

## Dependence of Adhesion Strength of Plasma Spray on Coating Surface Properties

Ajit Behera\*, S.C. Mishra

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

### ABSTRACT

*It is well known that thermal plasma spray coatings have diversified utilizations, but not vividly in use, mainly because of high cost of spray grade powder, for development of thick coating > 50  $\mu\text{m}$  on metal substrates. Present investigation aims at depositing plasma spray coating on metals using low-cost/waste materials. A mixture of fly-ash + 20 weight percent of Quartz + 20 weight percent of illmenite is used for coating on copper and mild steel substrates. The powder mixture is plasma sprayed by different operating power levels (11 to 21 kW) of the plasma torch. The coatings are characterized for their adhesion strength, porosity and deposition efficiency. It is observed that with increasing operating power level, the adhesion strength increases up to a certain power level, and then it remains nearly steady with further increase in torch input power. A maximum of 6.56 MPa adhesion strength is obtained on the coating developed on mild steel. It is observed that coating adhesion is more in case of mild steel than that of copper substrate. The amount of porosity in the coating decreases with increase in operating power level. Coating deposition efficiency also increases with increase in operating power of the plasma torch, up to a certain power level, and then becomes stagnant with further increase in input power. The substrate morphology of the coating observed under scanning electron microscope indicates the difference in particle size and inter-particle adhesion, which collaborate to the mechanical properties, i.e., adhesion strength and level of porosity.*

**Keywords:** Plasma spray coating, adhesion strength, porosity

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

### 1. INTRODUCTION

Coatings greater than 50 micron in thickness are used for a remarkable number of applications, viz., wear/erosion and corrosion resistance, thermal barriers, etc. [1]. Coating adhesion usually considers the result of a combination of three fundamental mechanisms, related to the nature of the interface bonding, i.e., via mechanical, chemical and physical forces [2].

A major challenge in development of plasma coating technology is to meet the requirement for new materials to sustain in progressively

more severe operating conditions. Different plasma spraying process variants allow their working with all kinds of materials, from low melting temperature, i.e., plastics to high melting temperature metals and ceramics. Plasma spraying is part of thermal spraying, a group of processes in which finely divided metallic and non-metallic materials are deposited in a molten or semi-molten state on a substrate [3]. Plasma spraying is one such route that involves projection of selected powder particles into the zone of high thermal density, where they are melted, accelerated and directed on to the substrate surface [4, 5]. Since a few decades, research has been

continuing for developing thermal plasma spray coatings by using different types of low-cost materials. It utilizes the exotic properties of plasma medium to effect physical, chemical or metallurgical reactions to produce new materials or impart new functional properties to conventional materials [6]. The chemical analysis of a mixture of fly-ash, quartz and illmenite shows that major constituent in these industrial wastage and low grade ore is iron oxide ( $\text{Fe}_3\text{O}_4$ ), titanium oxide ( $\text{TiO}_2$ ), silicon oxide ( $\text{SiO}_2$ ), aluminum oxide ( $\text{Al}_2\text{O}_3$ ), etc. Since all these are metal oxides, it was felt that the mixture of fly-ash + quartz + illmenite can possibly be spray coated. Since fly-ash, quartz and illmenite are available in plenty from industrial waste and low grade ore, the cost involvement will spring down. In 1995, Mishra and Ananthapadmanabhan used fly-ash as a coating material for plasma spray coating in their experiment [7] and the experiment is elaborated by fly-ash mixing with other industrial wastes as coating materials [8–10]. Mishra has also attempted to spraycoat raw fly-ash + illmenite mixture on metal substrate through plasma spraying [11]. He found that the adhesion strength was not increasing satisfactorily. Further, in 2009, Mishra repeated the plasma spraying process with fly-ash + quartz [12]. Here the problem was lower adhesion strength in both copper as well as mild steel substrate and deposition efficiency was also low.

Against this background, the present study is undertaken to increase adhesion strength with

increase in deposition efficiency and coating thickness. This investigation has been carried out to produce fly-ash + quartz + illmenite composite coating on metal substrates by conventional atmospheric plasma spray technique.

## 2. EXPERIMENTAL METHODS

Plasma spraying of fly-ash, quartz, illmenite mixture in 60:20:20 weight ratio was carried out on mild steel and copper substrates, having dimensions 1 in diameter and 3 mm thickness. The powders are mechanically milled in a FRITSCH-planetary ball mill for 3 hr to get a homogeneous mixture. The substrates were grit blasted at a pressure of  $3 \text{ kg/cm}^2$  using alumina grit to make the surface roughness  $\sim 5.00 \text{ Ra}$ . After grit blast, substrate surface was cleaned by acetone and spraying process immediately carried out. Spray coating was done using a 40 kW plasma spray system at the Laser & Plasma Technology Division, BARC, Mumbai. This is a typical atmospheric plasma spray system working in the non-transferred arc mode. The major subsystems of the set-up include the plasma spray torch, power supply, powder feeder, plasma gas supply, control console, cooling water and spray booth. A current-regulated DC power supply was used. The plasma input power was varied from 11 to 21 kW by controlling the gas flow rate and arc current. The powder was deposited at spraying angle of  $90^\circ$ . A four-stage closed loop centrifugal pump at a pressure of  $10 \text{ kg/cm}^2$  supplied cooling water to the system. A

plasma spray coating of fly-ash + quartz + illmenite (60:20:20 weight%) in the size range of 40–100  $\mu\text{m}$  was deposited over metal substrates. The adhesive strength of interface of the coating powder and the substrate plays an important role for plasma spray coating. In deciding on a particular coating material and method, generally the level of interface bond strength is estimated. Despite the fact that in most cases coating substrate interface bond strength is the main characteristic that proves the coating efficiency, the bond strength between the coating and the substrate depends not only on the bond character, the level of internal stresses and the dependence of coating thickness is also of great importance. The adherence strength of the coating is measured using coating pullout method [13]. The coating pull-out test was carried out using the set-up Instron 1195 at a crosshead speed of 1 mm/min. The test was performed as per ASTM-633. The morphology of coatings is observed using a JOEL T-330 scanning electron microscope. Operating parameters used for coating deposition are given in Table I.

### 3. RESULTS AND DISCUSSION

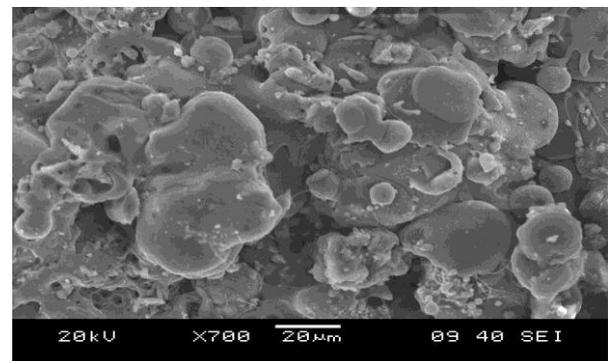
#### 3.1. Surface Morphology

The interface adhesion of the coatings depends on the coating morphology and inter-particle bonding of sprayed powders. The surface morphology of the coatings is examined under

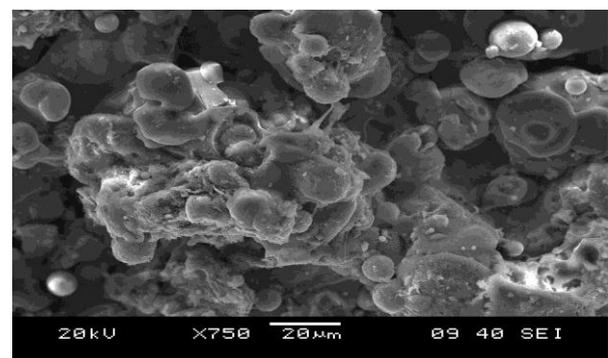
scanning electron microscope, shown in Figure 1.

**Table .I:** Operating Parameters during Deposition of Fly Ash+Quartz+ Illmenite Coatings.

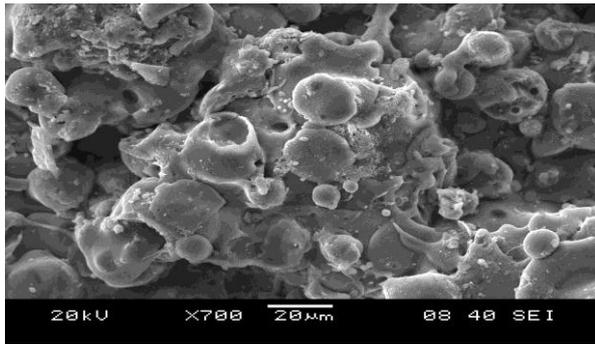
Operating Parameters	Values
Plasma arc current (amp)	260–500
Arc voltage (volt)	40–44
Torch input power (kW)	11,15,18, 21
Plasma gas (argon) flow rate (IPM)	28
Secondary gas (N <sub>2</sub> ) flow rate (IPM)	3
Carrier gas (Ar) flow rate (IPM)	12
Powder feed rate (gm/min)	15
Torch to base distance(TBD)(mm)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



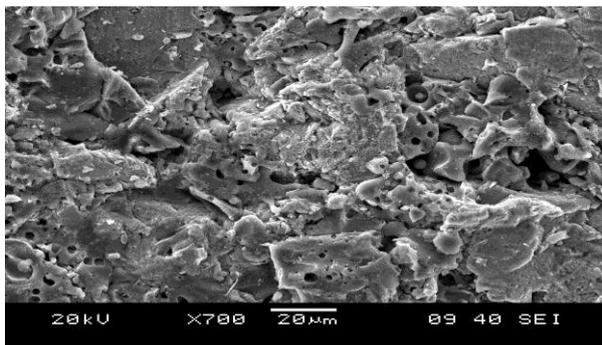
(a)



(b)



(c)



(d)

**Fig. 1:** Surface Morphology of Mild Steel Substrate at Different Power Levels  
(a) 11 kW, (b) 15 kW, (c) 18 kW, (d) 21 kW.

The coating deposited at 11 kW power level (Figure 1(a)) on mild steel substrate shows a uniform distribution of molten/semi-molten particles, which have agglomerated to form laths. More amount of cavitation's are observed at this lower power level. Some open pores are found on the inter-particle boundaries and at triple particle/grain junctions. These may have originated due to the inadequate flow of molten particles during their solidification [14]. The coating deposited at 15 and 18 kW (Fig 1(b) and (c)), forms a different morphology, i.e., some spheroidal splats are found, indicative of complete melting of particles during in-flight traverse

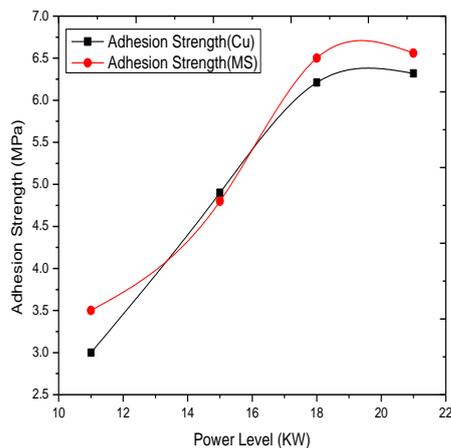
through plasma jet. Less cavitation is observed in inter-granular boundaries. In case of coating made under 21 kW power level (Figure 1(d)), the particles get more thermal energy, so during solidification from molten state, they agglomerate to form splats, i.e., flattened region. Here very less amount of cavitation is observed. This may be the reason for better interface adhesion of the coating onto the substrate, leading to increase of adhesion strength. This morphology also shows a uniform distribution of molten/semi-molten particles; hence no more increase in adhesion strength.

### 3.2: Influence of Different Parameters with Adhesion Strength

The variation of adhesion strength with operating power level for mild Steel and copper substrate is shown in Figure 2. It is found that with increase in operating power level, there is an increase in adhesive strength up to a certain level of operating power of the torch. It is found that for different types of substrate, the magnitude of adhesion strength differs. For mild steel, the strength has varied from 3.5 to 6.56 MPa, in mild steel, a maximum of 6.56 MPa at the power level of 21 kW, for copper substrate this value ranges from 3.00 to 6.32 MPa. Deposition efficiency improves to a certain extent with increase in arc power since it is associated with an enhanced particle melting [15].

Again by comparing adhesion strength of mild steel against copper (Figure 2), it is found that

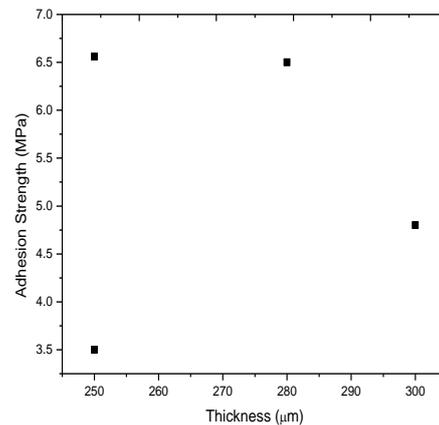
at 11 kW, 18 kW and 21 kW power levels, adhesion strength of mild steel is greater. Coating made after 21 kW power level, the adhesion strength, of mild steel and copper substrates tends to decrease.



**Fig. 2:** Comparison of Adhesion Strength of Mild Steel and Copper with Respect to Power Level.

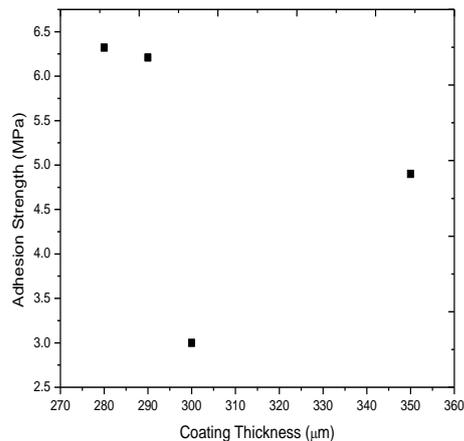
The variations of adhesion strength with coating thickness are shown in Figure 3(a, b). It is found that in both substrates, the adhesion strength increases up to a certain coating thickness and further increase in thickness adhesion strength of the coating tends to decrease. Thickness of plasma sprayed coatings of few hundred microns is sufficient in many cases to protect engineering surfaces. Because of higher residual stress states in thick plasma sprayed coatings, the security of the bond to the substrate reduces with the increasing thickness of the coating. In mild steel, the highest adhesion strength 6.56 MPa is observed at 250  $\mu\text{m}$  thicknesses and it decreases to 4.8 MPa at 300  $\mu\text{m}$  thickness

(Figure 3 (a)). In case of copper at maximum of 6.32 MPa, adhesion strength occurs at thickness of 280  $\mu\text{m}$  and shifts to 3.00 MPa, at a thickness value to 300  $\mu\text{m}$  (Figure 3 (b)). In case of copper, it observed that increasing thickness from 300 to 350  $\mu\text{m}$ , there is an increase in adhesion strength from 3 to 4.9 MPa. When there is increase in power level from 11 to 15 kW. Coating thickness might be playing a role in decreasing thermal gradient between coating and substrate, which is the adhesion strength [16, 17].



**Fig. 3:** (a) Variation of Adhesion Strength with Coating Thickness (Mild Steel).

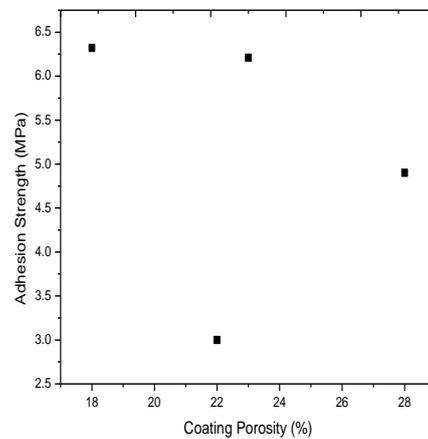
In case of mild steel, adhesion strength increases with decreasing coating thickness, by increasing power level from 15 to 21 kW. But the only exception is adhesion strength at 11 kW. Here, even if there is less thickness, the adhesion strength is lower. So it appears that 11 kW power level is not sufficient to transfer heat from coating surface to substrate for good adhesion, although there is increase in thickness [16, 17].



**Fig. 3:** (b) Variation of Adhesion Strength with Coating Thickness (Copper).

The adhesive strength is also affected by coating porosity. The extent of porosity in the sprayed coating may be controlled either through the use of the proper particle size of spray grade powders, by introducing the powder far downstream in the plasma jet, or by using a low-energy plasma. The pore interconnectivity in the structure also influences the cohesion of the deposit [18]. It has been reported that the thicker topcoat is mechanically weakened with increasing pores and residual stresses. It is known that the higher hardness, lower porosity and lower surface roughness can be obtained at lower coating thickness [19–22]. The dependency of adhesive strength with coating porosity of copper substrate is described in Figure 4. It reveals that with decrease in coating porosity, adhesion strength increases. In case of copper substrate, coating porosity decreases from

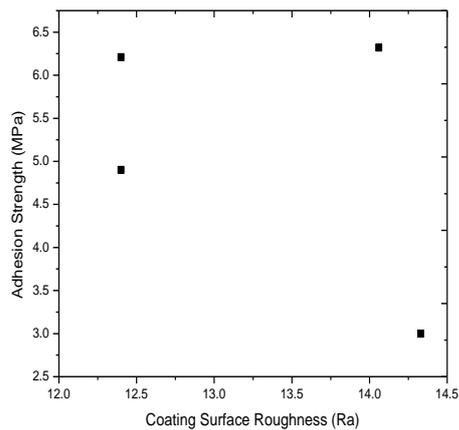
28% to 18% with increase in adhesion strength from 4.9 to 6.32 MPa.



**Fig. 4:** Variation of Adhesion Strength with Coating Porosity for Copper Substrate.

It is known that the rougher coating surface causes a higher mechanical anchorage. In our experiment, the effect of surface roughness on adhesion strength is plotted in Figure 5. It is found that when there is decrease in coating surface roughness from 14.33 Ra to 12.4 Ra, then adhesion strength increases from 3 to 4.9. But in case of 18 and 21 kW, the bond strength increases with surface roughness, only due to high power level, which transfers sufficient energy to fasten at the interface. Therefore, as per the kinetics of thermal spraying, the surface roughness has little effect on the interface bonding and in most cases the mechanical anchorage in micron/millimeter length-scale is not a dominant mechanism for coating build-up [23]. However, a new problem on sample preparation for lower

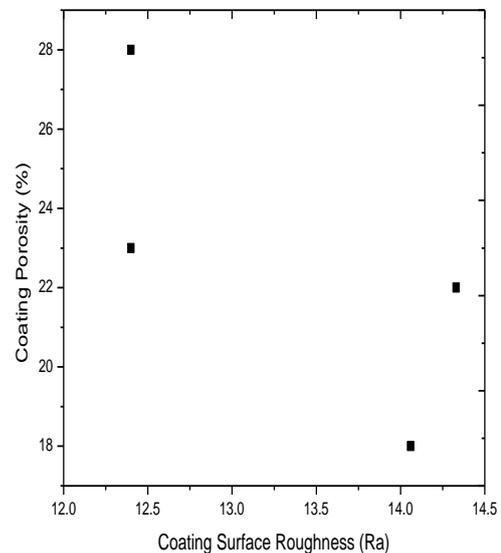
surface roughness appears, i.e., not only more care but also longer time has to be spent on it. Furthermore, it is very hard to prepare some kind of coating samples with very low roughness [24].



**Fig. 5:** Variation of Adhesion Strength with Coating Surface Roughness for Copper Substrate.

The inter-dependence of surface roughness and porosity is plotted in Figure 6. From the figure it can be visualized that when there is increase in power level from 11 to 15 kW, the surface roughness decreases but porosity increases. It is due to the presence of inter-connecting pores inside the surface of the coating. At 18 kW power level, coating porosity decreases due to higher energy. At 21 kW power level, the porosity decreases but with increase in coating surface roughness. It is due to large amount of inter-splat spacing on the surface. It is necessary that the composition and particle size of the sprayed powder employed should have adequate flow properties so as to stack into various pores and

crevices of the substrate in order to reduce the porosity and roughness of the substrate [25].



**Fig. 6:** Variation Coating Surface Roughness with Coating Porosity for Copper Substrate.

#### 4. CONCLUSIONS

Quartz, illuminate mixed with fly-ash, was found to be a suitable material for depositing thermal spray coating on metals. These coatings have wide range of applications including thermal barrier and chemical barrier. It is observed that the adhesion strength is affected widely with operating conditions, coating thickness and porosity and found to vary between 3.00 and 6.56 MPa. A maximum coating adhesion strength of 6.56 MPa is observed on mild steel substrate coated at the power level of 21 KW. Coating surface roughness and porosity have some effect on coating deposition and adhesion.

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