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Research Article

Evaluation and Characterization of Plasma Sprayed Cu Slag-Al Composite Coatings on Metal Substrates

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Copper slag is a waste product obtained during matte smelting and refining of copper. The present work explores the coating potential of copper slag by plasma spraying. This work shows that copper slag is eminently coatable. An attempt has been made in the present investigation to use the composites coatings of copper slag and Al powder in suitable combination on aluminium and mild steel substrates in order to improve the surface properties of these ductile metal-alloy substrates. When premixed with Al powder, the coating exhibits higher interfacial adhesion as compared to pure copper slag coatings. Maximum adhesion strengths of about 23 MPa and 21 MPa are recorded for the coatings of copper slag with 15 wt% of Al on aluminium and mild steel substrates, respectively. The input power to the plasma torch is found to affect the coating deposition efficiency and morphology of the coatings. It also suggests value addition of an industrial waste.

1. Introduction

Among different surface modification techniques, plasma spraying stands out as one of the most versatile and technologically sophisticated thermal spraying techniques [1–4]. The advantages of plasma spraying include formation of ceramic microstructures with fine, equiaxed grains without columnar defects, deposition of graded coatings with a wide compositional variety, formation of thick coatings with only modest investment in capital equipment, and design capability for free-standing thick forms of monolithic and mixed ceramics in near-net shape configuration. Plasma spray coating is a typical thermal spraying process that combines particle melting, quenching, and consolidation in a single operation. It utilizes the exotic properties of the plasma medium to process different materials. But high cost of spray grade powders limits the adoption of this technique leading to a scope for research on new cost-effective and cheap materials suitable for plasma spray coating.

In thermal plasma, it is possible to spray all kind of metallic and nonmetallic materials like metal oxides, carbides, nitrides, and silicides, and so forth [5–9]. Many of these conventional coating materials are relatively expensive, to the extent that cost of spray-grade powders alone can account for 50–60% of the cost of operating plasma spray unit. In recent years, although a large number of investigations have been carried out on production of ceramic coatings using these metal oxides, little efforts have been made in plasma processing of cheap and naturally occurring materials/minerals [10].

Copper slag is such an industrial waste obtained during matte smelting and refining of copper. The common management options for copper slag are recycling, recovering of metal, production of value added products such as abrasive tools, roofing granules, cutting tools, tiles, glass, road-base construction, railroad ballast, and asphalt pavements. One of the greatest potential applications for reutilization of copper slag is in cement and concrete industries. Many researchers have investigated its utility in the production of cement, mortar, and concrete as raw materials for clinker, cement replacement, coarse and fine aggregates [11, 12]. This provides potential environmental as well as economic benefits for all related industries, particularly in areas where a considerable amount of copper slag is produced. Despite increasing rate of reutilization of copper slag, the huge amount of its annual

Table 1: Operating parameters for coating deposition.

Parameter	Operating range
Operating power	10–25 kW
Arc current	400–600 A
Primary Plasmagen gas (argon) flow rate	40 Lpm
Secondary Plasmagen gas (helium) flow rate	8 Lpm
Carrier gas (argon) flow rate	10 Lpm
Nozzle to substrate distance (stand-off distance)	100 mm

production is disposed in dumps or stockpiles to date. Recently, while considerable emphasis has been placed on the processing of low-grade minerals through thermal spray techniques, fly ash was found to be a cost-effective substitute for conventional extenders in high performance industrial coatings [13]. The development of plasma technology, especially plasma processing, has been commercially successful mostly in the field of spray coating. Today, plasma spray coatings find wide applications for improving wear, thermal, and corrosion resistances of various machine components. Ceramic coatings on relatively ductile metal-alloy substrates behave in a complex way under static mechanical loads and/or thermal cycling conditions and hence result in poor interfacial mechanical properties. Recent studies have shown improvement of coating properties with premixed metal—ceramic powders [14–16]. In the view of this, the present study explores the coating potential of Cu Slag-Al composite for different engineering and structural applications.

2. Experimental

The raw material (copper slag) is collected from the plant site of Hindustan Copper Limited, Ghatsila, located in the eastern part of India. These slag particulates are sieved to obtain a particle size in the order of 50 μm. The composition of copper slag used in this work is found to be 35.3% Fe_2O_3, 36.6% SiO_2, 10% CaO, 8.1% Al_2O_3, 0.37% CuO, 4.38% MgO, 0.47% Na_2O, 3.45% K_2O, 0.12% PbO, and 0.97% Zn. Coating deposition is carried out using a 80 kW plasma spray system (M/s metallization make) at CSIR-IMMT, India. This is a typical atmospheric plasma spray system working in the nontransferred arc mode. The major subsystems of the setup include the plasma spray torch, power supply, six-axis robot, mass flow controller, powder feeder, plasma gas supply, control console, cooling water, and spray booth. A current regulated DC supply is used. A four-stage centrifugal pump at a pressure of 10 kg/cm^2 supplies cooling water for the system. Argon and helium taken from normal cylinders at an outlet pressure of 4 kg/cm^2 are used as plasma gas and carrier gas, respectively. Plasma sprayed copper slag coatings are thus deposited over aluminium and mild steel substrates of dimensions 60 × 20 × 3 mm. Before deposition, all the substrates are sand blasted to get surface roughness of about 4–6 μm. The powder feed rate is kept constant at about 20 gm/min. The operating parameters used in the experiments are presented in Table 1.

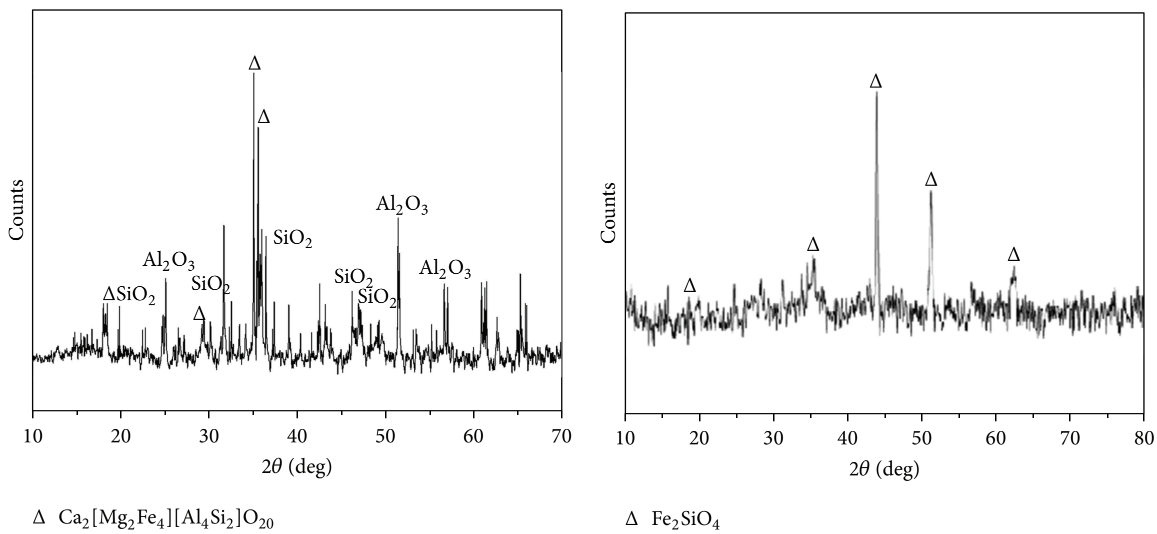
3. Results and Discussions

3.1. Phase Identification Analysis Using XRD. The X-ray diffraction patterns were recorded using a diffractometer (PAnalytical, X'Pert Pro) equipped with CuKα radiation. Figure 1 depicts the effect of plasma spray process on the X-ray diffraction patterns of raw copper slag powder and the copper slag coating. The pattern of raw copper slag indicates the presence of a number of phases among which Fe_2O_3, SiO_2, CaO, Al_2O_3, and MgO are the major matrixes. The diffraction pattern is well matched with the JCPDS file number 41-1483 which corresponds to a complex of all the said phases, that is, $Ca_2[Mg_2Fe_4][Al_4Si_2]O_{20}$. This is naturally expected as raw copper slag is a waste product from alumina ores via a high temperature process. However, the X-ray diffraction pattern of copper slag coating is well matched with the standard pattern of iron silicate (Fe_2SiO_4: JCPDS no. 83-2074), which indicates phase transformation from original phases like SiO_2, Fe_2O_3, and so forth.

3.2. Surface Morphology. Figures 2(a)–2(d) represent the surface microstructures of copper slag-Al composite coatings obtained from FESEM at different power levels. From the micrograph, splat boundaries of the molten and semimolten particles can be easily distinguished which indicates that the powder is spherodized during interaction with plasma plume. After impingement on the substrate, molten particles form splats and solidify. The morphology of coatings reveals regions of fully molten ceramic particles and in some cases microcracks are observed. These cracks seem to relieve thermal stresses during coating formation and fillers like aluminium seem to bridge the different phases. With addition of Al, coating seems to be homogeneous and uniformity in particle distribution along the coating surface is found to have improved. Typical micrograph of this coating at higher magnification is illustrated in Figures 3(a) and 3(b).

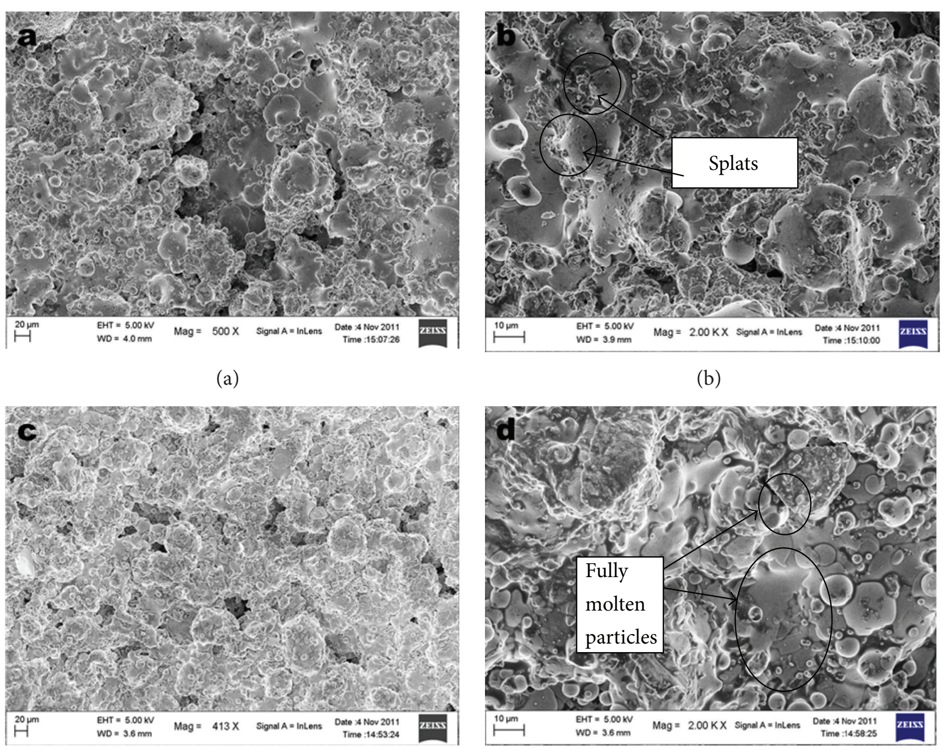
3.3. Coating Thickness. Thicknesses of the copper slag coatings on different substrates are measured on the polished cross-sections of the samples, using an optical microscope. Five readings are taken on each specimen and the average value is reported as the mean coating thickness. The mean coating thickness is found to be 200 μm. The variation of coating thickness with input power level is shown in Figure 4. It is evident that with increase in torch input power the thickness of the coating increases. Copper slag premixed with 15 wt% of aluminium powder resulted in a greater coating thickness, with a maximum of 257 μm at 25 kW power level. This may be due to melting of aluminium particles during plasma-particle interaction resulting in better intersplat bonding and hence higher rate of deposition.

3.4. Coating Adhesion Strength. The interface bond strength of the coating is evaluated by the coating pull-out method as per ASTM C633 standard, as shown in Figure 5. In all



(a) (b)

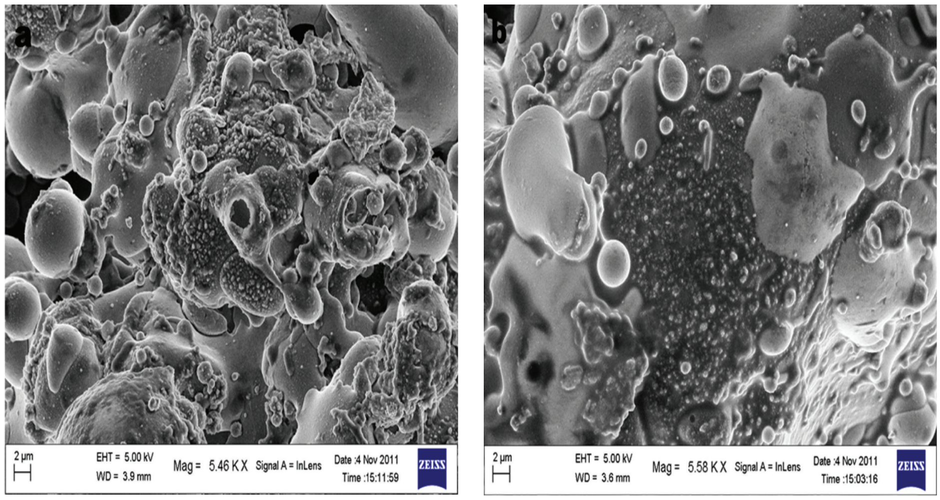
Figure 1: X-ray diffraction patterns of (a) raw copper slag powder and (b) plasma sprayed copper slag coating on MS plates.



(a) (b) (c) (d)

Figure 2: Scanning electron microscopic view (lower magnification) of the surface of plasma spray coatings on MS plate deposited with 10, 15, 20, and 25 kW power levels.

cases, fracture occurred at the coating-substrate interface stating the fracture mode to be adhesive. The variation of coating adhesion strength is shown in Figures 6 and 7 for aluminium and mild steel substrates, respectively. In both the figures, the increase in adhesion strength with increase in weight proportion of Al in the feed stock is clearly stated. The strength has increased with power level up to 15 kW and maximum value of 27 MPa is recorded with Cu slag premixed with 15 wt% of aluminium metal powder on Al substrates and 23 MPa is recorded with the same feed stock composition on mild steel substrates. Further increase in power level has a detrimental effect on its adhesion strength.



(a) (b)

FIGURE 3: Scanning electron microscopic view (higher magnification) of the surface of plasma spray coatings on MS plate deposited with 10 and 25 kW power.

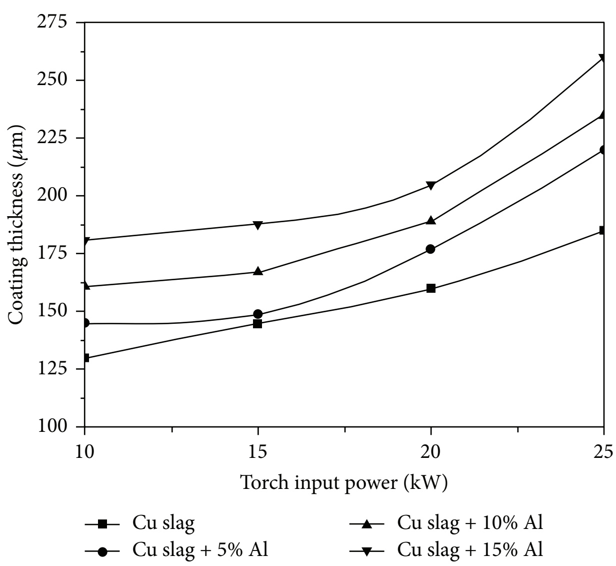


FIGURE 4: Variation of coating thickness with plasma torch input power.

Initially, when the torch input power level increased from 10 kW to 15 kW, it seems, there is better melting and mechanical interlocking of molten particles on the substrates which probably contributes to increase in adhesion strength. But at higher power levels (at 20–25 kW), lower adhesion strength attributed to vaporization of particles; as a result, small particles fly off during spraying. The presence of suitable proportion of alumina, which formed from Al powder, is also found to have helped in interparticle bonding as well as interface bonding. An increase of adhesion strength is observed in case of 15% of aluminium premixed with raw Cu slag. During the plasma-particle interaction, some amount of Al from Al_2O_3 may be diffusing to iron silicate which enhances the cohesive forces between the splats which in turn establishes true metallurgical bonds and mechanical interlocking between splat-splat and splat-substrate. Helene Ageorges and Pierre Fauchais reported such an increase of adhesion strength from 50 MPa to 80 MPa by adding Al_2O_3 to stainless steel particles [17]. Similar types of observations have also been reported by previous investigators for coatings of red mud, another industrial waste [18–20]. Coating adhesion strength was also found to be higher in case of aluminium substrate than that in case of mild steel substrate. This may be attributed to the dependence of heat dissipation at the coating-substrate interface on thermal conductivity of the substrate material, which is higher in case of aluminium than that of mild steel.

3.5. Coating Hardness. Microhardness measurement is made using Leco Microhardness Tester equipped with a monitor and a microprocessor based controller, with a load of 0.493 N and a loading time of 20 seconds. About ten or more readings were taken on each sample and the average value is reported as the data point. Maximum hardness of 637 HV has been recorded on the coating. For the coatings made with raw copper slag, the coating microhardness is found to increase from 528 Hv to 623 Hv (about 20%) as the plasma torch input power increases from 10 kW to 25 kW. It is also observed that the addition of aluminium to Cu slag in the feed stock results in marginal increase in the bulk hardness of the coatings. Influence of power level in terms of improvement of coating hardness is clearly seen from the results shown in Figure 8. However, the rate of this improvement with the operating power increasing from 10 kW to 15 kW is found to be relatively higher than during the increase from 15 kW to 25 kW.

3.6. Deposition Efficiency. Deposition efficiency depends on many factors that include the input power to the plasma

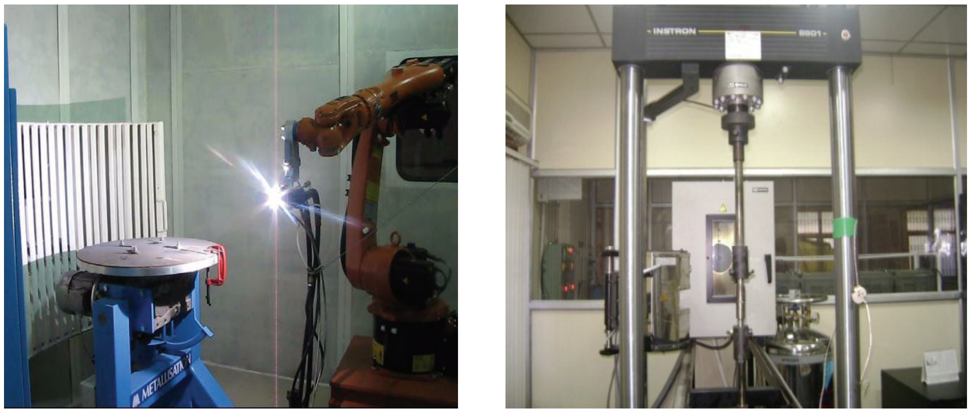


FIGURE 5: Plasma spraying torch and adhesion test setup as per ASTM C-633 standard.

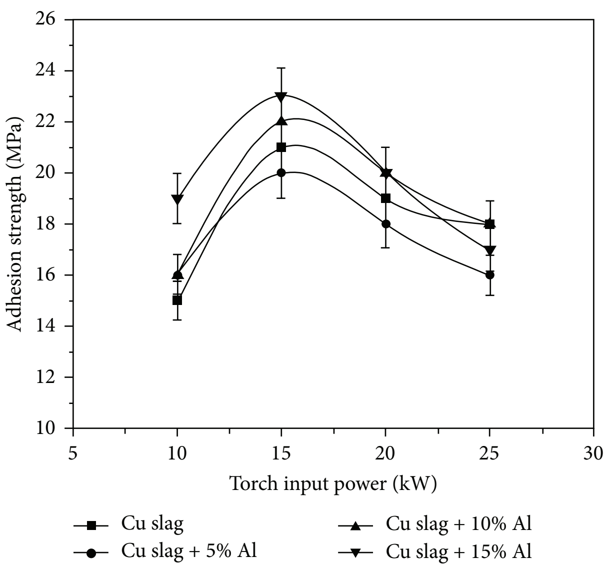


FIGURE 6: Variation in coating adhesion strength with plasma torch input power on Al substrates.

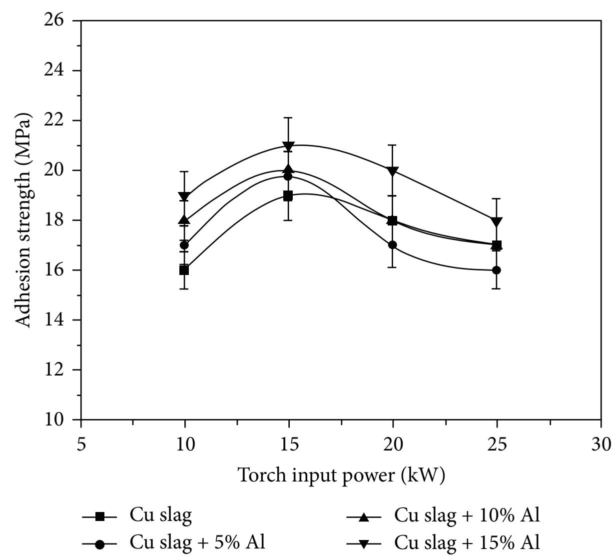


FIGURE 7: Variation in coating adhesion strength with plasma torch input power on MS substrates.

torch, material properties such as melting point, particle size range, heat capacity of the powder being sprayed, and the torch to base distance (TBD) [21]. For a given TBD and a specific coating material, torch input power appears to be an important factor for the deposition efficiency. Deposition efficiency values of copper slag premixed with Al coatings made at different operating powers (on aluminium substrate) are presented in the graph plotted in Figure 9. As the power level increases, the net available energy in the plasma jet increases leading to a better in-flight particle melting and hence to higher probability for the molten particles to flatten. The deposition efficiency reaches a plateau for the highest power levels due to the plasma jet temperature increasing which in turn increases both the particle vaporization ratio and the plasma jet viscosity.

4. Conclusions

This work establishes copper slag as a potential cost-effective coating material on metallic substrates and, thereby, suggests a value added utilization of this industrial waste. The adhesion strength of the coating is found affected significantly 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. The adhesion strength of the coating is also found to be increasing with addition of aluminium to copper slag. This is attributed to the formation of Al-diffusion layers within the splats, thereby enhancing the cohesive forces between splats. The as-sprayed coating has reasonable hardness and may be recommended for tribological application such as conveyer belts, pipes carrying particulates.

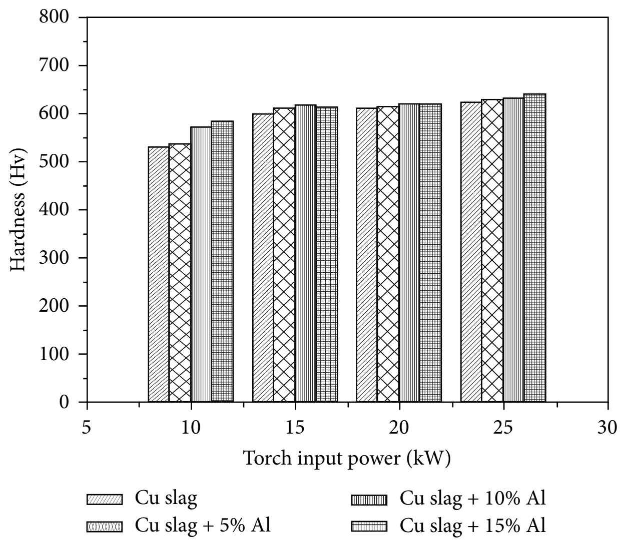


Figure 8: Variation in hardness with plasma torch input power on MS substrates.

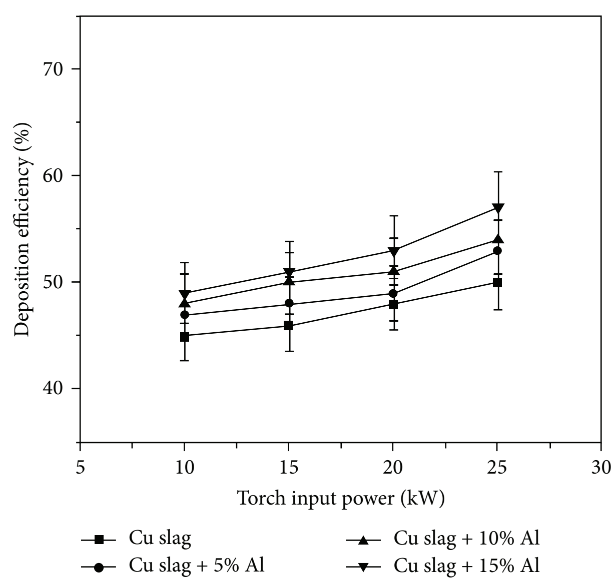


Figure 9: Coating deposition efficiency of various mixtures at different power levels.

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