

Article

Influence of Ni on the Organization and Properties of AlCoCrFeMn High-Entropy Alloys by Laser-Sintering Technique

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Abstract: In order to investigate the effect of the Ni element on the properties of AlCoCrFeMn HEAs, this experiment prepared AlCoCrFeMn and AlCoCrFeNiMn HEAs by using a laser-ignition self-propagation sintering technique with an equal molar ratio. And analyzed the effect of the Ni element on the microstructure of AlCoCrFeMn HEAs by using a metallurgical optical microscope (OM), scanning electron microscope (SEM), energy spectroscopic analysis (EDS), X-ray diffraction (XRD), and other experiments. Characterization equipment was used to analyze the effect of the Ni element on the microstructure, physical phase structure, wear resistance, compressive properties, and corrosion resistance of AlCoCrFeMn HEA materials. The results show that after the addition of the Ni element, the AlCoCrFeNiMn HEA changes from a single BCC phase to one consisting of BCC and a small amount of an FCC phase, with an equiaxial organization, and the yield strength reaches 780 MPa and the compressive strength is 3920 MPa. The corrosion rate is 2.08×10^{-3} mm/a, and the corrosion resistance and mechanical properties are greatly increased.

Keywords: laser sintering; HEA; wear resistance; corrosion protection; compressibility



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1. Introduction

HEAs [1] are composed of 5–13 principal elements with atomic ratios between 5% and 35% of each element [2]. The emergence of multi-principal element HEAs has overturned people's perception of traditional alloys and led scholars into a completely new field. Due to the high-entropy effect [3], lattice distortion effect [4], hysteresis diffusion effect [5], and cocktail effect [6] of HEAs, their performance in terms of the mechanical properties [7], thermal stability [8], corrosion resistance [9], and other aspects compared with other alloys has notable excellent characteristics.

At the end of the 20th century, Yeh Junwei from Taiwan, after a large number of experiments and accumulating a large amount of data, proposed the concept of HEAs and synthesized AlFeCrCoCuNi HEAs with a simple phase structure, which are of great interest because of their excellent mechanical, physical, and chemical properties [10–12]. Sriharitha [13] prepared Al_xCoCrCuFeNi ($x = 0.45, 1, 2.5, 5$) HEA powder materials and analyzed the effect of different Al contents on the phases of HEAs using XRD. Nong et al. [14] prepared cast AlCrFeNiTi HEAs using vacuum arc melting with argon as a protective gas and studied the hardness changes in the alloys under different heating temperatures and holding times. Wang et al. [15] prepared AlCoCrFeNi HEAs using laser machining and studied the phase organization, microstructure, mechanical properties, and corrosion resistance of the specimens at different aging temperatures. Sharma et al. [16] investigated the molecular dynamics simulation of the atomic origin of the structural phase transition in the AlCrFeNiCo series of HEAs using the classical method. Because the Mn element can make the alloys have enough toughness and high strength and hardness, there are many reports about Mn element HEAs nowadays. Based on this, AlCoCrFeMn alloys were prepared by the laser-ignited self-propagation sintering technique. On this basis,

the effect of the Ni element on the organization, compression properties, and corrosion resistance of the alloy was investigated.

At present, HEAs are mainly prepared by vacuum arc melting, which cannot avoid the staining of refractory materials. In addition, there are defects such as looseness and segregation in the melted samples. Laser-ignited self-propagating sintering technology can effectively overcome these shortcomings. The principle of laser-ignited self-propagating sintering is shown in Figure 1. The laser-ignition self-propagating sintering technology used in this paper is characterized by simple equipment and an easy process. In addition to the energy required for the ignition reaction, no external heat is needed, saving energy, reducing manufacturing costs, shortening the reaction time of self-propagating high-temperature synthesis, and high production efficiency. And the reaction can generate highly active substable products. These products are difficult to obtain by traditional processing technology and are favorable for the preparation of HEAs [17,18].

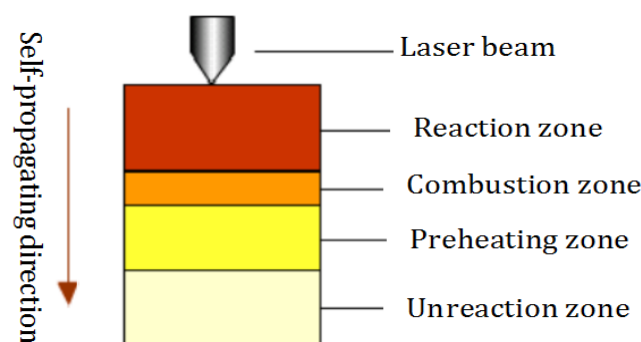


Figure 1. Schematic diagram of laser-ignited self-propagating sintering.

The purpose of this study is to investigate the effect of the Ni element on the organization and properties of AlCoCrFeMn through an improved technique (laser-ignition self-propagation sintering), which provides a theoretical basis for the preparation of new HEAs containing the Mn element.

2. Experimental Materials and Method

Pure Al, Co, Cr, Fe, Mn, and Ni powders with a purity of 99.9% (atomic fraction) and particle size of 75 μm were used for the tests [19]. AlCoCrFeMn and AlCoCrFeNiMn hybrid powders were prepared according to the equimolar ratio. The powder was mixed in a 200 r/min QM-3SP2 planetary ball mill (Changsha MITR Instrument Co., Changsha, China) grinding for 8 h and powder ball mill for 1 h. The ball milling process can refine the aggregate powder particles, ball milling the powder particle size to 0.1 μm , improve the chemical activity of the powder, and promote the reaction between the powder to form a new phase. After ball milling for 9 h, the machine was shut down to remove the ball milling jar, dissipated for 25 min, and then required vacuum-drying for 8 h to obtain the ball-milled aggregates. This experiment adopts the agglomeration method to granulate the aggregates after ball milling and the binder addition amount is 0.7% of the aggregate powder; they are then placed in the drum of the turntable granulator for continuous tumbling granulation, and after completing the agglomeration in the drying box, they are dried and cured. The granulation process solves the problem that the composite metal powder particle size is too small and easy to fly during compression, improves the homogeneity of the composite powder, and promotes the reaction between the composite powder. Figure 2 shows the morphology of the dried mixed powders. Among them, the powder particles of the Fe, Ni, Co, and Mn elements were spherical, the powder particles of the Al elements were nearly spherical, and the powder particles of the Cr elements were irregularly shaped. The ball-milled sintered powder was poured into a homemade mold and pressed into a cylindrical press billet of $\varnothing 20\text{ mm} \times 15\text{ mm}$ on a WE-300B liquid crystal digital display universal testing machine (Jinan KAYI Testing Machine Manufacturing Co., Jinan, China) at a pressure of

150 kN. The extruded billet was irradiated with an HL-1500 helium-free transverse flow CO₂ laser (Shenyang Continental Laser Technology Co., Shenyang, China) with a power of 1500 W and a spot diameter of 20 mm for 12 s, which triggered the self-propagation reaction. The laser-ignition self-propagation sintering process is shown in Figure 3.

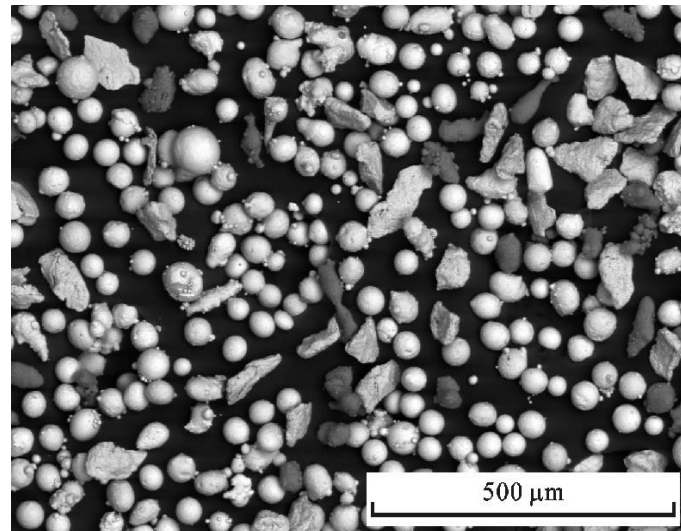


Figure 2. Particle morphology of mixed powder after drying.

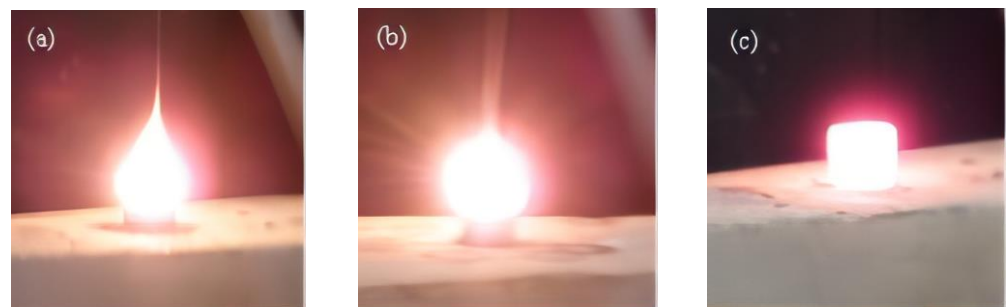


Figure 3. Laser-ignited self-propagating sintering process: (a) laser ignition; (b) self-propagating reactions; (c) sintering complete.

The physical phase analysis was carried out using a D8-ADVANCE X-ray diffractometer (Bruker, Karlsruhe, Germany) with the following experimental parameters: tungsten target, scanning angle of 10~100 degrees, scanning speed of 10°/min. The morphology was observed by a Verios 5 XHR SEM ultra-high-resolution scanning electron microscope (Thermo Fisher Scientific, Waltham, MA, USA), and the composition was analyzed by EDS. A room temperature dry sliding wear test was carried out on an MS-200 sliding friction wear tester (AVIC DINGLI Instrument Co., Beijing, China) to check the wear resistance of the alloy. The experiment was carried out with 600-mesh-thick sandpaper, load of 20 N, and effective wear time of 5 min. The wear rate of the alloy was calculated by measuring the wear area of the specimen and the mass of the specimen before and after wear, and the wear rate was calculated by the formula shown in Equation (1) as follows:

$$\omega = \frac{m_0 - m_x}{s} \quad (1)$$

In the formula, ω is the wear rate ($\text{mg} \cdot \text{cm}^{-2}$), m_0 is the mass before wear (mg), m_x is the mass after wear (mg), and s is the wear area (cm^{-2}).

In order to study the electrochemical corrosion performance of the alloy, a CHI-630E electrochemical workstation (Shanghai Hongyi Instrument Co., Shanghai, China) was used

for the test, as a three-electrode system, in which the HEA was the research electrode, the saturated glycomercury electrode was the reference electrode, and the platinum sheet was the auxiliary electrode. First of all, the metal specimen to be tested had an exposed surface area of 1 cm^2 , the rest of the surface sealed with epoxy resin, and was then placed in the mass fraction of 3.5% NaCl solution for 30 min, to be stable after the open-circuit potential impedance test and electrochemical polarization curves to evaluate the corrosion resistance of the alloy, and ZsimpDemo 3.20 software was used to fit the test data. The scanning speed of the kinetic potential polarization curve was 2 mV/s , the scanning potential range was -1.0 – 1.0 V , and the frequency range of the electrochemical impedance spectroscopy test was 10^5 – 10^{-2} Hz .

3. Results and Analysis

3.1. Phase Analysis of HEA

The XRD diffractograms of the two alloys are shown in Figure 4. From the figure, it can be seen that the AlCoCrFeMn alloy consists of a BCC phase structural solid solution without intermetallic compound diffraction peaks. It is shown that the addition of Ni has a promoting effect on the formation of the FCC phase, but the BCC phase is still dominated by the FCC phase solid solution.

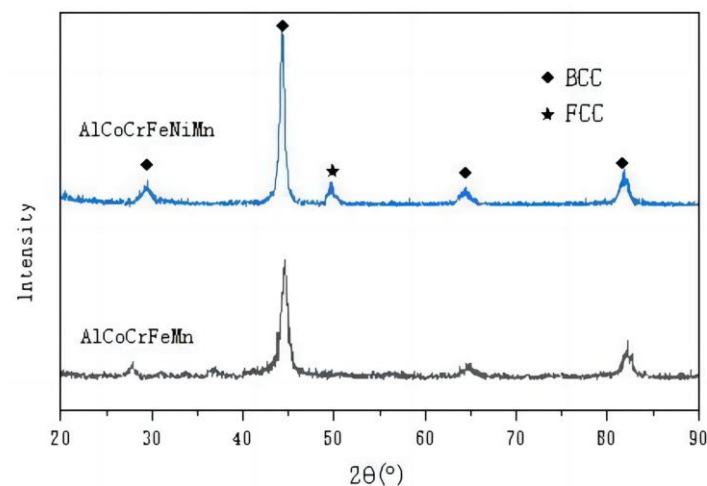


Figure 4. XRD results of HEA.

According to the Gibbs phase law, the number of equilibrium phases produced by the mixing of n elements in a general alloy system is $n + 1$, and the number of equilibrium phases produced by nonequilibrium solidification is greater than $n + 1$. However, in the HEA system, the number of phases produced by solidification is much smaller than that of general alloys, and instead of complex intermetallic compounds, the phases formed are simple BCC and FCC phases. According to Boltzmann's assumption, the mixing entropy [20] of the system is obtained as shown in Equation (2):

$$\Delta S_{conf} = -R \sum_{i=1}^n C_i \ln C_i \quad (2)$$

In the formula, R is the ideal gas constant ($R = 8.3145 \text{ J/K} \cdot \text{mol}$), n is the number of components, and C_i is the content of element i .

When the compositions of the AlCoCrFeMn and AlCoCrFeNiMn alloys satisfy the equimolar ratios, the maximum enthalpies of mixing are $1.61 R$ and $1.79 R$, respectively. The atoms in the alloys are very disorganized because of their high entropy of mixing. Therefore, atoms that diffuse can only diffuse in concert with different kinds of atoms, and the energy required for diffusion and the potential barriers required to cross are much larger. Therefore, the diffusion of atoms in the alloy becomes very difficult, which greatly

reduces the free energy of the alloy system and promotes the formation of randomly intercalated solid solutions. Finally, the alloys solidify to form simple BCC and FCC structured solid solutions.

3.2. Organization Analysis of HEA

The alloy morphology of AlCoCrFeMn and AlCoCrFeNiMn is shown in Figure 5. As shown in Figure 5a, there are many holes in the alloys and the distribution of the holes is not uniform. As shown in Figure 5c, there are almost no voids in the alloy, and it is relatively flat. The results show that Ni improves the porous defects in AlCoCrFeMn. Figure 5b shows the petal-like organization, which is aggregated and many fine cellular tissues appear. As shown in Figure 5d, after the addition of the Ni element, the structure changed significantly to an equiaxed crystal structure.

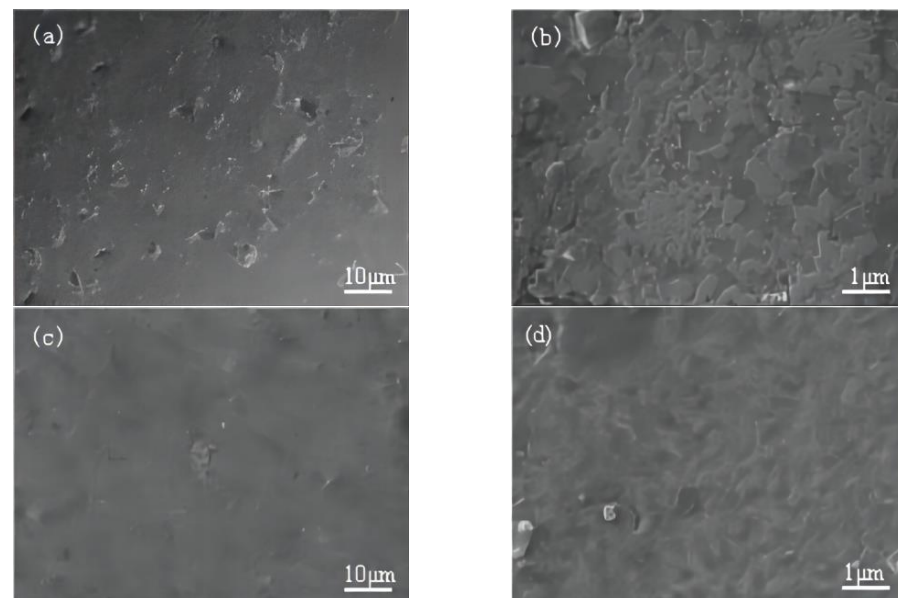


Figure 5. Micro-morphology of HEA: (a) AlCoCrFeMn 100 times; (b) AlCoCrFeMn 1000 times; (c) AlCoCrFeNiMn 100 times; (d) AlCoCrFeNiMn 1000 times.

From the SEM image of the HEA structure in Figure 6a, it can be seen that the composition of the AlCoCrFeMn alloy has undergone significant segregation, and the petaloidal organization appears. From the EDS spectrum analysis results in Table 1, it can be found that the three elements Cr, Fe, and Mn are mainly distributed in the petal-like organization. The thermodynamic analysis shows that the mixing enthalpy of the three elements is low, the affinity is good, and it is easy to form a solid solution. The Cr element is heavily enriched in the cellular structure. Fe and Cr are difficult to form a strong bonding and homogeneous solid solution, and therefore are easy to be segregated.

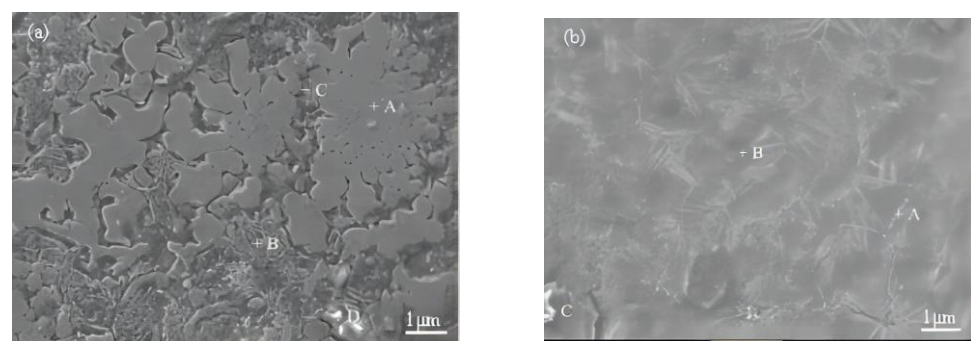


Figure 6. SEM results of different organization regions of HEA: (a) AlCoCrFeMn; (b) AlCoCrFeNiMn.

Table 1. EDS AlCoCrFeMn HEA analysis results.

Element	Al	Co	Cr	Fe	Mn
Nominal	20.00	20.00	20.00	20.00	20.00
A	8.94	12.68	27.51	24.46	26.41
B	2.68	3.46	60.45	13.66	19.75
C	5.08	7.56	44.78	19.76	22.82
D	6.98	9.15	39.58	20.35	23.94

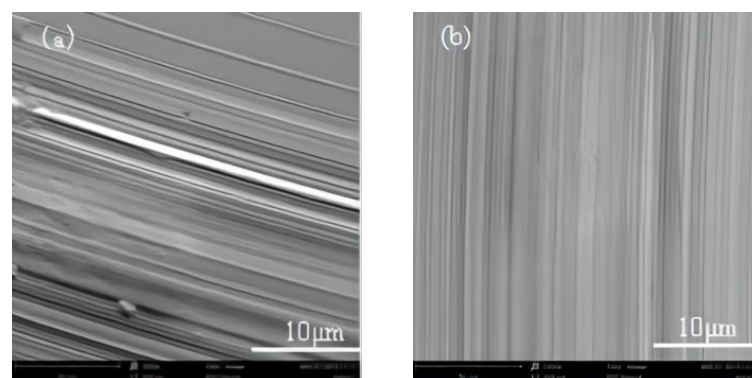
The EDS spectral analysis results of the gray-white organization (region A), gray organization (region B), and petal-like organization (region C) in Figure 6b are analyzed, as shown in Table 2, which shows that the alloy has a homogeneous composition with elemental ratios close to the nominal composition. It is shown that the mixing enthalpy of Ni atoms with other atoms in the alloy is between -40 kJ/mol and 10 kJ/mol, which is in accordance with the third condition of the solid solution formation principle of multi-major HEAs. Therefore, the Ni element is well compatible with other elements to form isometric crystals.

Table 2. EDS AlCoCrFeNiMn HEA analysis results.

Element	Al	Co	Cr	Fe	Mn	Ni
Nominal	16.66	16.66	16.66	16.66	16.66	16.66
A	18.04	16.14	16.45	13.96	19.56	15.85
B	17.94	15.88	15.77	14.78	18.69	16.94
C	17.02	15.65	16.06	14.97	18.82	17.48

3.3. Wear Resistance Analysis of HEAs

Figure 7a,b show the wear morphology of AlCoCrFeMn and AlCoCrFeNiMn, respectively. It is observed that the matrix surface of Figure 7a undergoes severe wear and a large number of furrows are generated on the surface, indicating that the matrix particles are dislodged due to fatigue under the action of stress during the wear process. The plough furrows in Figure 7b are shallower, and the surface has not been completely destroyed, which is due to the particles on the surface of the matrix, which fall off in the repeated friction of the sandpaper and are pressed between the sandpaper and the surface of the sample during the process of falling, which causes microcracks or even deformation on the surface of the sample. In comparison, AlCoCrFeNiMn has a higher wear resistance than AlCoCrFeMn.

**Figure 7.** (a) Wear morphology of AlCoCrFeMn. (b) Wear morphology of AlCoCrFeNiMn.

As can be seen from Figure 8, the wear curve of AlCoCrFeMn is located at the top and has the worst wear resistance; the wear curve of AlCoCrFeNiMn is located at the bottom and has the best wear resistance. And with the prolongation of the wear time, the increase

in the AlCoCrFeMn wear is larger than that of AlCoCrFeNiMn. The results show that the wear rate of the alloy decreases with the addition of Ni. Combined with the XRD analysis, it was found that a small amount of the FCC phase in the homogeneous equiaxed grain organization hinders the movement of dislocations after the addition of the Ni element, which is similar to the strengthening mechanism of the second phase in conventional alloys. Therefore, the two phases complement and promote each other in an appropriate ratio, thus reducing the wear rate of the alloy.

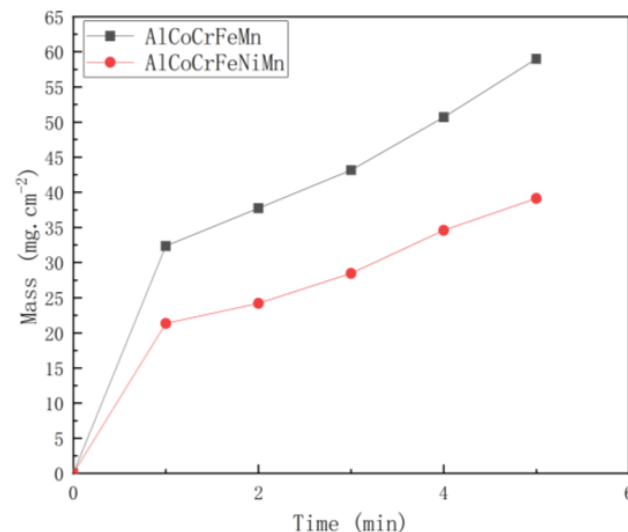


Figure 8. Abrasive wear curves of different HEAs.

3.4. Corrosion Resistance Analysis of HEAs

Figure 9 shows the electrochemical test diagrams of AlCoCrFeMn and AlCoCrFeNiMn HEAs prepared by the laser-sintering technique in NaCl solution with a mass fraction of 3.5%, respectively. Figure 9a shows the polarization curves, from which it can be seen that the self-corrosion potential of the AlCoCrFeNiMn HEA material prepared by laser-sintering technology is higher and the corrosion current density is smaller compared to those of AlCoCrFeMn, and the pitting potential is greatly increased, which enlarges the passivation interval. From Figure 9b, it can be seen that both materials, AlCoCrFeMn and AlCoCrFeNiMn, exhibit typical capacitive arc characteristics, but the radius of the impedance semicircle of AlCoCrFeNiMn HEA is larger. Figure 9c,d show the Bode phase angle spectra and the Bode impedance spectra, respectively, from which it can be seen that both the phase angle and the total impedance of AlCoCrFeNiMn are larger compared with those of AlCoCrFeMn, which indicates that the corrosion resistance of AlCoCrFeNiMn is better. Table 3 shows the electrochemical parameters of the two materials, and it is not difficult to find that the corrosion rate of the AlCoCrFeNiMn HEA in the mass fraction of 3.5% NaCl solution is smaller than that of AlCoCrFeMn, because in the HEA system, the addition of the Ni element promotes the formation of a dense passivation film, which protects the alloy material from the destruction of Cl^- in the 3.5% NaCl solution, and thereby shows excellent corrosion resistance.

Table 3. Electrochemical parameters of AlCoCrFeMn and AlCoCrFeNiMn HEAs.

Alloy Name	E_{corr}/V	$I_{\text{corr}}/(\text{A} \cdot \text{cm}^{-2})$	Corrosion Rate/ $(\text{mm} \cdot \text{a}^{-1})$
AlCoCrFeMn	−0.718	3.413×10^{-5}	9.68×10^{-3}
AlCoCrFeNiMn	−0.325	1.643×10^{-5}	2.08×10^{-3}

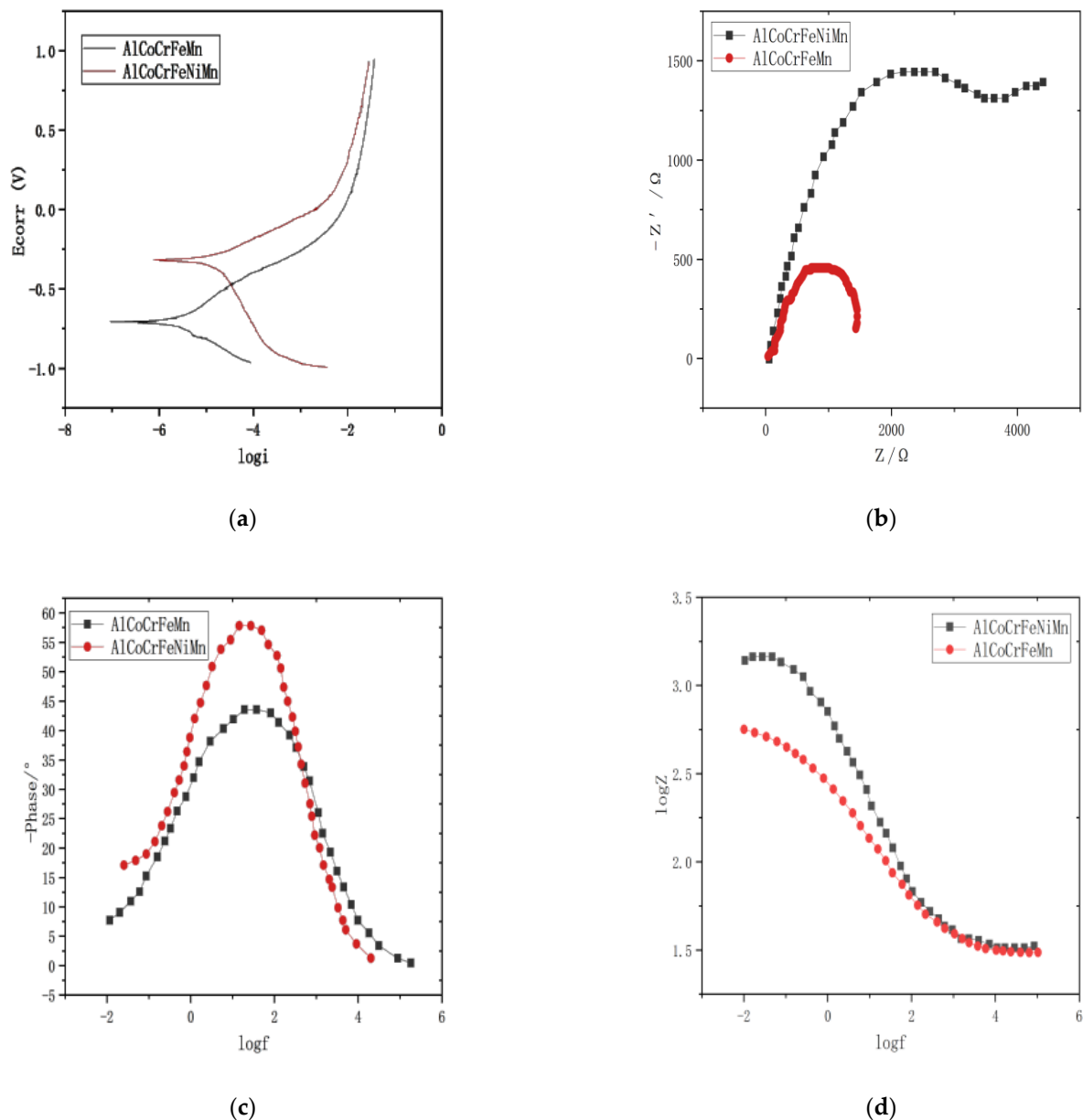


Figure 9. Electrochemical test results of AlCoCrFeMn and AlCoCrFeNiMn HEAs: (a) polarization curves; (b) Bode phase angle spectra; (c) Nyquist diagram; (d) Bode impedance spectra.

3.5. Compression Property Analysis of HEAs

Figure 10 shows the compression curves of the AlCoCrFeMn and AlCoCrFeNiMn HEAs at room temperature, and Table 4 shows their yield and compressive strengths.

Combined with Figure 10 and Table 4, the analysis shows that, compared with AlCoCrFeMn, no matter the strength or plastic toughness, AlCoCrFeNiMn has a good performance and the comprehensive mechanical properties are better. The reason is that in the region of the lower Ni content in AlCoCrFeNiMn, the difference in the atomic radius in the alloy is large, leading to lattice distortion, so that the solid solution strengthening effect is enhanced; while in the region of a higher Ni content, a variety of elements in the high-entropy alloy diffuse synergistically, the nucleation and growth of phases are delayed considerably, and the formation of an equiaxial crystalline structure, the area of the grain boundary, is enlarged, which leads to the dislocation motion being bound and the alloy strength being improved.

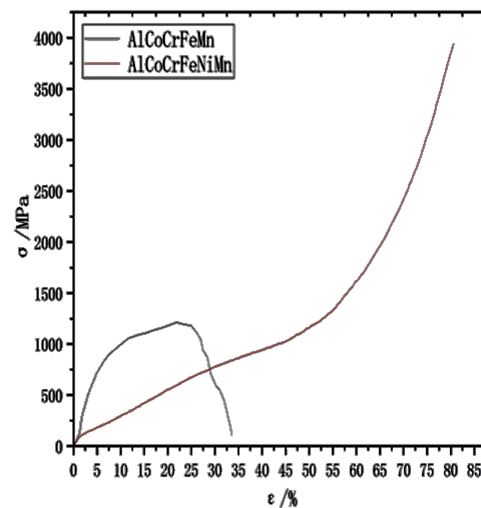


Figure 10. Room temperature compression curves of HEAs.

Table 4. Compression performance parameters of HEAs.

Alloy Name	Compression Rate/%	Yielding Strength/MPa	Compressive Strength/MPa
AlCoCrFeMn	34	150	1280
AlCoCrFeNiMn	83	780	3920

4. Conclusions

- (1) The organizational structure of AlCoCrFeNiMn HEA materials is the equiaxial crystal structure; compared with AlCoCrFeMn, the surface is more dense, the metal composition of the segregation phenomenon has been suppressed, and a small amount of the FCC phase appears in the composition of the alloy.
- (2) Compared with AlCoCrFeMn HEAs, the self-corrosion potential of AlCoCrFeNiMn is more positive, the pitting potential is higher, and the corrosion current density is smaller, thus expanding the passivation zone, and showing better corrosion resistance in 3.5% NaCl solution.
- (3) The addition of Ni promotes the formation of the FCC phase in the alloy, which greatly improves the wear resistance and yield strength of the HEA, making its comprehensive mechanical properties better.

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Conflicts of Interest: We declare that we have no financial and personal relationships with other people or organizations that can inappropriately influence our work, there is no professional or other personal interest of any nature or kind in any product, service, and/or company that could be construed as influencing the position presented in, or the review of, the manuscript entitled.

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