

Article **Evolution of Microstructure and Mechanical Properties of the CoFeNiMnMox High-Entropy Alloys**

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Abstract: In this study, the microstructure evolution and mechanical properties of CoFeNiMnMox high-entropy alloy after adding Mo were investigated. With the increase in Mo content, Mo atoms occupied lattice sites and the microstructure changed from hypoeutectic of primary FCC-phase to Laves phase particles of FCC-phase, and Vickers microhardness increased steadily from 193 to 357. The yield strength increased from 187 MPa when the Mo content was 0.25 to 537 MPa when the Mo content was 1.0. The microstructure formation can be explained by atomic size difference δ and parameter $\Delta \chi_A$. $\delta \ge 3.87\%$ and $\Delta \chi_A \ge 5.24\%$ criterion is proposed to better predict the microstructure formation of the coexistence of (FCC + Laves) phases.

Keywords: high-entropy alloy; laves phase; microstructure; mechanical properties

1. Introduction

High-entropy alloys (HEAs), firstly proposed by Yeh et al. [1,2], are a novel structural material. They are defined as alloys composed of at least 5 principal elements, and each of which has an atomic percentage concentration between 5% and 35% [3–7]. Some face-centered cubic (FCC) alloys, especially CrCoNi-based HEAs, have been proved to have higher strength and special damage tolerance, especially at low temperature [8–11]. Fu et al. [12] tested the tensile properties of as-cast CrMnFeCoNi alloy prepared by vacuum arc melting and found that the yield strength, tensile strength and fracture elongation of the alloy at room temperature were 138 MPa, 338 MPa and 25%, respectively. ABDUKADIR A-Mar et al. [13] prepared CrMnFeCoNi alloy with relatively uniform element distribution by laser melting deposition, and tested its mechanical properties at room temperature. The results showed that the yield strength of the alloy was 231 MPa and the fracture elongation was 55%. CrMnFeCoNi alloy prepared by different methods has low room-temperature strength, especially the yield strength is less than 300 MPa, which is the key factor limiting its use at room temperature.

Based on the above reasons, researchers began to consider the method of replacing elements to improve the microstructure and properties of CrMnFeCoNi alloy. Zuo et al. [14] systematically studied the microstructure and properties of CoFeNiMn alloy by adding Al, Ga and other elements, respectively. The results show that the addition of Al, Ga and Sn transformed the alloys from disordered-phase FCC to ordered phase body-centered cubic (BCC), which makes the alloys show high strength and low ductility. Cui et al. [15] also studied the microstructure and properties of CoFeNiMn-based high-entropy alloys containing Ti and Al elements, and discussed the formation law of BCC phase and FCC phase. With the increase in the Ti content and the decrease in the Al content, the crystal structure of CoFeNiMnTixAl1-x HEAs changes from BCC to FCC + BCC, then to FCC + BCC + Laves, and then to FCC + Laves phases.

Recently, in order to expand and enrich the field of multiprincipal component alloys, some researchers have studied the microstructure and compressive properties of Mocontaining alloys such as AlCoCrCuFeNiMox and AlCoCrFeNiMox [16]. When Mo atoms



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occupy lattice sites, the lattice distortion energy will increases significantly and the effect of solid solution strengthening enhances; thus, the alloy strength greatly increases with the decrease in ductility. Zhao et al. [17] prepared a high-entropy alloy coating by directly adding Mo to CrMnFeCoNi alloy using the direct current Magnetron sputtering method. The results show that with the increase in Mo content, the film is densified, and the phase structure gradually changes from a single FCC phase to a mixture of FCC and BCC phases. The grain size decreases, the lattice constant increases, from no Mo to Mo = 1, and the hardness increases from 8.5 GPa to 12 GPa, an increase of 41%. Element Mo is often regarded as one of the important elements in the design of high-entropy alloys, but there are few related experiments studied based on CoFeNiMn. Motivated by this fact, the alloy studied in this paper does not contain Al for strongly forming the BCC phase, but CoFeNiMnMox alloys are prepared. The expectation is to form microstructures containing FCC and Laves phases.

In the present study, CoFeNiMnMox high-entropy alloy was prepared by arc melting, in which *x* ranged from 0.25 to 1.0. The microstructure evolution and mechanical properties of the CoFeNiMnMox high-entropy alloy with Mo additions were investigated.

2. Experimental Details

Alloy ingot with nominal composition of CoFeNiMnMo_x (x = 0.25, 0.50, 0.75, and 1.00, in molar fraction; labeled as Mo0.25, Mo0.50, Mo0.75, and Mo1.00, respectively) alloys were arc-melted. Purity higher than 99.9% wt% of pure metal was raw materials. In order to ensure the uniformity of the alloy composition, the alloy was repeatedly melted six times with the help of electromagnetic stirring.

The specimens were cut from the samples, and polished for metallurgical observation. The phase composition was identified by X–ray diffractometer (XRD; Bruker D8 Advance) with Cu K α as a radiation. It was examined with a diffraction angle (2 θ) from 20° to 100° at a scanning rate of 4° min⁻¹. The microstructure was characterized by scanning electron microscopy (SEM; FEI Quanta 400) operated in back-scatter electron (BSE) mode. Local components were analyzed using energy dispersive spectrometry (EDS; Oxford Inca 350). The compression tests were carried out on an electronic universal testing machine (Instron 5985) at a strain rate of 2 × 10⁻⁴ s⁻¹ at room temperature. The samples with a dimension of 5 mm × 5 mm × 10 mm were prepared for compression testing and wire electrode cutting method is used. The Vickers microhardness (HV) of the cross-section surface was measured with a 402 MVD Vickers microhardness tester under 100 g load for 15 s. Each Vickers microhardness value is the average of 15 points at random locations.

3. Results and Discussion

3.1. Crystal Structure Characterization of the CoFeNiMnMo_x Alloys

According to the literature [18], CoFeNiMn alloy is a single FCC structure with a lattice constant of 3.5985 A. Figure 1 displays the XRD patterns taken from the as-cast CoFeNiMnMo_x alloys. According to the XRD results, it is found that FCC and Laves phases coexist in CoFeNiMnMo_x alloys with different Mo content [19,20]. The Laves phase is identified as a Fe₂Mo type with the lattice parameters of a = 4.962 A and c = 16.150 A. With the increase in Mo content, the diffraction peak of FCC solid solution structure shifts towards to a lower 2θ peak. According to the Bragg's law " $2d\sin\theta = n\lambda$ ", the lattice constant of the FCC phase can be deduced. The lattice constant (a) of the FCC phase is estimated to be 3.593 A, 3.601 A, 3.603 A, and 3.612 A for Mo0.25, Mo0.50, Mo0.75, and Mo1.00 alloys, respectively. Because Mo has a larger atomic radius (1.390 Å) than other constituent elements, including Co (1.251 Å), Fe (1.241 Å), Ni (1.246 Å) and Mn (1.350 Å). The results indicate that Mo element can be partially dissolved into FCC solid solution structure, thus leading to the lattice expansion. Figure 2 displays the variation in lattice strain of FCC phase as a function of Mo content in CoFeNiMnMo_x alloys. Lattice strain (ε) is expressed as $\varepsilon = |a-a0|/a0$, where a0 is the lattice parameter of FCC phase in Mo0.25 alloy. With the increase in Mo content, the lattice strain of FCC phases increases steadily.



Figure 1. XRD patterns of CoFeNiMnMo_x (x = 0.25, 0.5, 0.75 and 1.0) alloys.



Figure 2. Lattice strain of FCC phase as a function of Mo content in CoFeNiMnMo_x (x = 0.25, 0.5, 0.75 and 1.0) alloys.

3.2. Microstructure Observation in the CoFeNiMnMo_x Alloys

The microstructure of CoFeNiMnMo_x alloys was characterized using the backscattering electron mode under SEM. The typical microstructure of CoFeNiMnMo_x alloys under as-cast condition is presented in Figure 3. Table 1 lists the chemical compositions of the various phases identified by EDS. As shown in Figure 3a,b, when the Mo content is 0.25, the alloy evolves from a single FCC matrix to FCC+Laves phase, in which Laves phase is distributed on the FCC matrix in granular form. When the Mo content is 0.5, it can be seen from Figure 3c,d that the microstructure of the alloy is still composed of FCC phase and Laves phase, but interestingly, the laves phase turns into slate-like, similar to dendrites distributed on the FCC matrix. When the Mo content is 0.75, the microstructure of the alloy consists of a primary dendrite phase and Laves phase distributed between dendrites, as show in Figure 3e,f. Furthermore, when the Mo content is 1.0, the dendrite FCC structure becomes more obvious, and the Laves phase is embedded in the dendrite, as show in Figure 3g,h.



Figure 3. SEM BSE images of CoFeNiMnMo_{*x*} alloys. (**a**,**b**) x = 0.25, (**c**,**d**) x = 0.50, (**e**,**f**) x = 0.75 and (**g**,**h**) x = 1.00.

. 11		Elements (at. %)						
Alloys	Kegion –	Со	Fe	Ni	Mn	Мо		
Mo0.25	FCC	22.09	19.08	23.44	23.67	11.73		
	Laves phase	19.87	18.78	24.72	24.99	11.97		
Mo0.50	FCC	19.04	17.25	22.33	21.45	19.93		
	Laves phase	20.25	19.58	20.29	10.31	19.57		
Mo0.75	FCC	20.87	19.79	20.38	17.72	21.25		
	Laves phase	20.53	20.65	18.53	18.80	21.51		
Mo1.00	FCC	21.85	21.11	18.93	17.89	20.21		
	Laves phase	23.65	21.10	21.70	16.23	18.20		

Table 1. Chemical compositions of various phases in CoFeNiMnMo_x alloys in atomic percentage.

3.3. Phase Stability of CoFeNiMnMo_x Alloys

In order to predict the phase formation of HEAs, several criteria have been proposed [21]. The mixing enthalpy (ΔH_{mix}), the entropy of mixing (ΔS_{mix}), parameter Ω , atomic size difference (δ), valence electron concentration (VEC), Pauling electronegativity ($\Delta \chi_{\text{P}}$), and Allen electronegativity ($\Delta \chi_{\text{A}}$) are defined as (1)–(7) [21],

$$\Delta H_{mix} = \sum_{i=1, j \neq j}^{n} 4\Delta H_{mix}^{ij} c_i c_j \tag{1}$$

$$\Delta S_{mix} = -R \sum_{i=1}^{n} c_i lnc_j \tag{2}$$

$$\Omega = \frac{\left[\sum_{i=1}^{n} c_i(T_m)_i\right] \Delta S_{mix}}{|\Delta H_{mix}|} \tag{3}$$

$$\Omega \delta = 100 \sqrt{\sum_{i=1}^{n} (1 - r_i / \sum_{i=1}^{n} c_i r_i)}$$
(4)

$$VEC = \sum_{i=1}^{n} (VEC)_i \tag{5}$$

$$\Delta x_p = \sqrt{\sum_{i=1}^{n} c_i (\chi_i^P - \sum_{i=1}^{n} c_i \chi_i^P)^2}$$
(6)

$$\Delta \chi_A = \sqrt{\sum_{i=1}^n c_i (1 - \chi_i^A / \sum_{i=1}^n c_i \chi_i^A)^2}$$
(7)

where, c_i and c_j represent the mole fraction of the *i*th and *j*th element, respectively; *R* is an ideal gas constant; r_i is the atomic radius; $(T_m)_i$, $(VEC)_i$, χ_i^P , and χ_i^A are the melting point, valence electron concentration, Pauling electronegativity and Allen electronegativity of the *i*th element, respectively.

Table 2 lists the melting point, atomic radius, and mixing enthalpy of different atom pairs in CoFeNiMnMo_x alloys. Using Equations (1)–(7), the values of ΔH_{mix} , ΔS_{mix} , Ω , δ , VEC, $\Delta \chi_P$ and $\Delta \chi_A$ of CoFeNiMnMo_x alloys are calculated and listed in Table 3. With increasing Mo content, the atomic size difference δ increase and the VEC decreases, indicating that the addition of the Mo will lead to the severe lattice distortion and structural instability.

It is demonstrated that the BCC phases are stable when VEC < 6.87, while the FCC phases are stable when VEC ≥ 8 [22]. The ΔH_{mix} values are in the range from -4.00 kJ mol⁻¹ for CoFeNiMn alloy to -3.84 kJ mol⁻¹ for CoFeNiMnMo alloy. Ω is in the range from 4.92 for CoFeNiMn alloy to 6.24 for CoFeNiMnMo alloy. All the given

values satisfy the criteria $-20 < \Delta H_{mix} < 5 \text{ kJ mol}^{-1}$ and $\Omega \ge 1.1$ are required for solid solution formation.

Table 2. Enthalpy of mixing (Unit: kJ/mol) of different atom pairs in the CoFeNiMnMo_x alloys.

Elements (Melting Point, Atomic Radius)	Fe	Со	Ni	Mn	Мо
Fe (1811 K, 1.241 Å)	-	-1	-2	0	0
Co (1768 K, 1.251 Å)		-	0	-5	-5
Ni (1728 K, 1.246 Å)			-	-8	-8
Mn (1519 K, 1.350 Å)				-	+5
Mo (2883 K, 1.390 Å)					-

Table 3. Calculated values of valence electron concentration (VEC), enthalpy of mixing (ΔH_{mix}), mixing entropy (ΔS_{mix}), Ω , atomic size difference (δ), Allen electronegativity ($\Delta \chi_A$), and Pauling electronegativity ($\Delta \chi_P$) in the CoFeNiMnMo_x alloys.

Alloys	Phases	VEC	$\Delta H_{ m mix}/ m kJ~mol^{-1}$	$\Delta S_{ m mix}/J$ mol $^{-1}$ K $^{-1}$	δ(%)	$\Delta\chi_{ m A}$ (%)	Ω	$\Delta \chi_{ m P}$	Reference
CoFeNiMnMo _{0.25}	FCC + Laves	8.35	-3.98	12.71	3.87	5.24	5.67	0.158	This work
CoFeNiMnMo _{0.50}	FCC + Laves	8.22	-3.95	13.15	4.39	6.63	5.91	0.177	This work
CoFeNiMnMo _{0.75}	FCC + Laves	8.11	-3.90	13.33	4.63	7.61	5.99	0.187	This work
CoFeNiMnMo _{1.00}	FCC + Laves	8.00	-3.84	13.38	4.80	8.32	6.19	0.195	This work
CoFeNiMn	FCC	8.5	-4.00	11.53	3.55	2.65	4.92	0.143	[14]

According to the δ - $\Delta\chi A$ criterion proposed by references [20], the Laves phase is formed when $\delta \ge 3.87\%$ and $\Delta\chi A \ge 5.24$. The parameters δ and $\Delta\chi A$ of RHEAs reported in a large number of literatures [15] are summarized, and the δ - $\Delta\chi A$ plot are shown in Figure 4. The single FCC-phase lies in the left bottom corner of the graph ($\delta < 3.87\%$) and the (FCC + Laves) double-phase are formed when $\delta \ge 3.87\%$ and $\Delta\chi A \ge 5.24$. Therefore, $\delta \ge 3.87\%$ and $\Delta\chi A \ge 5.24$ criterions are credible parameters for correctly predicting the coexistence stability of the (FCC + Laves) zone.



Figure 4. Plots of $\Delta \chi_A$ vs. δ for predicting the evolution of FCC and Laves phase in the studied CoFeNiMnMo_x (*x* = 0.25, 0.5, 0.75 and 1.0) alloys and other reported HEAs [14,15,22].

3.4. Mechanical Properties

Figure 5 displays the compressive stress-strain curves of CoFeNiMnMo_x alloys at room temperature. The enlarged scan for CoFeNiMnMo alloy is shown in the bottom right

corner of Figure 5. Obviously, Mo content has a remarkably influence on the compressive properties of the alloys. The yield strength ($\sigma_{0.2}$), fracture strength (σ_f) and fracture strain (ε_f) of the alloys are also listed in Table 4. It can be seen from Figure 4 that all these alloys show significant plastic properties, and no compression fracture occurs when the fracture strain reaches 80%. Figure 5 shows that the yield strength of the alloy increased steadily with the increase in Mo content. When Mo with larger atomic radius was dissolved in FCC solid solution structure and occupied the lattice site, the lattice distortion energy increased significantly, which enhances the solid solution strengthening effect. The yield strength increased from 187 MPa when Mo content was 0.25 to 537 MPa when Mo content was 1.0. According to the analysis [20], the appearance of the Laves phase will increase the strength of the alloy, but also reduce the plastic properties of the alloy. However, in this study, the experimental results that do not conform to this law are obtained, which may be due to the influence of the distribution form and phase morphology of the Laves phase in the FCC matrix.



Figure 5. Room-temperature compressive stress-strain curves of CoFeNiMnMo_x (x = 0.25, 0.5, 0.75 and 1.0) alloys and enlarged scans for the CoFeNiMnMo alloy.

Table 4. Room-temperature compressive properties of the studied CoFeNiMnMo_x alloys and other reported CoCrFeNi-based and CoCrCuFeNi-based alloys.

Alloys	$\sigma_{0.2}$ (MPa)	σ_f (MPa)	ε_f (%)	Reference
CoFeNiMnMo _{0.25}	187	-	>80	This study
CoFeNiMnMo _{0.50}	261	-	>80	This study
CoFeNiMnMo _{0.75}	394	-	>80	This study
CoFeNiMnMo _{1.00}	531	-	>80	This study
CoFeNiMn	155.8	-	>50	[14]

Figure 6 shows the variation in Vickers hardness (HV) of CoFeNiMnMo_x alloys. The microhardness of Mo0.25 alloy is 193 HV. The addition of Mo significantly enhances the microhardness of the alloys. As the Mo content increases, the Vickers microhardness increases steadily from 193 HV for Mo0.25 alloy to 357 HV for Mo1.00 alloy. The increase in microhardness can be explained by the following factors. Firstly, the addition of Mo leads to obvious lattice distortion in the FCC matrix, resulting in solid solution strengthening. Secondly, the microhardness is directly related to the microstructure of the alloys. The FCC phase matrix exhibits excellent plasticity but poor strength. However, the formation

of a hard and brittle Laves phase is conducive to improving dislocation density and hindering the dislocation movement. The volume fraction of the Laves phase increases with the increase in Mo content in the alloys. Thus, it will enhance the degree of hindering dislocation movement, thereby improving their hardness.



Figure 6. Relationship between Vickers microhardness and Mo content in CoFeNiMnMo_x (x = 0.25, 0.5, 0.75 and 1.0) alloys.

4. Conclusions

- (1) With the increase in Mo content, the atomic size difference δ increased and the VEC decreased, indicating that the addition of the Mo element leads to the severe lattice distortion and structural instability of CoFeNiMnMo_x alloys.XRD and SEM analysis indicate that the microstructures of CoFeNiMnMo_x alloys are composed of FCC and a Laves phase.
- (2) The microstructure formation can be explained by atomic size difference δ and parameter $\Delta \chi A$. $\delta \geq 3.87\%$ and $\Delta \chi_A \geq 5.24$ criterions are more reliable parameters for correctly predicting the (FCC + Laves) coexistence region of CoFeNiMnMo_x alloys.
- (3) With the increase in Mo content, the hardness and yield strength of CoFeNiMnMo_x alloys increased from 193 to 357 and from 187 MPa to 537 MPa, respectively, but there was no compression fracture which occurred when the plastic deformation reached 80%.

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