Tensile Properties and Impact Toughness of AlCo$_{x}$CrFeNi$_{3.1-x}$ (x = 0.4, 1) High-Entropy Alloys

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In this work, AlCo$_{x}$CrFeNi$_{3.1-x}$ (x = 0.4, 1) high-entropy alloys (HEAs) were investigated. The effects of different Co and Ni content (both of which are FCC stabilizers with similar atomic radii) on the microstructure and mechanical properties of the system were studied with the goal of improving the cost-effectiveness of the alloys. AlCo$_{0.4}$CrFeNi$_{2.7}$ and AlCoCrFeNi$_{2.1}$ were prepared via vacuum induction melting. X-ray diffraction and scanning electron microscope results demonstrated that an FCC+B2 structure formed. Additionally, tensile tests at room temperature and impact tests at 77, 200, and 298 K were also carried out. From the results, it could be determined that the tensile properties of the tested samples were similar. The impact toughness of AlCo$_{0.4}$CrFeNi$_{2.7}$ with a V-notch was 21.77 J at 298 K, and this was more than three times larger than that of AlCoCrFeNi$_{2.1}$. Compared with the Al$_{0.3}$CoCrFeNi and Al$_{0.1}$CoCrFeNi FCC structured HEAs, AlCo$_{x}$CrFeNi$_{3.1-x}$ alloys showed low impact toughness but had a high yield strength.

Keywords: high-entropy alloys, eutectic, microstructure, tensile properties, impact toughness

INTRODUCTION

The development of materials tends to increase the complexity of their constituent components and increases entropy. High-entropy alloys (HEAs) are a type of advanced material that were first reported in 2004 (Yeh et al., 2004). Conventional materials are generally based on one or two elements, with further elemental doping being used to tune their properties. HEAs overcome the limitations of their constituent components and generally refer to a class of alloys that contain >5 elements (between 5–35 at.%) that tend to form solid solution phases (Yeh et al., 2004). For example, this class of materials includes Al$_{0.3}$CoCrFeNi alloys with a face-centered-cubic (FCC) structure (Li et al., 2017), AlCoCrFeNi alloys with a body-centered-cubic (BCC) structure (Manzoni et al., 2013), and GdHoLaTbY alloys with a hexagonal-close-packing (HCP) structure (Zhao et al., 2016).

Over the last few years, the HEA family has gradually expanded and second-generation HEAs have now been developed. These materials contain >4 main elements and form two-phase or multi-phase structures (Zhang W. et al., 2018), such as AlCoCrFeNi$_{2.1}$ eutectic HEAs (Lu et al., 2014) and Fe$_{50}$Mn$_{30}$Cr$_{10}$Co$_{10}$ TRIP HEAs (Li et al., 2016). Research in the HEAs field is not limited to three-dimensional bulk alloys, but also extends to thin-films (two-dimensional) and fibers (one-dimensional) (Zhang, 2019). Because of the solid solution strengthening effect, HEAs exhibit excellent properties that surpass other conventional alloys, such as high hardness and strength, excellent corrosion resistance, good wear resistance, and excellent high temperature resistance (Zhang, 2019).
Casting is an important and widely used technique for almost all engineering materials. Because of the compositional complexity of HEAs, as-cast HEAs with excellent properties can be obtained via tuning of the composition. For instance, Liu et al. (2018) added C to CrFeCoNi HEAs to obtain refined grains. Lv et al. (2019) used Fe\(_{50}\)C\(_{20}\)Cr\(_{10}\)Fe\(_{10}\) to obtain refined grains. For instance, Liu et al. (2018) added C to CrFeCoNi HEAs to obtain refined grains. Lv et al. (2019) used Fe\(_{50}\)C\(_{20}\)Cr\(_{10}\)Fe\(_{10}\) to obtain refined grains. For instance, Liu et al. (2018) added C to CrFeCoNi HEAs to obtain refined grains. Lv et al. (2019) used Fe\(_{50}\)C\(_{20}\)Cr\(_{10}\)Fe\(_{10}\) to obtain refined grains.

The tensile stress-strain curves of these two alloys at room temperature are shown in the Figure 4. There was no obvious yielding stage in the tensile curve, so \(\delta_{0.2}\) was chosen as the yield strength. The tensile properties of the Ni2.7 and Ni2.1 alloys are similar to each other at room temperature. The yield strength

**RESULTS**

**Microstructure**

Macroscopic, OM, and SEM images of the Ni2.7 and Ni2.1 alloys are shown in the Figure 2. Figure 2A shows the macroscopic picture of bulk Ni2.1 and Ni2.7 ingots. Figures 2B, C are OM images of Ni2.7 and Ni2.1, respectively. It can be clearly seen that the Ni2.1 alloy forms a uniform lamella structure that is consistent with the characteristics of eutectic alloys. The Ni2.7 alloy has many large primary phases (white part in the Figure 2B). Figures 2D, E show SEM images of the two alloys. The coupled two phase growth displays a clear compositional contrast, with the regions marked as A (A\(_1\) and A\(_2\)) in Figures 2D, E being rich in Co, Cr, and Fe, while the regions marked as B (B\(_1\) and B\(_2\)) were rich in Ni (detailed compositional information given in Table 1). In the Ni2.7 alloy, large A\(_1\) regions can be observed, and the other regions were filled by an A\(_1/\)B\(_1\) eutectic structure. In the Ni2.1 alloy, the formation of uniform and fine A\(_1/\)B\(_2\) lamella could be observed.

Figure 3 shows XRD patterns of the alloys. It can be seen that both FCC and B2 structures exist inside the two alloys. Therefore, a change in the ratio of Co and Ni did not have much influence on phase formation in this system.

**Experimental**

AlCoCrFeNi\(_{2.1}\) and AlCoCrFeNi\(_{2.7}\) were made via vacuum induction melting, with high purity elements (Al, Co, Ni: 99.9 wt.%; Cr, Fe: 99.5–99.6 wt.%). All the raw materials were placed in a ZrO\(_2\) crucible, heated to 600°C, and held for 1 h to remove water vapor. The pouring temperature was set at 1500°C and the temperature was monitored with an absolute accuracy of ±2°C using an IRTM-2CK infrared pyrometer. Approximately 2.5 kg of metal was melted, superheated, and poured into a high-purity graphite crucible with a length of 220 mm, an upper inner diameter of 62 mm and a bottom inner diameter of 50 mm. In all cases, the furnace chamber was first evacuated to 6 × 10\(^{-2}\) Pa and then backfilled with high-purity argon gas to reach 0.06 MPa.

The crystal structure was identified via X-ray diffraction (XRD) (D8-ADVANCE, Karlsruhe, Germany) with Cu Kα irradiation (40 kV, 300 mA) at a scanning speed of 2°/min. The scanning range was set between 20–100°. For microstructural characterization, the ingots were sectioned, ground, polished, and etched with marble solution (CuSO\(_4\)·5H\(_2\)O = 1:5:3). Then, they were observed using an optical microscope (OM) (Axio imager A2m, Jena, Germany) and a field-emission scanning electron microscope (SEM) (ZEISS SUPRA 55, Jena, Germany), equipped with an X-ray energy spectrometer (EDS) (ZEISS SUPRA 55, Jena, Germany).

The mechanical properties were studied in terms of the tensile and impact properties. Tensile testing was carried out on a CMT Model 4305 Universal Electronic Tester at room temperature with a strain rate of 0.5 mm/s. A non-standard sample size was used, as shown in Figure 1A. The impact tests were conducted on an ASTM standard E-23 setup (size: 10 mm × 10 mm × 55 mm; with a 2 mm deep, V-notch or U-notch at the center, as shown in Figure 1B) at T = 298, 200, and 77 K on a Tinus Olsen impact tester with 450 J impact energy.
was \( \sim 540 \) MPa, while the fracture strength and elongation were 941.1 MPa, 8.5\%, and 1068.8 MPa, 8.2\%, respectively.

The fracture surface morphology of the Ni2.1 and Ni2.7 alloys are shown in Figure 5. Both the fracture surfaces mainly exhibited a trench-like microstructure, which was caused by the FCC/B2 eutectic structure. The bottom of the trench was smooth, and the edge was a bright white line composed of dimples. Figures 5A, B were nearly similar. There were more bright lines for the Ni2.7 alloy than for the Ni2.1 alloy, which is consistent with the increased elongation of the Ni2.7 alloy.

Impact Properties

Figure 6 shows the impact energy of these two alloys with V- and U-notches at 298, 200, and 77 K. The impact energies reduced with decreasing temperature for both the U-notch and V-notch samples. Additionally, all the samples completely broke into two pieces, suggesting little resistance against crack propagation.

For the same alloy, the impact energy of the U-notch samples was slightly higher than for the V-notch samples. The V-notch was sharp and stress concentrated at the center, which made plastic deformation difficult and most of the impact energy was spent in crack propagation. However, the impact energy in the U-notch samples was primarily expended on crack formation. This resulted in the impact energy of the V-notch samples being lower.

For the different alloys, the impact energy of the Ni2.7 alloy was significantly higher than that of the Ni2.1 alloy. Since the V-notch sample is used more widely, it was the subject of the subsequent analyses unless otherwise stated. For the Ni2.7 alloy, the impact energy at room temperature (298 K) was 21.77 J. When the temperature declined to 77 K, the impact energy also reduced to 8.14 J. The impact energy of the Ni2.1 alloy did not exceed 10 J and was \( \sim 3.76 \) J at 77 K, and it finally reached 6.33 J at room temperature. The microscopic images of the impact fractures display similar features as the tensile fractures, i.e., a trench-like microstructure. The difference being that more white lines were present in the Ni2.1 alloy and it clearly showed cleavage fracture.

DISCUSSION

Phase Formation

Based on the various parameters that influence the phase formation of HEAs, several thermodynamic parameters have been proposed from experimental results and theoretical calculations. Solid solutions can be formed if these parameters are distributed in a certain range. Among these parameters, the atomic size difference (\( \delta \)), mixing enthalpy (\( \Delta H_{\text{mix}} \)), and mixing entropy (\( \Delta S_{\text{mix}} \)) are the most widely used and they can be calculated as follows (Guo and Liu, 2011; Yang and Zhang, 2012; Zhang et al., 2014):

\[
\delta = \sqrt{\bar{r} \left(1 - \frac{r_i}{\bar{r}}\right)^2},
\]

\[
\Delta H_{\text{mix}} = \sum_{i \neq j} 4c_i c_j \Delta H_{ij}^{\text{mix}},
\]

\[
\Delta S_{\text{mix}} = -R \sum c_i \ln c_i,
\]

\[
\Omega = \frac{T_m \Delta S_{\text{mix}}}{|\Delta H_{\text{mix}}|},
\]

where \( \delta \) is the average atomic radius of each constituent element, \( r_i \) is the atomic radius of the \( i \)-th element, \( \Delta H_{ij}^{\text{mix}} \) is the mixed enthalpy of the \( i \)-th and \( j \)-th elements, \( R \) is the gas constant (\( R = 8.314 \) J·mol\(^{-1}\)·K\(^{-1}\)), and \( T_m \) is the melting temperature of the alloy.

Table 2 shows some of the thermodynamic factors of the Ni2.7 and Ni2.1 alloys. Because of the similar atomic radii of Co and Ni, adjusting the ratio of Co and Ni would not affect the \( \delta \) of the system when the other element contents were kept constant. Compared with the Ni2.1 alloy, there was more Ni but less Co in the Ni2.7 alloy, which led to a smaller \( \Delta S_{\text{mix}} \). As shown in Figure 7, with the increasing entropy value, the properties of the alloy first increased and then decreased, while the trend in the cost was different (Zhang Y. et al., 2018). Hence, the cost-effective alloys are
most likely to appear within an entropy interval of 0.69–1.61 \( R \). The \( \Delta S_{\text{mix}} \) of the Ni2.7 and Ni2.1 alloys are plotted in Figure 7. It can be seen that both the \( \Delta S_{\text{mix}} \) values are within this interval, so these two alloys could be highly cost-effective.

Guo and Liu (2011) provided a range for forming solid solutions: \( -22 < \Delta H_{\text{mix}} < 7 \text{ kJ/mol}, \delta < 8.5\%, \) \( 11 < \Delta S_{\text{mix}} < 19.5 \text{ J/(K mol)} \). In addition, Zhang et al. (2014) proposed that the transition zone between the mixed ordered solid solution and disordered solid solution is: \( -20 < \Delta H_{\text{mix}} < 0 \text{ kJ/mol and } 5\% < \delta < 6.6\% \). The \( \Delta H_{\text{mix}}, \delta, \) and \( \Delta S_{\text{mix}} \) of the Ni2.7 and Ni2.1 alloys meet these requirements and form a solid solution of the FCC+B2 phase, as observed experimentally.

A new parameter \( \Omega \) (Yang and Zhang, 2012) has been proposed to compare the effects of entropy and enthalpy. \( \Omega \geq 1.1 \) and \( \delta \leq 6.6\% \) are considered as the requirements for forming a solid-solution phase. It is clear that the \( \Omega \) parameters of both alloys are above 1.1.

### TABLE 1 | Chemical composition of the AlCo\(_x\)CrFeNi\(_{3-1-x}\) alloys (at. %).

| Alloys  | Composition | Al  | Cr  | Fe  | Co  | Ni  |
|---------|-------------|-----|-----|-----|-----|-----|
| Ni2.7   | Nominal     | 16.39 | 16.39 | 16.39 | 6.56 | 44.26 |
|         | Actual      | 15.69 | 17.02 | 16.93 | 6.41 | 43.94 |
|         | A1          | 12.72 | 17.62 | 19.50 | 8.22 | 41.95 |
|         | B1          | 27.75 | 9.86  | 9.30  | 4.23 | 48.86 |
| Ni2.1   | Nominal     | 16.39 | 16.39 | 16.39 | 16.39 | 34.43 |
|         | Actual      | 15.09 | 17.14 | 17.03 | 15.94 | 34.81 |
|         | A2          | 10.16 | 19.94 | 19.70 | 18.02 | 32.17 |
|         | B2          | 25.51 | 10.35 | 11.51 | 12.63 | 40.01 |

FIGURE 2 | Images of the AlCo\(_x\)CrFeNi\(_{3-1-x}\) alloys obtained using different methods. (A) Macroscopic picture of the bulk ingot; (B) OM image of the Ni2.7 alloy; (C) OM image of the Ni2.1 alloy; (D) SEM image of the Ni2.7 alloy, and (E) SEM image of the Ni2.1 alloy.
Both Co and Ni can promote the formation of the FCC phase (Zhu et al., 2017; Cao et al., 2019). In this work, with decreasing Co and increasing Ni, there was increased FCC phase formation in the Ni2.7 than Ni2.1 alloys. The reason for this could be that the FCC phase formation ability of Ni is stronger than that of Co. Ke et al. (2006) reported that for FCC stabilizers, 1.11 Co is equivalent to 1 Ni, and for BCC stabilizers, 2.23 Cr has the same effect as 1 Al. Therefore, the percentage of Co-FCC in Ni2.7 was ~55.69%, which is slightly larger than in Ni2.1 (55.18%); hence, Ni2.7 has more FCC phase.

**Mechanical Properties**

According to the experimental results, the yield strengths of the Ni2.7 and Ni2.1 alloys are almost the same. However, the different FCC and B2 phase contents lead to varying fracture strengths and elongations. To be specific, Ni2.7, which contained more of the
FIGURE 5 | Fracture surface morphology of (A) AlCo$_{0.4}$CrFeNi$_{2.7}$ and (B) AlCoCrFeNi$_{2.1}$ at room temperature.

FIGURE 6 | The impact energy as a function of different test temperatures.

| Table 2 | $\delta$, $\Delta H_{\text{mix}}$, $\Delta S_{\text{mix}}$, and $\Omega$ of the AlCo$_x$CrFeNi$_{3-1-x}$ alloys. |
|---------|--------------------------------------------------|
| Alloys  | $\delta$ (%) | $\Delta H_{\text{mix}}$ (kJ mol$^{-1}$) | $\Delta S_{\text{mix}}$ (J mol$^{-1}$ K$^{-1}$) | $\Omega$ |
| AlCo$_{0.4}$CrFeNi$_{2.7}$ | 5.17 | -12.39 | 11.89 | 1.62 |
| AlCoCrFeNi$_{2.1}$ | 5.17 | -11.94 | 12.89 | 1.83 |

FCC phase, showed increased elongation. Similarly, the higher fracture strength of the Ni2.1 alloy was caused by the richer B2 phase inside it.

The presence of the FCC/B2 eutectic structure in the alloys can explain the formation mechanism of the trench-like microstructures (Lu et al., 2017). The B2 phase is hard to deform during the tensile process, while the soft FCC phase could be stretched easily. Then, the FCC phase is necked and gradually thins to form dimples, which gather to form bright lines. The B2 phase is then left at the bottom of the trench and is almost undeformed. From Figure 5, it can be seen that the Ni2.7 alloy with more lines composed of dimples has better plasticity.

The impact performance of the two alloys also shows a clear difference. Similar to the tensile test results, the Ni2.7 alloy with the higher FCC phase content has better impact performance. At low temperatures and high strain rates, the difference in the impact toughness between the two alloys is more obvious. The FCC phase content in the Ni2.7 alloy is larger, which may lead to its good impact performance at low temperatures and high strain rates. The results of impact studies on the HEAs are summarized and plotted in Figure 8A. The impact properties of the HEAs were relatively dispersed. The impact energy of...
$\text{Al}_{0.1}\text{CoCrFeNi}$, $\text{Al}_{0.3}\text{CoCrFeNi}$, $\text{CoCrFeNi}$, and $\text{CoCrFeMnNi}$ with FCC structures were very high and above 150 J. In addition, phase transformation occurred in $\text{CoCrFeNi}$ during the impact process, so it also has an impact energy of $\sim 100$ J. However, the impact energy for the other samples were very low at $< 10$ J. The impact energy values for the Ni2.7 and the Ni2.1 alloys are placed in the mid-region of the data obtained in this work. As the temperature decreased, the impact energy did not change significantly, and the ductile to brittle transition was not observed between 77–298 K.

The relationship between the yield strength and impact energy is shown in Figure 8B. The impact energies of $\text{Al}_{0.1}\text{CoCrFeNi}$ and $\text{Al}_{0.3}\text{CoCrFeNi}$ exceeded those of most reported metallic materials, while their yield strength was a little lower. In this work, the impact performance of the $\text{AlCo}_x\text{CrFeNi}_{3.1-x}$ alloys was poor, but it was still better than the AZ91C alloy, Be, Cr, and some carbon steels. In particular, the impact energy of the Ni2.7 alloy was almost equivalent to that of the alloy steel. The yield strength of the $\text{AlCo}_x\text{CrFeNi}_{3.1-x}$ alloys was higher than most materials (such as low alloy steel, X70 steel, and X80 steel), as shown in Figure 8B.

CONCLUSION

In summary, this work studied the changes in the microstructure and properties of the $\text{AlCo}_x\text{CrFeNi}_{3.1-x}$ ($x = 0.4, 1$) alloy on varying the Co and Ni content with the goal of preparing cost-effective alloys. Compared with the perfect eutectic FCC/B2 structure of the $\text{AlCoCrFeNi}_{2.1}$ alloy, the $\text{AlCo}_{0.4}\text{CrFeNi}_{2.7}$ alloy had the structural characteristics of the FCC primary phase and the FCC/B2 eutectic structure. The tensile properties of two alloys were similar, with the FCC phase forming a sharp bright white line when stretched, while the B2 phase was left as a trench with minimal deformation. In terms of the impact performance, the $\text{AlCo}_{0.4}\text{CrFeNi}_{2.7}$ alloy was much better than the $\text{AlCoCrFeNi}_{2.1}$. Additionally, the two alloys did not display characteristics of a ductile-brittle transition within the tested temperature range. Compared with other HEAs, the impact toughness values of the $\text{AlCo}_x\text{CrFeNi}_{3.1-x}$ alloys were in the middle of the range, and they were far below those of the $\text{Al}_{0.3}\text{CoCrFeNi}$ and $\text{Al}_{0.1}\text{CoCrFeNi}$ alloys. Finally, the yield strength of the $\text{AlCo}_x\text{CrFeNi}_{3.1-x}$ alloys were higher than most of the compared materials.
DATA AVAILABILITY STATEMENT

All datasets generated for this study are included in the article/supplementary material.

AUTHOR CONTRIBUTIONS

LZ prepared the high-entropy alloys and wrote the manuscript. YZ offered theoretical guidance. Both authors contributed to the general discussion.

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Acknowledgments

The authors are grateful for support by Professor Lu of Dalian University of Technology for providing the high-entropy alloys.

FUNDING

YZ would like to thank the National Natural Science Foundation of China (Grant Nos. 51671020 and 51471025), and the Fundamental Research Funds for the Central Universities (Grant Nos. FRF-MP-19-013 and FEF-MP-18-003) for financial support.

Conflict of Interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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