#### NATIONAL SEMINAR ON WASTE TO WEALTH

Organized by Indian Institute off Metals-Bhubaneswar Chapter and SGAT-Bhubaneswar



# Application of Fly-ash Composite in Plasma Surface Engineering

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## Abstracts

Fly-ash produced from iron & steel industries contains rich amount of metal oxides which gives a tremendous potential as a coating material on structural components. This demand leads to develop and characterize different type of fly ash composite coatings by using plasma spray technique. Plasma spray technology has the advantage of being able to process various low-grade ore minerals to obtain value-added products and also to deposit metals, ceramics and a combination of these, generating approximately homogeneous coatings with the desired microstructure. In the present investigation, coatings are developed on copper substrates using fly-ash+ quartz+illmenite composite (in a weight percentage ratio of 60:20:20) at various plasma torch input power levels ranging from 11 to 21 kW DC. Metallographic and chemical characterization of the produced composite coatings was performed with the aid of Scanning Electron Microscopy (SEM) and XRD Analysis. The adhesion strength was measured by using coating pull-out method. Maximum adhesion strength is 6.32MPa found in copper substrate. It was found that the quality and properties of the composite coating are significantly affected by the operating power level of the plasma spray torch. This work identifies fly-ash composite coating as a potential coating material for structural and engineering application.

Key words: composite; fly-ash composite; Plasma surface engineering; Power Level.

# **1. Introduction**

Fly-ash is a solid waste generated as in huge quantities in iron & steel industries. Only a small fraction of fly-ash is used in the development of high value products. New ways of utilizing fly ash are being explored in order to minimize the plant wastage and provide a safeguard to the environment. Fly-ash is a finely divided powder which can be use as a refractory in industry. Fly ash composite has a number of useful applications, are given in reference [1]. It is well known that fly-ash composite coatings are used extensively for the protection of mild steel and copper

substrates in various corrosion environments. However, the increasing and new demanding technological applications have led to the development of new fly-ash composite coatings. Flyash composite coatings, such as fly-ash+ Na-geopolymer [3], fly-ash+ Redmud [4] and fly-ash+ zinc coatings [5] have been extensively studied. According to recent investigations composite fly-ash coatings can obtain high corrosion resistant, in addition to increased wear resistance. Some of these recent reports concerning the development and surface properties of this type of fly-ash composite coatings are presented below. In present experiment, plasma spray of fly-ash+ quartz+ illmenite composite coatings was produced on copper substrates. The surface and crosssectional morphology, the crystal structure, the surface roughness and the microhardness of these coatings were studied. In addition, the adhesion strength and coating deposition efficiency were thoroughly examined. The results were compared with those of fly-ash+illmenite and fly-ash+ quartz composite coating. It should also be noted that this research investigation also aims in expanding the technological application of massive fly ash produced in industries. Therefore this research investigation aims in utilizing low cost fly ash, by expanding its technological applications on the important field of coatings technology. It is believed that fly-ash might possibly replace other expensive commercial oxides in the production of composite coating materials.

## 2. Experimental Procedure

Flay-ash, Quartz and illmenite mixture was taken with their weight percentage ratio of 60:20:20 and mechanically milled in a FRITSCH-Planetary ball mill for 3 hour to get a homogeneous mixture. This composite used as feed stock for plasma spraying was sieved and the range of 40 µm to 100 µm is separated out for plasma spraying. This powder composite is used as a coating material on the Copper substrates. The substrate materials have dimensions of 1 inch diameter and 3 mm thickness. The substrate were grit blasted at a pressure of 3 kg/cm<sup>2</sup> using alumina grit to make the surface roughness ~5.00 Ra. After grit blasting substrates surface were cleaned by acetone and then immediately plasma spraying was carried out. The spraying process carried out by using a 40 kW dc power supply plasma spray system at the Laser & Plasma Technology Division, BARC, Mumbai. The plasma input power level was varied from 11kW to 21 kW. This is a typically atmospheric plasma spray process, which is working in the non-transferred arc mode. The injection of the powder from the torch nozzle directed perpendicular to the plasma flow and parallel to the torch trajectory. The torch was operated using argon (Ar) and Nitrogen (N<sub>2</sub>) plasma mixture gas. For cooling the system, a water cooling system used which is regulated at a pressure of 10kg/cm<sup>2</sup> supply. Operating parameters used for coating deposition are given in Table-1. Flow rate of plasma gas (argon) and Secondary gas (N<sub>2</sub>) are kept constant. Powder feed rate, Powder Size and Torch to base distance (TBD) are varied with respect to increase in power level. The coated samples were subjected to various analyses. Surface and interface morphologies were studied using a SEM (JEOL JSM-6480LV). The coating pull-out test was carried out on the specimen to evaluate the coating adhesion strength as per ASTM C633 [14]. Phase identification study was done by XRD using a Phillips X-ray Diffractometer with Nifiltered Cu Ka radiation.

*Table-1: Operating parameters used during deposition of Fly-ash+ Quartz+ illmenite coatings.* 

Operating parameters	values
Plasma arc current (Amp)	260-500
Arc voltage (Volt)	40-44
Torch input power (kW)	11,15,18,21
Plasma gas (Ar) flow rate (IPM)	28
Secondary gas (N <sub>2</sub> ) flow rate (IPM)	3
Carrier gas (Ar) flow rate (IPM)	12
Powder feed rate (gm/min)	15
Torch to base distance (TBD) (mm)	100

# **3. Results and Discussion**

#### 3.1 scanning electron microscopy study of surface and interface:

The composite coating material and substrate interface plays most important role in the adhesion of coatings [15-16]. The surface microstructure of composite coating is studied by using SEM show the presence of different phases, typical examples of which are shown in Figure 1(a) & (b). At 11KW power level coating, there is presence of some open pores. These may have originated due to the inadequate flow of molten particles during their solidification [17]. But in case of 21KW power level, the composite particles become fully molten and uniformly spread on the surface. Here there is no open pore but there is some close pore present in between the splat layer. Close pore generate due to much vaporization, at higher torch input power.



Figure 1. SEM surface morphology of Fly-ash+quartz+illmenite Composite coated at (a) 11kW and (b) at 21kW.

The surface morphology of the coatings cannot predict the interior (layer deposition) structures and their importance/acceptability. Thus, the polished cross-sections of the samples were examined under scanning electron microscopy and are shown in Figure 2 (a) & (b). From the micrographs, it is evident that the coating deposited at 11 kW shown in figure 2 (a) has a lamellar structure with small number of cavitations at the interface between the lamellae. Here splats are of small diameter. But Splats formed at 21kW power level are larger in dimension and equi-axed type shown in figure 2(b). Other than the mechanical interlocking of the sprayed coating with the metal substrate, some metallurgical bonding might have occurred at the interface which is evident from the presence of some inter-diffusion zones.



(a) (b) Figure 2. SEM interface morphology of Fly-ash+quartz+illmenite Composite, coated at (a) 11kW and (b) at 21kW.

### 3.2 XRD analysis:

Figure-3 and figure-4 represents X-ray diffraction analysis to examine the presence of various phases in the raw fly ash and in the resulting fly-ash+quartz+illmenite composite coatings respectively. The X-ray diffraction pattern for raw fly ash particles (shown in Figure 3) exhibits distinct peaks which are assignable to the various metal oxide phases present, such as SiO<sub>2</sub>, TiO<sub>2</sub>, Ti<sub>4</sub>O<sub>7</sub>, FeTiO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>SiO<sub>5</sub>. It is clear that, this composite powder contains oxide elements. However, the XRD pattern of the composite coating suggests the presence of crystalline phases like SiO<sub>2</sub>, Ti<sub>3</sub>O<sub>5</sub>, and Al<sub>2</sub>SiO<sub>5</sub> along with additional phases such as Fe<sub>3</sub>O<sub>4</sub> FeTiO<sub>3</sub>, Fe<sub>2</sub>TiO<sub>5</sub> and Al<sub>2</sub>SiO<sub>5</sub>, Ti<sub>4</sub>O<sub>7</sub>. This suggests that, during plasma spraying at higher torch input power some element combined with other element or transformed to its higher stable state.



Figure 3. X-ray Diffractogram of the raw fly-ash composite.



Figure 4. X-ray Diffractogram of the composite coating on Copper substrate.

#### 3.3 Adhesion Strength and Deposition Efficiency of Composite Coating:

The interface bond strength of the coating is evaluated by the coating pull-out method. It is seen that, in all cases, fracture occurred at the coating-substrate interface. However, it has been stated by Lima & Trevisan that the fracture mode is adhesive; if it takes place at the coating-substrate interface and that the measured adhesion value is the value of practical adhesion, depending exclusively on the surface characteristics of the adhering phase and the substrate surface conditions [7]. Adhesion strength increased with power level up to 18 kW and a maximum value of 6.18MPa and further increase in the operating power level exhibited a detrimental effect on the interface strength (shown in figure-5). Initially, when the operating power level is increased from 11 kW to 18 kW, the melting fraction and velocity of the particles also increase. Therefore, there is better splashing and mechanical interlocking of molten particles on the substrate surface leading to an increase in adhesion strength. But, at much higher power levels (beyond 18 kW), the amount of fragmentation and vaporization of the particles are likely to increase. There is also a greater chance of smaller particles (during in-flight traverse through the plasma) to fly off during spraying. This results in poor adhesion strength of the coatings. During in-flight traverse through the plasma, a fly ash particle would melt either partially or fully depending on the temperature and the flame residence time of that particular particle.



Figure 5. Adhesion strength between composite coating material and the Copper substrate with increase in power level.

# 4. Conclusions

Fly-ash composite can be gainfully used as a potential cost-effective material for deposition of plasma spray coatings on metallic substrates. Premixing of quartz & illmenite powder with fly ash can produce metal-ceramic composite coatings of improved interfacial adhesion. Maximum adhesion strength of about 6.18 MPa was recorded. The adherence strength was significantly affected by the plasma torch input power level. The operating power level of the plasma torch also affects the coating deposition efficiency and morphology of the coatings.

## **5. References**

- Satapathy A., Sahu S. P., Mishra D (2009) Development of protective coatings using fly ash premixed with metal powder on aluminium substrates. Waste Management & Research 1-7, doi: 10.1177/0734242X09348016.
- [3] Temuujin J., Minjigmaa A., Rickard W., Lee M., Williams I., Riessen A. v (2010). Fly ash based geopolymer thin coatings on metal substrates and its thermal evaluation. Journal of Hazardous Materials, Vol180, 748-752.
- [4] Mishra S.C. and satpathy A., (2003) Plasma spraying of red mud-fly ash mixture on metals: an experimental study. Presented in National Conference on Materials and Related Technology, TIET, Patiala, India, http://dspace.nitrkl.ac.in/dspace/bitstream/2080/315/1/ALOK-NCMRT-2003.pdf.
- [5] Panagopoulos C.N., Georgiou E.P., Gavras A.G (2009) Composite zinc-fly ash coating on mild steel. Surface & Coatings Technology, 204, 37-41, doi:10.1016/j.surfcoat.2009.06.023
- [14] ASTM C633-01 (2008) Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings. ASTM International, DOI: 10.1520/C0633-01R08.
- [15] Mitta K. L. (1995) BOOK: Adhesion measurement of films and coatings. VSP, Netherlands, ISBN 90-6764-182-0, 15-40.
- [16] Joseph R. Davis (2004) Handbook of thermal spray technology. ASM International, Thermal Spray Society Training Committee, 49-53.
- [17] Ctibor P. et al (2007) Plasma Sprayed Ceramic Coatings without and with Epoxy Resin Sealing Treatment and Their Wear Resistance. Wear, volume 262, 1274-1280.