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Fabrication and Characterization of Five-Element High Entropy Alloys

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ABSTRACT

High Entropy Alloys (HEAs) are an emerging category of materials that have attracted considerable attention due to their unique combination of properties, such as exceptional corrosion resistance, high strength, and thermal stability, compared to traditional alloys, which are typically composed of one or two main elements, HEAs are consist of five or more principal elements in equiatomic or near-equiatomic proportions. Graded materials are particularly promising for their enhanced mechanical properties, including improved ductility and strength. This study introduces a functionally graded Alx-Zn-Cd-Mn-Sb alloy, produced in situ for the first time using powder metallurgy. The alloy exhibits a gradient in aluminum concentration along the building direction. The investigation focused on the microstructure and mechanical properties of the alloy. Results showed that increasing aluminum content transformed the structure from a dual-phase of face-centered cubic (FCC) and body-centered cubic (BCC) phases into a single BCC phase. The precipitation temperature range of the disordered BCC phase expanded, aiding its formation. As the phase ratio changed, the alloy's hardness increased from 342 HV to 397 HV. It exhibited an optimal combination of properties, with an elongation of 42.3% and a compressive strength of 827.4 MPa. Superior strength and toughness were attributed to grain refinement and high-density dislocations. However, the segregation of chromium at grain boundaries led to increased brittleness. This study introduces a new technique for creating high-entropy alloys, opening the way for gradient materials that are suitable for a variety of applications.

1. INTRODUCTION

The potential of high entropy alloys (HEAs) offers a significant change in the design and characteristics compared with traditional alloys with the same materials. High Entropy Alloys are alloys that contain five or more main elements in equal proportions, generally ranging between five and thirty-five atomic percent [1, 2]. HEAs' special structure may make the solid solution more stable than their complex intermetallic structures, thereby producing a super combining mechanical and physical characteristics [3]. Moreover, these special features of HEAs show potential utility in nuclear and aerospace [4] sectors. HEAs have demonstrated low density and great strength at high temperatures when compared between the Nickel superalloys and the Al-Mo-Nb-Ta-Zr. By means of the variations in the manufacturing technique of five constituents of HEAs, the microstructure and the characteristics of HEAs undergo diverse modifications as well. Although generating and describing five characteristics of HEAs generates a lot of research, the manufacturing of HEAs still suffers a major knowledge vacuum. This void must be closed so that we may better grasp HEAs and benefit from their many uses. Due to its impact on the industrial uses and the efficiency for High-Energy alloys [5], the microstructure and the resulting properties are more significant.

There are four primary parameters that distinguish high-entropy alloys from traditional alloys: slow diffusion rates, high entropy, significant lattice distortion, and the combined impact of multiple components. These characteristics contribute to their unique stability at high temperatures, incredible hardness, excellent resistance to corrosion, and superior durability. However, the complicated interactions among the five constituent elements make it a challenge to precisely define and maintain their microstructure and physical properties [5, 6].

There are several methods to create high-entropy alloys (HEAs) including arc melting, powder metallurgy, additive manufacturing, and other advanced techniques [7]. Each technique has unique advantages and limitations. For example, arc melting is a common option because it's easy to use and efficient in alloy production, but it often leads to inconsistent composition and microstructures. However, additive manufacturing can be used for producing complex shapes and customized designs but presents challenges related to porosity and residual stresses [4].

Powder metallurgy has attracted considerable attention due to its ability to create uniform microstructures and provide precise compositional control, which makes it especially valuable for producing functionally graded materials (FGMs). This process also optimizes raw material usage and provides better control over phase distribution and porosity than the previous methods. However, there is still a lack of knowledge on how specific processing parameters, including particle size distribution, sintering temperature, and pressure, influence the formation of microstructure and mechanical properties in HEAs [5, 6].

Research on HEAs made using functionally graded materials has mainly explored the microstructure of mechanically alloyed (MA) powders. While these studies provide significant information, they often ignore the relationship between processing methods, gradient composition, and their influence on phase transformations and mechanical behavior. This demonstrates an important need for further investigation to improve processing techniques and promote the performance of these advanced materials [7].

X-ray diffraction, transmission electron microscopy, and scanning electron microscopy were employed to investigate sintered specimens. On the other hand, their mechanical features were investigated by utilizing micro and nano hardness methods. The single-edge V-notch beam (SEVNB) technique was employed to complete the fracture toughness measurements [8, 9]. BCC (κ) and FCC (τ) solid solution phases are found in the MA powder. The phases in the MA powder remain unchanged after extensive ball milling (up to 60 hours) [10]. For the specimen sintered between 973 and 1273 K, the sintered pellets exhibited a phase-separated microstructure with Al-Ni-rich L12 phase, α′, and tetragonal Cr-Fe-Co based σ phase in addition to Al-Ni-Co-Fe FCC solid solution phase (ε). Evidence from experimentation demonstrates that the solid solution of BCC (κ) turns into a eutectoid during the sintering process, leading to the creation of L12 ordered α′ and σ phases. Still, the FCC (τ) phase stays the same, but the lattice parameter changes slightly. The hardness of the specimen increases with sintering temperature, and a sudden rise in hardness is observed at 1173 K. The specimen with the most excellent hardness, about ~8 GPa, was sintered at 1273 K. The elastic modulus mapping shows three phases with elastic moduli of around 300, 220, and 160 GPa. Due to the existence of brittle nanosized σ phase precipitates, the fracture toughness determined by the SEVNB method indicates a maximum magnitude of 3.9 MPa $m^{1/2}$ [11]. It is hypothesized that enhanced densification at a higher sintering temperature and increased hardness are caused by a significant increase in the proportion of σ phase precipitates and eutectoid transformation of the τ phase [12, 13].

The main objective of this research is to figure out the impact of the powder technology method on the microstructure and properties of the constituent that is used in the preparation of high-entropy alloys (HEAs). This work seeks to discover the complicated connections between processing parameters, microstructural characteristics, and the resulting properties of five-element HEAs, with a particular focus on their production and properties.

Understanding and awareness these interactions are important for improving the performance of high entropy alloys and maximizing their important applications. Moreover, this research aims to contribute to the growing knowledge of complex multi-component alloy systems, opening the way for creating and developing innovative materials with better properties for high-performance technical applications.

The selection of Al-Zn-Cd-Mn-Sb as the alloy system was chosen by the exceptional contributions of each element to the needed mechanical and physical properties of the alloy. Aluminum was selected due to its ability to enhance the strength and thermal stability, while zinc was chosen due to its contribution to improve the corrosion resistance and ductility. Cadmium, in spite of its potential toxicity, plays a significant role in enhancing wear resistance and improving the overall mechanical properties of the alloy under particular conditions. Manganese and antimony were added for their ability to stabilize the microstructure and refine grain boundaries, improving hardness and strength.

2. FABRICATION PROCESS FOR ENTROPY ALLOYS (EAS)

The study developed five-element high-entropy alloys with superior characteristics utilizing the powder technology method. The proposed HEAs consist of equiatomic proportions of five elements selected based on their potential contribution to obtaining desirable features. As illustrated in Figure 1, a 35g mixture of metal powders was prepared according to the ratios identified in Table 1.

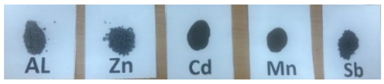


Figure 1. Five types of powder elements utilized

Table 1. Weight percentage of the HEAs

Symbol	Alloys	Al (%)	Zn (%)	Cd (%)	Mn (%)	Sb (%)
AE_1	$Al_{38}ZnCdMnSb$	38	32	9	8	13
AE_2	$Al_{42}ZnCdMnSb$	42	28	9	8	13
AE_3	$Al_{46}ZnCdMnSb$	46	24	9	8	13

The powder mixture was mixed manually using a mortar and pestle. For wet mixing, to prevent the metal powders from combining/aggregation and to avoid a sharp rise in temperature during the mixing process, 5 ml of methanol was utilized as the process control agent. The second mixing process was performed using mechanical alloying (MA) through a ball mill machine at (200) rpm for (1 hr).

After 60 min of MA, the resulting powder filled a 15mm diameter test tube. For the pressing process, a press machine with pressures of 5 and 8 tons/cm^2 was used to obtain the series of specimens, and for each series were produced twice samples to ensure reproducibility as demonstrated in Figure 2.

Homogenization heat treatments followed the fabrication process to ensure uniformity in the microstructure. The sintering process of the compacted specimens was carried out using the tube furnace. Inert Ar gas was introduced into the furnace chamber with a flow rate of 5 (l/min). The temperature utilized for the sintering process was slightly above the lower melting temperature of the Cd metal (330°C) for (30 min).

The specimens were cooled at a rate of 10°C/min after achieving the necessary temperature for the sintering process at a heating rate of 5°C/min.

Finally, the HEA specimens were removed from the furnace after the sintering process, as demonstrated in Figure 3 and prepared for density, hardness, and microstructure characterization.

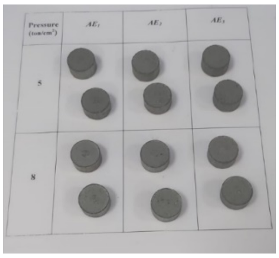


Figure 2. The compacted specimens

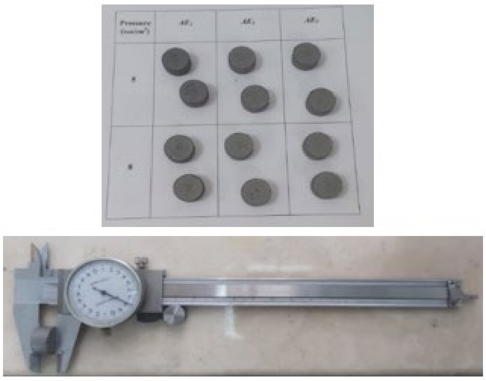


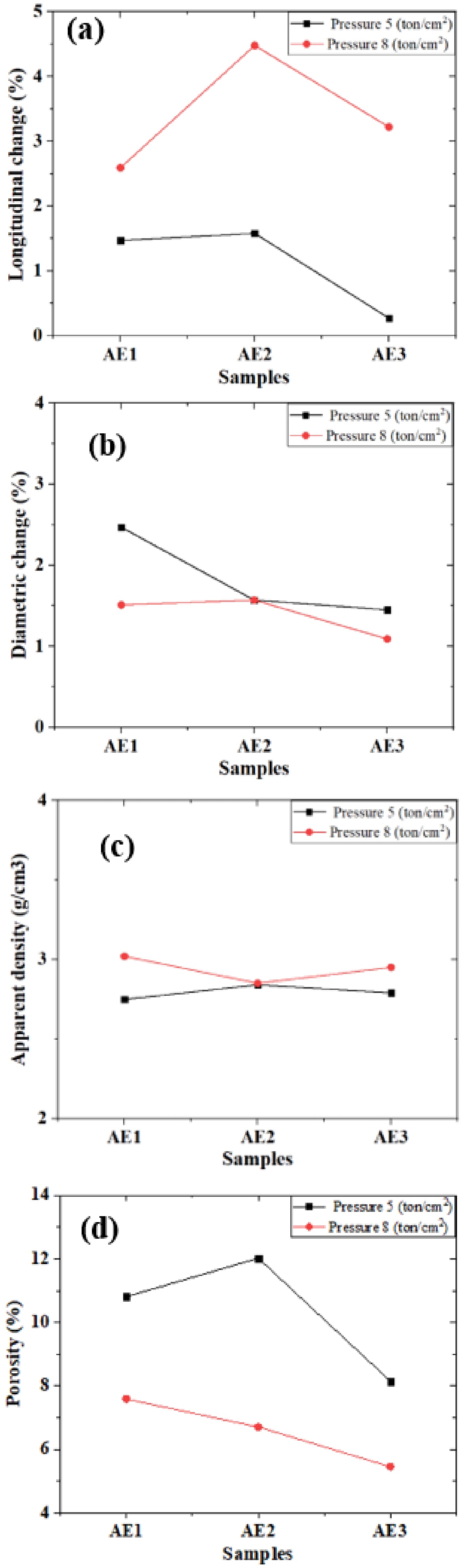
Figure 3. The compacted specimens

3. MEASUREMENTS

Characterization of the fabricated HEA was based on a quantitative evaluation of the HEA's mechanical and physical characteristics along with a qualitative description of its microstructure. The hardness property was measured three times for each sample, while the density, porosity, dimensional changes, and XRD analysis were measured once for each sample. The density of the HEA specimens and the porosity were evaluated using the Archimedes principle. Diametric and longitudinal changes of the specimens were calculated based on the dimensional measurement before and after the sintering process. The physical features of the prepared specimens are demonstrated in Table 2, and Figures 4 (a-e). The mechanical features of the HEAs were tested using the Vickers hardness test. The Vickers hardness measurements were performed using a digital microhardness tester with a load of 300 g and a dwell time of 15 sec.

Table 2. Physical and mechanical characteristics of the prepared specimens

Features	Pressure (ton/cm^2)	AE_1	AE_2	AE_3
Longitudinal change (%)	5	1.47	1.58	0.27
	8	2.59	4.48	3.22
Diametric change (%)	5	2.47	1.57	1.45
	8	1.51	1.57	1.09
Apparent density (g/cm3)	5	2.75	2.84	2.79
	8	3.02	2.85	2.95
Porosity (%)	5	10.81	12.02	8.15
	8	7.59	6.71	5.46
HV Hardness	5	69.1	55.04	48.95
	8	73	58.5	66.1



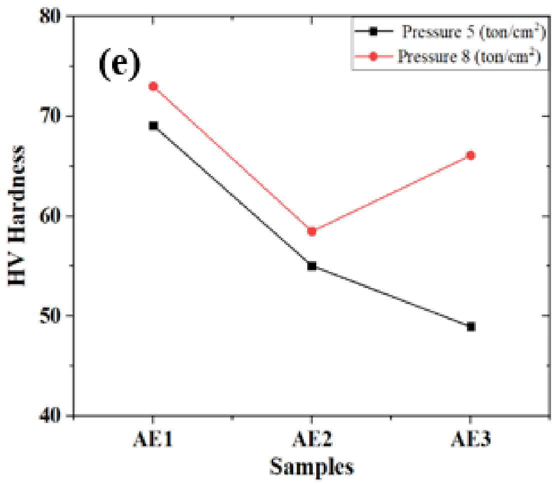


Figure 4. Mechanical and physical properties for HEA samples, (a) Longitudinal change, (b) Diametric change, (c) Apparent density, (d) Porosity, and (e) Microhardness

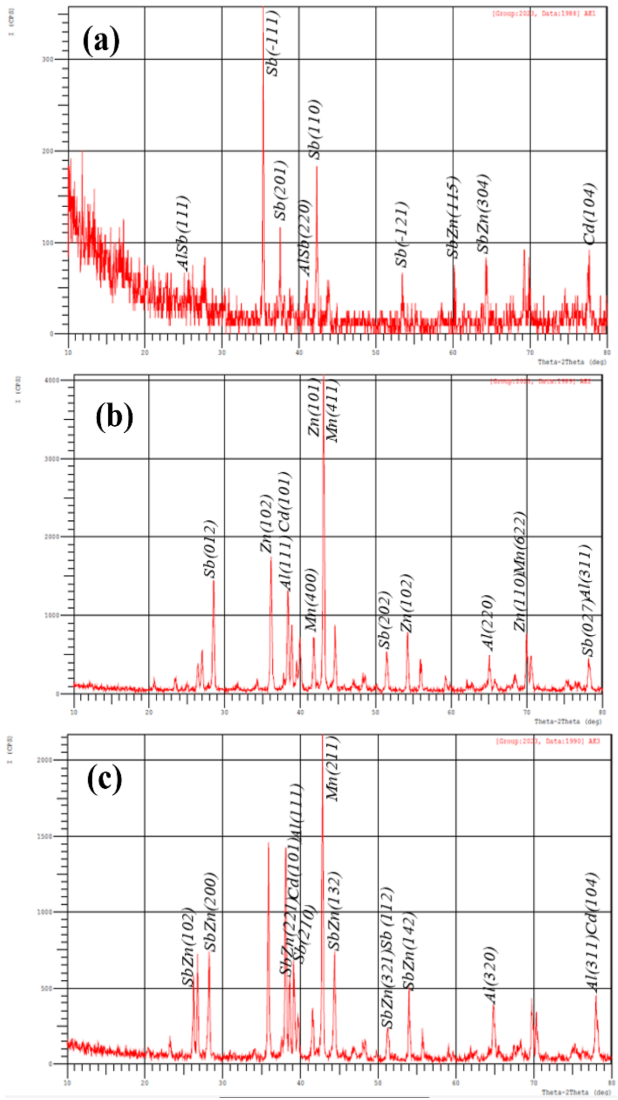


Figure 5. XRD for sample: (a) AE_1, (b) AE_2, and (c) AE_3

Finally, the HEA specimens were subjected to microstructural characterization analysis to examine the phase composition and grain structure utilizing X-Ray Diffractometer-6000 (XRD-6000) (SHIMADZU, Japan). The XRD test findings for specimens AE_1, AE_2, and AE_3 are illustrated in Figures 5 (a-c).

4. DATA ANALYSIS

The practical implications highlight that Aluminum-rich HEAs under high pressure exhibit high performance, in line with the study objectives, and providing insights for improving alloy manufacturing processes for real-world applications.

4.1 Mechanical properties

The mechanical properties of the high-entropy alloys (HEAs) were evaluated in terms of hardness, strain, and densification under varying pressures and aluminum concentrations.

4.1.1 Hardness trends

HEAs can exhibit superior mechanical features in comparison to conventional alloys.

The hardness of the alloys increased significantly with elevated pressure. At 8 tons/cm², improved densification and reduced porosity led to a more compact grain structure, enhanced interparticle bonding, and minimized structural defects. Specimen EA_3, initially showing the lowest hardness at 5 tons/cm², exhibited a substantial increase under 8 tons/cm², surpassing Specimen EA_2. This indicates that higher aluminum content, combined with increased pressure, positively influences the mechanical features of the alloy. Specimen EA_1 consistently demonstrated the highest hardness across both pressure levels, with the most pronounced improvement observed in EA_3.

4.1.2 Strain behavior

The differences in Aluminum amount which is related to the microstructure cause the variation in the longitudinal strain among the alloys. The sample EA_3 which contains the highest Aluminum amount showed higher resistance to deformation. On the other hand, the sample EA_2 has a bigger grain size and less Aluminum amount.

4.2 Microstructure analysis

In order to better understand the interaction between the processing parameters and physical properties the microstructure of the alloys was investigated.

4.2.1 Grain refinement and porosity

Higher pressures led to significant grain refinement and reduced porosity, particularly in Aluminum-rich alloys. This refinement contributed to enhanced hardness and strength through improved grain boundary interactions [14]. Under stress, EA_2, which has less Aluminum, has a weaker phase structure and a greater grain size, which facilitates deformation and increases strain [15]. As underlined in many researches on their deformation behavior and strengthening processes, the mechanical features of high-entropy alloys depend critically on the interaction of these microstructural features, including phase stability and grain size [16, 17].

4.2.2 Grain boundary strengthening

Aluminum content played a critical role in strengthening the grain boundaries, reducing dislocation movement and contributing to a stable phase structure. Specimens with higher aluminum concentrations displayed fewer defects and increased resistance to deformation.

4.3 Phase transformation

To understand precisely the mechanical properties of the alloys, we have investigated the phase transformation.

4.3.1 Impact of aluminum content

Higher aluminum content facilitated phase transformations, contributing to grain boundary strengthening and precipitation hardening. For instance, in the Al46 alloy [18, 19], a stable crystal structure was maintained, limiting dislocation movement and enhancing deformation resistance [20]. Which indicate that an increase in aluminum content improves the alloys' resistance to deformation by maintaining their crystal structure and restricting dislocation movement under compressive stress [21, 22].

4.3.2 Role of pressure in phase stability

Elevated pressures encouraged the formation of denser phases, reducing porosity and enhancing overall alloy stability. These transformations were most evident in specimens with higher aluminum concentrations, highlighting the interplay between pressure, composition, and phase stability.

Thus, the mechanical characteristics of these alloys directly relate to their microstructural qualities and aluminum concentration. At pressures of 5 and 8 tons/cm², the apparent density measurements for the high-entropy alloy specimens (EA_1, EA_2, and EA_3) helped one to grasp how composition and pressure influence the porosity and compaction features of a material. The measured densities, which range from 2.75 to 2.84 g/cm³, are very similar at a pressure of 5 tons/cm², with AE_2 displaying the most excellent density magnitude. Conversely, at a pressure of 8 tons/cm², AE_1 reveals the most pronounced increase in density (from 2.75 to 3.02 g/cm³), indicating that this alloy exhibits enhanced compaction efficiency under elevated pressure conditions, likely resulting in a reduction of porosity. AE_3 displays moderate augmentation in density, whereas AE_2 maintains relative stability, implying that the latter may already possess optimal compaction at lower pressure levels. The findings imply that higher Aluminum content (as observed in AE_3) correlates with improved stability and compaction efficacy. In contrast, alloys characterized by lower aluminum content may undergo more significant density variations with increasing pressure, attributable to their higher initial porosity.

4.4 Statistical analysis

Different statistical methods were employed to investigate the mechanical and physical characteristics of the fabricated samples of high-entropy alloys (HEAs) in order to verify the validity of the conclusions drawn from the data.

4.4.1 Analysis of Variance (ANOVA)

ANOVA was used to assess the significance of variations in hardness, density, and porosity across different aluminum concentrations and applied pressures (5 and 8 tons/cm²). The results showed statistically significant differences ($p < 0.05$), confirming that both aluminum content and pressure significantly influence these properties.

4.4.2 Linear regression analysis

To determine the relation between the Aluminum amount and the hardness we have used Linear Regression analysis. This analysis shows a strong positive correlation ($R^2 > 0.85$), according to this analysis the hardness improved by increasing Aluminum content.

4.4.3 T-tests

We have compared the mechanical characteristics such as density and hardness by utilizing paired T-tests at varies pressure levels (5 vs. 8 tons/cm²). The results presented impressive improvements in hardness and density at higher pressures.

4.4.4 Pearson correlation coefficients

The relationship between grain size, porosity, and hardness was evaluated using Pearson correlation. A strong negative correlation (-0.88) was found between porosity and hardness, showing that reduced porosity results in increased hardness.

4.4.5 Error analysis

Standard deviations and confidence intervals were calculated to evaluate the variability in the measurements. For example, the Vickers hardness values had a standard deviation of ±2.3 HV, indicating consistent and precise results.

5. CONCLUSION

The current work focused on the structure, microstructure, and mechanical features of EA_1, EA_2, and EA_3 high entropy alloy powder metallurgical processing. The following conclusions can be drawn from the current investigation: high-entropy alloys (HEAs) reveal a significant correlation between microstructural features and mechanical features, particularly hardness and density. The findings show that higher aluminum content improves the alloys' resistance to deformation mostly by means of better atomic bonding and a more stable phase structure, therefore restricting dislocation migration under pressure. Especially, using high pressures throughout manufacturing findings in improved densification and lower porosity, therefore contributing to a tight grain structure that strengthens the alloys even more. With consequences for their use in advanced materials, the findings highlight the relevance of microstructural features like grain size and phase stability in defining the mechanical performance of HEAs. This work emphasizes generally the possibilities of HEAs, especially those with more aluminium content, to surpass traditional alloys in mechanical uses. The results of the statistical analysis support the trends observed in the mechanical and physical properties of the high-entropy alloys (HEAs). ANOVA confirmed significant differences in hardness, density, and porosity across different aluminum concentrations and pressures ($p < 0.05$). Linear regression showed a strong positive relationship between aluminum content and hardness ($R^2 > 0.85$). Paired T-tests indicated significant increases in density and hardness at higher pressures ($p < 0.01$), and Pearson correlation revealed a strong negative link between porosity and hardness (-0.88). These findings confirm that aluminum content and pressure directly influence the mechanical properties of the HEAs. By including these statistical evaluations, the reliability and accuracy of the conclusions are reinforced, providing a solid basis for optimizing HEA production techniques.

While this study primarily examined the initial mechanical and physical properties of HEAs. Future research should focus on assessing their long-term behavior when subjected to high temperatures, corrosive environments, and cyclic loading. Such studies would offer a clearer understanding of their practicality and effectiveness in real-world applications.

Also expanding the scope to include different combinations of elements and compositions could identify new HEA systems with unique properties tailored for specific applications.

The high strength, hardness, and thermal stability of the HEAs produced in this study indicate their potential for use in aerospace applications, including turbine blades and structural supports, where exceptional performance under extreme conditions is essential. Additionally, their biocompatibility and robustness make them promising materials for medical applications, such as implants, surgical tools, and prosthetic devices.

The environmental implications of HEAs deserve attention. Their extended lifespan and superior mechanical properties can reduce the need for frequent replacements, leading to lower material consumption over time. However, the energy-intensive production processes and challenges associated with recycling multi-component materials must be addressed to ensure their sustainability. Future research should explore more energy-efficient manufacturing methods and recycling strategies to minimize the environmental footprint of HEAs, making them a more sustainable choice for industrial applications.

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