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Investigation on Composite Coating of Low Grade Minerals

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ABSTRACT: In this investigation, coatings of fly ash (an industrial waste) mixed with illmenite (a low grade ore mineral) have been deposited on mild steel and copper substrates using conventional atmospheric plasma spray technique. Micro-hardness measurement, phase composition analysis, coating porosity measurement, and surface and interface morphology are studied to characterize the coatings.

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INTRODUCTION

A UNIQUE WAY to tailor the surface properties of a component, suitable to a specific operating environment without sacrificing the bulk characteristics of the structure, is by deposition of a surface coating. Thermal spraying processes have been known for producing hard overlay coatings, especially ceramic coatings, to improve tribological properties [1,2]. Amongst the thermal spray techniques, conventional atmospheric plasma spray technique is widely used to develop coatings of ceramic materials [3–6]. In this work plasma spray deposition of alumino-silicate composite coatings onto metal substrates is carried out using industrial waste, i.e., fly ash with addition of illmenite, a titanium bearing ore mineral; the aim being to reduce the cost of the spray grade powders (i.e., raw material) required for coating. Generally metallic bond coat is provided onto the substrate for better interface adherence of ceramic coating to overcome the poor interface mechanical properties arising due to the mismatch of thermal expansion coefficients of ceramics and metals [7,8]. An improvement of coating properties when pre-mixed metal–ceramic powders are used (instead of using a bond coat) has already been reported [9]. It is well established that addition of titania to alumina provides dense coating and better adherence on substrate [10]. Therefore illmenite, a low grade ore mineral, plentily available in India, is pre-mixed with fly ash so as to further reduce the raw material cost. Using an atmospheric plasma spraying system coatings are deposited on metal substrates at different operating power levels of the plasma torch and then characterization of the coatings is carried out.

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Deposition efficiency is an important factor that determines the techno-economics of the process, so it is evaluated for the coatings deposited within the scope of this investigation. In view of tribological applications, coating hardness is one of the most required mechanical properties [11]. Coating porosity is measured by image analysis technique. For ascertaining the phases present and phase changes/transformation taking place during plasma spraying, XRD analysis is made. Coating surface and interface morphology is studied with scanning electron microscope.

EXPERIMENTAL DETAILS

Fly ash–40% illmenite are mixed in a planetary ball mill for 3 h to get a homogeneous mixture. The particle size of the powders, in the range of ~40 to ~100 μm is selected for coating deposition. Commercially available copper and mild steel are chosen as substrate materials. The circular disc specimens of 1 in diameter and 3 mm thickness are grit blasted at a pressure of 3 kg/cm^2 using alumina grits. The stand-off distance in shot blasting is kept between 120 and 150 mm to get an average substrates surface roughness of about 6.8 μm. The grit blasted specimens are cleaned with acetone and spraying is carried out immediately after cleaning. Conventional 40 kW atmospheric plasma spray (APS) system is used. The plasma input power is varied from 11 to 21 kW by controlling the gas flow rate, voltage and the arc current. The operating parameters during coating deposition are shown in Table 1.

Deposition efficiency is evaluated by weighing method [12] which is a widely accepted method;

$$\eta = \left(\frac{G_c}{G_p}\right) \times 100\% \tag{1}$$

where η is the deposition efficiency, G_c is the weight of coating deposited on the substrate, and G_p is the weight expended feedstock.

Micro-hardness measurement is done with Leitz micro-hardness tester using 0.4903 N load. The porosity is measured on the polished cross-section of the coatings using image analysis technique. X-ray diffraction is used to identify the different (crystalline) phases present in the coatings. XRD analysis is done using Ni-filtered Cu-KM radiation. The surface morphology of eroded coatings is investigated with JEOL JSM-6480 LV scanning electron microscope in secondary electron imaging mode.

Table 1. Operating parameters of the plasma spray unit.

Operating parameters	Values
Plasma arc current (amp)	80–500
Arc voltage (volt)	40–45
Plasma gas (Argon) flow rate (Lpm)	28
Secondary gas (N_2) flow rate (Lpm)	3
Carrier gas (Argon) flow rate (Lpm)	12
Powder feed rate (g/min)	15
Torch to base distance TBD (mm)	100

RESULTS AND DISCUSSION

Coating Deposition Efficiency

Figure 1 shows the variation of deposition efficiency of fly ash-illmenite coating with operating power level. It is interesting to note that the deposition efficiency is increased in a sigmoidal fashion with the torch input power which reveals that efficiency of coating deposition is significantly influenced by the input power of the torch. Plasma spray deposition efficiency of a given material depends on its melting point, thermal heat capacity and particle size of the powder, etc. At lower input power to the plasma torch, the plasma jet temperature is not high enough to melt the entire feed powder (particles) that enter the plasma jet. As the power is increased, the average plasma temperature increases, thus melting a larger fraction of the powder, etc. The spray efficiency therefore increases with plasma power. However, beyond a certain power level of plasma, the temperature of the plasma becomes high enough leading to vaporization/dissociation of the powder particles. Thus there is not much improvement, i.e., increase of deposition efficiency and coating thickness as well. This tendency is generally observed in plasma spray coatings. However, the plasma operating power above which the efficiency decrease depends on the chemical nature of the feed material, i.e., powder and its particle size, thermal conductivity, phase transformation, etc.

Coating Hardness

Microscopic observation revealed three distinct different visible regions/phases namely gray, dark and spotted/mixed. Micro-hardness measurement carried out on these optically distinguishable phases with a Leitz Micro-Hardness Tester using 0.4903 N load, is presented in Figure 2. The results show that these three structurally different phases bear three different ranges of hardness which is due to different phases present/formation

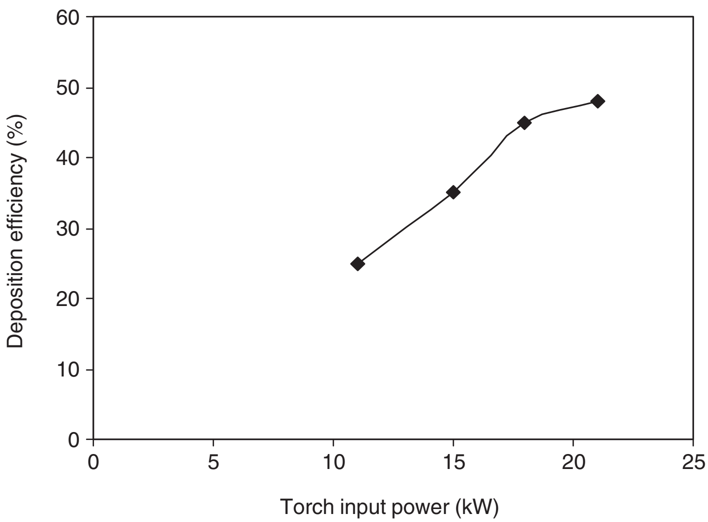

Figure 1. *Variation of deposition efficiency of fly ash-illmenite coating.*

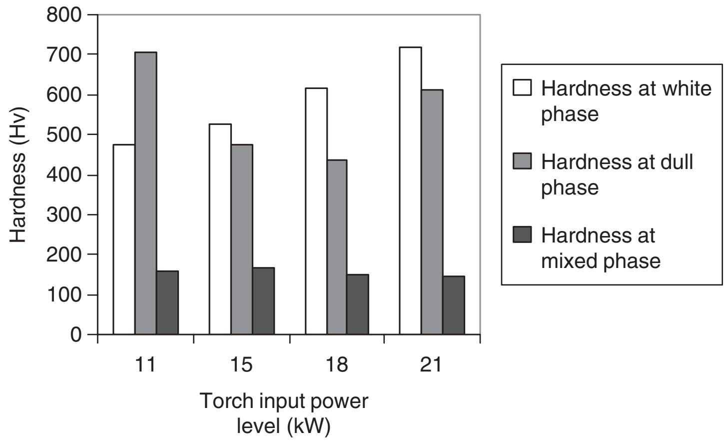

Figure 2. *Hardness on the different phases in the coating.*

during coating deposition, which is clear from X-ray diffraction analysis. High hardness values may be due to the presence of different phase forms (i.e., allotropic transformations) of alumina phase.

Low hardness values may be of the titania phases. With increase in power level there is an increase in hardness values of some phases, which may be due to the formation of different allotropic forms and their composition variations during spray deposition.

Coating Porosity

Porosity measurement is done using the image analysis technique, shown in Figure 3. It is observed that the amount of porosity is more in the case of the coatings made at lower (11 kW) and higher (21 kW) power levels. However, porosity is minimum for the coatings deposited at 18 kW. The increased amount of porosity may be the reason for low adhesion strength of the coatings deposited at low and at high power level.

XRD Phase Composition Analysis

The XRD of the feed material shows the presence of SiO_2, $FeTiO_3$, etc. phases. Coating made at lower power level, i.e., at 11 kW shows the presence of M- Al_2O_3, SiO_2, $FeTiO_3$, Ti_2O_3 and some new phases such as TiO and mullite. With increase in power level the amount of inter-oxide phases, i.e., TiO_3, Ti_2O_5 and mullite are found to increase. It may be due to the availability of high temperature which has accelerated the phase transformation, with increase in torch input power during coating deposition. Generally Al_2O_3 transform to different allotropy phase forms and TiO_2 reduces to Ti_3O_5, Ti_2O_3, Ti_2O, TiO depending on enthalpy/environment and transformation conditions [13,14]. During plasma spraying the different phase transformations are taking place, so it is expected that a ceramic composite coating from low grade materials could be made which can have better wear resistance properties.

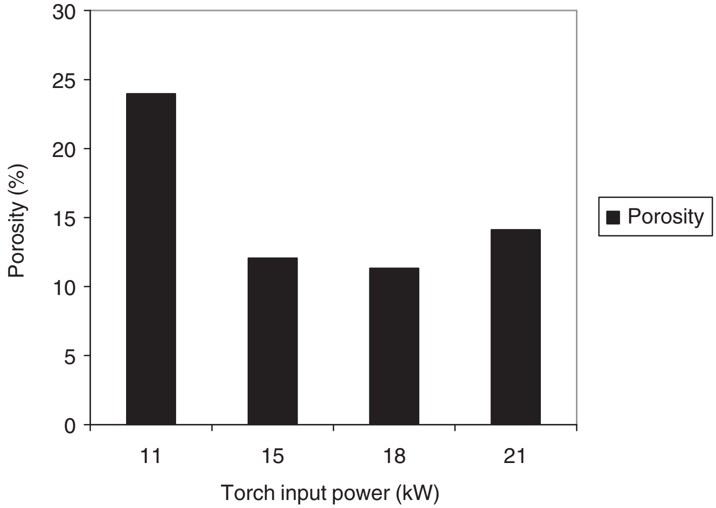

Figure 3. *Variation of coating porosity of fly ash-illmenite coating with torch input power.*

Surface Morphology

Surface morphology of fly ash-illmenite coating eroded at different impact angles are shown in Figure 4. It can be seen from the Figure 4(a) and (b) that when the erodent impacted at 90°, at any point the crack is initiated, spreads in all directions along the splats/grains. When the impact is 45°, Figure 4(c) and (d), crack propagation is mainly at grain/splat boundaries. Load is transferred to lower layer, and cracks have originated and spread over that layer instead of the top layer where it is impacted; this may be due to the dominancy of the tangential force exerted by the impinging particles/erodent. A similar type of structural evidence has also been observed by Westergard et al. [15]. They have reported that at 45° angle of impact of the erodent, the erosion rate is higher due to higher amount of plastic deformation. At an impact angle of 30° Figure 4(e) and (f), chipping away of layers occurs and hence more cavities are formed. Some cracks are observed and appear to have originated and spread along grains/splats boundaries. The detailed erosion wear mechanisms studies of these coatings are in progress.

CONCLUSIONS

From this study it is found that fly ash with low grade ore mineral (illmenite) additions can be used to provide plasma spray ceramic composite coating on metal substrates. Inter-oxide transformations of the raw materials do occur and their amount varies with operating power of the plasma torch which affects the coating hardness. The operating power level of the plasma torch affects the coating morphology and mechanical properties of the coating. The angle of impact plays a major role in crack development, propagation and material removal from the coating surface.

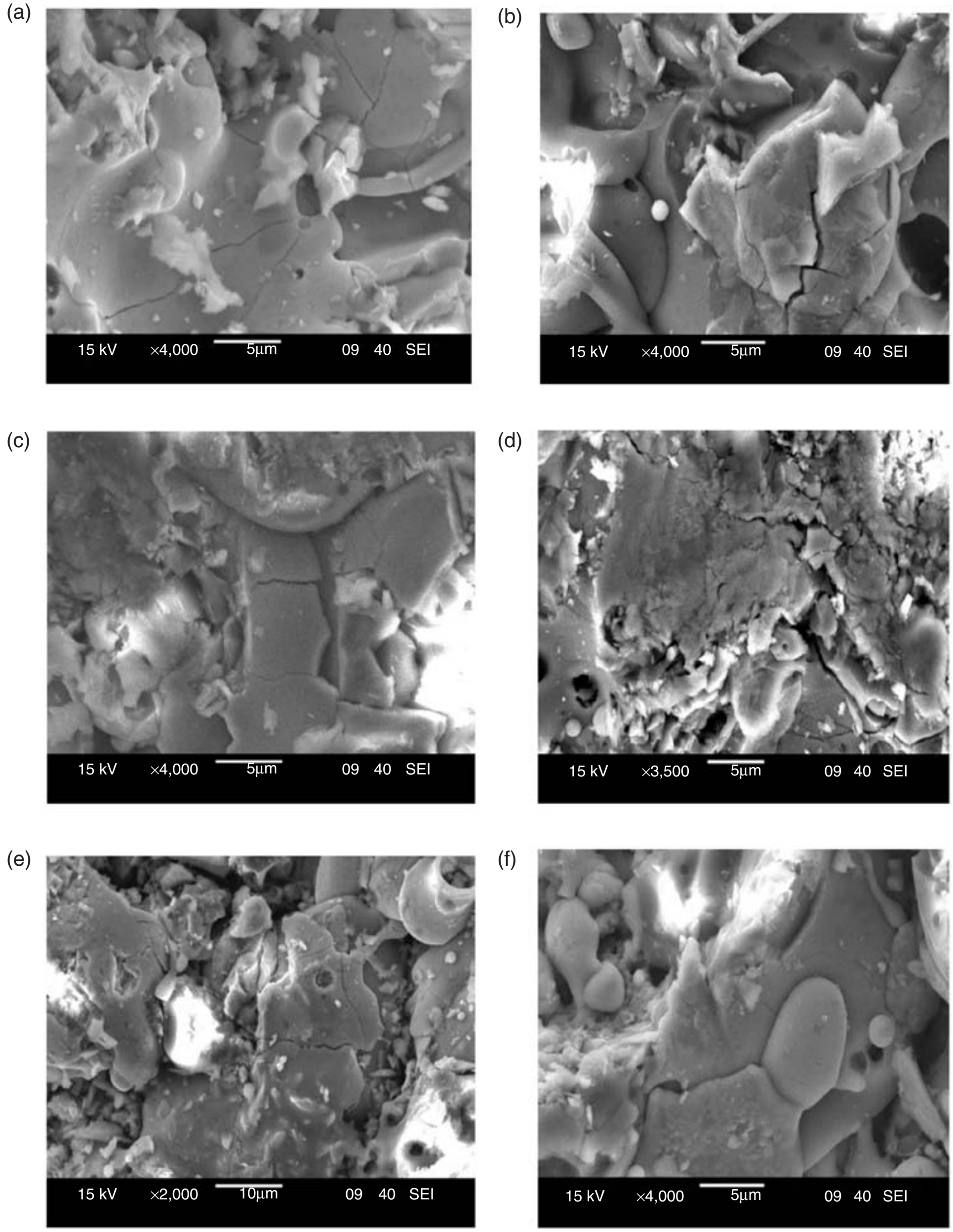

Figure 4. *Micrographs of eroded surfaces of coatings deposited at: (a) 11 kW and (b) 18 kW for 90° angle of impact; (c) 11 kW and (d) 18 kW for 45° angle of impact; and (e) 11 kW and (f) 18 kW for 30° angle of impact.*

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