Effect of Cr content on microstructures and properties of CoNiCuCrᵸ medium entropy alloys

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Abstract: CoNiCuCrₓ (ₓ=0,0.4,1) medium entropy alloys (MEAs) were prepared by powder metallurgy technology, and the effects of Cr content on the microstructures and properties of the alloys were studied. The results show that the CoNiCu MEA consists of FCC1 solid solution phase and FCC2 solid solution phase. With Cr addition, some new phases appear in the CoNiCuCrₓ MEAs, and they are FCC3 solid solution phase, BCC solid solution phase and a small number of NiCoCr intermetallic compound. The new structural phase improves the hardness, compressive yield strength, compressive strength and wear resistance of the MEAs, with the increase of Cr content. NiCoCuCr MEA shows good comprehensive mechanical properties, and the hardness, compressive yield strength, compressive strength and specific wear rate are 331 MPa, 511.87 MPa, 1142.71 MPa and 4.09×10⁻⁴ mm³/N·m respectively.

1. Introduction
Since 2004 [1], high entropy alloys (HEAs), after nearly 20 years of development, have been widely studied due to its excellent performance. The earliest studied HEAs are Cu-Co-Ni-Cr-Al-Fe alloys [2], and all subsequent studies have been developed on this HEAs series. The most representative one is CoCrFeMnNi HEA with good strength and plasticity, its tensile strength and elongation can greatly be improved to 1500 MPa and 51.23% respectively by adding trace C and rare earth elements [3,4]. In order to pursue more extreme performance, certain research results have been obtained in the fields of refractory HEAs and light weight HEAs, such as NbTaWMo refractory HEA [5], whose microhardness and compressive strength are 7684 MPa and 1460 MPa respectively. In the field of light HEAs, the density of Al₂Li₂₀Mg₁₀Sc₂₀Ti₃₀ HEA is as low as 2.67 g/cm³, while the AlCoCrFeNiSn₀.₁ HEA has a high compressive strength of 2950 MPa [6~8].

According to the ΔSₓₓ(xx₧) = -klnn, Where ΔSₓₓ(xx₧) is mixed entropy, k is Boltzmann constant, n is mixed complexity, R is gas constant R=8.314J/k mol, and n is the number of elements)
and the mixed entropy, all alloys are divided into three categories: low-entropy alloys (LEAs) when $S_{\text{mix}} \leq R$, medium-entropy alloys (MEAs) when $R < S_{\text{mix}} \leq 1.5R$, and high-entropy alloys (HEAs) when $1.5R < S_{\text{mix}}$ [9,10]. The principal component elements number of MEAs is $2 \leq n \leq 4$. Recently, the research on MEAs is gradually developed. The most typical MEA is CoNiCr which exhibits higher inoxidizability than those of CoCrFeMnNi and SS 304, and slow diffusion and high entropy effects that inhibits outward diffusion of metal cation and vacancy formation during oxidation [11]. The mechanical properties of the alloy can be improved by adding elements such as Ti, Al, W [12~14].

This paper designs a new CoNiCu MEA and adds different contents of Cr to explore the performance change of CoNiCuCr MEAs.

2. Methods

2.1 Composition Design

Based on empirical design criteria: $\Omega$ (Entropy enthalpy ratio of alloy) $\geq 1.1$, $\delta$ (Atomic radius difference) $\leq 6.6\%$, the atomic ratio of the alloy was calculated and designed [15]. $\Omega$ and $\delta$ are calculated by the following Formula (1) and Formula (2):

\[
\Omega = \frac{T_m \Delta S_{\text{mix}}}{\Delta H_{\text{mix}}} \quad (1)
\]

\[
\delta = \sqrt{\sum_{i=1}^{n} c_i \left(1 - \frac{r_i}{r}\right)} \quad (2)
\]

$\Omega$ is the ratio of entropy and enthalpy of alloy, $\delta$ is atomic radius difference, $T_m$ is theoretical melting point of alloy, $\Delta S_{\text{mix}}$ is entropy of mixing, $\Delta H_{\text{mix}}$ is enthalpy of mixing. The results are shown in Table 1 and the enthalpy of mixing of each element is shown in Table 2.

Table 1 Entropy enthalpy ratio and atomic radius difference of CoNiCuCr MEAs

| Alloys     | $T_m$ (K) | $\Delta S_{\text{mix}}$ (J/K) | $\Delta H_{\text{mix}}$ (kJ/mol) | $\Omega$ | $\delta$ (%) |
|------------|-----------|-------------------------------|----------------------------------|---------|--------------|
| CoNiCu    | 1604.67   | 9.13                          | 4.44                             | 3.30    | 1.23         |
| CoNiCuCr0.4 | 1666.47  | 11.07                         | 3.60                             | 5.13    | 1.19         |
| CoNiCuCr  | 1736.00   | 11.53                         | 2.75                             | 7.28    | 1.13         |

Table 2 Enthalpy of mixing of CoNiCuCr, HEAs [16]

| Enthalpy of mixing (kJ·mol$^{-1}$) | Co | Ni | Cu | Cr |
|-----------------------------------|----|----|----|----|
| Co                                | -  | 0  | 6  | -  |
| Ni                                | -  | -  | 4  | -  |
| Cu                                | -  | -  | -  | 12 |

2.2 experimental

The raw Co, Ni, Cu, and Cr powder (purity > 99%, diameter < 48μm) of CoNiCuCr$_x$ MEAs was milled by QM-3SP4 ball grinder under argon atmosphere protection, and the ball milling process was as follows: the milling time was 30 h, the milling speed was 150 r/min, and the mass ratio of ball and powder was (6~8):1. After milling, CoNiCu, CoNiCuCr$_{0.4}$ and CoNiCuCr bulk MEAs were prepared under the condition of 950°C, 30MPa and insulation time of 6 min by SMVB80 vacuum hot pressure sintering machine.

The phase structure was characterized by X ray diffractometer (D/max 2500V). The scanning speed was 8°/min, and 20 was 20°~100°. The density values of the MEAs were measured by Archimedes drainage method. The microstructures of the samples after corroded by aqua regia were analyzed by scanning electron microscope (SEM) (Hitachi S-3400N, with energy dispersive spectrometer (EDS)). The hardness was tested by HVS-1000 microhardness tester, and the compression performance was tested by Instron 8801 universal testing machine. The compression fractures were analyzed the fracture characteristics of the MEAs. Friction coefficient and wear resistance of the MEAs were studied by high speed reciprocating friction and wear tester (HSR-2M), and the wear morphologies were analyzed.
3. Results and discussion

3.1 Density and microstructure

The MEAs are processed into samples of 5 mm × 15 mm × 25 mm. The density measurement formula of the drainage method is shown as Formula (3). Table 3 shows the values of actual density, theoretical density and relative density of CoNiCu, CoNiCuCr0.4 and CoNiCuCr alloys (denoted by Cr0, Cr0.4 and Cr1 respectively). It can be seen from the Table 3 that the alloys with high density were obtained after sintering. The relative densities of the three alloys are about all 98%, and the highest relative densities reaches 98.88%. With the increase of Cr element, the density of the alloy decreases gradually, because the density of Cr element is smaller than that of other elements.

\[ \rho_s = \frac{m_1 \times \rho_l}{m_1 - m_2} \]  

\( \rho_s \) is the actual density of the alloy, \( m_1 \) is the mass in air, \( m_2 \) is the mass when suspended in water, \( \rho_l \) is the density of water, \( \rho_l = 0.9958 \text{ g/cm}^3 \).

| Alloys | Actual density (g/cm³) | Theoretical density (g/cm³) | Relative densities (%) |
|--------|-------------------------|-----------------------------|------------------------|
| Cr0    | 8.71                    | 8.90                        | 97.82                  |
| Cr0.4  | 8.59                    | 8.69                        | 98.88                  |
| Cr1    | 8.35                    | 8.46                        | 98.70                  |

Fig.1 shows the XRD patterns of NiCoCuCr\( x \) MEAs. On the whole, after 30h ball milling and sintering, the massive alloy obtained good solid solution and formed an alloy structure dominated by solid solution. In fact, high energy ball milling can promote the formation of supersaturated solid solution in alloy powder. The nanocrystalline phase produced by ball milling greatly increases the number of grain boundaries, and the grain boundaries store a large amount of lattice distortion energy, which can provide power for the phase transition. Lattice recombination occurs in the alloy during vacuum hot pressing sintering. In Cr0 alloy, the alloy forms a dual-phase solid solution structure, and mainly is composed of face-centered cubic structure 1 (FCC1) solid solution and FCC2 solid solution. With the increase of Cr content, the alloy gradually forms a four-phase structure. In the Cr0.4 alloy, the alloy is composed of FCC1 solid solution, FCC2 solid solution, FCC3 solid solution and a small amount of body-centered cubic structure (BCC) solid solution. In addition to the above four solid solutions, NiCoCr intermetallic compound is formed in Cr1 alloy, which can be explained by mixing enthalpy. It can be seen from Table 2 that the mixing enthalpy of Cr, Ni and Co is negative, and it is easier to form intermetallic compound because of the low mixing enthalpy with the increase of Cr.

Fig.2 shows the SEM photographs of CoNiCuCr\( x \) MEAs, and EDS analysis results are listed in Table 4. As can be seen from Fig.2, the different contrast of the phases can be clearly seen. The microstructure of CoNiCu MEA is relatively uniform, which is composed of two main phases. With the addition of Cr
element, the phases of the alloy gradually increase, and finally a four-phase structure appears. This result is consistent with the XRD results. The dark gray phase gradually increases, and the microstructure becomes nonuniform.

As can be seen from Fig.2(a1) and Table 4, region 1 is the matrix phase, and region 2 is the Co-rich phase, and region 3 is the black granular oxide phase. Combined with XRD analysis results, region 1 is FCC1 solid solution, and Co-rich phase in region 2 is FCC2 solid solution. The first reason for the formation of this Co-rich phase is as following: the melting point of Co element is higher than that of the other two alloy components, and it has a low diffusion coefficient, which leads to the slow alloying rate of Co, and Co does not fully diffuse into the new phase, so significant segregation phenomenon occurs after sintering. The second reason is the hysteretic diffusion effect which impedes the diffusion of elements, and results in the segregation of elements and prompts the precipitation of fine even nano-sized precipitates in the alloy. Region 3 is rich in O element, and it indicates that part of the alloy structure has obvious oxidation phenomenon, and the black region can be judged as oxide phase. In general, the structure of entropy alloy blocks in CoNiCu is relatively uniform.

According to Fig.2(a1), (b1) and (c1), obvious element segregation phenomenon appears in the alloy after adding Cr element, and the microstructure of the alloy is mainly composed of four phases with different contrast, which is consistent with the results of XRD analysis. In Cr0.4 MEA (such as Fig.2(b1)), the Ni-rich phase in region 1 is FCC2 solid solution, the Cu-rich phase in region 2 is FCC1 solid solution, the Cr-rich phase in region 3 is BCC solid solution, and the Co-rich phase in region 4 is FCC3 solid solution. Clear interface between different phases, there are two possible reasons for this: firstly, ball solid solution after milling is not sufficient; secondly, the sintering holding time is short, which results in insufficient diffusion and solution between atoms, and black oxide phase precipitates mainly at the interface of Cr-rich phase and Co-rich phase. The four major constituent phases of Cr1 MEA are the same as those of Cr0.4 MEA. In Fig.2(c1), region 1 is Ni-rich phase, region 2 is Cu-rich phase, region 3 is Cr-rich phase, and region 5 is Co-rich phase. The four phases correspond to FCC2, FCC1, BCC, and FCC3 in the XRD pattern, respectively. With the increase of Cr content, the massive dark gray Cr-rich phase increases greatly and is evenly distributed in the alloy. The size and content of black oxide phase (such as region 4 in Fig.2(c1)) increase. The change of microstructures will affect the mechanical properties of MEAs. Studies have shown that the addition of Cr to AlTiCrFeCoNiCu alloy forms Cr-rich BCC phase, which improves the microhardness and compressive strength of the alloy [17].

Fig.2 SEM photographs of NiCoCuCr_x MEAs
a, a1: Cr0; b, b1: Cr0.4; c, c1: Cr1
Table 4 EDS analysis results of CoNiCuCr$_x$ MEAs (at.%)

| Alloys | Region | Co  | Ni  | Cu  | Cr  | O   |
|--------|--------|-----|-----|-----|-----|-----|
| Cr0    | 1      | 34.50 | 34.46 | 31.04 | 0  | 0   |
|        | 2      | 88.56 | 8.15  | 3.29  | 0  | 0   |
|        | 3      | 41.85 | 4.07  | 4.28  | 0  | 49.79 |
| Cr0.4  | 1      | 2.97  | 93.79 | 2.04  | 1.19 | 0   |
|        | 2      | 10.08 | 11.09 | 74.86 | 3.97 | 0   |
|        | 3      | 3.17  | 3.06  | 1.75  | 92.02 | 0   |
|        | 4      | 56.16 | 29.35 | 8.28  | 6.21 | 0   |
| Cr1    | 1      | 21.72 | 61.56 | 7.31  | 9.41 | 0   |
|        | 2      | 5.54  | 6.27  | 86.00 | 2.19 | 0   |
|        | 3      | 2.34  | 2.15  | 1.60  | 93.91 | 0   |
|        | 4      | 1.93  | 1.69  | 0.77  | 27.23 | 68.38 |
|        | 5      | 55.67 | 13.90 | 7.02  | 23.40 | 0   |

3.2 Hardness and compressive performance

Fig.3 shows the hardness change trend of the CoNiCuCr$_x$ MEAs, and Fig.4 shows the compressive stress-strain curves of the MEAs at room temperature. The compressive performance and hardness are listed in Table 5. It can be concluded from Fig.3 that the hardness values of the MEAs increase with the increase of the Cr content, but the error also gradually increases. The maximum hardness of Cr1 alloy is 331 MPa. This shows that the addition of Cr increases the hardness of the alloy, but makes the uniformity of the alloy structure worse. The reason is that when Cr is added, the alloy forms a BCC phase with high hardness and generates a small amount of NiCoCr intermetallic compound. The second phase dispersion strengthening improves the hardness of the alloy. According to Fig.2(b) and (c), it can be seen that the microstructures of the MEAs become nonuniform after Cr is added, which results in the increase of hardness error.

As shown in Fig.4, the stress-strain curves of the MEAs have a relatively obvious yield plateau. With the increase of the atomic percentage of Cr element, the yield stress and the plateau stress of the MEAs gradually increase, while the fracture strain gradually decreases. The highest yield strength among the three CoNiCuCr$_x$ alloys are 511.87 MPa but its plasticity is poorer. The reason is that with the increase of Cr content, the fine precipitated phases playing a second phase strengthening role in the alloy increase. Meanwhile, the addition of Cr makes the alloy form a Cr-rich BCC structure solid solution. Compared with the FCC structure, the BCC structure has less slip systems and is not easy to deform. The increase of BCC phase is conducive to the improvement of the compressive yield strength and compressive strength of the alloy.

Fig.3 Hardness of CoNiCuCr$_x$ MEAs
Fig. 4 Compressive stress-strain curves of CoNiCuCr$_x$ MEAs at room temperature

Table 5 Compressive performance and hardness of CoNiCuCr$_x$ MEAs

| Alloys | Yield strength (MPa) | Compressive strength (MPa) | Fracture strain (%) | Hardness (MPa) |
|--------|----------------------|---------------------------|-------------------|---------------|
| Cr0    | 400.09               | 1177.80                   | 41.89             | 221           |
| Cr0.4  | 447.14               | 1096.76                   | 36.33             | 234           |
| Cr1    | 511.87               | 1142.71                   | 28.64             | 331           |

Fig. 5 shows the compressive fractures of the CoNiCuCr$_x$ MEAs. It can be seen from Fig. 5(a) that dimples and cleavage steps appear in the fracture of Cr0 MEA, which indicates that the alloy has plastic deformation and quasi-cleavage fracture in the compression process. It can be seen from Fig. 5(b) and (c) that there are more cleavage steps in the fracture of Cr0.4 MEA and Cr1 MEA, and they are brittle fracture. This is due to the increase of BCC solid solution and the formation of intermetallic compounds, which makes the alloy brittle.

3.3 Friction and wear performance

Fig. 6 shows the change curves of the friction coefficient of CoNiCuCr$_x$ MEAs with time. It can be seen from the figure that the friction coefficients of Cr0 and Cr0.4 MEAs gradually stabilize after five minutes, and the friction coefficients increase slightly after 15-20 minutes. This indicates that the structures of the two alloys are relatively uniform. However, the friction coefficient of Cr1 MEA increases obviously at 12 min and 22 min, which may be related to the presence of uneven distribution of BCC phase and intermetallic compound in the alloy. This result is consistent with the XRD and SEM results. By calculating, the average friction coefficients of Cr0, Cr0.4 and Cr1 MEAs is 0.952, 0.850 and 0.761 respectively. With the increase of Cr content, the average friction coefficient of the MEAs decreases, which indicates that the addition of Cr reduces the friction coefficients of CoNiCuCr$_x$ MEAs and has the effect of reducing friction and lubricating.

A distance sensor was used to measure the amount of wear. Fig. 7 shows the wear profiles of the CoNiCuCr$_x$ MEAs, and the wear loss and wear rates calculated are listed in Table 6. It can be concluded from Fig. 7 and Table 6 that with the increase of Cr content, the wear rates of the
CoNiCuCr, MEAs gradually decrease. This indicates that the addition of Cr improves the wear resistance of the MEAs and the Cr1 MEA exhibits the best wear resistance with only 0.37mm$^3$ wear (specific wear rate only $4.09 \times 10^{-4}$ mm$^3$/N·m). When the ratio of material hardness ($H_m$) to abrasive hardness ($H_a$) is greater than 0.8 ($\frac{H_m}{H_a} > 0.8$), the wear resistance of the material increases with the increase of hardness [18]. The addition of Cr increases the hardness of the MEAs, so the wear resistance of the material is improved.

![Fig.6 Friction coefficient diagrams of CoNiCuCr MEAs](image)

![Fig.7 Wear profiles of CoNiCuCr MEAs](image)

**Table 6 Results of friction and wear test**

| Alloys | Wear width (mm) | Wear depth (μm) | Wear loss (mm$^3$) | Specific wear rate ($\times 10^{-4}$ mm$^3$/N·m) |
|--------|-----------------|-----------------|-------------------|-----------------------------------------------|
| Cr0    | 2.02            | 165.41          | 2.14              | 11.6                                          |
| Cr0.4  | 1.85            | 147.77          | 1.80              | 9.10                                          |
| Cr1    | 1.16            | 49.97           | 0.37              | 4.09                                          |

The friction and wear morphologies of the CoNiCuCr, MEAs are shown in Fig. 8. It can be seen from Fig.8 (a) that the materials have obvious plastic deformation and spalling. When the alloy does not contain Cr, the hardness of the alloy with mainly FCC structure is only 221 MPa. Therefore, the plastic deformation occurs obviously during the wear process. The spalling results from cold welding during the wear process, which causes the alloy to be pulled and result in the increase of friction coefficient. According to Fig.8 (b), when the Cr content is 0.4, the spalling area reduces and the plastic deformation reduces. This is because the addition of Cr makes the alloy form the BCC solid solution, which increases the hardness of the alloy and improves the wear resistance of the alloy. From Fig.8(a),
(b) and (c), it can be seen that both Cr0 and Cr0.4 are mainly adhesive wear, but as the content of Cr increases, the wear form gradually changes from adhesive wear to abrasive wear, and the main wear form of Cr1 alloy is abrasive wear.

**Fig. 8 Friction and wear surface morphologies of CoNiCuCr\(_x\) MEAs**

(a) \(x=0\); (b) \(x=0.4\); (c) \(x=1\)

4. Conclusions
In this paper, CoNiCuCr\(_x\) MEAs were prepared successfully by powder metallurgy technology, and the influence of different Cr content on the microstructures and properties of the alloys were studied.

The results show that CoNiCuCr\(_x\) MEAs is mainly composed of FCC structure solid solution, and with the addition of Cr, new Cr-based BCC structure solid solution and a small amount of NiCoCr intermetallic compound are formed. The addition of Cr improves the hardness and compressive yield strength of the CoNiCuCr\(_x\) MEAs. CoNiCuCr alloy shows good mechanical properties; the fracture strain of the alloy decreases by about 13%, but its hardness and compressive yield strength have reached the larger value, namely: 331 MPa and 511.87 MPa respectively; at the same time, the addition of Cr also plays the role of reducing friction and improving lubrication, which reduces the friction coefficient and improve the wear resistance of the alloy. CoNiCuCr alloy shows the better wear resistance, and its friction coefficient and wear amount are 0.761 and 4.09×10\(^{-4}\) mm\(^3\)/N⋅m respectively.

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