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Proceeding Paper

Surface, Microstructure, and Wear Characterization of Annealed Cold-Sprayed HEA Coatings [†]

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Abstract: Surface coatings are essential for enhancing the mechanical and functional properties of materials. Among these, annealed high-entropy alloy (HEA) coatings have gained attention for improving wear resistance and durability. This study comprehensively analyzes HEA-annealed coatings, focusing on their surface roughness and wear behavior. A systematic and thorough approach is employed to examine the impact of annealing on coating characteristics. The research involves depositing Al_{0.1–0.5}CoCrCuFeNi and MnCoCrCuFeNi coatings using the cold spray (CS) method, followed by a controlled annealing process. Surface roughness is evaluated through profilometry and microscopy techniques to assess modifications due to annealing. Tribological tests are conducted to investigate the wear performance of the coatings, and the findings are correlated with roughness measurements, offering insights into the relationship between surface texture and wear resistance.

Keywords: cold spray (CS); high-entropy alloy (HEA); surface characterization; wear



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1. Introduction

Advancements in metallurgical science have led to the creation of alloys that exhibit enhanced and more tailored properties than single metals. Traditional alloys are typically composed of a single primary element, which restricts the compositional flexibility of the alloy. A novel alloy concept introduced by J. W. Yeh and B. Cantoris is known as high-entropy alloys (HEAs). These alloys contain a minimum of five principal elements, each present in atomic percentages ranging from 5% to 35%. In contrast, secondary alloying elements constitute less than 5% of the composition [1–11].

Currently, the majority of research focuses on HEAs that incorporate aluminum (Al), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), and nickel (Ni), along with derivatives that involve the addition or substitution of other elements. However, a vast array of alloy compositions remains to be explored. In terms of structural and functional applications, HEAs demonstrate significant potential [1,3].

High mixing entropy is a key factor that drives the formation of solid solutions with simple microstructures, leading to remarkable material properties [1,3]. The high

strains and stresses within crystal lattices enhance strength and hardness while minimizing sensitivity to temperature fluctuations [1–3,10,11]. High-entropy alloys (HEAs) stand out for their exceptional characteristics thanks to their carefully balanced chemical composition of multiple primary elements. These alloys deliver outstanding performance, including superior strength and hardness, impressive wear resistance, remarkable stability under high temperatures, and exceptional resistance to corrosion and oxidation. Furthermore, their versatility is evident because HEAs can be produced using existing manufacturing technologies without requiring specialized techniques or facilities, making them a practical choice for various applications. The presence of multiple primary elements in high-entropy alloys (HEAs) leads to “core effects” that significantly influence their microstructural characteristics and exceptional properties. These effects result in high mixing entropy, considerable crystal lattice distortion, sluggish diffusion, and a cocktail effect [1–3,6,7,10,11].

According to reference [1,12], samples of the Al_{0.5}CoCrCuFeNi alloy quenched in water and cold-rolled demonstrated better performance than many conventional alloys, with fatigue strength limits ranging from 540 to 945 MPa. This HEA system is potentially valuable for future applications where fatigue strength is crucial. Additionally, the chemical composition of HEAs plays a vital role in determining their tribological properties. Adding elements such as Al, Fe, and Nb leads to refined grains and the formation of phases with a body-centered cubic (BCC) structure [1,6].

Manganese (Mn) and chromium (Cr) play a significant role in forming hard phases in materials. As the concentrations of carbon (C), nitrogen (N), boron (B), and silicon (Si) increase, various compounds such as carbides, silicates, and boron compounds may form, or a phase transition can occur from face-centered cubic (FCC) to body-centered cubic (BCC) or B2 structures. Heat treatment methods similar to those used for AISI 304 stainless steel can enhance the hardness of molybdenum (Mo) more effectively than for AISI 304 stainless steel itself. In high-entropy alloys (HEAs), wear resistance is influenced by the type of phase in the material. Alloys composed solely of simple and disordered phases (“SDPs”) typically exhibit wear resistance that is not superior to conventional alloys with comparable hardness. Conversely, when the prevailing phase is complex and ordered (known as “COPs”), the wear resistance of the alloy can significantly surpass that of conventional alloys with similar hardness levels [1,3,13]. Consequently, one of the primary applications of HEAs is the development of coatings that possess high wear resistance, which can be applied using various deposition techniques. Coatings that incorporate particles of ceramic materials within a metal matrix can enhance the overall hardness of the coating and, as a result, reduce its wear rate [1,2,4,7–9,13–19].

This paper presents a characterization of annealed HEA coatings produced through cold spray technology. It emphasizes detailed surface and microstructural analysis and an evaluation of the coatings’ mechanical properties (HV) and wear resistance applied to a magnesium substrate. Additionally, we investigated the formation of two distinct HEAs with the compositions Al_xCoCrCuFeNi and MnCoCrCuFeNi, analyzing their performance at three targeted deposition temperatures: 650 °C, 750 °C, and 850 °C.

2. Experimental Procedure

2.1. Materials and Post-MA+CS Vacuum Annealing

This study utilized several pure elemental powders, including Al, Mn, Co, Cr, Cu, Fe, and Ni, all boasting high purity levels exceeding 99.9%. The mechanical alloying (MA) technique formulated the octonary high-entropy alloys (HEAs) Al_{0.1–0.5}CoCrCuFeNi and MnCoCrCuFeNi. For more details on the processes of mechanical mixing (MM) and mechanical alloying (MA), refer to reference [20]. As detailed in Table 1, calculations were carried out using pure elemental powders of Al, Cu, Co, Cr, Fe, and Ni to attain the

targeted compositions of Al_{0.1–0.5}CoCrCuFeNi and MnCoCrCuFeNi. After mixing and homogenizing, the materials underwent MA for 5.5 hours. To avoid contamination from the ambient environment, the MA powders were kept in sealed containers filled with argon gas until they were processed via cold spray (CS). For a more comprehensive discussion on the effects of MA on powders, see reference [20].

Table 1. Composition of HEA powders in nominal units.

Sample Designation	Nominal Chemical Composition						
		Al	Cu	Cr	Co	Fe	Ni
Al _{0.1} CoCrCuFeNi	at%	4.22	17.38	21.2	18.67	19.74	18.79
	wt%	1.96	19.61	19.61	19.61	19.61	19.61
Al _{0.2} CoCrCuFeNi	at%	8.13	16.65	20.33	17.91	18.95	18.02
	wt%	3.85	19.23	19.23	19.23	19.23	19.23
Al _{0.5} CoCrCuFeNi	at%	18.3	14.8	18.09	15.96	16.84	16
	wt%	9.15	18.1	18.1	18.1	18.1	18.1
MnCoCrCuFeNi	at%	-	15.62	16.81	13.6	13.6	16.86
	wt%	-	16.7	16.7	16.7	16.7	16.7

In the cold spray deposition process, a magnesium (Mg) substrate was coated with high-entropy alloys (HEAs) such as Al_{0.1–0.5}CoCrCuFeNi and MnCoCrCuFeNi at different nitrogen gas temperatures: specifically, 650 °C, 750 °C, and 850 °C. For this study, Trinity College in Dublin, Ireland, utilized the cold spray deposition system referenced in [20] and [21]. The MA and CS Al_{0.5} HEA coatings achieved various microstructures and properties by carefully regulating the cold spray process parameters. The cold spray process employed nitrogen at a pressure of 30 bar, utilizing a type NZ1 nozzle with a standoff distance of 47 mm, a powder feed rate of 9%, a nozzle velocity of 15 millimeters per second, a beam distance of 2 millimeters, and four layers of coating applied in a circular configuration. The magnesium substrate plate measured 50 mm by 50 mm, and the powder feedstock for the cold spray consisted of -63 mm fractions of MA Al_{0.5} HEA powder. Additionally, vacuum annealing was performed at 600 °C for one hour under a pressure of 0.05 bar in a batch furnace at the National Heat Treatment Center in Kildare, Ireland. Specific pressures and powder feed rates were selected to prevent nozzle clogging. The cold spray (CS) temperatures were chosen based on the existing literature, which typically employed higher temperature ranges, leading to moderate temperatures to reduce the risk of oxidation in the high-entropy alloy (HEA) powder. The 47 mm standoff distance was selected as it provided the optimal experimental configuration for our cold spray (CS) apparatus, facilitating the best feed rate and achieving an effective buildup rate for regulating coating thickness efficiently. Tables 1 and 2 display the coatings' nominal chemical composition and the sample designations.

Table 2. Al_{0.1–0.5}CoCrCuFeNi and Al_{0.1}CoCrCuFeNi chemical composition samples were prepared by cold spray.

Al _{0.1} CoCrCuFeNi	Cold spray temperature [°C]	650	750	850
	Denomination	A	B	C
Al _{0.2} CoCrCuFeNi	Cold spray temperature [°C]	650	750	850
	Denomination	D	E	F
Al _{0.5} CoCrCuFeNi	Cold spray temperature [°C]	650	750	850
	Denomination	G	H	I
MnCoCrCuFeNi	Cold spray temperature [°C]	650	750	850

2.2. Microstructure Characterization

The sample preparation and microstructural characterization procedures are detailed in Ref. [22]. The HEA coating and Mg substrate were deliberately removed, wholly and partially, to examine their interface from a direct plane-on view. Cross-sectional samples were prepared following standard metallographic techniques and polished using 0.06 mm colloidal silica to evaluate the coating microstructure. An energy-dispersive X-ray spectrometer (EDS) integrated with a scanning electron microscope (SEM) was utilized to analyze the coating elements. Figure 1 presents macro photographs of annealed MA+CS Al_{0.1–0.5} (Mn)CoCrCuFeNi HEA coatings at three different process gas (N₂) temperatures: 650 °C, 750 °C, and 850 °C.

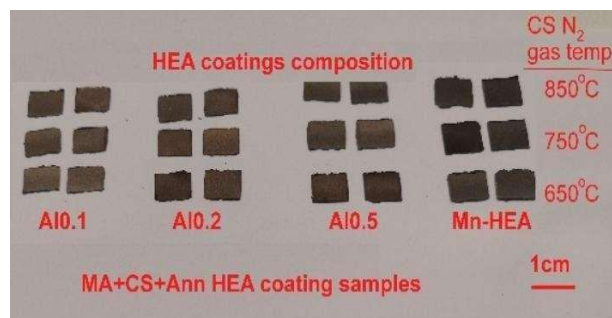


Figure 1. A macrophotograph of MA+CS Al_{0.1–0.5} HEA and Mn-HEA annealed coatings on magnesium.

2.3. Surface Characterization Measurement

The coating's surface roughness was meticulously measured using the cutting-edge RTP80 roughness tester from SM-Instruments, ensuring precise and reliable results. As detailed in Table 3, all specimen coatings thoroughly evaluated the surface roughness, with measurements taken parallel and perpendicular to the deposition direction. Employing a cut-off dimension of 0.25 mm and a measuring length of 1.5 mm, each coating sample was subjected to five rigorous roughness measurements, focusing specifically on three critical parameters: Ra, Rq, and Rt. These parameters, defined by the standard UNI EN ISO 4287 (2011), are key indicators of surface quality, representing the amplitude of roughness that denotes the distance between peaks and valleys. Specifically, Ra indicates the arithmetical mean deviation, providing insight into the average surface irregularities. Rq, the root mean square deviation, offers a robust measure of the average amplitude along the height direction. At the same time, Rt captures the total height of the profile, measuring the distance between its highest peak and lowest valley. This comprehensive analysis ensures a thorough understanding of the coatings' surface characteristics, reinforcing their quality and performance.

Table 3. Detailed specifications for the RTP80 roughness tester.

Characteristic	Description/Value
Measuring range [μm]	± 500
Resolution [μm]	0.001
Cut-off length [mm]	0.25
Measuring length [mm]	1.5

2.4. Microhardness Measurement

Innovate Vickers microdurometers were employed to precisely measure the HV30 microhardness profiles of the coating samples, utilizing a controlled preload of 3 N and a standardized indentation time of 15 seconds. To begin with, the annealed coatings were securely bonded to magnesium substrates using a reliable adhesive. The microhardness

of each specimen was determined by averaging the results of three carefully executed indentations on the polished-side cross-section, ensuring accurate and consistent data.

2.5. Tribological Analysis

ZCHBWear tests were conducted under ambient conditions using a pin-on-disk tribometer (Anton Paar TriTec, TRB, Turin, Italy) at the Polytechnic University of Turin. A dedicated pin was developed to ensure a tight clamping of the samples during testing. The counterparts used during the wear tests were quenched 100Cr6 steel with a grinding surface. Figure 2 illustrates the experimental setup used during the wear tests. Table 4 demonstrates the wear testing parameters.

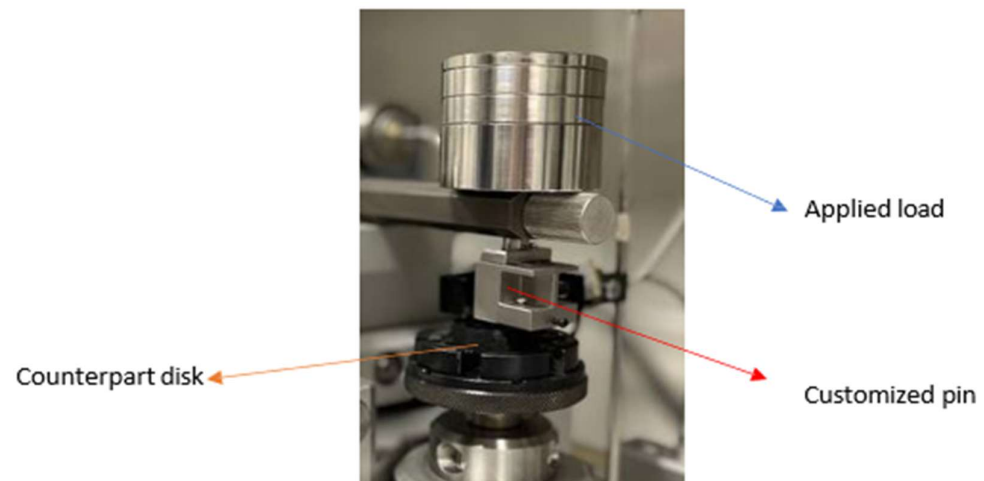


Figure 2. Experimental setup.

Table 4. Parameters wear testing.

Wear Testing Parameters	
Rotational speed [rpm]	250
Normal load [N]	10
Sliding length [m]	200
Linear speed [mm/s]	235.6

3. Results and Discussion

3.1. Microstructural Analysis

Figure 3a illustrates SEM-SE imaging of Al_{0.5}CS850°C annealed at 600 °C. There is a similar level of roughness on the surface of the coating as in the MA+CS sample. According to [22], the porosity level is very low compared to the MA+CS sample. The surface roughness at the interface between the HEA coating and the customized pin counterpart disk magnesium substrate applied to the customized load indicates good bonding and adhesion. Figure 3b shows the same results for the 600 °C-annealed Mn-HEA CS850 °C sample. The top surface roughness is also similar to that of the MA+CS counterpart. Porosity levels are also very low, interfaces are rough, and the first direct and plane-on surface imaging (viewing) of the interface following mechanical removal of the Mg substrate from the HEA coating following annealing processes indicates the roughness of the interface that is responsible for its excellent bonding with the Mg substrate by the HEA coating.

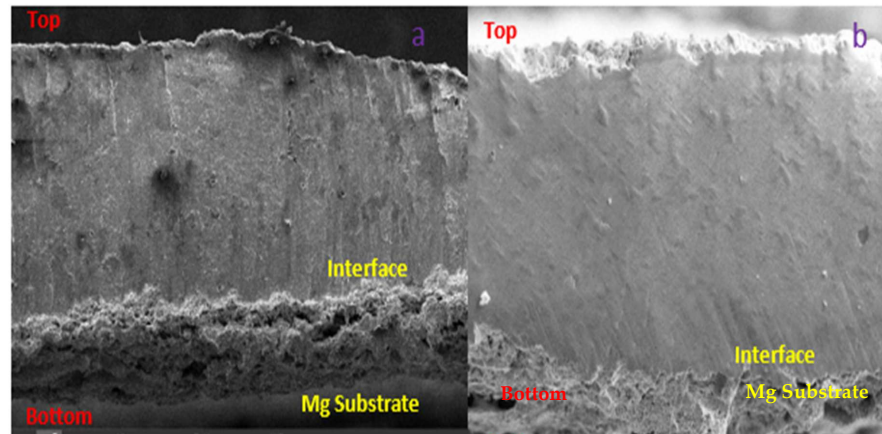


Figure 3. SEM micrograph: (a) Al_{0.5} HEA CS 850 °C, (b) Mn-HEA CS 850 °C.

3.2. Surface Characterization
Roughness Measurements

Figure 4 analyzes the roughness values obtained for samples with the same chemical composition at different deposition temperatures. For Al_{0.1–0.5} CoCrCuFeNi, all roughness parameters decrease with increasing deposition temperature. However, experimental scattering cannot establish a monotonic relationship between roughness parameters and deposition temperature for other specimens.

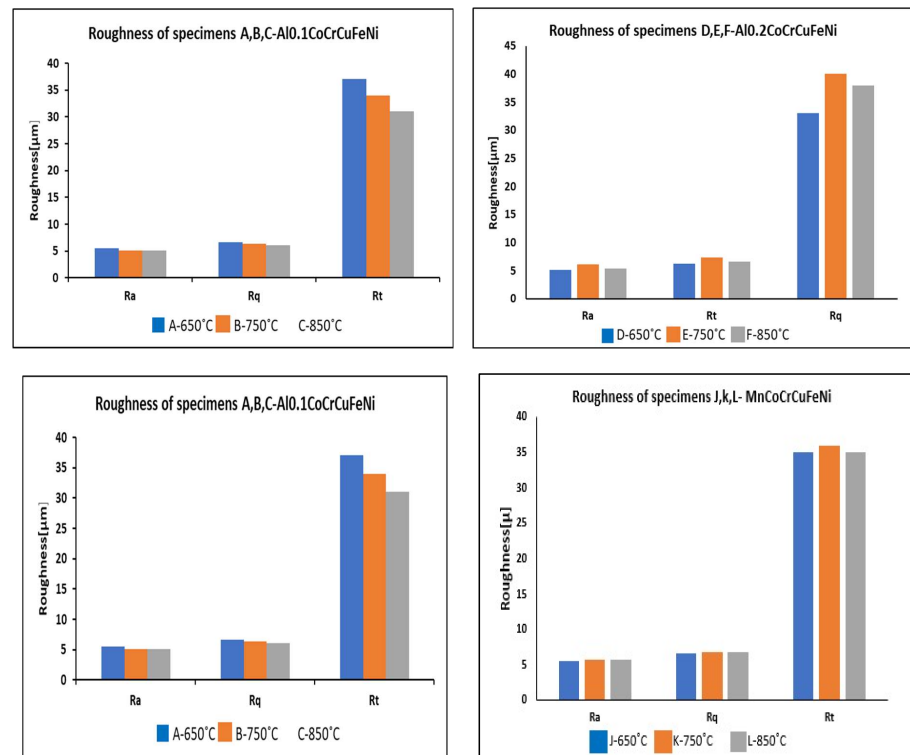


Figure 4. Roughness values of Al_{0.1–0.5}CoCrCuFeNi and MnCoCrCuFeNi coatings deposited at different cold spray temperatures. (a) Al_{0.1}CoCrCuFeNi, (b) Al_{0.2}CoCrCuFeNi, (c) Al_{0.5}CoCrCuFeNi, and (d) MnCoCrCuFeNi.

3.3. Microhardness Test

After carefully polishing the lateral surfaces of the samples, indentations were made at the interface close to the substrate. Based on three measurements, a mean value was calculated for each position. Table 5 indicates that the microhardness of a coating is highly dependent on its chemical composition. Microhardness averages for samples with

$Al_{0.1}CoCrCuFeNi$ composition (A–C) are the lowest, whereas mean microhardness averages for samples with $Al_{0.5}CoCrCuFeNi$ (G–I) composition are the highest.

Table 5. Vickers microhardness measurements for all compositions.

		Coating	
$Al_{0.1}CoCrCuFeNi$	A (650 °C)	Average	113.26
		Std dev	4.55
	B (750 °C)	Average	79.48
		Std dev	7.76
	C (850 °C)	Average	95.22
		Std dev	9.42
$Al_{0.2}CoCrCuFeNi$	D (650 °C)	Average	151.57
		Std dev	15.33
	E (750 °C)	Average	80.427
		Std dev	14.73
	F (850 °C)	Average	91.21
		Std dev	19.96
$Al_{0.5}CoCrCuFeNi$	G (650 °C)	Average	92.34
		Std dev	4.76
	H (750 °C)	Average	171.49
		Std dev	14.17
	I (850 °C)	Average	131.96
		Std dev	25.78
$MnCoCrCuFeNi$	J (650 °C)	Average	91.8
		Std dev	18.56
	K (750 °C)	Average	84.123
		Std dev	3.7
	L (850 °C)	Average	152.03
		Std dev	13.09

3.4. Wear Test

Figure 5 illustrates the average friction coefficient observed in each test. The $Al_{0.1-0.5}CoCrCuFeNi$ composition at 750 °C shows the highest friction coefficient. In contrast, for the $MnCoCrCuFeNi$ composition, an increase in the cold spray temperature decreases the friction coefficient.

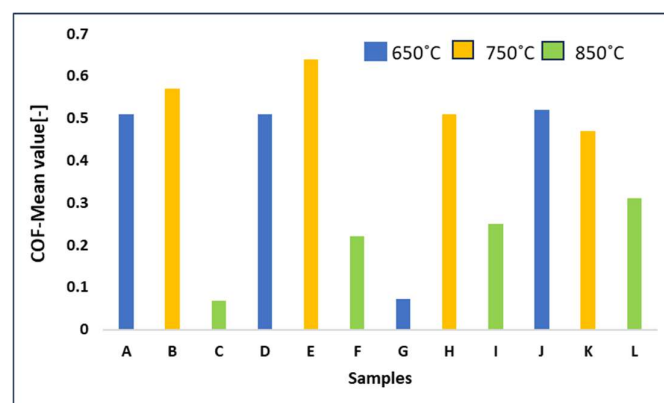


Figure 5. The average coefficient of friction for the coated samples.

4. Conclusions

The study examined the microstructure, roughness, hardness properties, and wear results for Al_{0.1–0.5} (Mn) CoCuCrFeNi high-entropy alloy (HEA) coatings deposited on a magnesium substrate. Nitrogen was used as the process gas at three different temperatures. Vacuum annealing was performed in a batch furnace at a pressure of 0.05 bar for one hour at 600 degrees Celsius. The findings of this study lead to several conclusions.

The experimental data indicate that the chemical composition of the high-entropy alloy does not significantly influence the coating's roughness. However, the composition does have a noteworthy impact on the layer's microhardness. Specifically, a clear trend shows that microhardness increases as the aluminum content in the samples rises.

Annealing leads to significant microstructural changes, such as reduced porosity and improved bonding at the interface between the coating and the magnesium substrate. These changes enhance the coating's overall cohesion.

Annealing results in smoother surfaces by reducing roughness levels compared to non-annealed, cold-sprayed samples, while microhardness decreases after annealing. Due to the softer surface, wear resistance improves, which reduces friction and enhances material contact conditions. These smoother profiles enhance tribological performance by minimizing friction.

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