



(RESEARCH ARTICLE)



EFFECT OF Al_2O_3 ADDITION ON THE SURFACE ROUGHNESS, ADHESION STRENGTH, AND MICROSTRUCTURE OF FLAME-SPRAYED CU-AL-NI-FE ALLOY COATINGS

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ABSTRACT

This study aims to synthesize (Cu-Al-Ni-Fe) alloy composite coatings reinforced with alumina (Al_2O_3) particles at weight fractions of 0 wt%, 3 wt %, and 6 wt%, deposited on AISI 304L stainless steel substrates using the flame thermal spraying technique. The coatings were evaluated by measuring surface roughness and adhesion strength, alongside microstructural and elemental characterization via Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS). The SEM results revealed that the surface roughness of the samples increased from 5 to 8 μm and the adhesion strength increased from 22 ± 0.655 Mpa to 26 ± 0.818 Mpa As the Al_2O_3 content was increased, SEM micrographs showed reduced porosity and microcracks and improved homogeneity of the microstructure. Energy-Dispersive X-ray Spectroscopy (EDS) the presence of the main constituents of alloy (Cu,Al,Ni,Fe) and elements associated with the Al_2O_3 reinforcement. The incorporation of Al_2O_3 particles significantly contribute to the structural and mechanical properties of developed composite coatings as a whole

Keywords: Flame Spray, Cu-Al, Ni-Fe alloy, Surface Roughness, Aluminum Oxide, Adhesion Strength

1 INTRODUCTION

In engineering environments, metallic surfaces are exposed to extreme operational stresses such as friction, wear, mechanical loading, and temperature variations, which result in the deterioration of their surface characteristics and shortening of their service lifetime. Applied surface engineering is a viable method to overcome these hurdles since it provides a unique approach for altering properties of parts without bulk changes in the substrate metal. Protective surface coatings are one of the predominant technologies in this regard, with sophisticated barrier layers that directly enhance wear and corrosion resistance and contribute in optimizing mechanical performance of critical components [1]. Thermal spray methods represent one of the leading advanced coatings technology known within the spectrum of surface engineering. These approaches are based on the thermal melting or semi-melting of the feedstock and the impact of the particles accelerate on the surface of the substrate that generates a cohesive and lamellar microstructure (splats). These approaches are characterized by a rich variety of energy sources and acceleration mechanisms, allowing tailoring for different materials and meeting the demands posed by many engineering and industrial applications [2,3]. Flame thermal spraying is a method that is commonly used for covering metals surface by heating and projecting the metallic material, with a flame for instance (combustion flame). This approach is relatively easy, efficient and

inexpensive to operate, leading to its use in many surface engineering applications, and it has been shown to enhance morphological as well as mechanical properties. [4]. Nevertheless, thermal spray coatings generally show structural defects such as high porosity, inhomogeneous microstructures, incomplete inter-particle bonding and interface defects. As these defects may affect the mechanical properties, structural stability and in-service performance, control over the coating microstructure and the minimization of defects continue to be an important challenge for the development of high-performance thermal coatings [5,6]. Bond coats are often used as intermediate layers to improve the interfacial bonding between the coating and substrate and reduce residual stresses caused by mismatches in thermal and mechanical properties. These layers are essential to increase adhesion, stability of the system and preventing degradation when operating under severe conditions [7,8]. Thermal spray coatings are key to applications needing enhanced wear and high friction resistance, and also better resistance to corrosion, because their make-up can be designed to suit different operative conditions. Therefore, they are extensively used to shield components from severe degradation, and thus improve performance and usage lifetime [9,10]. Motivated by the rapid growth of thermal spraying technology, the recent development is to develop surface microstructure and coating performance via a combination of the hybrid spray process or strict control of deposition parameters to fulfil the demanding requirement of current engineering applications. [11].

This study focused on the surface and mechanical properties (surface roughness and bond strength) of (Cu .Al .Ni .Fe) alloy coating with and without reinforcement of aluminum oxide (Al_2O_3) particles deposited by flame thermal spraying. In addition, scanning electron microscopy (SEM) was used to analyze the related microstructural evolution. The objective of this work is to clarify the structure-properties relationships of coatings developed by adding Al_2O_3 and varying the microstructural architecture. Alumina (Al_2O_3) is a rational selection for the use as ceramic reinforcement in both coating engineering and composite systems due to its unique physical and chemical properties. Alumina has impressive or highly improved toughness, a high extent of resistance to chemical corrosion and mechanical wear, impressive thermal stability and a melting point of over 2000 °C, chemical inertness and high electrical insulation efficiency. Now, these properties make Al_2O_3 a promising constituent phase for thermal spray composite coatings, especially for systems where the surface performance enhancement and high protection under the extreme working conditions are desired. [12,13,14].

2 EXPERIMENTAL PROCEDURE

In this study, composite coatings were synthesized using a copper-based alloy matrix reinforced with aluminum oxide (Al_2O_3) particles. The chemical composition of this matrix alloy was 83.42 wt.% Cu, 10.66 wt.% Al, 4.61 wt.% Ni, and 1.31 wt.% Fe. Three distinct volume fractions of (Al_2O_3) particles were investigated: 0 vol.% (the unreinforced reference alloy), 3 vol.%, and 6 vol.%. All composite formulations were deposited onto AISI 304L stainless steel substrates. The macroscopic appearance of a representative prepared coating specimen is shown in Figure 1. Before the deposition process, the substrate surfaces were prepared using a standardized protocol. First, the substrates were degreased with an alcohol solution to remove surface contaminants and oils. Second, a controlled mechanical grooving process was performed using an engraving tool to increase the effective surface area and create physical bonding sites. To enhance mechanical interlocking and metallurgical adhesion while preventing delamination, a commercial bond coat consisting of 80 wt.% Nickel and 20 wt.% Aluminum was flame-sprayed onto the roughened substrates prior to the main coating application. Both the bond coat and the composite layers were deposited using an atmospheric oxy-acetylene flame spraying system; a schematic representation of the setup is shown in Figure 2. To guarantee stable particle melting and splat morphology, the following operating parameters remained constant: oxygen pressure at 0.7 bar, acetylene pressure at 0.4 bar, spraying angle of 90° (perpendicular to the surface), and standoff distance fix and equal to 16 cm. Following the deposition, the coated specimens underwent a post-treatment diffusion sintering process in an electric furnace. This heat treatment was performed across a temperature range of 1000 °C with a dwell time of 90 min to promote interfacial diffusion and relieve residual thermal stresses. The synergistic effects of the (Al_2O_3) reinforcement and the subsequent heat treatments were evaluated through multiple characterizations. Surface roughness profilometry was used to evaluate surface quality, adhesion tests were performed to determine bonding strength, and scanning electron microscopy (SEM) was utilized to analyze the cross-sectional microstructural evolution



Figure 1: Macroscopic photograph of a representative flame-sprayed composite coating specimen.



Figure 2: Photographic view of the commercial Rototec 80 flame spray torch utilized in the deposition process.

3 CHARACTERIZATION TECHNIQUES

3.1 Surface Roughness Test

Surface roughness is the phenomenon of surface non-smoothness that may be noticed when touched. Roughness was measured with the portable MarSurf PS1 device, which is a German-made instrument which is made by Mahr GmbH and available at the Technological University / Department of Applied Sciences / Materials Branch. The device works by measuring the length of a sample's surface using a fine-touch probe that slides along the surface, and displays the reading on the top screen, recording and tracking the difference in fine protrusions and depressions on the surface length.

3.2 Adhesion Strength Test

The adhesion test for the coating layers has been done using the tensile testing machine which was provided by the Directorate of Materials Research / Ministry of Science and Technology. This machine is equipped with a very accurate computer (WDW-50E) provided by German company (Time Group Inc.) based on ASTM A370 standard.

3.3 Scanning Electron Microscope (SEM)

The coating surfaces were investigated to characterize the surface morphology and microstructure, revealing the changes resulting from adding different percentages of Al_2O_3 to the Cu-Al-Ni-Fe alloy. The examination allowed for monitoring the homogeneity degree, agglomerations, pores, voids, and cracks, providing an accurate description of the microstructural variations associated with the thermal flame spraying process. The analysis was performed using an SEM instrument (TESCAN MIRA3) equipped with an EDS system at Aria Laboratory, Republic of Iran, to capture high-resolution micrographs and verify the chemical composition.

4 RESULTS

4.1 Surface Roughness (Ra)

Particle size of bond coat is one of the most sensitive parameters that influences the structure of the thermal spraying coating, and as a result, the mechanical interlocking of the coating with the substrate surface. Experimental results (Table (1) and Figure (3)) show a steadily increasing pattern of surface roughness values. In particular, a (Ra) of around $5\text{ }\mu\text{m}$ has been reported for the unreinforced, pure metallic matrices (0 wt. % Al_2O_3). Upon the addition of 3 wt. % Al_2O_3 , where it eventually peaked near $8\text{ }\mu\text{m}$ for the highest reinforcement content of 6 wt. % Al_2O_3 . This results from the very large disparity of thermal properties and melting points of the reinforcing ceramic phase ($\text{Al}_2\text{O}_3 \approx 2072\text{ }^\circ\text{C}$) versus all the metallic matrices used (from the low-melting point Al metal to Cu, Ni and Fe) [15]. This thermal gradient could explain why, in contrast to the super heated (fully molten or highly softened) alloy matrix components formed during the spray deposition stage, the Al_2O_3 particles might not reach full melting during transit in to the flame. These non-molten or partially molten alumina particles exhibit constrained splatting behavior when striking the substrate, thereby preventing full flattening and stacking with the continuous metallic matrix. As a result, irregular surface morphology, protrusions with micro-roughness, and abrupt steps on the surface are created, which directly account for the discontinuous values of (Ra) that increase from $5\text{ }\mu\text{m}$ to $8\text{ }\mu\text{m}$ between the metal matrix composites systems considered herein [16]. As the Al_2O_3 content increased gradually from 0 to 3 and 6 wt. %, the packing density of the ceramic particles associated with layer formation is enhanced, resulting in a greater influence on deposition mechanism and uniformity of the resultant surface. The build-up of these unmelted particles changes the flow behaviour during the deposition, thus inducing greater prominent protrusions and surface unevenness, and these are presented as the constant increase of (Ra) values. In fact, these results suggest an upward correlation between the surface roughness of the coating and the content of reinforcement phase in the investigated range [17].

Table 1: Surface Roughness (Ra) Values of the Prepared Coatings

Al_2O_3 Reinforcement Content (wt.%)	Arithmetic Average Roughness, Ra(μm)
0	5
3	6.5
6	8

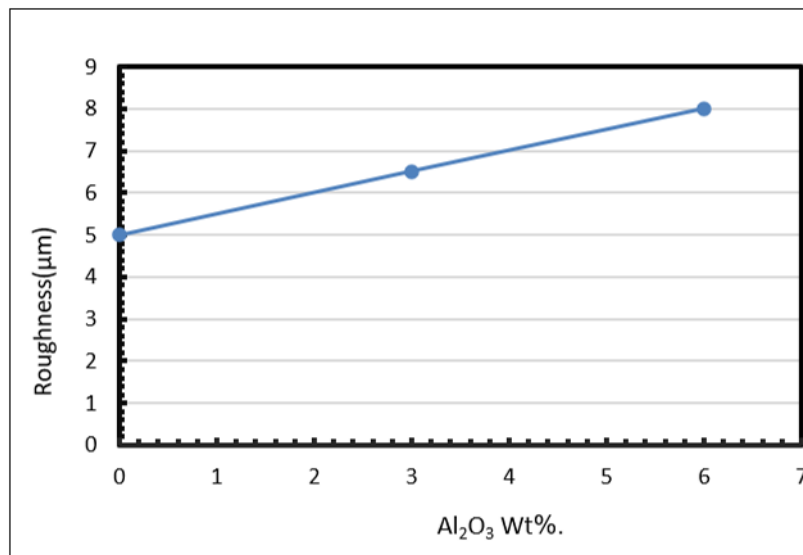


Figure 3: The relationship between Surface roughness (Ra) and (Al_2O_3).

4.2 Adhesion Strength

Adhesion strength is one of the essential mechanical properties to assess the efficiency of thermally sprayed coatings, which is defined as the ability of the deposited coating to resist substrate separation under the load application. Results indicated that the addition of alumina (Al_2O_3) particles resulted in enhanced adhesive bonding. In particular, the strength of unreinforced alloy was measured to be $22 \pm 0.655\text{ MPa}$. Upon introducing 3 wt. % and 6 wt. % of the ceramic reinforcement, the adhesion strength progressed to $23 \pm 0.754\text{ MPa}$ and reached its maximum value of $26 \pm 0.818\text{ MPa}$, respectively, indicating a higher resistance to peel forces at the interface, as systematically summarized in Table 2 and

pictorially demonstrated in Figure 4. This steady increase may be justified primarily on the basis of improved mechanical interlocking arising from the revision of the fabrication technique of the substrate surface topology [18]. The surface roughness results showed significant increases in roughness values as Al_2O_3 content was increased, leading to a better profile for mechanical bonding [19]. Thermally sprayed particles flatten and adapt within the micro-valleys and peaks of the surface as a result of impact events with the substrate. This approach increases the apparent contact area, and keeps the deposited layer locked within the topographical features of the substrate, thereby increasing its resistance to delamination. According to the observations from scanning electron microscopy (SEM), it also confirms the microstructure changes as a result of the alumina addition. Homogeneous distribution of ceramic particles in a metallic matrix refines internal structure of the coating, increasing its resistance to localized plastic deformation. Such structural changes limit the crack origin and crack propagation in feeble interfacial regions [20]. In the end, this achieving better mechanical interlocking in addition with microstructural refinement fundamentally contributor to the overall enhancement of adhesion from 22 ± 0.655 MPa to 26 ± 0.818 MPa.

Table 2: Results of the adhesion strength of the added percentages of (Al_2O_3)

Al_2O_3 wt.%	Mean and standard deviations of Adhesion Strength
0	22 ± 0.655
3	23 ± 0.754
6	26 ± 0.818

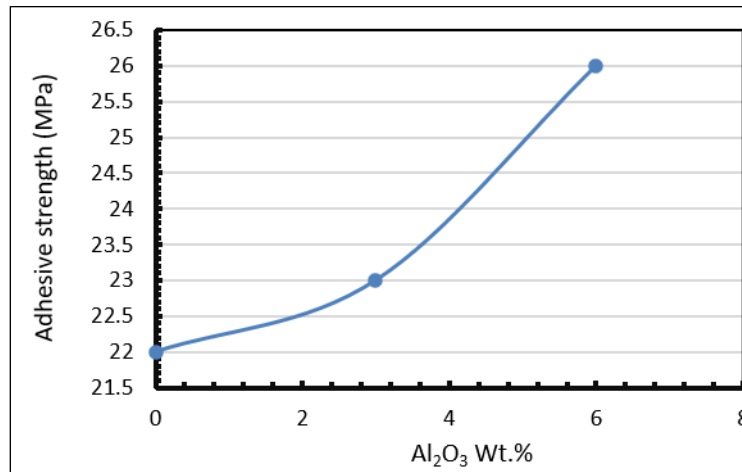


Figure 4: The relationship between adhesion strength and (Al_2O_3).

4.3 Scanning Electron Microscope (SEM)

Scanning Electron Microscopy (SEM) is an indispensable method for surface morphology inspection of coatings, which is also used for homogeneity and uniformity tests and surface defect detection. High magnification scanning electronic microscopy (SEM) micrographs (left and middle in Figure 5) demonstrated that its surface morphology changed progressively as more Al_2O_3 was added to the Cu–Al–Ni–Fe alloy coating. For the unreinforced sample (0 wt [21,22]. For the coating with 3 % Al_2O_3), their surface looked more heterogeneous, with small cracks, agglomerations and irregular profiles, in correlation with the type of layer generated in thermal spraying and the different melting and flattening splats of the particles when impacting the substrate. Upon adding 3 wt.% Al_2O_3 , the surface became more homogeneous, showing a distinct reduction in agglomerations and visible defects, which indicates improved distribution uniformity of the coating components within the metallic matrix layer [23]. With increasing the Al_2O_3 content to 6 wt.%, the general continuity and uniformity of the surface further improved, while some localized protrusions remained; these can be partially attributed to the presence of Al_2O_3 particles with a high melting point and the difficulty of their complete melting during the thermal spraying process, in addition to the effect of their distribution within the metallic matrix. Energy-dispersive X-ray spectroscopy (EDS) results support the presence of the primary alloying elements (Cu, Al, Ni, and Fe), with the Cu element exhibiting the highest percentage among the detected elements, which aligns with the metallic matrix being the main component of the coating. Furthermore, Al and O elements were detected in the reinforced coatings, as presented in Table 3. Given that Al is inherently present in the alloy composition, its standalone appearance in the EDS results does not provide sufficient evidence for the Al_2O_3 addition; however, the simultaneous detection of Al and O in the reinforced samples supports the presence of the added ceramic component, without causing any fundamental change in the base elemental composition of the alloy. This indicates that the coating retained its primary metallic components alongside the presence of the added ceramic

component. The observed morphological evolution correlate well with the surface roughness measurement. Hard ceramic particles and their localized protrusions associated with their distribution can elevate local microtopographical roughness, warming the roughness values with increased Al_2O_3 content. This increase in surface roughness due to sandblasting and the irregularity of topography on the surface of the substrate could possibly increase mechanical interlocking between coating and substrate (the good adhesion of the two layers) which agree with the clear increase of adhesion strength [24]. SEM and EDS examination corroborated by roughness and adhesion measurements indicated that these changes in surface morphology occurred without changing the major constituents of the metallic matrix at ratios of Al_2O_3 addition. This fact reflected on the surface and mechanical responses of the coating at the investigated ratios.

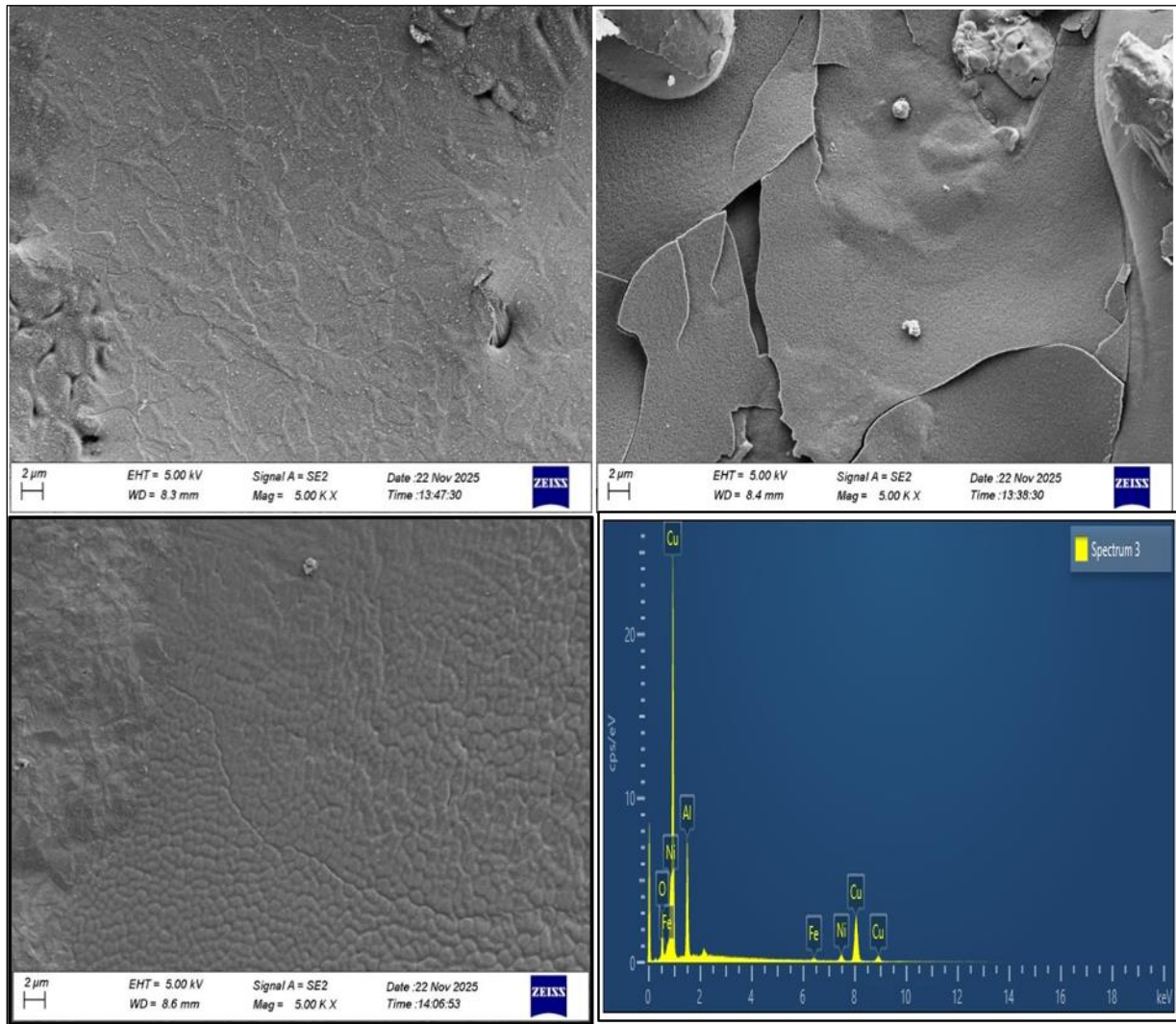


Figure 5: SEM, EDS results for the paint samples after adding different amounts of Al_2O_3 (0%,3%,6%)

Table 3: EDS results for a coating layer with 6wt.% Al_2O_3

Element	Weight %
O	9.04
Al	13.39
Cu	70.89
Ni	1.28
Fe	5.40
Total	100.00
Element	Weight %

O	9.04
Al	13.39
Cu	70.89
Ni	1.28
Fe	5.40
Total	100.00

5 DISCUSSION

The experimental findings show a distinct linear relationship among the evaluated properties, as increasing amounts of alumina (Al_2O_3) particles (0 wt%, 3 wt% and 6 wt%) resulted a corresponding elevation in surface roughness and adhesion strength. The increased level of surface roughness from here are mainly due to the presence of hard ceramic dispersoids that disturb the surface growth kinetics during deposition processes and yield topography in finer detail than that of the unreinforced matrix. This morphological change occurs critically at the layer boundary of the deposited layer and AISI 304L substrate. Here, the improved surface roughness can effectively increase the real contact area and mechanical interlocking through anchoring sites and micro-geometric irregularities, allowing for more profound and secure mechanical interlocking. The mechanical mechanism derives a logical rationalization for the increased adhesion strength observed consistent with the incorporation of alumina within the investigated range effectively increasing interfacial cohesion. This interpretation is also further consolidated by the high matching of mechanical measurements with structural characterizations. In the SEM micrographs, this results in the surface microstructural features progressing continuously which correlates very well to the measured roughness values. At the same time, support by Energy-Dispersive X-ray Spectroscopy (EDS) analysis indicates the retention of the main matrix elements (Cu,Al,Ni,Fe), while separate peaks corresponding to aluminum and oxygen indicates the successful addition and distribution of the added particles in the composite. By integrating all of these findings, we can coherently establish a connection between Al_2O_3 reinforcement and a surface structural alteration leading to increased surface roughness and improved bonding properties.

6 CONCLUSION

- Flame thermal spraying demonstrated high efficiency in fabricating homogeneous and stable composite coatings reinforced with alumina particles.
- Surface roughness exhibited a progressive increase directly proportional to the higher reinforcement content of the hard ceramic alumina phases.
- A significant enhancement in adhesion strength between the sprayed layer and the substrate was achieved by increasing the alumina reinforcement ratio.
- SEM micrographs revealed a well-defined and coherent microstructure, showing a marked reduction in both porosity and agglomerations at higher reinforcement levels.
- EDS analysis confirmed the presence of the primary alloy elements, while the detection of the oxygen peak provided definitive proof of the successful incorporation of alumina within the coating matrix.

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

Disclosure of conflict of interest

The authors declare no conflicts of interest regarding this manuscript.

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