: Wear scar of cast Al A 356.0 (soft) for 3 N applied load

Notes: The higher hardness and higher flow stress values lead to lower plowing and also due the presence of the MML the wear rate is lower. Dark patches of SiC are seen.

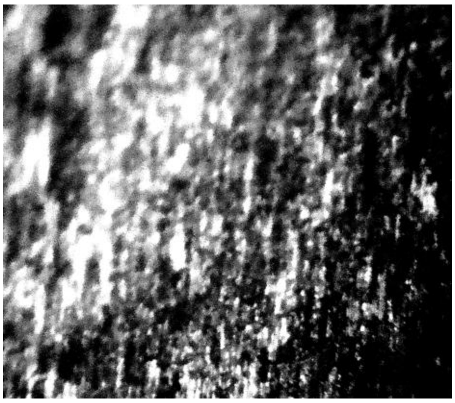
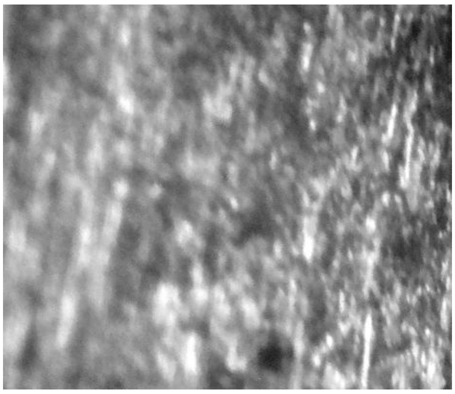
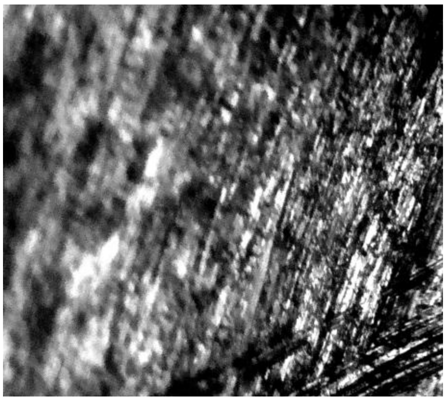


Figure 5: Wear scar of cast Al A 356.0 T 6 treated, for 3 N applied load

Notes: Due to higher hardness and flow stress values, the wear rate is lower and the wear mechanism is mainly cutting and fragmentation. The MML improves the wear resistance. The dark patches are that of SiC.



(a)



(b)

Figure 6: Wear scars of Al T6 tempered A 356.0 with bond coat and without bond coat

Notes: Wear rates are lower and wear mechanism is mainly cutting and fragmentation due to the higher hardness of the coating composite phases present.

4.1Properties of materials and wear conditions correlated to wear behavior (Raymond, 2002**):**

1. The wear rate depends on the hardness of the specimens, as the depth of indentation for a given load by the abrasive particle is a function of the hardness of the material. The results show ploughing wear mechanism in the case of Al 6063-O with deeper grooves when compared to cast Al A 356.0 in the tempered condition. As the hardness increases, the ductility reduces resulting in a change in the abrasion mechanism from predominantly ploughing/cutting to fragmentation. The wear rate of coated specimens was better with T6 treated specimens being the best.
2. Wrought Al being more ductile than cast Al, plowing takes place and the probability of wear debris formation is high which get embedded in the grooves and also the proportion of ploughing will be more than cracking or fragmentation.
3. As the contact pressure increases, the wear rate increases.
4. The contact conditions of velocity and impingement angle of the abrasive play a major role in the wear rate. As the sliding velocity increases, the wear rate also increases. In this study the sliding velocity has been kept constant. The impingement angle is 0° in this study. For higher angles, wear rate will be more.
5. The abrasive particle characteristics such as size and hardness ratio of wear material and the abrasive are important parameters. With an increase in the abrasive size, the wear rate also increases as in this case. Lower the ratio of H/Ha,(H being the hardness of the material tested and Ha the hardness of the abrasive) as in the case of wrought Aluminum 6063, plastic deformation takes place, with deeper grooves.
6. The microstructure plays a major role in the wear rate of the materials. In the T 6 treated condition the microstructure is more homogenous and hence the wear rates are lower. The coated specimens having an amorphous microstructure have still lower wear rates.
7. For materials of equivalent hardness, the plastic flow behavior depends on the E/σ_y ratio (E being the Young's modulus and σ_y being the flow stress of the material being tested). Higher the ratio, higher will be the ploughing action. Flow stress σ_y for Al 6063-O is lower and E/σ_y is higher and hence a plowing action results. E/σ_y is lower for Cast Al and less ploughing occurs. The flow stress of Mullite is lower but the composite microstructure contributed to fragmentation and chipping.

4.2 Discussion of the wear factor results.

1. The wear loss is lower for higher applied loads, due to the MML formation.
2. The wear factor decreases for all the applied loads and increasing sliding distances for the materials considered.
3. The wear factor for the 2 N and 3 N applied loads is lower than the wear factor for 1 N load, which shows the better wear performance of the materials at higher applied loads.
4. The wear factor is the highest for the Al 6063 alloy and the lowest for the Cast Al 356.0 T6 tempered condition revealing the better performance of the cast alloy in the heat treated condition. The results are still lower for the coated specimens with bond coat. A mechanically mixed layer with higher load bearing capacity due to the combined ceramic phases and the bare Al is the reason. The wear scars show the polished microstructure.
5. The morphology of the wear scars of 6063 –O Al and cast A 356.0 Al, and the coated specimens for the applied load of 3 N, sliding speed of 1.3 m/s and sliding distance of 246 m are shown in the figures.

For 1 N load, 6063 Al exhibited a rough wear surface with deeper grooves, with the major wear mechanism being ploughing (plastic deformation due to higher ductility). Similarly for the cast specimens, the scars are rough due to cutting/fragmentation and less ploughing. For 2 N and 3 N loads, the microstructure is less rough due to the mechanically mixed layer formation comprising of Al and SiC phases for Al 6063 and Al 356.0 cast and the combined ceramic phases of Al_2O_3–SiO_2, SiC and Al for the coated specimens. The mechanically mixed layer has a higher load bearing capacity resulting in lower wear rates. The MML played a vital role in dry sliding wear in the range of loads (Li and Tandon, 2000). It provided a surface protection before critical condition was reached.

6. In general, the wear loss of the materials increases linearly with increasing sliding distance and applied load, but the wear rate decreased with increasing sliding distance since wear rate is the ratio of wear volume to sliding distance in a certain wear condition.

5. CONCLUSIONS

1. Wear rate of any material depends on the hardness of the material and its ductility. Cast Aluminum A 356.0 T6 treated showed a better wear performance than Aluminum A 356.0 in the as cast condition and wrought Aluminum alloy 6063-O in the annealed condition. Coated samples performed still better.
2. The wear performance of the materials selected showed a better result for higher loads applied due to the formation of a mechanically mixed layer with a higher load carrying capacity. This is applicable for the low stress range and the sliding distances selected and also for the wear situation described.
3. The wear loss increases as a function of sliding distance and applied load initially and then flattens for the materials tested.
4. In this study, the wear is found to be severe in all cases; though it is better in the case of heat treated cast alloys and coated samples. The wear rate being in the range 10^{-2} to 10^{-5} mg/Nm falls in the regime of severe wear. Critical load is the applied load when the transition from mild to severe wear takes place. In this study, for the wear situation considered 1 N may be considered well above the critical load.
5. Though applied stresses are lower, the wear situation, especially the abrasive grain size of 75 microns of SiC, has contributed for the severe wear.
6. The coating with bond coat is a functionally graded duplex coating with an amorphous layer of Mullite plasma sprayed as the top coat. The ceramic layer acts as a composite by virtue of its porosity. The amorphous structure has no lamellar and intra lamellar cracks or grain boundary defects and imparts good hardness and wear resistance to the coating.
7. For a duplex microstructure with two or more phases, as in the coating, the softer matrix is removed by ploughing and the harder phases are left unsupported and a pull out mechanism acts by fragmentation.

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