

Article

Modification of Cold-Sprayed Cu-Al-Ni-Al₂O₃ Composite Coatings by Friction Stir Technique to Enhance Wear Resistance Performance

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Abstract: An innovative hybrid process combining two effective surface modification techniques, cold spray (CS) and friction stir processing (FSP), was proposed to refine the microstructure of Cu-Al-Ni-Al₂O₃ composite coating material. FSP was performed under constant rpm using extensive cooling conditions to remove heat generated during the operation. Microstructural characterizations such as optical micrography (OM), scanning electron microscopy (SEM), Electron Backscatter Diffraction (EBSD), Energy-dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) were performed to evaluate the microstructural evolution of the coatings before and after FSP treatment. Mechanical characterizations such as microhardness and elastic modulus were measured using micro-depth sensing techniques. Furthermore, sliding wear tests were performed to study the wear resistance of the as-sprayed and processed coatings. The findings suggest that after FSP, there is an improvement in microstructure of the coating layers with the elimination of particle boundaries, micro-pores and micro-cracks, and processed coatings showed an improvement in mechanical properties. Furthermore, there was a slight reduction in the wear rate of the deposited CuAlNi-Al₂O₃ composite coatings. Among all the test coatings, friction stir processed S1 coating showed the lowest wear rate, which was an almost two times lower wear rate than its unprocessed counterparts.

Keywords: cold spray coating; friction stir processing; metal matrix composites

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1. Introduction

Bearings are considered crucial components of internal combustion engines and other equipment that deal with power transmission [1]. The primary function of the bearing is to facilitate the smooth rotation of the connecting rod, which connects the piston to the crankshaft, and support the rotation of the crankshaft inside the engine block [2]. In order to minimize the energy loss in the engine, proper selection of bearing materials is of paramount importance, since a considerable portion of the energy generated is lost to overcome frictional force. The bearing material should meet tribological requirements and have low friction, good corrosion and wear resistance, conformability and embeddability [3]. Bearing materials such as a Cu- or Al-based alloys containing a slight amount of lead such as Al-Sn or Al-Pb alloys are most commonly used by bearing manufacturers to produce engine bearings. Owing to the low-friction characteristics and excellent embeddability and conformability behavior of lead, materials containing Pb such as Pb-based and Cu-Pb based alloys have been extensively used in the bearing industry. Moreover, these Pb-based layers protect the bearing linings from corrosive damage caused by the degrading lubricating oil and improve the bearing material's seizure resistance [4–6]. In the recent decade, Pb-based alloys have been replaced with Pb-free bearing materials due to the growing environmental and health hazard and bioaccumulation caused by Pb poisoning [6,7]. The bearings used

for internal combustion engines are being manufactured based on Cu-based alloys with a softer phase such as Bi, Al, Sn [8–11].

The degradation in load-bearing components can also lead to premature failure of the machinery and thus may lead to heavy revenue losses resulting from the poor efficiency and higher maintenance cost [12]. Surface modification techniques such as thermal spray, cold spray, laser claddings and microwave claddings are generally used to develop wear-resistant surfaces for various industrial applications [13–17]. Of all the coating development techniques, the thermal spray technique is the most widely adopted; however, still there are some drawbacks associated with it, such as high porosity, oxide formation, splat boundaries and microcracks [18,19]. However, the use of microwave and laser cladding is not recommended since it could potentially alter the microstructure of the underlying substrate material due to substrate melting during cladding development, which makes it more susceptible to other modes of failures [20–22]. In recent years, cold spray deposition has gained significant attention among all these technologies. Cold Spray is a solid-state material deposition technology used for depositing thick or thin coating layers on metal surfaces as additive manufacturing or repair. This technique has recently attracted much attention and is widely adopted in the automotive, aerospace, power-generation, marine and offshore oil and gas industries. In the cold spray process, the spray particles are accelerated using a supersonic gas jet by a de Laval-type nozzle to a velocity of about 500–1200 mm/s, and these particles, which have an average particle size of less than 50 μm , are made to impact onto a substrate material [23]. Coatings with extremely low porosity and low oxygen content can be quickly developed, since these particles experience a temperature below the melting point of the spray powder. Due to this, metals such as Al, Cu, Ni, Sn, etc., or metal-matrix composite coatings (MMC) such as Al-Al₂O₃ or Cu-Al₂O₃ and others can be easily developed using cold spray [24–26]. Furthermore, cold-sprayed coatings tend to have superior mechanical and microstructural properties, corrosion and wear resistance; as well as thermal conductive properties [27,28]. These peculiarities of cold spraying technology have gained significant attention in the research community in recent years. The primary advantage of cold-sprayed coatings over their thermally sprayed counter parts is that metallic and metal–ceramic composite coatings provide low oxygen content and porosity [29]. Furthermore, high coating density with negligible microstructural defects of cold-sprayed coatings is a criterion for excellent corrosion resistance. These coatings also show higher adhesion strength, hardness and extremely low residual stresses [30,31]. Moreover, the temperatures used for the cold-sprayed coatings are significantly lower than that of the melting point of the feedstock material, thus eliminating the chance for the formation of new oxides and phase transitions which could alter the electrochemical properties of the coatings [32,33]. Yin et al. [34], in their work, developed cold-sprayed CoCrFeMnNi coatings that exhibited higher hardness (332 HV) with superior wear resistance compared to laser-cladded CoCrFeMnNi claddings. Further, the authors reported that the improvement in hardness was mainly due to the high-strain plastic deformation during the cold spraying process. Another study investigated by Lehtonen et al. [35] studied gas-atomized MnCrFeNi HEA coatings deposited using high-pressure cold spray system by varying carrier gas pressure and feed rates. Their study showed that coating hardness decreased as a function of feed rate and improved linearly with gas pressure. This was attributed to the high coating porosity obtained at a higher feed rate, which significantly hampered particle deformation. Melendez et al. [36] fabricated tungsten carbide-based metal matrix composite coatings using a low-cost, low-pressure cold spray system. Their study showed that WC-based composite coating with the highest WC content and highest hardness showed the lowest wear rate. Even though the hardness was low for cold-sprayed coatings in comparison to thermally sprayed counterparts (WC-based HVOF coating), the wear rate was comparable to coatings layers developed by both processes. These studies confirm the potential of using cold spraying for fabricating metal matrix composites with good microstructural properties and wear resistance.

In recent times Cu-Al-Ni-based alloys have been widely used as sliding bearing materials in the shipping and automobile industry [37,38]. The traditional manufacturing used for producing bearings involves casting and thermo-mechanical processing to bond the Al-Sn layers onto steel back. During this process, careful control parameters and intermediate heat treatment are required to control the microstructure property of the developed coating layer. Cold spray technology can deposit such coating layers on the steel back without facing the difficulties associated with undesirable phase transformations.

Taking in into account the advantages of the cold spray process while developing MMC coating compositions, controlling the distribution of secondary particles is still a complicated task. Thus, there is an uneven distribution of secondary particles in the formed coating matrix. Additionally, the bonding interface remains low-valued between the particle and the matrix, resulting in the poor cohesion strength of coating materials. This further degrades the wear resistance of the coatings [39–41]. Moreover, the cold-sprayed coating has little to no ductility for as-sprayed coating materials [42]. Therefore, post-spray treatments such as heat treatment of cold-sprayed coatings are used to overcome the issues mentioned above and enhance the microstructural and mechanical characteristics of the coatings that can be comparable to the bulk material. Few previous studies report on the utilization of various post-processing techniques including laser remelting [43,44], microwave annealing [45,46], furnace annealing [47,48] and friction stir processing [49–51] for coatings. Among them, friction stir processing (FSP) is a relatively new processing technique which was developed by Mishra et al. [52] for microstructural modifications based on the basic principles of friction stir welding. FSP is considered to be one of the most effective post-treatments. Treatment is performed by a rotating tool with a probe and/or a shoulder into the deposited coatings for microstructural rejuvenation [53]. FSP is a very effective solid-state processing technique which can provide control of microstructures and localized modification in the near-surface layers of the processed material. Compared to other post-processing routes, FSP has some clear advantages over the other techniques. First, by controlling the parameters such as temperature and speed, depth of penetration, and rpm, the microstructure and mechanical properties of the workpiece can precisely controlled. Moreover, FSP is a solid-state post-processing technique and in a single step it achieves densification, microstructural refinement and homogeneity. Further, the thickness of the processed zone can be optionally controlled by altering the length of the tool pin, and it is very difficult to attain an optionally adjusted processed depth using other metal processing techniques [54].

The main limitation of the precursor materials applicable as feedstock for cold spray comes from its bonding properties. Precursor materials must possess a good degree of ductility at high strain rates so that it deforms upon impact with the substrate material, which subsequently results in material bonding and coating build up. Therefore, cold spray technology cannot be considered a convenient technology for developing hard coatings made from ceramic particles and intermetallic compounds. To improve the anti-wear properties of the coatings hard particles with enhanced hardness are required. Since these particles are highly brittle in nature they cannot be deposited directly using cold spray technique. To solve this problem, hard particles were added to a deformable metallic matrix and it appeared to be an appropriate solution [55]. The main idea of MMC was adopted to facilitate deposition of brittle materials and the development of hard-wear-resistant coatings [56]. Morisada et al. [53] performed FSP of WC-CrC-Ni MMC deposited using a WC-Co tool. The processed layers showed nearly 1.5 times improvement in the hardness and significant improvement in the bonding between coating and substrate. This could be attributed to the enhanced diffusion of different phases during FSP. Hodder et al. [40] used FSP for improving the for homogenization of alumina particles in Al matrix developed using low-pressure cold spraying technique. Further, Peat et al. [41] studied the effect of FSP on erosion properties of cold-sprayed metal-matrix composite coatings and, from their study, a significant reduction in the volume loss as a result of post-treatment (almost 40% reduction) was reported. This improvement in wear resistance was mainly attributed

to the enhancement in the microhardness and significant reduction in the size of SiC particles in the matrix. Yang et al. [57] adopted FSP for microstructural homogenization of AA2024-Al₂O₃ coating deposited using cold spray technique. The improvement in the mechanical properties of the coating's layers were attributed to Orowan strengthening and enhancing interparticle bonding. In this study, the mechanically mixed powders were used for developing the coatings owing to their advantages of convenience and low cost, and thus the hybrid process of cold spray and the post-treatment aimed to achieve the element alloying in the coating [58]. FSP has been carried out on the as-sprayed cold-sprayed MMC coatings to improve the distribution of secondary alumina particles and eliminate the clear interface between the matrix and reinforcement particles. Rani et al. modified the thermally sprayed Ni-Cr alumina composite coating via FSP, and found that the microstructures, mechanical and corrosion properties were greatly improved after post-treatment [59]. Furthermore, other researchers have also reported that there was significant improvements in the microstructures and mechanical properties of cold-sprayed coatings [58,60], thermally sprayed coatings [61] and laser cladding [62,63] after FSP.

Prior to the current work, very few researchers studied the effect of FSP on coatings developed using a medium-pressure cold spray setup and on the sliding wear behavior of friction stir post-processed coatings relative to their as-sprayed counterparts. In the present work, FSP has been carried out on the as-sprayed cold-sprayed MMC coatings to improve the distribution of secondary alumina particles and eliminate the clear interface between the matrix and reinforcement particles. However, there is a significant gap in understanding the underlying microstructural features of metal-matrix composite coatings with varying weight fraction of alumina particles fabricated through cold and post-treated using friction stir processing. The main objective of this study is to develop a coating of the Cu-Al-Ni-based alloy on an Al substrate using in-house-developed medium-pressure cold spray technique and study the effect of FSP on the microstructural and mechanical properties along with the wear resistance of these newly developed coatings and to investigate the wear mechanism using SEM studies.

2. Materials and Methods

All the feedstock powder materials used in this study were analyzed, and were procured from Obninsk Center for Powder Spraying (OCPS, Obninsk, Russia). The powder feedstock blends were mechanically mixed for 5 h and dried in a hot-air oven at 413 K for 2 h to eliminate any potential moisture content. Point EDS analysis were performed on the feedstock powders to study their elemental compositions and the results are given in Table 1. The coatings are formed onto an AA2024 substrate material at a constant feed rate of 0.4 gm·s⁻¹ and by the in-house-built medium-pressure cold spray system (MPCS) using optimized parameters at a total pressure of 1064 kPa and total temperature of 723 K, where the constant stand-off distance of 30 mm and travel speed of 100 mm·s⁻¹ were maintained. Prior to the coating development, AA2024 blocks (substrate) of dimension (100 × 50 × 3 mm) were machined, and an average surface roughness (Ra) of 6–8 µm was attained with the aid of grit-blasting, using 500 kPa pressure and F24 alumina particles.

Table 1. Elemental compositions of the coating feedstock powders.

Coatings	Elements in wt. %			
	Cu	Al	Ni	Al ₂ O ₃
S1	33	14	30	23
S2	26	11	23	40

FSP of the as-sprayed MMCs coatings was performed using a universal milling machine (Proma, Ilfov, Romania, model: FNS-55PD). The coated aluminum plates were fixed onto the machine bed using a mechanical clamping system to arrest any movement during the FSP (Figure 1). A tool with flat end of 10 mm diameter, manufactured from tungsten car-

bide, was rotated clockwise continuously at a constant speed of 360 rpm and 20 mm/min^{-1} transverse speed and four continuous passes were made with a plunge depth of $200 \text{ }\mu\text{m}$.

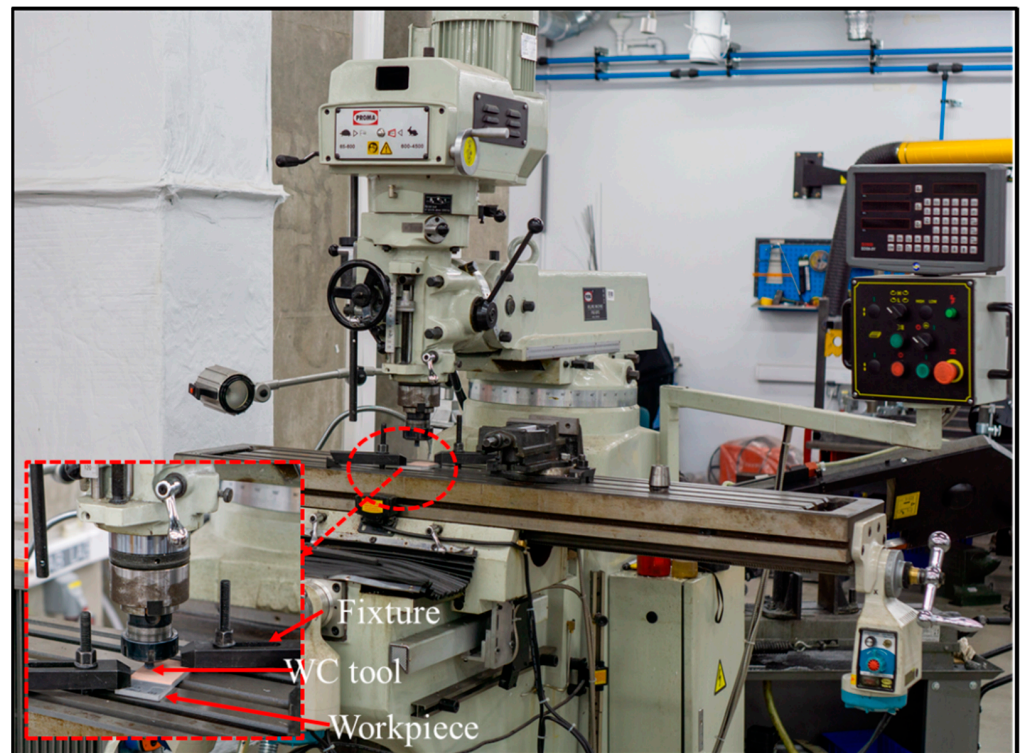


Figure 1. Experiential setup for friction stir processing.

To assess evolution of coatings microstructure, the following characterization techniques are used: optical microscope (Olympus GX71, Olympus, Beijing, China); and scanning electron microscopy (SEM) in a backscattered electron mode (QuattroS, Thermo Fisher, Waltham, MA, USA) and energy-dispersive X-ray analysis (EDS) were performed to evaluate the coatings microstructure and elemental composition, respectively, with a stand-off distance of 8 mm and a working voltage of 15 kV. The distribution of the grain boundary misorientations and grain diameters were determined using electron backscatter diffraction (EBSD) technique. The EBSD scans were in areas of $50 \times 50 \text{ }\mu\text{m}$ with a $0.1 \text{ }\mu\text{m}$ step size. Optimized scanning parameters were used so that the high-angle grain boundary was defined, if the misorientation between adjacent points of measurement was higher than 15° . The X-ray diffraction (XRD) using a Bruker D8 Advance system (Bruker, Billerica, MA, USA), scanned from 20 to 100 degrees with a step size of 0.02° , using a Cu-K α radiation with a wavelength of 0.154 nm. Additionally, the micro-depth-sensing technique was performed to evaluate material plasticity index (H/E) by measuring micro-hardness and contact elastic modulus of deposited materials (Nanovea PB100, Nanovea, Irvine, CA, USA). All micro-indentations were performed by using a varying load of 0 to 20 N with a constant loading and unloading rates of 10 N/min; load-displacement results were used for estimating the contact elastic modulus of the coating layers.

The sliding wear resistance of the cold-sprayed and processed coatings were estimated using Anton-Paar THT1000 tribometer (Anton-Paar, Graz, Austria) with a ball-on-disc configuration as per ASTM standard G-99, where a constant load of 2 N at a constant temperature of 298 K and relative humidity of around 45% was used. For all the tests, a constant disc speed was maintained at 200 rpm, while AISI 52100 100Cr6 chrome steel ball of diameter 6 mm was used as a counter body. The total sliding distance for each test was 100 m, during the test, the coefficient of friction was recorded and analyzed. After each test, the width of the wear track was measured by optical profilometer (Nanovea PB100) and

volume loss, and the specific wear rate of the coatings was calculated accordingly using Equations (1) and (2) [64].

$$V_{loss} = 2\pi R \left[r^2 \sin^{-1} \left(\frac{d}{2r} \right) - \left(\frac{d}{4} \right) \sqrt{(4r^2 - d^2)} \right] \quad (1)$$

$$W = \frac{V_{loss}}{R_N \times L} \quad (2)$$

where r = radius of the ball (mm); R = radius of the track (mm); d = width of the track (mm); R_N = Normal load (N); L = sliding distance (m); V_{loss} = volume loss of the sample (mm³); and W = specific wear rate (mm³/N·m). At least three samples were tested in each case to ensure repeatability. The all-worn surface morphologies of the coatings were also analyzed to estimate the damage mechanisms.

3. Results and Discussion

3.1. Microstructural and Mechanical Characterization

All the feedstock powder materials used in this study were analyzed using SEM and XRD and the results are shown in Figure 2. From the XRD results, it was found that the Al, Cu, Ni and Al₂O₃ powders were free from other impurities (Figure 2). The Al powder showed a spherical morphology, and the other feedstock particles showed an irregular and bulky morphology due to the method of powder feedstock manufacturing.

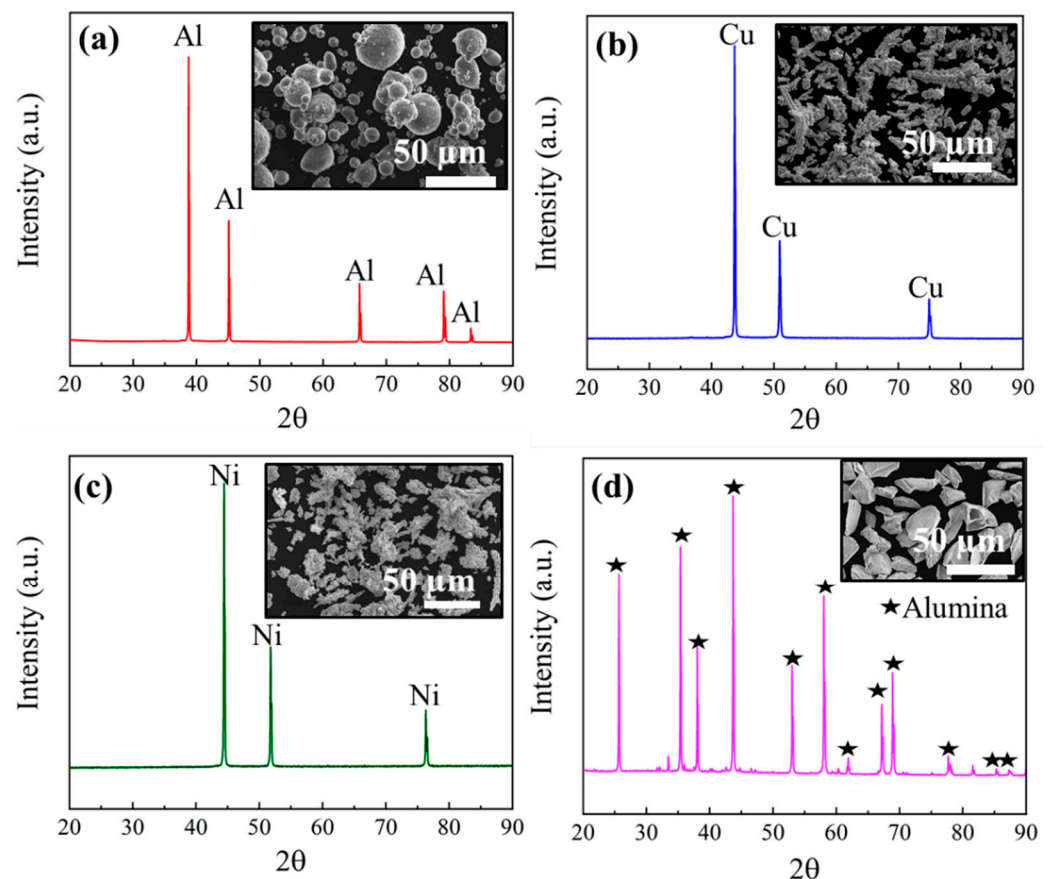


Figure 2. Morphology and phase distribution of the feedstock powders used in the study (a) Al, (b) Cu, (c) Ni, and (d) Alumina.

Optical micrographs of the polished cross-sections from the as-deposited and friction-stirred specimens are displayed in Figure 3. The patterns of light and dark regions in the stir zone relate (processed layer) to the matrix and traces of reinforcing

particles within aluminum, respectively. The optical micrographs of the coatings in Figure 3 represent different microstructures before and after FSP, respectively, where analysis of the cross-sectional images demonstrates an average coating thickness of $600 \pm 10 \mu\text{m}$.

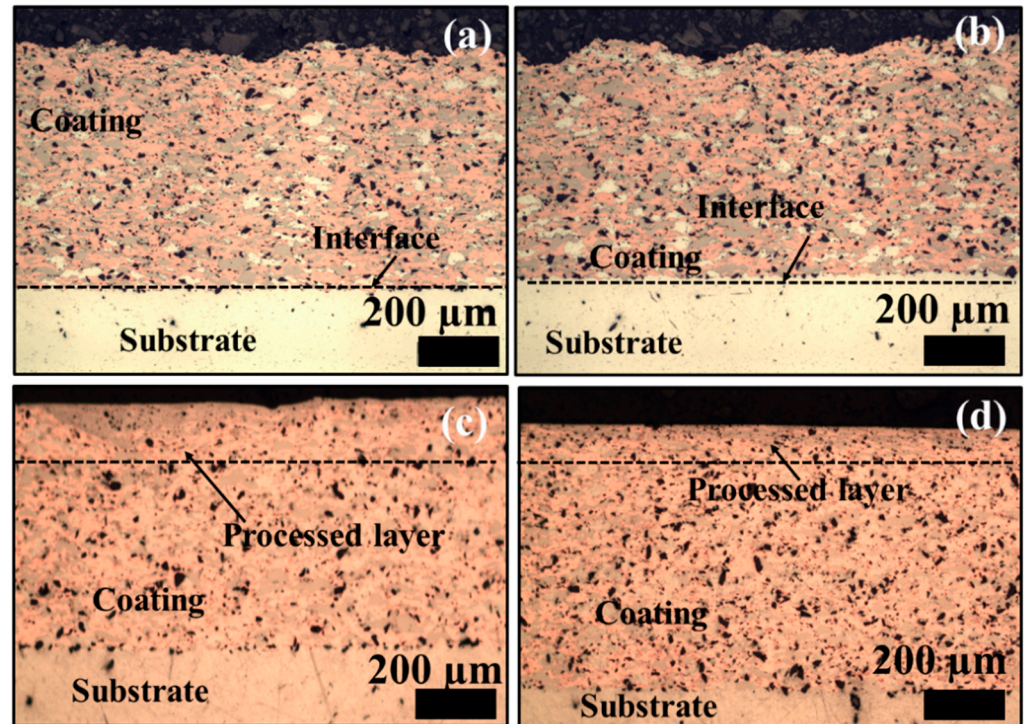


Figure 3. Optical micrographs of the cross-sectional cold sprayed coatings (a) S1, (b) S2, (c) FSP-S1, and (d) FSP-S2.

Figure 3a,b shows that Al and Ni is located along the grain boundaries of the Cu particles and formed almost continuous layers. This could contribute towards improving the cohesion strength of the coating layer [65]. Further, alumina particles are also found to be uniformly distributed in the matrix material, without any change in shape or size; this could be attributed to the lack of plasticity of alumina particles [66]. From the image analysis, it was estimated that the as-sprayed coatings demonstrate the formation of a dense microstructure with a low level of porosity and micro-cracks, showing an average porosity of around $1.22 \pm 0.13\%$. From Figure 3c,d, significant refinement in the microstructure was observed for FSP processed coating layers with a slight reduction in the coating porosity to $1.05 \pm 0.17\%$. The processed coatings exhibited an average coating thickness of $600 \pm 10 \mu\text{m}$ and there was no significant variation in the coating thickness after processing. This refinement in the microstructure of the coating with the reduction in micro-cracks and pores could be associated with the shear forces exerted by the WC tool as it stirs the plasticized material; prior studies also report similar findings [42,67,68]. It was determined that FSP aids in the uniform dispersion of individual oxide particles, since both the agglomerates and the binder phases are plastically deformed during the processing.

From the high magnification SEM images (Figure 4), it can be observed that the distribution of alumina particles across the matrix-binder phase has considerably improved after FSP. In this regard, it is evident that some of the secondary particles have been broken up into finer particles and mixed with Cu-Ni-Al matrix phase presented within the coatings. Prior studies also showed a significant improvement in the particle refinement with the increase in the number of passes during the friction stir processing [41]. Therefore, it is expected that additional FSP passes through the cold sprayed coating would uniformly distribute almost all the secondary particles and other constituent elements in the as-deposited coating to a greater extent within the coating layers. Additionally, from Figure 4

a significant improvement in the bonding between the coating's binder phase and the secondary alumina particles is observed, attributed to the elevated processing temperature caused by mechanical mixing and high strain rate plastic deformations during FSP. More importantly, the micro-reinforcement mechanisms could occur due to improved dispersion of the secondary particles and the dissolution of different phases present in FSP coatings similar to the findings in [69].

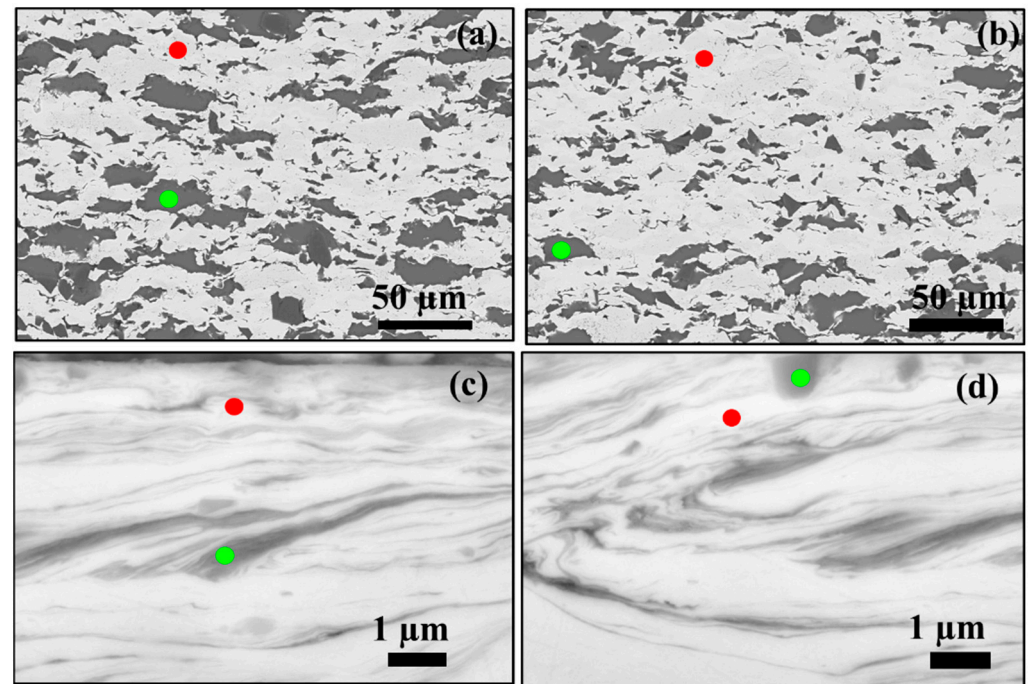


Figure 4. High-magnification SEM images of the cold-sprayed coatings (a) S1, (b) S2, (c) FSP-S1, and (d) FSP-S2. EDS of the highlighted points are shown in Table 2.

Table 2. The elemental composition of the highlighted regions of all the coatings shown in Figure 4.

Coatings		Elements Are in wt%			
		Ni	Cu	Al	O
S1	●	-	-	44.75	55.17
	●	12.17	21.01	66.13	0.69
S2	●	-	-	44.83	55.17
	●	11.17	67.13	21.01	0.69
FSP-S1	●	-	-	97.07	2.93
	●	96.66	0.81	1.63	0.9
FSP-S2	●	-	1.41	97.57	2.92
	●	89.66	2.81	4.63	0.9

The elemental composition of as-sprayed and processed coatings was also analyzed using EDS analysis; highlighted regions are depicted in Figure 4, and the results of which are summarized in Table 2. The results show that the secondary particles in the as-sprayed coating and FSP are uniformly distributed within the coating layers. Moreover, it was observed there is a slight improvement in the distribution of secondary particles on the retreating side of the stir zone.

Figure 5 shows the coating microstructure in a magnified view taken from the cross section and the corresponding EDS maps that were obtained. The EDS mapping results suggest that the matrix comprises Al-Cu and Ni-Al phases which make up the majority share of the coating. Moreover, the mapping results show that the Al-O-rich phase corresponded to alumina particles, which are embedded in the coating matrix. As a note, the

formation of new oxides phases can largely be excluded by the EDS mapping analysis. In this regard, the image analysis performed by using ImageJ software reveals an average Al_2O_3 content within an S2 coating of $13.4 \pm 1.0\%$ by area, which has significantly reduced compared to the original composition of the feedstock power mixture. This is attributed to the rebound of hard alumina particles upon impact and at the moment of deposition onto the pre-deposited coating layers or substrate; it could also be due to the poor bonding between the metallic matrix and alumina particles [66]. This demarcation between the phases is evident from the EDS mapping results.

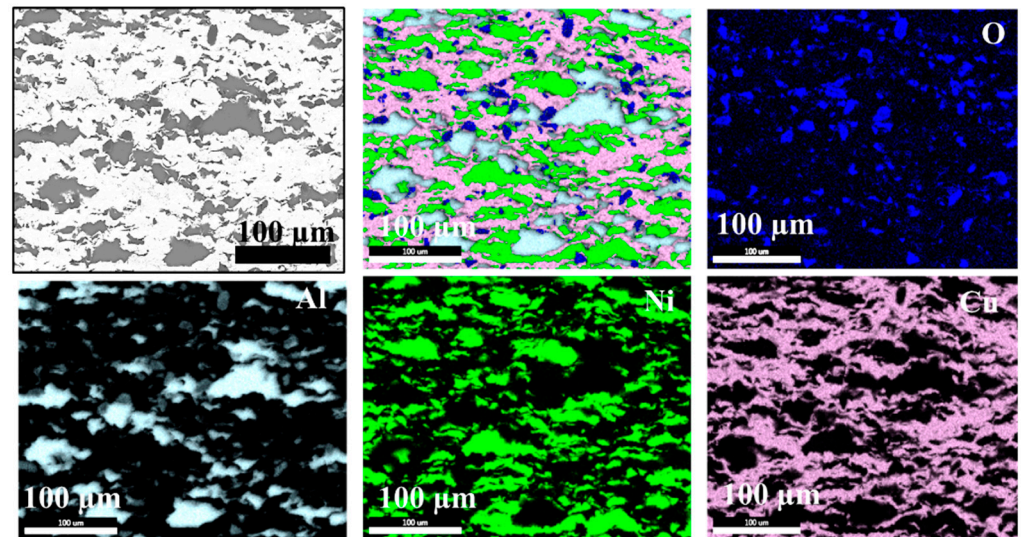


Figure 5. EDS mapping showing the elemental distribution of the S2 coating.

From Figure 6, it could be observed that all the constituent elements in the FSP-treated area such as Ni, Cu, Al and Al_2O_3 are well distributed across the coating layer, while all elements existed in the individual phase in the as-sprayed coating layers. The proper mixing of the elements in the FSP layer could be attributed to the constant tool rotation speed, which leads to the mechanical mixing of all elements across the processed region, as highlighted in Figure 6. The shear forces also aided in the breaking of large alumina particles in the matrix and further aided in its uniform distribution across the coating layer.

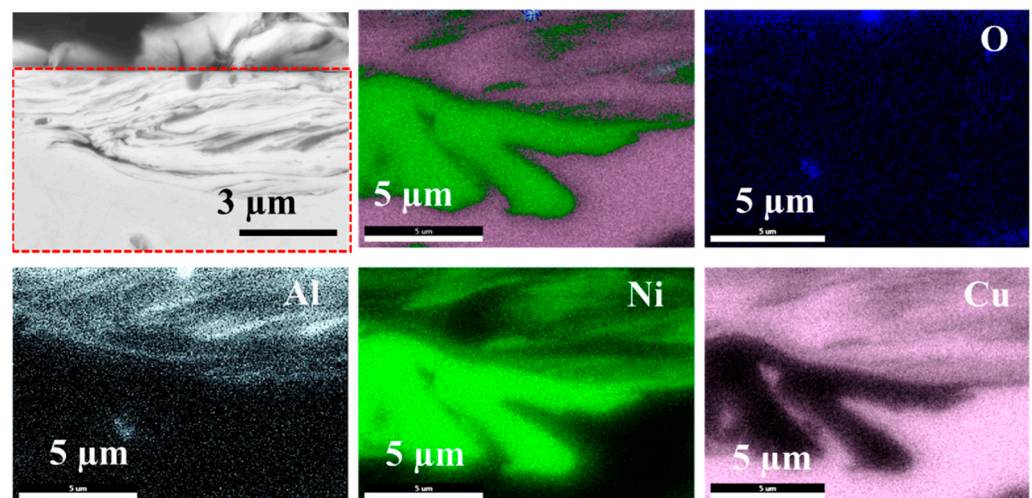


Figure 6. EDS mapping showing the elemental distribution of the FSP-S2 coating.

Figure 7 displays the EDS line scanning results of the cross-section FSP-S2. From Figure 7 left, the processed layer is found to be denser and more homogeneous with a significant reduction in porosity. The thickness of the processed region was measured to be in the range of 40 to 45 μm . According to the Figure 7 right, distribution of alloying elements was nearly equal at the processed region when compared to the unprocessed region. This improvement in the elemental distribution could be attributed to the microstructural refinement dissociation of individual phases (Cu, Al, and Ni) during the FSP. EBSD orientation contrast and the corresponding grain boundaries of the as-sprayed S2 and FSP-S2 coatings are shown Figure 8. The poor EBSD quality could be attributed the patterns overlapping or mixing near or upon the sub-grain boundaries or corresponding grain boundaries [70]. Furthermore, in addition to lattice strain and dislocation density, the low pattern quality might be attributed to the intermixing and overlapping of EBSD patterns near the grain boundaries [71]. From Figure 8a it can be noted that the deformation in the as-sprayed coatings is nearly heterogeneous, as shown by the mixture of equiaxed and columnar grains and long wide grains, i.e., some particles remain equiaxed while others are clustered.

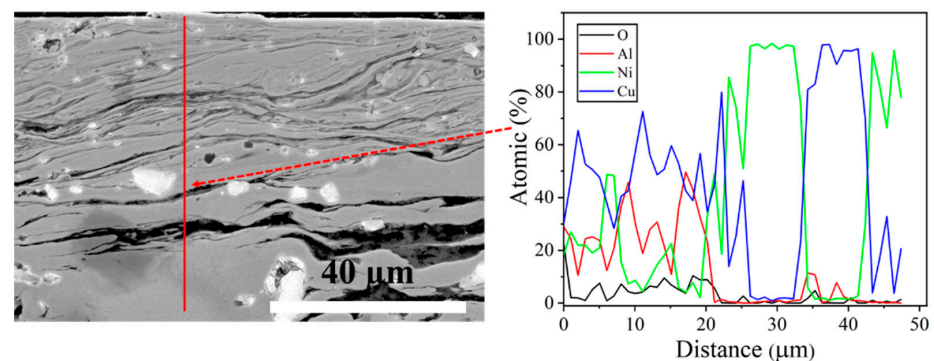


Figure 7. EDS line mapping showing the elemental distribution across the interface of the FSP region and the unprocessed FSP-S2 coating.

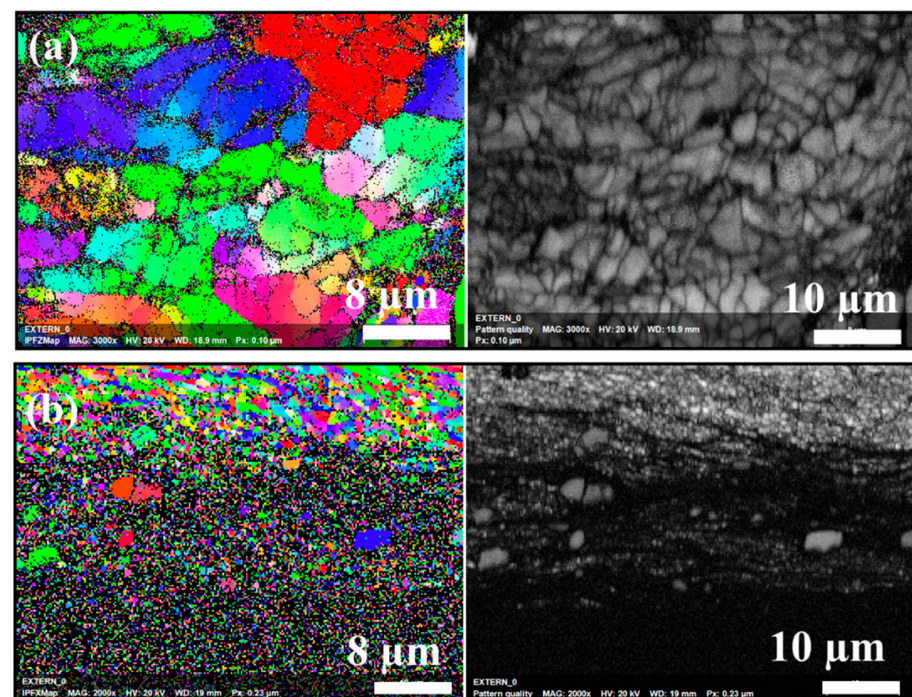


Figure 8. (a) S2 and (b) FSP-S2 coatings' Inverse pole figure (IPF) images and the corresponding gray-scale images with large-angle grain boundaries.

From Figure 8b, it can be observed that after FSP, there is a significant reduction in the grain size and that the deformation in the grains is highly inhomogeneous, denoted by a mixture of micro- and ultra-fine grains. Regardless of whether the constituent particles were heavily deformed or remained equiaxed, a large number of ultrafine grains were observed in the FSP region along with alumina particles in the processed region. As shown in the pattern quality map, the matrix and particle interfacial regions have a relatively poor pattern quality compared to the unprocessed regions.

Figure 9a shows the XRD spectra of the as-sprayed S1 and S2 coatings layers; it is noticeable that there is no phase transformation observed in as-sprayed coatings and all the phases that are present in the feedstock powders remain intact. However, after FSP treatment, a significant amount of phase transformation occurs in the coating with the evolution of new phases such as Al-Cu (ICDT #00-026-0016), and intermetallic NiAl_3 (ICDT #03-065-0140). This could have evolved due to the high strain rate deformation and excessive heat generated during the processing, similar to prior studies [69]. In all FSP-treated coatings, predominantly, two phases of NiAl_3 and Al-Cu are formed in approximately equal amounts determined from the analysis of XRD results. Certainly, during the friction stir processing, owing to the increase in temperature, the atomic mobility increased, leading to strain relaxation and the formation of homogeneous phases in the processed region [72].

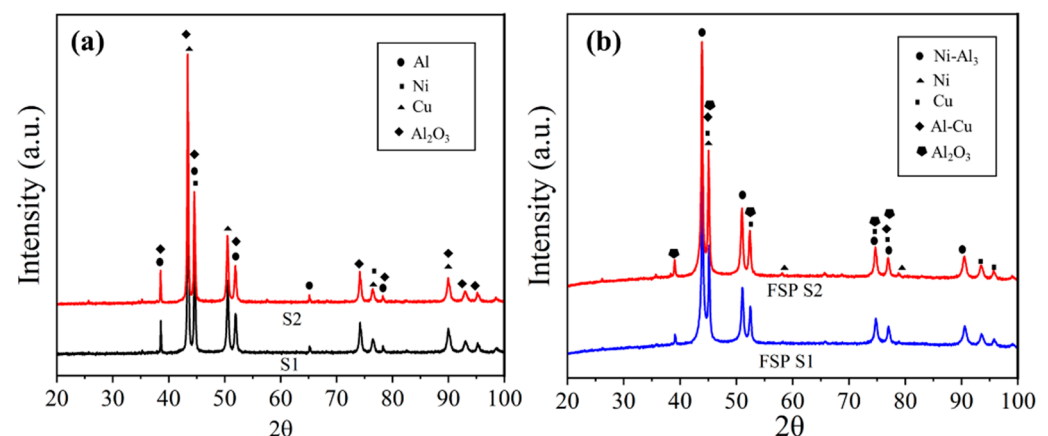


Figure 9. (a) XRD spectra of as-sprayed S1 and S2 coating and (b) of the friction stir processed FSP-S1 and FSP-S2 coatings.

The results of the microhardness investigation at the cross-section of as-deposited and friction stir processed coatings are shown and summarized in Figure 10. It is observed that microhardness for the as-sprayed coating is comparable to the AA2024 aluminum substrate ($\sim 110 \text{ HV}_{0.3}$), while the friction stir processed coating showed values around $160 \text{ HV}_{0.3}$, which is an improvement when compared to the unprocessed counterparts or the substrate material. There was also noticeable improvement in hardness values for S1 and S2 coatings after FSP by 40% and 30%, respectively. This improvement in hardness could be attributed to the finer microstructure and due to the formation of hard secondary phases which further hindered the dislocations; similar results were also reported in prior studies [69]. Moreover, coating hardness deviates slightly across the treated area, whereas it could be explained by the presence of hard alumina particles within the coating matrix and a lack of cohesion strength and bonding between particles forming the coating. This also supported by the high magnification images depicted in Figure 4, confirming that the coating microstructure plays a critical role in microhardness values distribution.

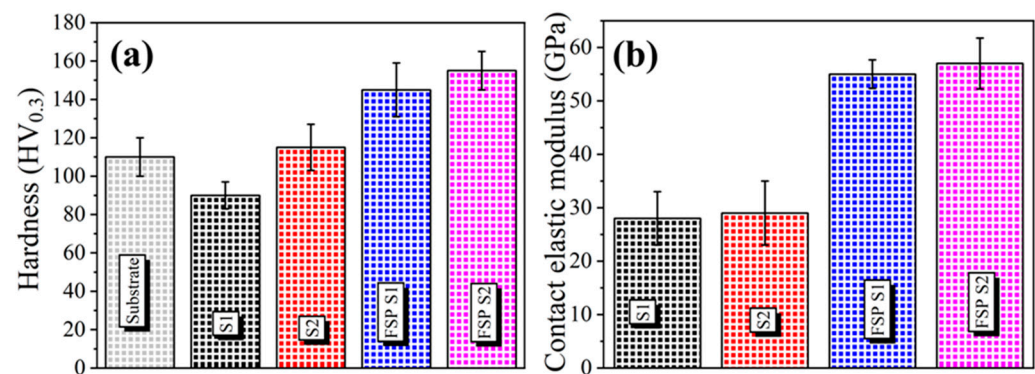


Figure 10. Mechanical properties of the as-sprayed and friction stir processed coatings (a) hardness, (b) contact elastic modulus.

Further, there was a significant improvement in the contact elastic modulus and, therefore, the plasticity index (H/E) of the coating layers; the processed S1 and S2 samples showed almost two times higher contact elastic modulus values compared to untreated ones. The increase could lead to the explanation of behavior in materials wear rates where plasticity index is known to have significant importance [15,73,74]. However, the improvement in the hardness and contact elastic modulus could be attributed to microstructural refinement during post-treatment operation leading to disintegration of splat boundaries in the as-deposited microstructure. Further, the coating layer densification is a consequence of the elimination of pores, the more homogeneous distribution of the secondary particles and the constituent elements across the coating layer [69].

3.2. Sliding Wear Behavior

Studying the mechanical properties of as-sprayed and friction stir processed coatings is essential to evaluate and predict the wear resistance of these coatings under operational conditions. Nevertheless, in some situations, the mechanical responses of these coatings depend on their operating conditions and the intrinsic properties under which these coatings work during their indented service life. In the case of the wear resistance, the material of the counter body, temperature, load rate, surface roughness and the type of relative movement between the surfaces could affect the properties, and, thus, could be investigated further.

The coefficient of friction (COF) was plotted as a function of sliding time for as-sprayed and FSP-processed coatings in Figure 11a. The initial spike of COF at the starting of the sliding wear could be attributed to a localized adhering of the coating asperities to the steel ball which were in contact during the sliding motion [75]. Then, the COF attains a steady-state value. This could happen because as the process continued, the surface of the coatings became smoother and presented less or no asperities [75]. Likewise, durable strain-hardened layers could have formed on the coating layers; this could be attributed to the Ni-particles in the binder phase. This hard layer on the coating surface further aided in maintaining a constant COF. From Figure 11a, it is evident that the processed coatings showed slightly lower COF than their unprocessed counterparts. This reduction in COF could be attributed to the improvement in the hardness and contact elastic modulus of the coating layers after processing, leading to an improved plasticity index of the treated coating materials. Prior studies also indicate that a high concentration of Cu (<11 wt%) can improve the elastic recovery and plasticity index [76]. FSP aided in the uniform distribution of all the constituent elements being distributed uniformly across the processed region; this furthered the load-bearing capacity of the processed coatings when compared to the as-sprayed coatings [76]. The specific wear rate at the end of test and after 100 m of a sliding distance were calculated using Equation (1) and (2), respectively. Among all the tested samples, the S1 FSP sample showed the lowest wear rate, which was almost two times lower than its unprocessed counterparts, and the friction stir processed S2 showed a nearly

25% lower wear rate than as-sprayed S2 (Figure 11b). Furthermore, the processed coatings showed lower COF than their unprocessed counterparts, along with a slight reduction in the specific wear rate. This improvement in the wear resistance could be associated with the improvement in the mechanical properties and the reduction of the microstructural defects in the coating.

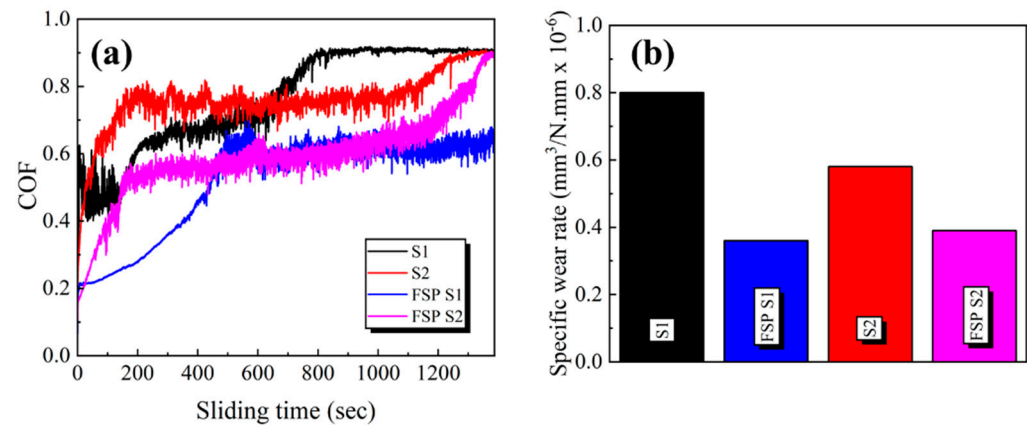


Figure 11. (a) Variation of coefficient of friction as a function of time, and (b) specific wear rate for a regular load of 2N of all developed coatings.

Analysis of the worn-out surfaces were performed to evaluate the wear mechanism, and surface images are depicted in Figure 12. Figure 12a,b show the low- and high-magnification SEM images of the wear track morphology of as-sprayed S2 coating layer. A significant amount of adhesive wear due to plastic flow deformations could be observed on the wear track. The formation of lips-like structures and the subsequent removal of these patches through microcracking were observed for as-sprayed and processed coatings. From the high-magnification SEM image (Figure 12b,d) of the wear track, it is evident that the as-sprayed S2 coatings underwent extensive scoring of the surface in the sliding direction and plowing, which resulted in significant amount of coating delamination, while for the FSP coatings (FSP-S2) the wear track showed significantly lower damage morphology with a shallow wear track and lower numbers of microgrooves, galling and delaminated spots. This could be attributed to the improvement in the hardness of the metallic binder phase after FSP and, due to the uniform distribution of the secondary particles (alumina) in the Cu-Ni-Al matrix, this further improved resistance towards plastic deformation. From Figure 12d, it can be noted the binder phase was preferentially squeezed up against alumina particles and worn away due to lack of adherence. This could lead to the formation of stable tribolayers as compared to the as-sprayed coating layers. Moreover, these severely deformed layers can detach from the wear track, and these particles, after repeated plastic deformation, can undergo oxidation. Due to the adhesive forces between the finer-wear debris and due to surface energy, small debris were agglomerated at some points and formed a compact layer [77–79]. Further, from the low-magnification SEM image (Figure 12a,c), it can be seen that there is a reduction in the width of the wear track after FSP. The primary mode of coating failure was observed through the coating delamination and could be attributed to the poor hardness and the low cohesion strength of the as-sprayed coating. This is evident from the high magnification SEM images (Figure 12b,d). The formation of micro-grooves and micro-cracks could also be observed for only the FSP-processed samples and could be attributed to a change in the microstructure after FSP, where the processed layer in the coatings showed a nearly homogeneous microstructure. Moreover, all the constituent elements were uniformly distributed and this layer exhibited no defects, such as porosities and microcracks. Owing to the uniform mixing of all the phase the coatings, work hardening capacity could have improved. This could aid in formation of a much more stable work-hardened layer in FSP coatings, while for as-sprayed coatings no such layers were formed. This could have resulted in high amount of coating

delamination of as-sprayed coatings during sliding wear. Additionally, the FSP-processed samples showed a shallower and narrower wear track compared to their unprocessed counterparts. Moreover, friction stir processed coatings showed significantly lower wear marks where a large portion of the coating remains intact with minimal surface damage. The primary mode of coating failure during sliding wear for the as-sprayed and friction stir processed coatings could be attributed to the delamination, micro-cracks, galling and groove formation.

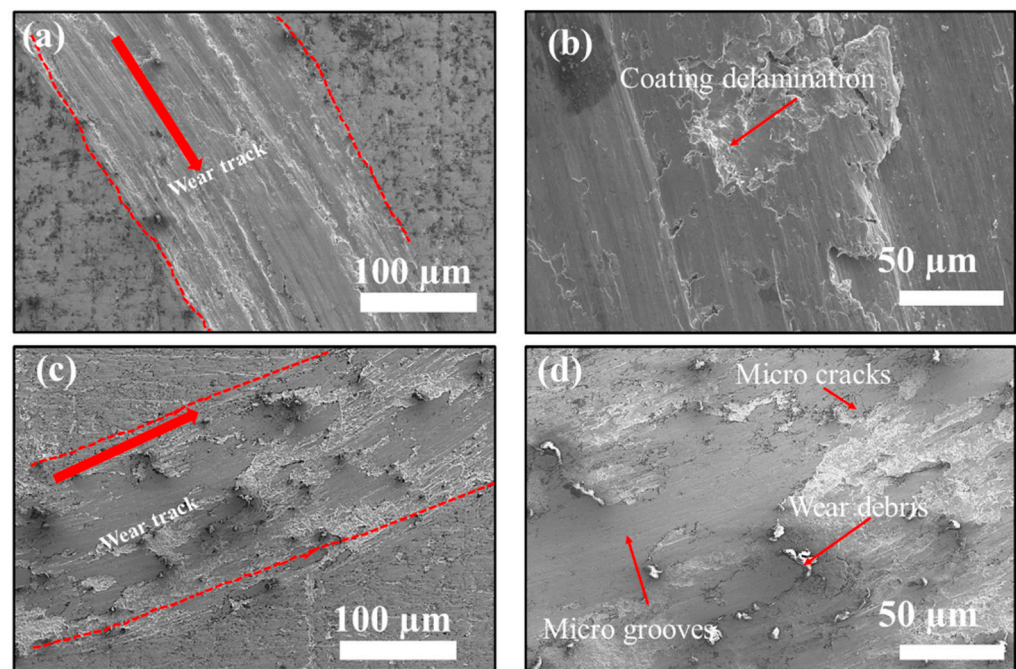


Figure 12. (a,b) low- and high-magnification SEM images of the wear track of S2 coating and (c,d) low- and high-magnification SEM images of the wear track of FSP-S2 coating.

4. Conclusions

Using the cold spray process, thick and dense Cu-Ni-Al-based metal matrix composites with varying weight fraction of alumina particles were deposited onto aluminum substrate using in-house-developed medium-pressure cold spray system.

- These coatings showed a well-bonded substrate interface without the presence of any interfacial cracking, and with a very low level of porosity. The developed coatings had an average coating thickness of around $600 \pm 10 \mu\text{m}$. The as-sprayed coatings showed areas that were either rich in Cu, Ni and Al, while the distribution of alumina particles was nearly uniform across the coating layers.
- Post-processing of the cold-sprayed Cu-Ni-Al alumina composite coatings was achieved using a high strain rate deformation technique during a friction stir processing operation with the aim to enhance the microstructural and mechanical properties. FSP aided in a significant amount of grain refinement and better dispersion of fine alumina particles across the processed layer.
- The processed coating showed a nearly 2-times enhancement in the contact elastic modulus compared to its unprocessed counterpart, along with a 1.4 times increase in microhardness. Furthermore, the FSP samples showed a slightly lower wear rate than their unprocessed counterparts.
- Friction stir processing leads to a nearly three-times reduction in the specific wear rate of the as-sprayed coating during sliding wear. This improvement in wear resistance could be attributed to the processed coating layers' superior microstructure and mechanical properties.

- From the SEM studies, it was evident that as-sprayed coatings layers showed deeper wear marks where the large portion of the coating surface were delaminated from the surface, while in the processed coating, a large area of the surface remains intact with negligible amount of coating damage. The primary mode of coating failure during sliding wear for the as-sprayed and friction stir processed coatings could be attributed to the delamination, micro-cracks, galling and groove formation.

Thus, the present study showcases that friction stir processing is a versatile and highly effective tool for significantly improving the sliding wear resistance of cold-sprayed coatings through microstructural refinement.

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