

Effect of Mo Content on the Microstructure and Properties of FeCoCrNiMo_x High-Entropy Alloy Coatings Prepared by High-Velocity Oxy-Fuel Spraying

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High-entropy alloy coatings (HEA) were applied to key components such as cutting tools, molds, and valves. In this research, FeCoCrNiMo_x high-entropy alloy coatings were deposited on 45 steel substrates using high-velocity oxy-fuel (HVOF) spraying. The influence of molybdenum (Mo) content on the coating properties was systematically investigated by characterizing the microstructure of the coating surfaces and cross-sections, as well as by evaluating the hardness, and wear resistance. The coating thickness ranges from 75 – 150 μm. During the spraying process, some powder particles with high Mo content have remained incompletely melted, leading to their embedding in the coating in a solid state. By adding Mo element, the coatings hardness reached 750 – 800 HV0.3, more than twice higher than that of the steel substrate. The increased Mo content potentially enhances the solid solution strengthening effect within the high-entropy alloy system, thereby suppressing segregation tendencies of elements such as Fe and consequently improving overall coating homogeneity.

Keywords: high-velocity oxy-fuel spraying, FeCoCrNiMo high-entropy alloy coatings, microstructure, wear resistance, corrosion resistance.

1. INTRODUCTION

Owing to their unique solid solution structure formed by multiple principal elements, high-entropy alloys (HEAs) exhibit exceptional wear and corrosion resistance, attracting increasing research attention in recent years. High-entropy alloy coatings, which retain the superior properties of HEAs without compromising the characteristics of the substrate, show immense application potential and market value in surface strengthening for aerospace, high-end industries, and related fields [1 – 15].

Currently, many scholars have conducted studies on the wear resistance and corrosion resistance of high-entropy alloy coatings, as well as the influence of alloying elements on the properties of these coatings. Li et al. [16] studied the microhardness, wear resistance, and corrosion resistance of Al_xCrFeCoNiCu high-entropy alloy coatings on aluminum by laser cladding. The results revealed that the effect of Al on wear resistance followed a trend similar to that of hardness. Among the compositions, Al_{0.8}CrFeCoNiCu demonstrated the best corrosion resistance, which manifested as intergranular corrosion. This high-entropy alloy was identified as the most promising candidate for enhancing the surface properties of aluminum alloys. Xiao et al. [17] investigated the microstructure and wear behavior of FeCoNiCrMn high entropy alloy coating deposited by plasma spraying. Analysis of the wear tracks revealed that splat spalling was the dominant wear mechanism for the as-sprayed coatings. The enhanced wear resistance achieved by increasing the H₂ flow rate and through annealing was

attributed to the improved cohesive strength between individual splats. Zhang et al. [18] fabricated FeCuNiTiAl high-entropy alloy coating on Ti6Al4V substrate by laser metal deposition, and investigated the microstructure and wear resistance. The results showed the predominant wear mechanism for the Ti6Al4V substrate is abrasion, while the FeCuNiTiAl HEA coating primarily undergoes adhesive wear, with minor contributions from oxidation and abrasion. Tian et al. [19] prepared CrCoFeNiMn HEA coating on the surface of the substrate by laser cladding for surface strengthening, and studied the microstructure, mechanical properties, and friction behavior. The result showed that the wear characteristics of the CrCoFeNiMn HEA coatings involve abrasive, oxidative, and adhesive mechanisms. Relative to the substrate, the coating region exhibited a 41.44 % decrease in maximum wear depth.

In summary, most research was concentrated on the microstructure, wear resistance, and corrosion resistance of high-entropy alloy coatings. However, the influence of key alloy element contents on the structure and properties of these coatings was rarely reported, particularly the effect of Mo content on the microstructure and properties of the coatings fabricated by high-velocity oxygen fuel (HVOF) technology. In this work, the impact of Mo content on the microstructure, wear resistance, and corrosion resistance of FeCoCrNiMo_x high-entropy coatings prepared by HVOF were systematically investigated.

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2. EXPERIMENTAL

2.1. Experimental materials

The materials utilized in this study for preparing the FeCoCrNiMo_x ($x = 0, 0.5, 1$) high-entropy alloy coatings via high-velocity oxy-fuel (HVOF) spraying included FeCoCrNi , $\text{FeCoCrNiMo}_{0.5}$, and FeCoCrNiMo powders. The substrate material was AISI 1045 steel. The particle size range was 10–50 μm .

2.2. Substrate pretreatment

Prior to the high-velocity oxy-fuel (HVOF) spraying process, the 45# substrate surfaces were grit-blasted with alumina grit to remove surface oxides, rust, and other contaminants. This treatment was conducted to activate the surface and enhance its roughness for improved coating adhesion. The resulting surface roughness was controlled at approximately $R_a = 3.0\text{--}4.0\ \mu\text{m}$.

2.3. Coating deposition

The spray powders were first dried in a vacuum drying oven to remove moisture. Subsequently, the coatings were deposited onto the substrate surfaces using a high-velocity oxy-fuel (HVOF) spraying system to form dense coatings. During the spraying process, the spray gun was maintained perpendicular to the substrate and traversed at a constant speed to prevent localized overheating and to ensure uniform coating thickness. After deposition, the coated samples were allowed to cool naturally to room temperature.

The as-sprayed FeCoCrNiMo_x ($x = 0, 0.5, 1$) HEA coatings were then sectioned using a CNC wire-cut electrical discharge machine (EDM). From each coating, two small specimens of 10 mm $\times$ 10 mm and four large specimens of 20 mm $\times$ 20 mm were prepared.

All specimens underwent a meticulous cleaning procedure in an ultrasonic cleaner. They were first immersed in deionized water and ultrasonically cleaned for three minutes. This water cleaning cycle was repeated three times. Afterwards, the specimens were immersed in alcohol for a final ultrasonic cleaning step. Finally, all specimens were thoroughly dried using an electric air blower and stored in bags for subsequent analysis.

The 10 mm $\times$ 10 mm specimens with the coating surface of interest were mounted in resin for ease of handling during subsequent metallographic preparation. The mounted samples were then ground and polished sequentially using silicon carbide sandpapers with grit sizes of 600, 800, 1000, and 1200 to achieve a smooth surface suitable for microstructural examination.

2.4. Testing methods

The Vickers hardness of the high-entropy alloy samples was measured using the indentation hardness method. For each sample with a specific Mo content, eight hardness values with close agreement were obtained. A test force of 200.0 g and a dwell time of 10 s were applied during testing. The indentations were subsequently observed under an optical microscope, and the lengths of the impression diagonals were recorded.

To prevent interaction between adjacent indentations and minimize errors arising from local microstructural

variations, a sufficient spacing was maintained between successive hardness impressions on the sample surface. For each set of eight measurements obtained per sample, the highest and lowest values were excluded. The average Vickers hardness was then calculated from the remaining six values. The friction and wear tests were performed on 20 mm $\times$ 20 mm specimens of the three coatings with different Mo contents. The tests were conducted under a load of 60 N for a duration of 30 minutes, and the corresponding friction data were recorded.

The microstructural characteristics of the HEA powder and the resulting coatings were characterized using a Quanta 250 FEG scanning electron microscope (SEM). Phase analysis was performed via X-ray diffraction (XRD) on a Rigaku D/max-2550VB diffractometer with $\text{Cu K}\alpha$ radiation, scanning 2θ angles from 20° to 80° . Coating porosity was assessed quantitatively by analyzing at least fifteen randomly selected cross-sectional SEM images (at 1000 $\times$ magnification) with Image Pro-Plus 6.0 software.

3. RESULTS AND DISCUSSION

3.1. Powder morphology and structure

As shown in Fig. 1, the Mo-free FeCoCrNi high-entropy alloy powder exhibits a single FCC solid solution phase due to the mutual dissolution of the principal elements (Fe, Co, Cr, Ni). The corresponding XRD pattern displays only characteristic FCC peaks, such as (220), (330), and (111), with sharp profiles and no detectable secondary phases, indicating good compositional homogeneity of the powder. With the addition of Mo, however, the atomic radius of Mo, which is significantly larger than that of the other constituent elements, induces lattice distortion and an increase in the lattice constant during solid solution formation. This leads to a shift of the primary FCC peaks toward lower angles. The XRD pattern of the $\text{FeCoCrNiMo}_{0.5}$ powder still maintains symmetrical peak shapes, suggesting that the FCC solid solution remains the dominant phase. Nevertheless, a Mo-rich FCC solid solution phase may gradually form based on Co-Cr-Ni-Mo.

SEM imaging and EDS mapping were performed on the three powders with different Mo contents, and the results are presented in Fig. 2.

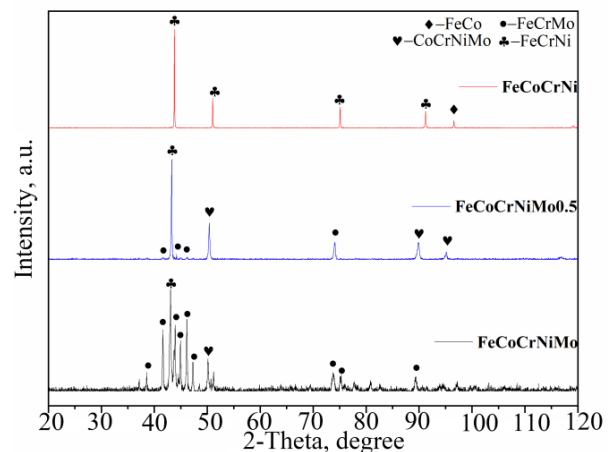


Fig. 1. XRD patterns of HEA powder

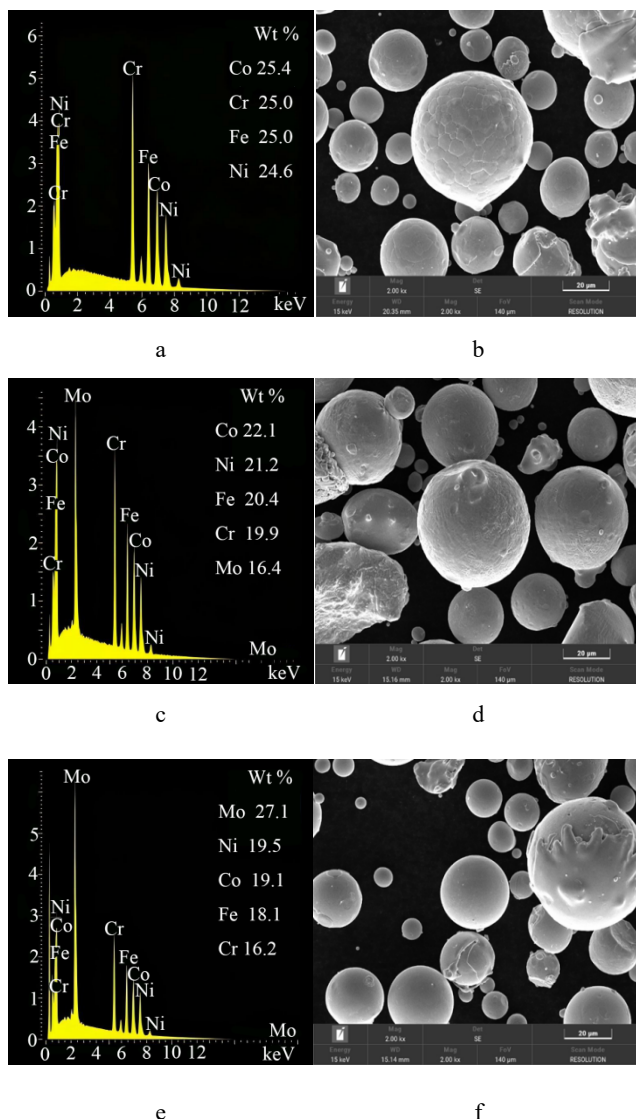


Fig. 2. EDS and SEM photos of HEA powders: a–EDS of FeCoCrNi powder; b–SEM of FeCoCrNi powder; c–EDS of FeCoCrNiMo_{0.5} powder; d–SEM of FeCoCrNiMo_{0.5} powder; e–EDS of FeCoCrNiMo powder; f–SEM of FeCoCrNiMo powder

As revealed by the EDS data in Fig. 2 a, the concentrations of the four principal elements Fe, Co, Cr, and Ni are all close to the designed equiatomic ratio. This result aligns well with the high-entropy alloy design concept, which emphasizes multiple principal elements in near-equiatomic proportions. Fig. 2 b shows the powder morphology, revealing particle sizes ranging approximately from 20 μm to 50 μm . Most particles exhibit relatively good sphericity, which generally facilitates subsequent powder processing by improving flowability and packing density [20]. However, as indicated in Fig. 2 c, the actual Mo content appears slightly lower than the nominal value. With the addition of Mo, local enrichment of Mo is observed in certain regions, as seen in Fig. 2 e, which may promote the formation of the σ phase. This finding is consistent with the XRD analysis of the FeCoCrNiMo high-entropy alloy powder.

3.2. Microstructure and XRD analysis of FeCoCrNiMo_x high-entropy alloy coatings

3.2.1. Microstructure of the FeCoCrNi high-entropy alloy coating

Fig. 3 presents the surface SEM images of the FeCoCrNi high-entropy alloy coating deposited by high-velocity oxy-fuel (HVOF) spraying at magnifications of 200 $\times$, 500 $\times$, 1000 $\times$, and 2000 $\times$. It is preliminarily concluded that the dark contrast arises because Mo likely exists in the form of nanoscale precipitates or is dissolved within the matrix [21]. In such cases, the atomic number contrast of Mo may be overshadowed by surrounding elements with lower atomic numbers (e.g., Fe, Cr), resulting in an overall darker appearance in SEM images. Nevertheless, EDS point analysis still detects locally high Mo content in these areas.

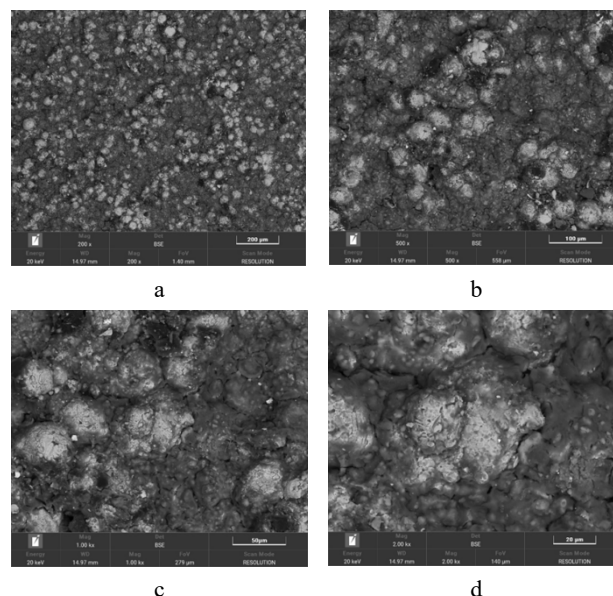


Fig. 3. SEM photos of the surface of FeCoCrNi coating: a–magnification of 200 $\times$; b–magnification of 500 $\times$; c–magnification of 1000 $\times$; d–magnification of 2000 $\times$

The coating thickness ranges from 75–150 μm . Although the white particles appear with high brightness in the SEM images (Fig. 3), EDS results show their Mo content is only 0.3 wt.% and 0.6 wt.%. In contrast, the dark gray regions, despite their lower image contrast, contain up to 7.5 wt.% Mo, as shown in Fig. 5. This confirms that the image brightness is not directly correlated with the distribution of Mo.

From the cross-sectional SEM image in Fig. 4 a, a small amount of light-gray phase can be observed within the FeCoCrNi coating.

Based on EDS point and mapping analyses of the cross-section (Fig. 6), combined with the prior surface analysis and the spraying sequence, it is inferred that the detected Mo originates from residual FeCoCrNiMo powder remaining inside the spraying equipment (e.g., the powder feeder) from previous experiments.

This residual material contaminated the current FeCoCrNi coating, leading to the localized presence of Mo in both the surface and cross-section.

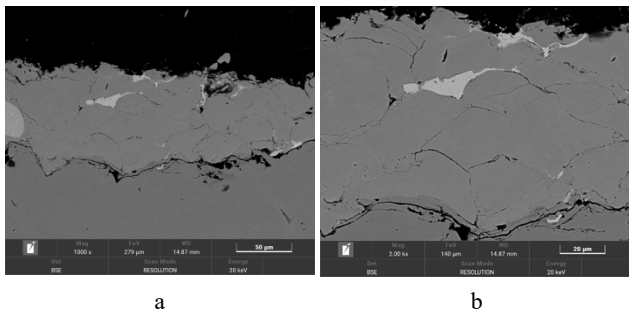


Fig. 4. SEM photos of cross-section of FeCoCrNi coating: a – magnification of 1000×; b – magnification of 2000×

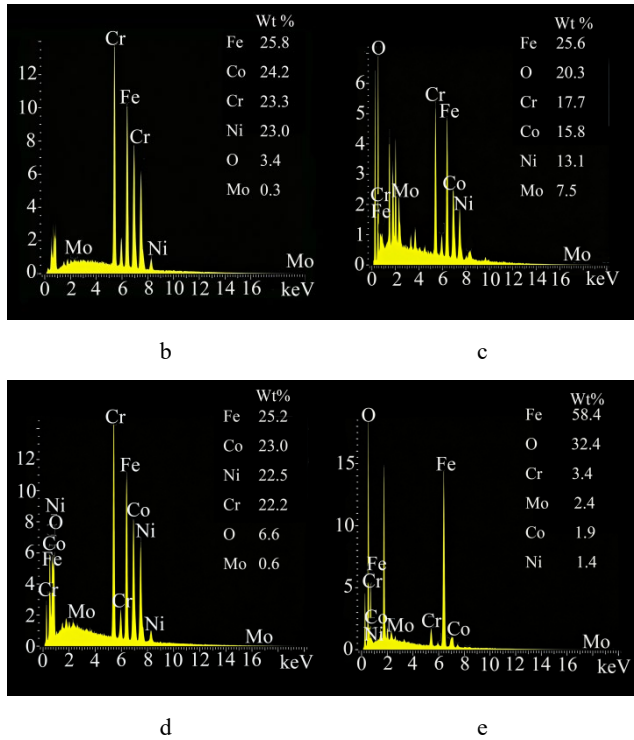
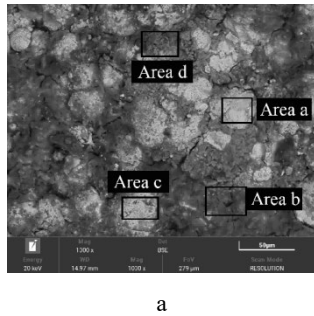


Fig. 5. EDS photos of FeCoCrNi HEA coating surface: a – coating surface; b – section a; c – section b; d – section c; e – section d

Subsequent EDS mapping of the coating cross-section shows an overall low average Mo content of about 0.8 wt.%. However, EDS point analysis on the isolated light-gray regions reveals a significantly high Mo content of 29.2 wt.%, whereas the surrounding areas contain only about 0.2 wt.% Mo. This confirms that the light-gray phase observed in both EDS and SEM results corresponds to residual FeCoCrNiMo powder that contaminated the FeCoCrNi powder feedstock.

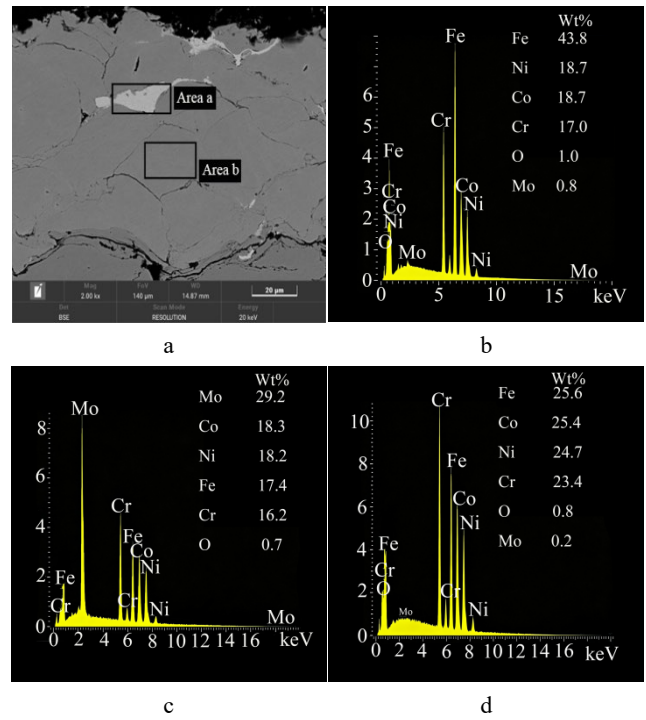


Fig. 6. EDS photos of cross-section of the coatings: a – cross-section of coating; b – surface scanning of coating; c – point scanning of section a; d – point scanning of section b

3.2.2. Microstructure of FeCoCrNiMo_{0.5} coating

SEM examination of the FeCoCrNiMo_{0.5} high-entropy alloy coating revealed the presence of highly bright, light-gray regions on the coating surface, as shown in Fig. 7.

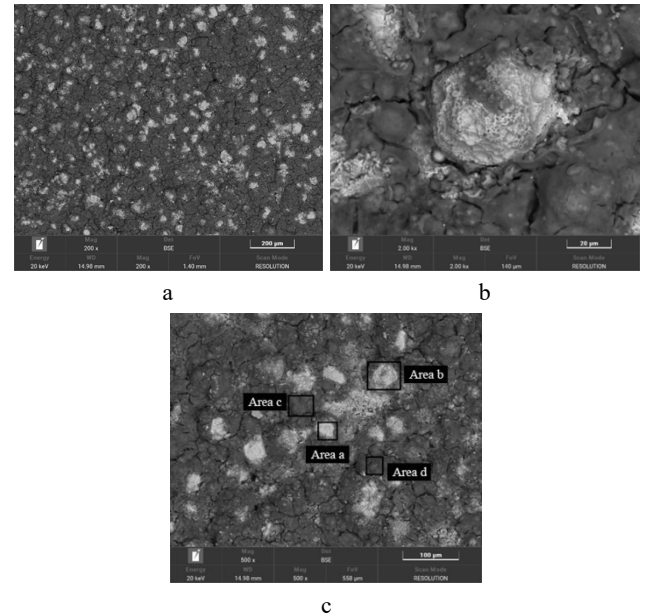


Fig. 7. EDS area of FeCoCrNiMo_{0.5} coating surface

At a higher magnification of 2000×, these bright areas exhibited a porous morphology with randomly distributed pores. In contrast, the darker regions showed a lamellar structure, though pores were also present on their surface. EDS point analysis was performed on selected surface areas, and the results are summarized in Table 1. The bright regions contained 17.1 wt.% Mo and 3.0 wt.% O, while

other elements such as Fe, Co, Ni, and Cr were present in nearly equiatomic proportions.

Table 1. Element content of FeCoCrNiMo_{0.5} coating

	Mo, wt.%	O, wt.%	Fe+Co+Cr+Ni, wt.%
Area a	17.1	3.0	79.9
Area b	17.5	3.8	78.7
Area c	22.3	55.0	22.7
Area d	22.6	37.2	40.2

In the dark regions, the Mo and O contents were significantly higher, reaching 22.3 wt.% and 55.0 wt.%, respectively, whereas the concentrations of other elements were notably reduced, for instance, Fe and Co dropped to 10.6 wt.% and 5.3 wt.%, respectively.

During the spraying process, some powder particles with high Mo content may have remained incompletely melted, leading to their embedding in the coating in a solid state. The weak interparticle bonding of these unmelted particles likely resulted in the formation of pores [22].

Fig. 8 shows the cross-sectional SEM images of the FeCoCrNiMo_{0.5} high-entropy alloy coating. The coating generally exhibits a uniform and dense lamellar structure with minimal visible porosity, dominated by gray regions with limited pore formation.

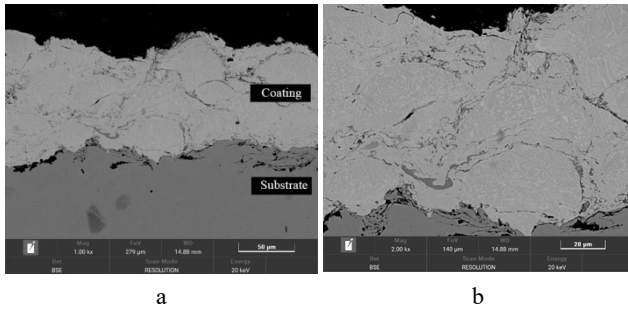


Fig. 8. SEM photos of cross-section of FeCoCrNiMo_{0.5} coating: a – magnification of 1000×; b – magnification of 2000×

Observation of the microstructure at a higher magnification of 2000× (Fig. 9) reveals the presence of some pores, which may be attributed to low spraying power or inappropriate spray distance during the high-velocity oxy-fuel (HVOF) process [23–25]. The formation of microcracks is preliminarily attributed to stress concentration: the surface temperature of the material increases during spraying, but non-uniform cooling rates across the coating result in incomplete contraction, leading to microcracking. These structural defects are likely to influence the coating performance.

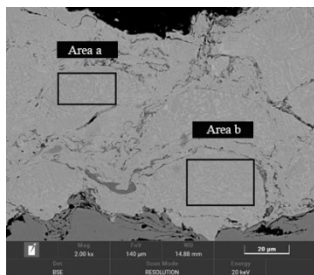


Fig. 9. Selected area of cross-section of FeCoCrNiMo_{0.5} coating

Fine grains are observed in localized areas, indicating that dynamic recrystallization or rapid solidification occurred during the coating preparation. EDS point analysis was conducted on selected regions of the coating cross-section, and the results are summarized in Table 2.

Table 2. Element content of cross-section of FeCoCrNiMo_{0.5}

	Fe	Co	Cr	Ni	Mo	O
	wt.%					
Area a	20.3	21.1	19.2	21.4	17.5	0.7
Area b	20.6	21.8	18.8	21.3	16.8	0.8

3.2.3. Microstructure of the FeCoCrNiMo high-entropy alloy coating

Fig. 10 displays the surface morphology of the FeCoCrNiMo high-entropy alloy coating. A significant number of spherical particles can be clearly observed on the coating surface. The coating exhibits a relatively homogeneous gray tone without distinct bright white or dark black regions, suggesting a uniform elemental distribution across the surface. This homogeneity is crucial, as contrast variations in SEM images primarily arise from differences in atomic number.

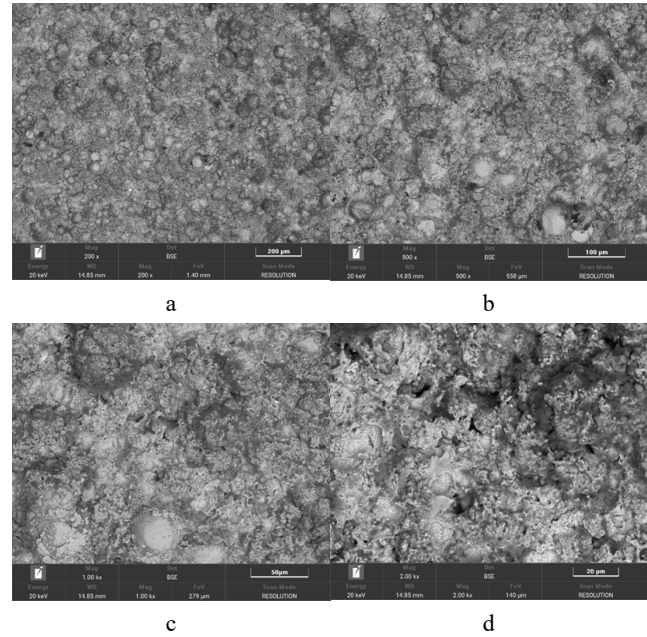


Fig. 10. SEM photos of FeCoCrNiMo coating surface: a – magnification of 200×; b – magnification of 500×; c – magnification of 1000×; d – magnification of 2000×

EDS point analysis was conducted on selected regions of the coating surface, with the corresponding results presented in Fig. 11 and Table 3.

Table 3. Element content in the selected areas on FeCoCrNiMo coating surface

	Fe	Co	Cr	Ni	Mo	O
	wt.%					
Area a	15.7	14.3	14.9	13.8	26.8	14.5
Area b	18.8	11.5	13.1	9.8	22.8	24.0
Area c	16.0	15.3	14.2	14.8	27.0	12.7
Area d	14.5	3.3	9.6	2.9	23.4	46.4
Area e	20.1	8.8	11.8	8.7	19.0	31.6
Area f	16.8	17.5	20.0	16.9	20.0	8.6

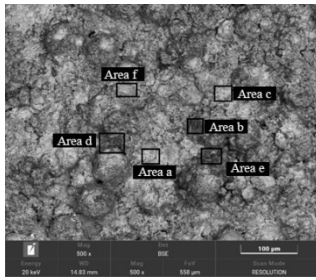


Fig. 11. Selected area of FeCoCrNiMo coating surface

The EDS spectra from most measurement points exhibit relatively minor fluctuations in the weight percentages of the principal elements Fe, Co, Cr, Ni, and Mo. Specifically, the Fe content ranges approximately from 14.46 wt.% to 20.09 wt.%, while the Mo content varies between 19.03 wt.% and 27.02 wt.%. However, spectra acquired from dark regions show a notably different composition, characterized by significant deviation in Mo concentration and substantially elevated oxygen content, suggesting the occurrence of localized oxidation in these specific areas.

The limited variation in principal element concentrations observed in the majority of the gray-white regions indicates that the overall coating composition is close to the designed FeCoCrNiMo equiatomic ratio. This consistency aligns with the solid solution characteristics typical of high-entropy alloys [26].

The relatively lower concentrations of Co and Ni detected in these oxidized regions may be attributed to signal suppression effects caused by the surface oxidation layer [27, 28]. The increased Mo content potentially enhances the solid solution strengthening effect within the high-entropy alloy system, thereby suppressing segregation tendencies of elements such as Fe and consequently improving overall coating homogeneity.

Fig. 12 presents the cross-sectional morphology of the FeCoCrNiMo high-entropy alloy coating.

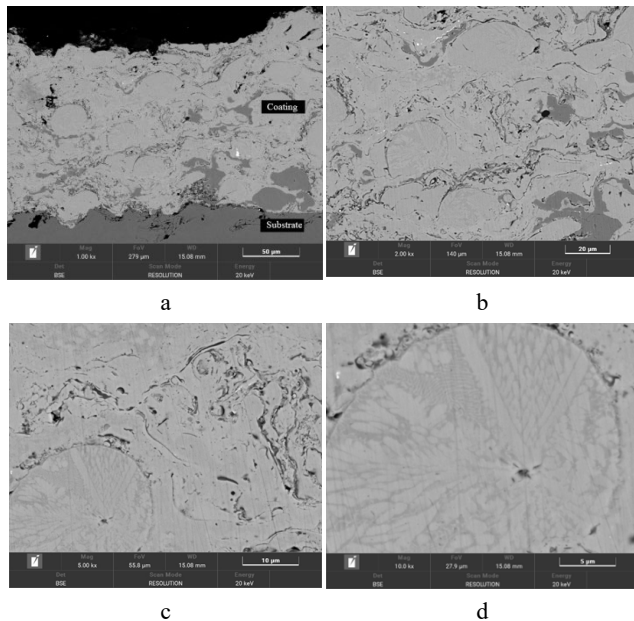


Fig. 12. SEM photos of cross-section of FeCoCrNiMo coating: a – magnification of 1000×; b – magnification of 2000×; c – magnification of 5000×; d – magnification of 10000×

As shown in Fig. 12 a, the coating adheres to the 45 steel substrate primarily through mechanical interlocking, which represents a typical interfacial bonding characteristic in thermal spray coatings. During the HVOF process, the molten high-entropy alloy particles experience rapid heating upon impact with the substrate followed by immediate cooling. This can lead to the entrapment of oxygen from the surrounding atmosphere, forming oxide inclusions. Simultaneously, insufficiently melted original powder particles may become "frozen" within the coating, creating isolated "island-like" unmelted particle regions.

At higher magnification (10,000×), distinct dendritic structures are clearly visible within the coating microstructure, as demonstrated in Fig. 12 d. The clear boundaries between the dendritic arms and inter-dendritic regions indicate that the solidification process involved dendritic growth.

Fig. 13 presents the EDS analysis of the FeCoCrNiMo high-entropy alloy coating cross-section. The dendritic structure shows significantly elevated Mo content in selected areas b and c, while the distribution of Cr, Fe, Co, and Ni remains relatively uniform. This suggests the segregation of Mo in the inter-dendritic regions.

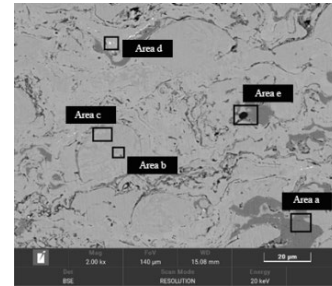


Fig. 13. EDS of selected area on cross-section of FeCoCrNiMo coating

Selected area d in Table 4 shows the unexpected detection of W, while the EDS mapping data in Table 5 shows no abnormal W distribution.

Table 4. Element content of selected cross-section of FeCoCrNiMo coating

	Fe	Co	Cr	Ni	Mo	O	W
	wt. %						
Area a	0.7	34.5	31.3	33.0	0.1	0.4	0
Area b	17.0	19.7	16.5	17.0	31.0	0.5	0
Area c	17.5	19.1	15.0	20.7	16.9	0.8	0
Area d	10.3	13.7	10.6	9.8	17.6	1.1	36.9
Area e	0.6	1.0	68.6	0.8	0.7	28.4	0

Table 5. Element content of surface scanning on cross-section of FeCoCrNiMo coating

	Fe	Co	Cr	Ni	Mo	O
	wt. %					
Surface scanning	16.1	19.1	17.0	18.5	26.8	2.6

This discrepancy is preliminarily attributed to either raw material contamination or instrumental interference. EDS point analysis of dark regions reveals significant enrichment of Co, Ni, and Cr in area a, with almost no detection of Fe and Mo, possibly representing incompletely mixed raw material particles. Area e shows extremely high Cr content with O reaching 28.4 wt.% and other elements at

minimal levels, indicating this region consists primarily of chromium oxide.

3.2.4. Phase composition and XRD analysis of FeCoCrNiMo_x high-entropy alloy coatings

Fig. 14 displays the XRD patterns of the FeCoCrNiMo_x ($x = 0, 0.5, 1$) high-entropy alloy coatings. The pattern of the FeCoCrNi coating ($x = 0$) is primarily indexed to an FCC structure, identified as a FeCrNi-based solid solution. The presence of minor peaks associated with Mo-containing phases is attributed to contamination from residual Mo-containing powder during the HVOF process. Characteristic FCC peaks are observed in the 2θ range of 40° to 70° .

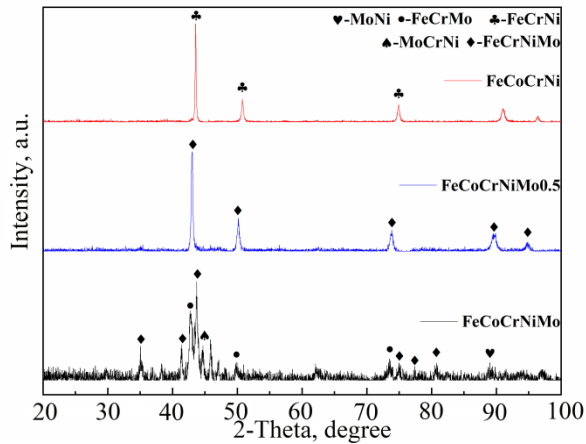


Fig. 14. XRD patterns of the HEA coatings

For the FeCoCrNiMo_{0.5} coating, a single FCC solid solution phase is identified without detectable secondary phases. Compared to the Mo-free coating, the main diffraction peaks of the Mo_{0.5} coating shift slightly toward lower angles, which is inferred to result from lattice expansion due to the dissolution of larger Mo atoms.

The XRD pattern of the FeCoCrNiMo coating ($x = 1$) shows that the primary phase remains an FCC solid solution; however, the peak intensities decrease and the peak widths further increase, suggesting a tendency toward amorphization or the formation of nanocrystalline structures.

3.3. Tribological properties of FeCoCrNiMo_x high-entropy alloy coatings

Fig. 15 shows the friction coefficient curves of the FeCoCrNiMo_x ($x = 0, 0.5, 1$) high-entropy alloy coatings.

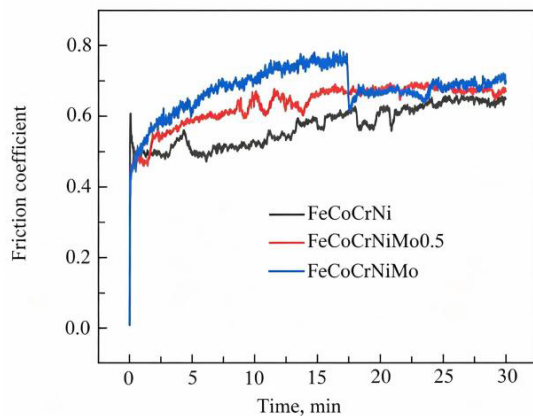


Fig. 15. The wear friction coefficient of the HEA coatings

The FeCoCrNi coating exhibits the lowest friction coefficient, while a noticeable increase in the friction coefficient is observed with increasing Mo content. The friction curve of the FeCoCrNi coating displays significant fluctuations over time, suggesting an unstable surface state during the wear process. The initial rapid rise in the friction coefficient may be related to surface roughness or the breakdown of the oxide layer, while the subsequent fluctuations reflect repeated embedding and detachment of abrasive particles. In contrast, the FeCoCrNiMo_{0.5} coating shows relatively minor fluctuations in its friction coefficient, which may be attributed to the formation of a stable protective layer on the coating surface.

The measured wear rates for the FeCoCrNi, FeCoCrNiMo_{0.5}, and FeCoCrNiMo high-entropy alloy coatings are $8.009 \times 10^{-5} \text{ mm}^3/\text{N}\cdot\text{m}$, $1.106 \times 10^{-4} \text{ mm}^3/\text{N}\cdot\text{m}$, and $1.125 \times 10^{-4} \text{ mm}^3/\text{N}\cdot\text{m}$, respectively, and the standard deviations range is $0.03 \times 10^{-6} \text{ mm}^3/(\text{N}\cdot\text{m})$.

The wear rate of the FeCoCrNi coating is significantly lower than those of the two Mo-containing coatings. The wear rates of the two Mo-containing coatings are very close to each other, showing no substantial difference.

Molybdenum is typically added to alloys to enhance hardness and high-temperature stability, which should theoretically improve wear resistance. By adding Mo element, the coating's hardness reached 750–800 HV_{0.3}, more than twice higher than that of the steel substrate. The standard deviations range 15–25 HV. However, the wear rate data indicate that, compared to the FeCoCrNi coating, the addition of Mo degrades the wear resistance. It is hypothesized that the introduction of Mo alters the original single FCC phase structure of FeCoCrNi, leading to the formation of a brittle σ phase. This brittle phase is more prone to fracture and spalling during the friction process, thereby increasing material loss.

Furthermore, the addition of Mo increases the residual stress within the high-entropy alloy coatings and introduces more micro-defects, such as pores and microcracks, which compromise the coating integrity and load-bearing capacity, as Fig. 16 exhibits.

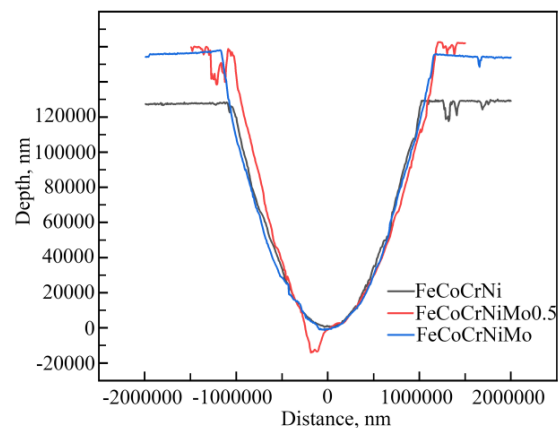


Fig. 16. The friction profile of HEA coatings

In conclusion, the FeCoCrNi high-entropy alloy coating demonstrates the best wear resistance among the tested samples. The wear resistance of the FeCoCrNiMo_{0.5} and FeCoCrNiMo coatings shows no significant difference, but

the addition of Mo increases the wear rate by approximately 38 % compared to the Mo-free coating.

SEM examination of the wear track on the FeCoCrNi coating (Fig. 17) reveals the presence of pits and dense, fine scratches, indicating mechanisms of adhesive wear and abrasive wear. EDS analysis of the worn surface (Table 6) detected regions enriched with oxygen and silicon, as well as iron-rich areas. Given that the counterface material was a silicon nitride ball, the presence of Si is attributed to material transfer from the ball during sliding rather than environmental contamination. The silicon may have oxidized to silica during friction, influencing the surface chemistry alongside the oxygen-rich regions.

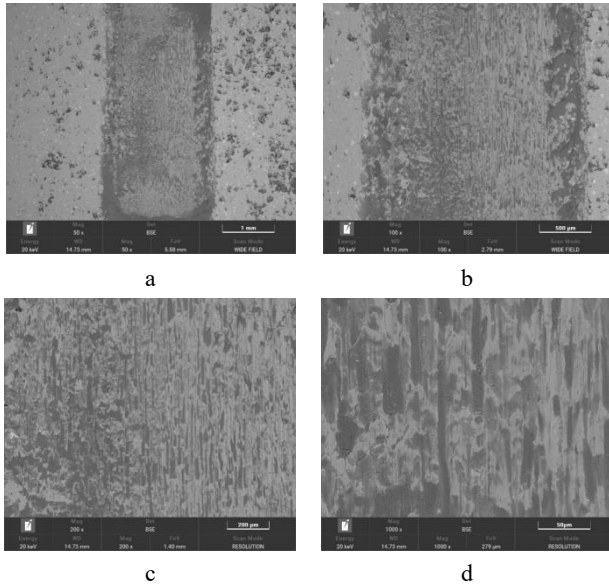


Fig. 17. The tribological surface morphology of FeCoCrNi coating: a – magnification of 50×; b – magnification of 100×; c – magnification of 200×; d – magnification of 1000×

Table 6. Element distribution on the friction surface of FeCoCrNi coating

	Fe	O	Si	Mo	Others
	wt. %				
Surface scanning	81.4	14.2	2.0	0.0	2.3
Area a	58.2	28.9	5.1	0.2	7.7
Area b	96.0	3.3	0.0	0.0	0.6

Fig. 18 presents the SEM image of the wear track on the FeCoCrNiMo_{0.5} coating. The measured width of the wear track is approximately 2.29 mm horizontally and 5.69 mm vertically. The SEM micrograph reveals abundant debris on the worn surface, while scratches are scarcely observed. It is preliminarily inferred that the addition of Mo enhances the resistance to plastic deformation of the high-entropy alloy coating through solid solution strengthening. Meanwhile, the heat generated during prolonged reciprocating sliding promotes the formation of an oxide film, which reduces direct contact between the coating and the counterball, thereby suppressing the generation of scratches.

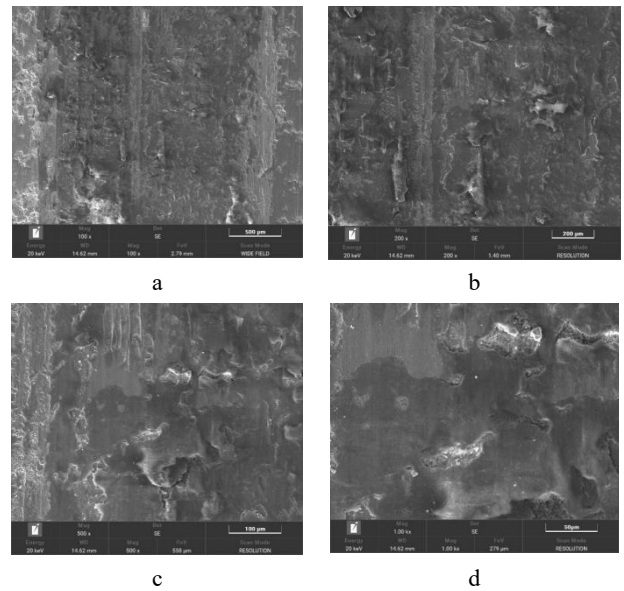


Fig. 18. Friction surface morphology of FeCoCrNiMo_{0.5} coating: a – magnification of 100×; b – magnification of 200×; c – magnification of 500×; d – magnification of 1000×

However, due to its brittle nature, the oxide film undergoes localized spallation under continuous friction, resulting in the observed debris. EDS point analysis within the wear track further reveals iron-rich and silicon oxide-rich regions similar to those detected in FeCoCrNi coating, as exhibited in Fig. 19 and Table 7.

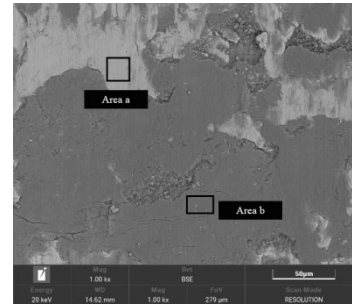


Fig. 19. Selected area of FeCoCrNiMo_{0.5} coating

Table 7. Point scanning of the friction surface on FeCoCrNiMo_{0.5} coating

	Fe	O	Si	Mo	Others
	wt. %				
Surface scanning	71.5	18.2	3.1	1.6	5.6
Area a	96.0	2.9	0.0	0.4	0.7
Area b	53.3	28.7	4.6	3.0	10.4

Fig. 20 shows the wear track of the FeCoCrNiMo high-entropy alloy coating after 30 minutes of reciprocating sliding at room temperature. The wear track measures approximately 2.43 mm in horizontal width and 5.61 mm in vertical width. As observed in Fig. 21, the worn surface exhibits significant debris-like structures with no visible scratches, suggesting that the primary wear mechanism is adhesive wear.

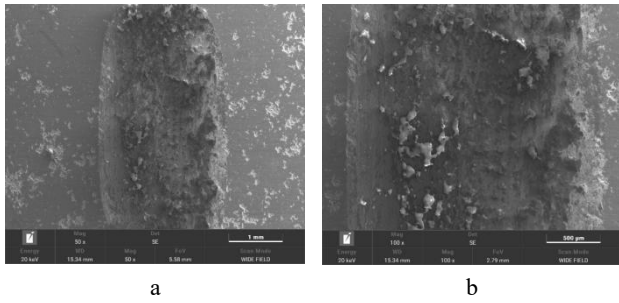


Fig. 20. Friction surface morphology of FeCoCrNiMo coating: a – magnification of 50x; b – magnification of 100x

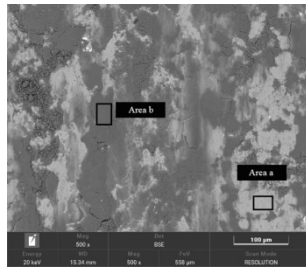


Fig. 21. Selected area of friction surface of FeCoCrNiMo coating

EDS analysis of this wear track reveals a different composition compared to the other two Mo-containing coatings as exhibited in Table 8. A Mo-enriched region is detected with an oxygen content of only 7.07 wt.%, indicating that significant oxidation likely did not occur in this localized area during the sliding process.

Table 8. EDS point scanning on friction surface of FeCoCrNiMo coating

	Fe	O	Si	Mo	Others
	wt%				
Surface scanning	13.2	21.2	1.9	21.8	41.8
Area a	16.3	7.1	0.0	28.0	48.7
Area b	11.8	28.3	3.2	19.7	36.9

4. CONCLUSIONS

1. FeCoCrNiMo_x ($x = 0, 0.5, 1$) high-entropy alloy coating samples were deposited on a steel substrate, significantly enhancing the wear properties.
2. The FeCoCrNiMo_{0.5} coating exhibits the superior corrosion resistance by achieving an optimal balance between Mo content and solid solution structure. With the addition of Mo, local enrichment of Mo is observed in certain regions.
3. The FeCoCrNi high-entropy alloy coating demonstrates the best wear resistance among the tested samples. The wear resistance of the FeCoCrNiMo_{0.5} and FeCoCrNiMo coatings shows no significant difference, but the addition of Mo increases the wear rate by approximately 38 % compared to the Mo-free coating.

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