# Friction and Wear Behaviour of Plasma Sprayed Fly Ash Added Red Mud Coatings

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6 Abstract: The present investigation aims at evaluating the effect of fly ash addition on sliding 7 wear behaviour of pure red mud. Plasma sprayed coatings composed of red mud and varying 8 percentage of fly ash were considered for the wear behavior study. Plasma spraying technique 9 was used with varying levels of power namely 6, 9, 12 and 15 kW. Investigations of the coatings focused on tribological properties like sliding wear behaviour, wear morphology, 10 11 wear mechanism and frictional force. Experimental investigations also include the effect of 12 varying percentage of fly ash on dry sliding wear behaviour of pure red mud. Fly ash with 10, 13 20 and 50 % by weight was mixed with red mud and sliding wear test performed using pin on 14 disc wear test machine. The wear test was performed for sliding distance up to 942 m with 15 track diameter of 100 mm and at sliding speed of 100 rpm (0.523 m/s); applying normal load 16 of 10 N for a maximum duration of 30 minutes. The variation of wear rate and frictional force 17 with that of sliding distance and time has been presented. Significant wear resistance was 18 visible with the addition of fly ash due to increase in bond strength and dense film at the 19 interface. Wear rate decreases with operating power up 12 kW thereafter declines initiating 20 other dominating parameters.

Key words: Red mud; Fly ash; Plasma Coating; Sliding wear; Wear morphology;
Frictional force; wear mechanism.

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# 24 **1.INTRODUCTION:**

25 Coating technologies have already gained a promising momentum for the creation of 26 emerging materials in the last few decades. Coatings with advanced wear properties claim 27 frequent use in tribological applications. Plasma spray is one of the most widely used 28 techniques involved in surface modification by improvement of wear resistance, which may 29 affirm the great versatility and its application to a wide spectrum of materials. The coatings 30 with considerable amount of hardness can protect against variety of wear mediums including abrasive, adhesive and corrosive. Basically, wear resistant coatings are fabricated from some 31 32 common conventional materials like nickel, iron, cobalt and molybdenum based alloys [1-2].

Extensive investigations of erosion wear behavior of plasma sprayed ceramic coatings using Taguchi Technique was reported by some experimenters [3]. The tribological properties of traditional manganese phosphate coatings and hBN composite coatings composed of nano hexagonal boron nitride (hBN) in layered manganese phosphate crystals on AISI 1040 steel were studied in [4].

38 Studies are also available regarding the wear behaviour of WC with 12% Co coatings 39 produced by Air Plasma Spraying method at different standoff distances [5]. Examinations of 40 the wear behaviour of Mo and Mo+NiCrBSi thermally sprayed coatings were performed for 41 the application as next generation ring face coatings [6]. Almost all plasma sprayed ceramic 42 coatings featured favorable tribological performance in linear contact at high temperatures: 43 high anti-wear resistance and easy to be lubricated owing to the oil storage of pores in 44 coatings [7-9]. But needful to say, plasma sprayed ceramic coatings exhibit some failure 45 mechanisms during sliding such as plastic deformation, brittle fracture and polishing effects 46 [10], which in turn demands a few additives, which could reduce the friction and wear of 47 plasma sprayed ceramic coatings [11].

48 Several factors may influence the tribological behaviour of a coated surface such as: the 49 geometry of the contact including macro geometry and topography of the surfaces; the 50 material characteristics; basic mechanical properties as well the microstructure and finally the 51 operating parameters controlling the coating deposition [12].

52 Red mud as an industrial waste material is considered to be the material of choice for coating 53 applications. It is behooved to mention here that, red mud in present decade should be 54 considered as an alternative for replacing some conventional expensive coating materials. 55 Utilization of red mud and its implications is available in literature [13] in great details. Few 56 results on the wear behavior of red mud were reported by some researchers. In addition to the 57 above, morphology and solid particle erosion wear behaviour of red mud and fly ash 58 composite were studied in [14]. Characteristics of plasma sprayed pure red mud coatings were 59 reported in [15]. Red mud as filling material is also found to be the wear enhancing agent for 60 metals [16]. Tribological aspects of thermally sprayed red mud-fly ash and red mud-Al 61 coatings on mild steel was reported [17]. Data pertaining to the sliding wear behavior of fly 62 ash based red mud composite coatings are not abundant and need to be addressed. The present 63 investigation aims to evaluate the wear behavior of varying percentage of fly ash with pure 64 red mud coating at different operating power subjected to normal laboratory conditions. This 65 paper may pave the path for extending the study to throw more light on fly ash based red mud 66 coatings.

# 68 2. MATERIALS AND METHODS OF EXPERIMENTATION

## 69 2.1 Preparation of coating powder

The present experimental work included the preparation of coating powder from the raw materials as red mud and fly ash powders. The powder mixture of red mud and different percentage of fly ash was prepared using V-shaped drum mixer. In addition, pure red mud powder was also used as coating material for the comparison on the basis of percentage of fly ash addition. Coating of the various combinations of mixed powders was conducted on one side cross section of the mild steel substrate. Data in Table.1 shows the different mixtures chosen for plasma spraying.

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Table. 1 Powders used for coating deposition.

Sl.No.	Coating material	Mixture composition (by weight %)	
1	Red mud (RM)	100	
2	Red mud + fly ash (FA)	90 + 10	
3	Red mud + fly ash	80 + 20	
4	Red mud + fly ash	50 + 50	

Red mud, as the primary raw material was collected in powder form from National Aluminium Company (NALCO) located at Damonjodi in the state of Odisha, India. The asreceived powder was sieved to obtain particles in the required size range of 80-100 µm. Raw fly ash was collected from the captive power plant of Rourkela steel plant, India and sieved to maintain same size range as that of fly powder. Powders having three different weight ratios of red mud and fly ash (Table 1) were prepared by mixing thoroughly.

# 84 2.2 Preparation of substrates

85 Commercially available mild steel rod was used as source for substrate preparation. The 86 rod was cut to pieces having one particular dimension (l = 40 mm and  $\emptyset = 12 \text{ mm}$ ) each. The 87 specimens were grit blasted from one side cross section (initial roughness 0.03 mm) at a 88 pressure of 3 kg/cm<sup>2</sup> using alumina grits of grit size 60. The stand-off distance in the shot 89 blasting was kept between 120-150 mm. Then the average roughness of the substrate was 6.8 90 µm. The grit blasted specimens were used for plasma spraying after cleaning in an ultrasonic 91 cleaning unit.

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# 93 2.3 Plasma spraying

The spraying process was performed at the Laser and Plasma technology division of Bhabha Atomic Research Centre, Mumbai, India by adopting conventional atmospheric plasma spraying (APS) set up. The plasma input power was varied from 6 to 15 kW by controlling the gas flow rate, voltage and arc current. The powder feed rate was maintained

98 constant at 10 gm/min by using a turntable type volumetric powder feeder. Plasma generation

99 used argon as primary and nitrogen as secondary gas agent. The mixtures of powders were

 $\frac{100}{100}$  deposited at spraying angle of 90° by maintaining the powder feeding external to the gun. The

101 operating parameters of the coating deposition process are shown in Table-2.

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#### Table 2 Operating parameters during coating deposition

<b>Operating parameters</b>	Values	
Plasma arc current (Ampere)	200,225,250,300	
Arc voltage (Volt)	30,40,48,50	
Torch input power (kW)	6,9,12,15	
Plasma gas(argon), (litre/min)	20	
Secondary gas(nitrogen),(litre/min)	2	
Career gas(argon) flow rate (litre/min)	7	
Powder feed rate (gm/min)	10	
Torch to base distance (mm)	110	
Arc length range (mm)	2,3,6,8,11	

# 103 2.4 Pin on disc wear testing

104 The above experiment was being conducted in the pin on disc type friction and wear 105 monitor (DUCOM; TR-20-M100) with data acquisition system. The machine was used to 106 evaluate the wear behavior of the coatings against hardened ground steel disc (En-32) having 107 hardness of 65 HRC and surface roughness (Ra) 0.5  $\mu$ m. The equipment is designed to study 108 the wear behaviour under un-lubricated sliding condition, which occurs between a stationary 109 pin and a rotating disc.

The disc of the machine rotates with the help of a D.C. motor having speed range of 0-200 rpm with wear track diameter 0-160 mm; this can yield sliding speed of 0-10 m/s. Load is applied on the pin (specimen) by dead weight through pulley string arrangement. The system has a maximum loading capacity of 500 N. For the present experimentation, pin specimen was kept stationary perpendicular to the disc, while the circular disc was rotated as shown in Figure 1.



120 **Figure 1** Schematic Representation of Pin on Disc Apparatus

# 121 3. **RESULTS AND DISCUSSION**

# 122 3.1 Scanning electron microscopy and compositional analysis

123 The characterization of red mud powder involved taking microstructures by the help of 124 scanning electron microscope (JEOL; JSM-6480 LV). The micro structural images captured 125 by SEM (scanning electron microscope) and EDS (energy dispersive spectroscopy) analysis 126 of pure red mud powder are illustrated in Figure 2. EDS experiment was performed by the above SEM with the required attached module. Data presented in Table 3 indicates the weight 127 128 as well the atomic percentage of elements comprising pure red mud powder. The EDS 129 analysis of red mud revealed the signature of elements like Fe, Al, Si, O and some other 130 minor constituents. The prominent constituent of red mud was found to be iron with its 131 oxides. The EDS analysis of red mud with 20 % fly ash coatings prepared at 9 kW of 132 operating power is shown in Figure.3. In addition, the analogous elemental analysis relating to 133 Figure-3 is reported in Table.4, indicating the increase in silica and iron constituents in the 134 composite coating.





Figure 2. (a) SEM and (b) EDS analysis of red mud

-	Tables. Elemental analysis of fed fild			
	Element	Weight%	Atomic%	
_	C K	24.59	33.29	
	O K	23.65	24.54	
	Al K	7.41	4.47	
	Si K	12.21	7.07	
	Fe K	32.14	30.62	





Table4.Elemental analysis of RM+20 % FA composite coated at 9 kW

Element	Weight %	Atomic %
Fe K	36.13	25.90
O K	21.74	42.61
Ti K	14.02	9.18
Si K	17.10	7.93
Al K	6.59	7.66
Cr K	2.14	1.29
СК	1.99	5.20
Ca K	0.29	0.22
Au K	0.00	0.00
Mg K	0.00	0.00
Totals	100 %	100

Table3. Elemental analysis of red mud

# 146 *3.2 Coating porosity*

147 Image analysis technique was adopted for the measurement of porosity of coating 148 materials. The polished surfaces of various coatings were kept under a microscope (Neomate) 149 equipped with a charge coupled-device (CCD) camera (JVC, TK 870E). Volume of interest 150 (VOIS) image analysis software paid an important role for the determination of porosity. The 151 software can measure accurately the total area captured by the objective of the microscope. 152 Hence the total area and the area covered by the pores are separately measured to report porosity. The "VOIS image analysis software" used in this analysis is being licensed by the 153 154 authors. The porosity data of three different coatings are shown in Table.5. A cross sectional 155 view of red mud coating prepared at 9 kW of operating power was captured by the help of 156 field emission scanning electron microscope (FESEM; Nova Nano SEM-450), as shown in 157 Figure 4.



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# 159 **Figure 4.** FESEM image of coating cross section of pure red mud at 9 kW

**Table 5.** Coating porosity for different coating type.

<b>Coating material</b>	Plasma torch	Porosity (%)
	input power(kW)	
	6	12.89
D - 1 1	9	12.02
Ked mud	12	11.87
	15	13.02
90 % Red mud	6	11.52
+	9	11.12
10 % Fly ash	12	10.90
	15	12.98
80 % Red mud	6	10.89
+	9	10.54
20 % Fly ash	12	10.17
	15	11.78

161 Approximately 8-13% porosity range was observed (Table-5) for all three coating 162 materials. Porosity amount was found to be ameliorated in case of all coating compositions 163 prepared at lower (6 kW) and at higher (15 kW) power levels. At 6 kW operating power level, 164 there is poor melting of particles subjected to relatively low plasma gas temperature 165 exhibiting non-uniform mixing of molten particles; which in turn causes reasonably porous coating laver. On the other hand, at highest operating power level (15 kW) the high plasma 166 167 gas temperature caused faster deposition of molten particles by creating thickened coating 168 layer with less hardness and high porosity.

Porosity level was found to be higher in case of pure red mud compared to the composite coatings made of the mixture of fly ash and red mud. About 3-10% porosity level was reported for the coatings prepared by conventional plasma spraying [18], which supports the porosity results obtained in the present investigation.

#### 173 3.3 Coating hardness

174 The polished section of the coatings put under optical microscope for the microscopic 175 observations, which revealed the presence of three distinguishable different phases namely 176 dull, white and spotted. The three different distinct phases were subject to micro indenting to 177 record micro hardness data with the help of Leitz micro hardness tester using 50 Pa (0.493 N) 178 on all samples. The results are summarized in Table-6. The three structurally different phases 179 of red mud coatings bear three different ranges of hardness values varying from 488 to 588 180 HV. Hardness values were found to be enhanced for the red mud and fly ash composite 181 coatings. This result is attributed to the increased content of alumina and silica in the 182 composition of feed material forming alumino-silicate (mullite phase) during spray deposition 183 [19].

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# Table 6. Coating hardness for different operating power level

	Plasma torch	Ν	Micro hardness (HV)		
Coating material	input power ( kW)	Dull	White	Spotted	
	6	540	488	496	
100% Red mud	9	532	498	511	
	12	586	513	508	
	15	555	502	510	
90% Red mud	6	638	632	628	
+	9	648	642	636	
10 % Fly ash	12	660	638	628	
-	15	651	640	632	
80 % Red mud	6	658	649	642	
+	9	669	658	649	
20 % Fly ash	12	699	689	652	
2	15	681	681	650	
50 % Red mud	6	696	679	658	
+	9	682	633	672	
50 % Fly ash	12	726	712	660	
•	15	719	679	668	

# 185 *3.4 Wear test study*

186 Prior to starting the wear testing experiment, the pin and the disc surface of the concerned 187 equipment were polished perfectly with emery papers for better ensuring of smooth contact 188 with the coating samples. Hereafter the surface roughness was reduced to 0.1 µm. The wear tests were carried out per ASTM G- 99 standard for maximum time period of 30 minutes 189 190 under un-lubricated condition in a normal laboratory ambience having relative humidity of 191 40-55% and the temperature range of 20-25°C. The weight of the specimens before and after 192 the wear experiment being recorded by using electronic weighing machine having accuracy of 193 (0.01 mg) for monitoring the mass loss occurrence in the coating samples. Specimens were 194 periodically cleaned with woolen cloth to avoid entrapment of wear debris and to maintain 195 uniformity in each set of experiments. The test pieces were cleaned with tetrachloroethylene 196 solution before and after each test. Wear rate was estimated by measuring the mass loss ( $\Delta m$ ) 197 of the specimen after each test. Wear rate relating to mass loss and the sliding distance (L) 198 was formulated below in equation (1).

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$$W_r = \frac{\Delta m}{L} \tag{1}$$

200 Where  $W_r$  = wear rate in N/m;

201  $\Delta m = \text{mass Loss in Newton (N)};$ 

202 L = sliding distance in meter (m).

The frictional force (F) was measured directly from the apparatus in 'kg' at each time interval.

The wear experiment was carried out at normal atmospheric temperature under a constant normal force of 10 N and a fixed speed of 100 rpm. The track diameter of the equipment was kept at 100 mm. The maximum duration of sliding was 30 minutes comprising of 10 intervals of 3 minutes each. Each sample was allowed for sliding for distinct time interval.

Initially, the experiment was performed with red mud coated samples and then continued for fly ash based red mud coating composites. Figure 5 illustrates the variation of wear rates with sliding distance for different operating power levels.

The wear results for pure red mud coating operated at 6 kW of operating power being visible in Figure 5 (a) disclose the variation of wear rate with minimum value of 0.11 N/m to maximum value of 0.45 N/m. The wear rate value was found to increase from 0.11 to 0.13 N/m for the first 6 minutes of sliding. After a drastic increase from 6 to 12 minutes of duration, the wear rate plot assumed a plateau. The evolution of wear rate value may be attributed to the variation of coating layer property. This fact indicates hardness of denser surface of top layer than that of bulk layer.

The wear rate was reduced for fly ash based (10%, 20% and 50%) composite coatings, as illustrated in Figure 5. The wear rate trend for fly ash composite coatings are quite similar to those of pure red mud coating. Initial slow increase in wear rate for the composite coatings was visible followed by a drastic increase. Henceforth, the wear rate was roughly constant for all composite coating type. The plots in Figure 6 represent the variation of wear rates of eachcoating type with that of sliding distance for different operating power level.

The effect of operating power level on wear rate is quite interesting. The wear rate is affected by the porosity and hardness. The wear rate was found to be decrease up to 12 kW and increase again slightly for 15 kW. The wear rate for 15 kW was found to lie between 9 and 12 kW. This might be due to the improper particle to particle bonding and poor stacking to the substrate, which in turn lowered the hardness as well as density due to poor interfacial bond strength. Figure 7 shows the trends of wear rate for all coating materials against operating power level for a particular sliding time (15 minutes).

An experimental study on coating thickness for fly ash and red mud composite with operating power is reported in [19]. An increase in coating thickness with increase in input power to the plasma torch; up to about 12 kW is observed and then with further higher input power no improvement in coating thickness is recorded.

236 The frictional force (F) in kg was measured directly from the wear apparatus. The variation 237 of frictional forces with sliding time is shown in Figure 8, which includes the picture for all 238 coating materials and also for operating power levels considered. As per the observations, 239 maximum frictional force is evidenced for pure red mud coating and decreases with the 240 addition of fly ash, akin to the results observed for the wear rate. An increase in frictional 241 force up to a maximum value of 0.63 kg for pure red mud coating at 12 minute sliding time is 242 observed followed by a fluctuating wavy response up to 21 minutes then a constant magnitude 243 up to 30 minute of sliding.

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Figure 9 compares frictional forces for the coating composites with 10% fly ash. The frictional force is found to be maximum at 6 kW and minimum at 12 kW operating power. At 15 kW of operating power, the frictional force was found to be in the range of values for the power levels between 9 to 12 kW. These results are in accordance with the findings observed for wear rates.

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251 Wear morphology for selected coating samples are highlighted in some images captured by 252 FESEM. Figure 10 represents the wear morphological images for red mud with 10% fly ash 253 coating (prepared at 6 kW operating power) allowed for sliding for the time intervals of 3, 6, 254 12 and 15 minutes. Owing to continuous sliding of counter surfaces, wear debris formed 255 which interlock within the sliding interfaces, causing pitting and eventually crack formation. 256 Wear scars, debris formed and cracked sections are clearly visible in Figure 10 (b) and (d) 257 indicating a fatigue failure on the worn surface. Figure 11 shows the worn surfaces for 50 % 258 fly ash based red mud coatings (prepared at 12 kW of operating power level) for the sliding 259 intervals 3, 6, 12, 15, 27 and 30 minutes. The wear morphology changes with increase in the 260 sliding distance impacting change in surface roughness leading to the interruption of its 261 contact mechanism. The change in wear characteristics may be attributed to the variation of 262 hardness of coating inter-layers with respect to the change in sliding distance. At incipient, a 263 slow increase in wear rate is observed and then attains a rapid increment, the 'break in' 264 situation, after traversing of certain sliding distance. The further increase in sliding distance 265 cannot change the contact area; causing a relatively steady wear rate. Hence, it can be concluded that the wear takes place by the phenomenon of adhesion and abrasive mechanism due to development of shear stresses between the hard asperities of the two surfaces in contact. After the "break in" phase, the trend of wear rate remains almost constant for coatings deposited at all power levels. The duration of this stage extends till the end of the test.



Figure 5. Wear rates obtained for different coating type with sliding distance. (a) 6 kW, (b) 9
kW, (c) 12 kW, (d) 15 kW.





280 Figure 6. Wear rate comparison for different operating power level. (a) red mud,

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(b) red mud +10 % fly ash, (c) red mud+20% fly ash, (d) red mud+50% fly ash.



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Figure 7. Variation of wear rate with operating power level at sliding time of 15 minutes.

285 286 6 kW

- 12 kW

1000

600

**(b)** 

800

- 15 kW



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- 290
- 291

292 Figure. 8. Frictional forces against sliding time for all coating type. (a) 6 kW, (b) 9 kW, (c) 12 kW, (d) 15 kW. 293

(c)

(d)







Figure.9. Comparison of frictional force values for 10 % fly ash coating.





Figure.10. Worn surfaces for red mud + 10 % fly ash coatings for 6 kW operating power level.; (a) 3, (b) 6, (c) 12 and (d) 15 minutes time interval.



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Figure.11. Worn surfaces for red mud + 50 % fly ash coatings for 12 kW operating power Level.; (a) 3, (b) 6, (c) 12, (d) 15, (e) 27 and (f) 30 minutes time intervals.

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#### 307 **4. CONCLUSION**

308 The present allow for some salient concluding remarks. Red mud, the waste generated 309 from alumina plants is coat able on metal substrates by employing thermal plasma spraying 310 technique with excellent wear resistance. The addition of fly ash with red mud reduces the 311 wear rate by enhancing the coating property. But the optimum percentages of fly ash required 312 for better coating material still impact a question mark for the researchers. It is observed that 313 for the early stage the wear rate increases slowly and then rises drastically with sliding 314 distance for all coating type and finally becomes stagnant. Operating power level proved to be 315 the remarkable variable for coating property. The coating wear resistance increases until an 316 optimum value at 12 kW indicating some other dominating parameters. The present work 317 leaves a wide spectrum of scopes for future investigators to explore many other aspects of red 318 mud coatings. Thermal stability of these coatings may be evaluated for better claiming in high 319 temperature applications. Corrosive wear behavior under different operating conditions may

320 be investigated to identify suitable application areas. Post heat treatment of these coatings

321 may also be implemented for furthering the study regarding the improvement in coating

- 322 quality and properties.
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# 324 **CONFLICT OF INTEREST**

325 The authors declare no conflict of interest exist for publishing this paper.

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