

## Improvement of Mild Steel Surface Properties by Fly-ash + Quartz + Illmenite Composite Coating

Ajit Behera\*, S. C. Mishra

Department of Metallurgical & Materials Engineering, National Institute of Technology,  
Rourkela-769008, Odisha, India

### ABSTRACT

Plasma spray of fly-ash composite gives a stable improvement of surface properties for industrially used materials/equipments, which has become a major technological research area encompassing studies in a broad range from medical field, chemical field to ceramic field. In the present investigation, coatings are developed on mild steel 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 6.56 MPa was found between coating and mild steel substrate. The adhesion strength was found to be higher than that of other fly-ash composite coatings. 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.

**Keywords:** Composite, fly-ash + quartz + illmenite, mild steel, plasma spraying, power level

\***Author for Correspondence** E-mail: [ajit.behera88@gmail.com](mailto:ajit.behera88@gmail.com)

### 1. INTRODUCTION

Nowadays, a considerable emphasis has been placed on the processing of low-grade ore minerals through plasma spray technologies. In the present investigation, the suitability of plasma spray coating by using fly-ash composite onto a mild steel substrate has been demonstrated. Fly-ash is the waste generated by iron and steel industry in ever-increasing quantities and is posing an environmental threat and also a disposal problem since adequate avenues for its utilization are not currently available. Somehow, fly-ash composite has a number of useful applications, which are described by Satapathy et al., Narayan et al., and Sudarshan et al. [1–3]. Fly-ash is available free of cost and presents itself as an attractive feed stock material for plasma spray coating, provided that spray

performance and coating performance are acceptable. In plasma spray coating, the materials in the form of powder mixture (Fly-ash + quartz + illmenite) are injected into a high-temperature plasma flame. The powder mixture (size from 40 to 100  $\mu\text{m}$ ) is then rapidly heated and accelerated to a very high velocity by the plasma flame, which impacts the surface of the substrate material in the form of molten or semi-molten state and very quickly cools to form a high-quality coating [4–7]. In this context, Mishra has investigated the spray of fly-ash composite on different types of metal by plasma spraying [8–12]. In the present experiment, plasma spray of fly-ash + quartz + illmenite composite coatings was developed on mild steel substrates. The surface and cross-sectional morphology and the crystal structure of these coatings were studied. In addition, the adhesion strength and

coating deposition efficiency were thoroughly examined. It should also be noted that this research investigation also aims at expanding the technological application of massive fly-ash produced in industries. It is believed that fly-ash might possibly replace other expensive commercial oxides in the production of composite coating materials.

## **2. EXPERIMENTAL PROCEDURE**

The substrate material used in this study was a commercially supplied mild steel alloy sheet. Its percent weight chemical composition was: 99.43% Fe, 0.07% C, 0.02% Si, 0.36% Mn, 0.01% P, 0.01% S, 0.01% Cu, 0.04% Al, 0.02% Cr and 0.02% Ni. The coating material is a composite of fly-ash, quartz and illmenite mixture. Fly-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 h to get a homogeneous mixture. This mixture used as feed stock for plasma spraying was first sieved and from 100 to 40  $\mu\text{m}$  powder size are separated out. The substrate materials have dimensions of 1 in diameter and 3 mm thickness. The substrate was grit blasted at a pressure of 3  $\text{kg}/\text{cm}^2$  using alumina grit to make the surface roughness  $\sim 5.00$  Ra. After grit blasting, substrate surfaces were cleaned with acetone and then plasma spraying was carried out immediately. The coating process was made 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 11 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 was directed perpendicular to the plasma flow and parallel to the torch trajectory. The torch was operated using argon (Ar) and Nitrogen ( $\text{N}_2$ ) plasma mixture gas. The major subsystems of the setup included the power supply, powder feeder, plasma gas supply, plasma spraying torch, and substrate to torch distance controller, cooling water and spray booth. For cooling the system, a water cooling system was used which was a four-stage closed-loop centrifugal pump, regulated at a pressure of 10  $\text{kg}/\text{cm}^2$  supply. Operating parameters used for coating deposition are given in Table I. Flow rate of primary plasma gas (Ar) and secondary gas ( $\text{N}_2$ ) were kept constant. Powder feed rate, powder Size and torch to base distance (TBD) were varied with respect to increase in power level. The thickness of the coatings was measured by a traveling microscope on a polished cross section of the specimens. Surface and interface morphologies were studied using a scanning electron microscope (JEOL JSM-6480LV). The coating pull-out test was carried out on the specimen to evaluate the coating adhesion strength as per ASTM C633 [13]. Phase identification study was done by X-ray diffraction using a Phillips X-ray Diffractometer with Ni-filtered Cu  $K\alpha$  radiation.

**Table I:** Operating Parameters 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 (argon) 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 (SEM)

##### Study of Surface and Interface

The composite coating material and substrate-interface plays the most important role in the adhesion of coatings [14, 15], which is studied by scanning electron microscopy (SEM). The surface microstructure of composite coating at highest power level (18 kW and 21 kW) is shown in Figure 1(a) and (b). In both cases, the presence of cavitations in between splat is nearly same.

The surface morphology of the coatings cannot predict the interior structures. Thus, the polished cross sections of the samples were examined under SEM and are shown in Figure 2(a) and (b). From the micrographs, it is evident that the coating deposited at 18 kW shown in Figure 2(a) has more metallurgical bonding/mechanical interlocking than that of 21 kW deposited surface shown in Figure 2(b). So, it is concluded that the highest adhesion

strength occurs at 18 kW power level and with further increase in power level, there is no more increase in the adhesion strength.

#### 3.2. X-Ray Diffraction Analysis

Figure 3 represents a comparative XRD analysis to examine the presence of various phases in the raw fly-ash (bottom pattern) and in the resulting fly-ash + quartz + illmenite composite coatings at 15 kW (middle pattern) and 21 kW (top pattern).

The X-ray diffraction pattern exhibits distinct peaks which are assignable to the various metal oxide phases present, such as SiO<sub>2</sub>, TiO, Ti<sub>2</sub>O, TiO<sub>2</sub>, Ti<sub>3</sub>O<sub>5</sub>, Ti<sub>4</sub>O<sub>7</sub>, FeTiO<sub>3</sub>, Fe<sub>2</sub>TiO<sub>5</sub>, Fe<sub>3</sub>O<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub> and some other inter-metallic compound. The presence of crystalline phases like SiO<sub>2</sub>, TiO<sub>2</sub>, FeTiO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> along with additional phases such as Fe<sub>3</sub>O<sub>4</sub>, FeMnTi<sub>3</sub>O<sub>10</sub>, NiMn<sub>2</sub>O<sub>4</sub> and Al<sub>2</sub>SiO<sub>5</sub>, suggests that during plasma spraying at higher torch input power, some element combined with other element or transformed to its higher stable state.

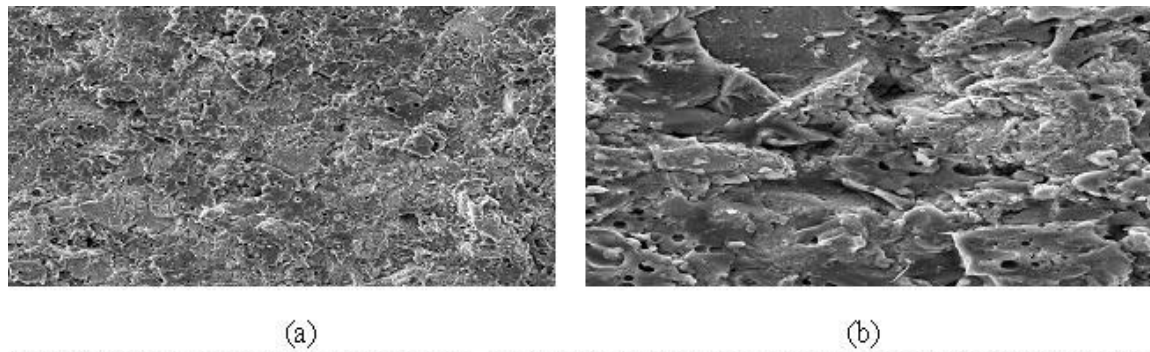


Fig. 1: SEM surface morphology of Fly-ash+quartz+illmenite Composite coated at (a) 18KW and (b) at 21KW on mild steel substrate.

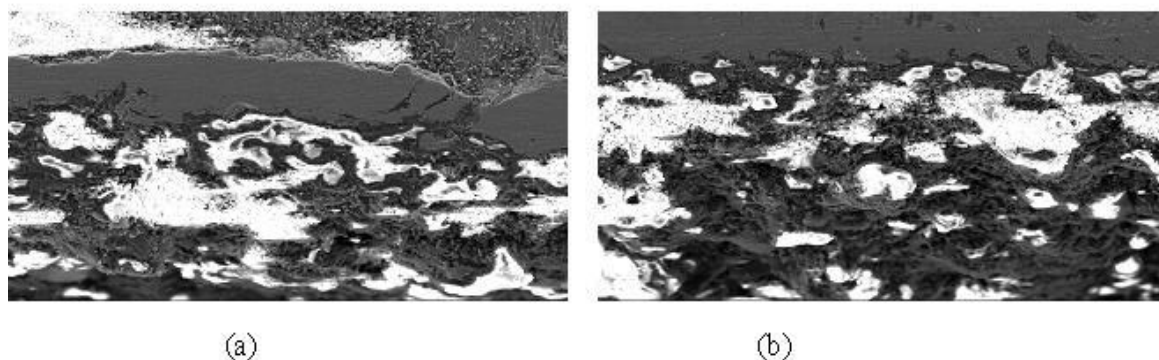


Fig. 2: SEM interface morphology of Fly-ash+quartz+illmenite Composite, coated at (a) 18KW and (b) at 21KW on mild steel substrate.

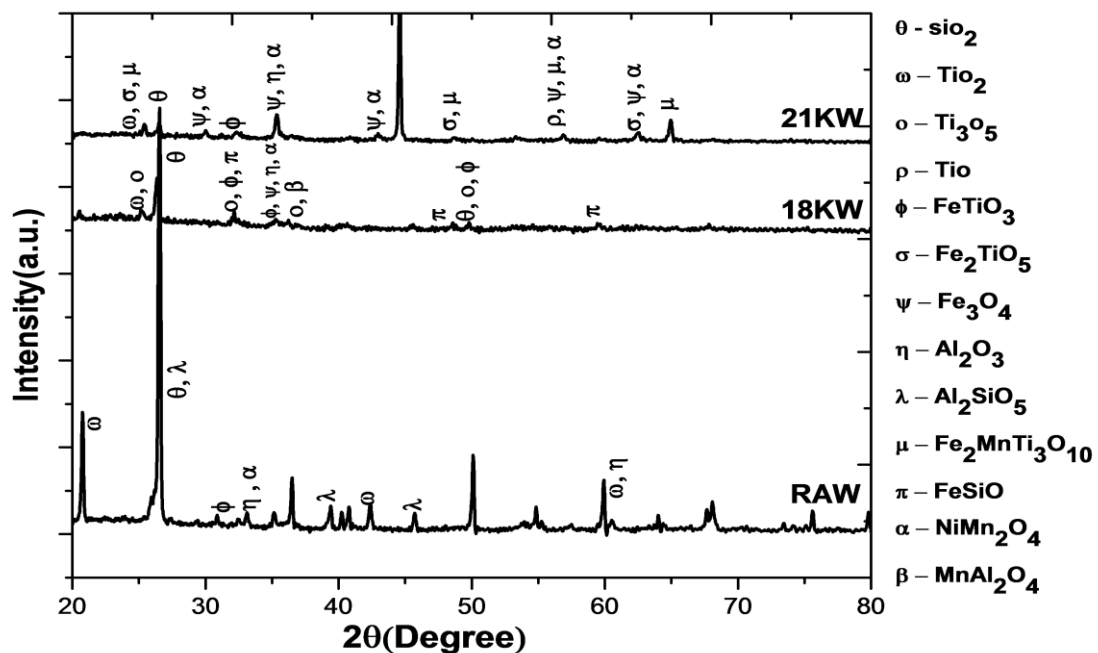
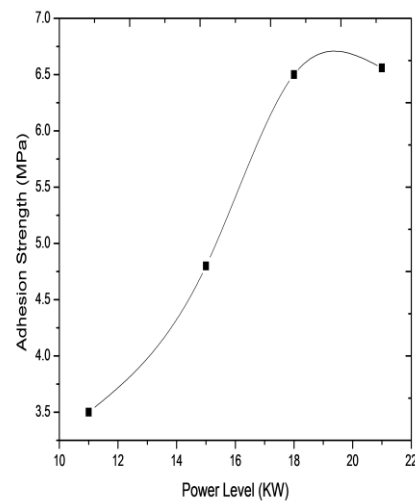


Fig. 3: X-ray Diffractogram of the Raw Fly-Ash Composite (Lower Pattern), Composite Coating on Mild Steel Substrate at 18 kW (Middle Pattern) and 21 kW (Upper Pattern).

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

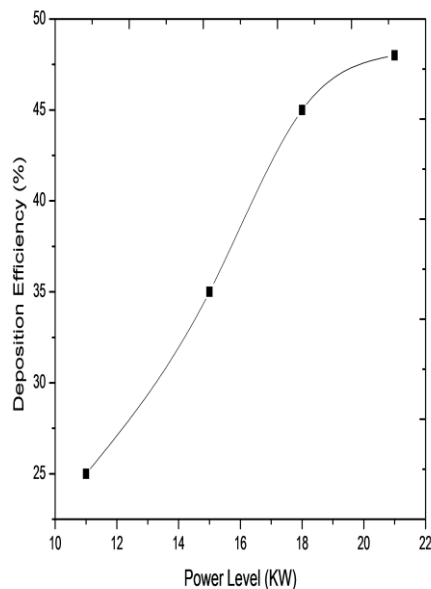
Figure 4 shows the interface bond strength of the coating, which is evaluated by the coating pull-out method. It is seen for all cases that fracture occurred at the coating-substrate interface. However, it has been stated by Lima and Trevisan that the fracture mode is adhesive; if it takes place at the 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 [16]. Adhesion strength increased up to power level 18 kW with a maximum value of 6.56 MPa adhesion strength and with further increase in the operating power level there is no increase in the adhesion strength. 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 a better mechanical interlocking of molten particles on the substrate surface leading to an increase in adhesion strength. But at much higher power levels, i.e., 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.



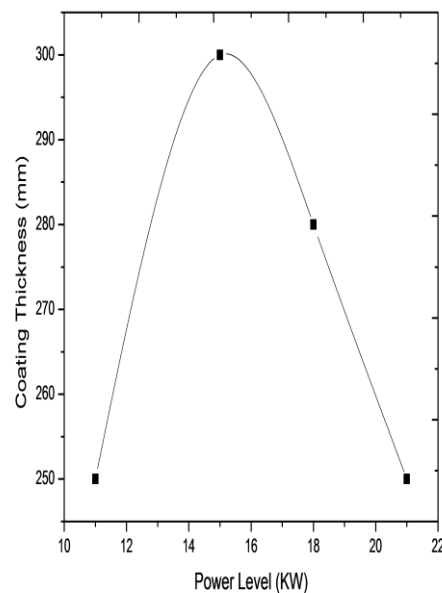
*Fig. 4: Adhesion Strength between Composite Coating Material (Fly ash + Quartz + Illmenite) and the Mild Steel Substrate with Increase in Power Level.*

Figure 5 shows deposition efficiency of fly-ash + quartz + illmenite composite on mild steel substrate. The deposition efficiency can be defined as the ratio of the mass of coating deposited on the substrate to the mass of the expended feedstock. In this investigation, the deposition efficiency gives a sigmoid-type evolution with the torch input power. As the power level increases, the net available energy in the plasma jet increases leading to a better in-flight particle molten state and hence, to higher probability for particles to flatten. The deposition efficiency reaches a plateau for the highest current levels due to the increasing plasma jet temperature which in turn, increases both the particle vaporization ratio and the plasma jet viscosity. It is found that the maximum deposition efficiency is 48% at 21 W power level.





**Fig. 5:** Deposition Efficiency of Composite Coating Material (fly ash+Quartz+ Illmenite) on Mild Steel Substrate with Increase in Power Level.



**Fig. 6:** Variation of Coating Thickness of Composite (Fly-Ash + Quartz + Illmenite) on Mild Steel Substrate with Respect to Power Level.

Figure 6 presents the dependency of adhesion strength on coating thickness. It indicates that with increase in torch input power, the thickness of the coating increases up to a certain point and with further increase in power level there is decrease in coating thickness because by gaining higher energy the composite powder becomes fully molten and spreads on the surface. The thickness of the coating varies between 250 and 300  $\mu\text{m}$  with change in operating power from 11 to 21 kW. At 15 kW power level, greater coating thickness of 300  $\mu\text{m}$  is obtained. Here there is increase in coating thickness with increase in coefficient of thermal conductivity, by decreasing thermal gradient between coating substrate. So, adhesion strength increases [17].

#### 4. CONCLUSIONS

Fly-ash composite can be efficiently used as a potential cost-effective material for deposition of plasma spray coatings on metallic substrates. Maximum adhesion strength of about 6.56 MPa was recorded for mild steel substrate. The adhesion strength was affected by the power level of plasma torch. A maximum 48% deposition efficiency was obtained at 21 kW power level. Operating power level also affected the coating deposition efficiency and morphology of the coatings. The coating thickness varied between 250 and 300  $\mu\text{m}$ .

## REFERENCES

1. Satapathy A., Sahu S. P. and Mishra D. *Waste Management & Research*. 2009. 1–7p.
2. Narayan H., Montano A. M., Hernandez M. L., et al. *Journal of Materials and Environmental Science*. 2012. 3(1). 137–148p.
3. Sudarshan and Surappa M. K. *Materials Science and Engineering: A*. 2008. 480(1-2). 117–124p.
4. Mishra S. C., Satapathy A. and Chaithanya M. *Journal of Reinforced Plastics and Composites*. 2009. 28(23). 2931–2940p.
5. Fauchais P. *Journal of Physics. D: Applied Physics*. 37. 2004. 86–108p.
6. Heimann R. B. *Plasma Spray Coating: Principles and Applications*. New York. WILEY-VCH. 2008. 8p. ISBN: 978-3-527-32050-9.
7. Davis J. R. *Handbook of Thermal Spray Technology*. Thermal Spray Society Training Committee. Materials Park, Ohio, USA. ASM Int. 2004. 5–10p.
8. Mishra S. C., Rout K. C., Padmanabhan P. V. A., et al. *Journal of Materials Processing Technology*. 2000. 102. 9–13p.
9. Mishra S. C., Das S., Satapathy A., et al. *Journal of Reinforced Plastics and Composites*. 2009. 28. 3061–3067p.
10. Mishra S. C., Praharaj S. and Satpathy A. *Journal of Manufacturing Engineering*. 2009. 4(2). 241–246p.
11. Mishra S. C. and Acharya S. K. *Journal of Reinforced Plastics and Composites*. 2007. 26. 1201–1202p.
12. Mishra S. C. and Satpathy A. *National Conference on Materials and Related Technology*. TIET, Patiala, India. 2003.
13. ASTM C633-01. *Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings*. ASTM International. 2008. DOI: 10.1520/C0633-01R08.
14. Mitta K. L. *Adhesion Measurement of Films and Coatings*. VSP, Netherlands. 1995. ISBN 90-6764-182-0, 15-40.
15. Davis J. R. *Handbook of Thermal Spray Technology*. ASM International. Thermal Spray Society Training Committee. 2004. 49–53p.
16. Lima C. R. C. and Trevisan R. E. *Journal Thermal Spray Technology*. 1997. 6. 199–204p.
17. Zhu D. and Robert A. M. *ASM International*. JTTEE5. 1999. 9. 175–180p.