Precipitation behavior of yttrium-rich nano-phases in AlCoCrFeNi2.1Yx high-entropy alloy

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Abstract
In this paper, rare earth element yttrium (Y) was selected to be doped into AlCoCrFeNi2.1Yx (x = 0, 0.1%, 0.3%, 0.5%, and 1.0%) high-entropy alloy in order to refine grain and increase yielding strength. The precipitation behavior of the Y-rich nano-phases in the face centered cubic (FCC (L12)) and the body centered cubic (BCC (B2)) phase was investigated by x-ray diffraction, scanning electron microscopy, high-resolution transmission electron microscopy and differential scanning calorimetry. Refined crystal grains were observed due to the high-density precipitation. The nano-phase within the BCC(B2) phase was (Al-Ni-Y)-rich phase and single cubic (SC) structure. The nano-phase formed within the FCC (L12) phase was (Fe-Co-Cr-Y)-rich phase and FCC structure. Fine (Al-Ni-Y)-rich nano-particles were formed due to the addition of Y and the amount of the nano-phase increased with increasing Y content. The adoption of Y promoted dispersed precipitation of (Al-Ni-Y)-rich nano-phase under deformation. The more Y content, the more nano-precipitates. When Y = 1.0 at.%, the lamellar structure was transformed into bamboo-like structure in the BCC phase due to the segregation of Y. Double yielding phenomenon occurred during the compression deformation of the AlCoCrFeNi2.1Yx alloys (when x ≥ 0.5 at.%) and caused an increase of yielding strength by 40%. It was since the barrier effect of both the (Al-Ni-Y)-rich nano-precipitation and the (Fe-Co-Cr-Y)-rich nano-phases on the dislocations within grains and grain boundaries led to the second yielding. With the further increase of Y content, the lamellar BCC phase was separated from the bamboo-like BCC phase owing to the super-saturated precipitation and segregation of Y at the solidification front of B2 phase, leading to a degradation of mechanical properties.

1. Introduction

Rare earth (RE) elements are a type of highly active metals, which were discovered in 1794 and have been used in metallurgy industries since the 1970s. They have good oxygen-accumulation and deoxidization effects, as well as microalloying and grain refinement characteristics [1, 2]. Apart from being commonly applied in metallic materials, such as steels, casting iron, and nonferrous alloys, RE elements have also been used in the functional materials, such as permanent magnetic, luminescent, and hydrogen storage materials, and have promoted the development of relevant industries and scientific research [3–5]. RE elements were widely used to tailor the microstructures and improve the mechanical properties of steels due to their positive effects on molten steel purification and modification of inclusions [6–8].

High-entropy alloys (HEAs) exhibit unique combinations of properties that include high strength, excellent wear resistance, good thermal stability, and excellent radiation resistance [9–11], etc, which have broad application prospects in aeroengines, armor materials, heat-resistant [12, 13] and wear-resistant molds, magnetic materials [14, 15], and other fields [16]. Lu et al [17] developed a eutectic high entropy alloy, which possessed the characteristics of high strength, high toughness, and good cast performance without obvious shrinkage defects in 2.5 kg ingot [18], provided a new way for industrial application of HEAs. reddy et al [19]
reported the deformation of AlCoCrFeNi2.1Y alloy by a novel hybrid-rolling process. The study demonstrated that dual structure-composition heterogeneities can be a novel microstructural design strategy for achieving outstanding strength-ductility combination in multiphase high entropy alloys Shi et al [20] have provided a pathway for strengthening eutectic alloys and widened the design toolbox for high performance materials based upon EHEAs. As the characteristics of micro-alloying, grain refinement and etc, rare earth elements have been used in HEAs. Several studies have reported the effect of rare earth elements on the phases and mechanical properties of HEAs. Yuan et al found evidence that RE-HEAs had great potential to be used as magnetic refrigerants [21]. Hu et al have prepared a series of AlCoCrCuNiTiY HEAs and revealed that the maximum compressive stress and strain of the HEAs were 1495 MPa and 7.68%, respectively [22]. Kong et al investigated the effect of RE elements on the Laves phase related BCC solid solution metal hydride alloys, and found that Y increased the reversibility of gaseous phase hydrogen storage [23]. Zhang et al suggested that principal elements with hexagonal close-packed (hcp) structure and suitable atomic size were beneficial to the formation of hcp dendrites, and the interdendritic region containing CuY-type phase was corroded preferentially [24].

2. Experimental procedure

High purity (>99.5 wt% purity) metals of Al, Co, Cr, Fe, Ni and Y were used to fabricate AlCoCrFeNi2.1Yx (x = 0, 0.1%, 0.3%, 0.5% and 1.0%) samples by arc-melting. The surfaces of the pure metals were cleaned with a wire brush and ethanol before being imported into the arc furnace. The alloys were remelted three to four times in a Ti-gettered argon atmosphere in order to ensure compositional homogeneity. For the tensile test, compression test, x-ray diffraction (XRD) and differential scanning calorimetry (DSC), rod samples with 5 mm in diameter and 60 mm in length, were prepared by non-consumable arc-melting furnace. The structure of the obtained alloys was examined by XRD with Cu Kα radiation. The diffraction angle was in the range between 10° and 90° and the step length was 0.065. The High Score Plus phase analysis software was used to analyze the diffraction patterns. The thermal stability was investigated by DSC (SETSYS -1750 CS Evol) at a heating rate of 30 K min−1 and a thermal peak of 1623K under argon atmosphere. Tensile and compression tests were conducted using an Instron5582 testing device at a constant strain rate of 0.001 mm s−1. At least three samples of each rod were tested to ensure reliability. The samples were eroded in aqua regia for 5–10 s, and the morphology and composition of the samples were analyzed by SEM (HITACHS-3400N) with energy dispersive x-ray spectroscopy (EDS). The samples were thinned manually and then two jets thinning with 5% perchloric acid under 55 mA cm−2 and 60 V, and the morphology and composition of the samples were analyzed by TEM (TEM: JEM-2100 and JOEL 2100F).

3. Results and discussion

3.1. AlCoCrFeNi2.1Yx alloys characterization

SEM images of the AlCoCrFeNi2.1Yx (x = 0, 0.1%, 0.3%, 0.5% and 1.0%) alloys with different Y content were shown in figures 1(a)–(d), respectively. For Y = 0, the morphology of the alloy was composed of dendrite and interdendrite. The dendrite was coarse, while necking was observed in the secondary dendrite. The dendrite was (Fe-Co-Cr)-rich primary phase and the liquid metal surrounded the primary dendrite nucleus, forming a (Al-Ni)-rich phase of the eutectic structure (figure 1(a)). It is noting that a fine structure with a flower-like helical morphology was formed when Y was 0.1%. With the increase of Y content, (Fe-Co-Cr)-rich phase and (Al-Ni)-rich phase of the eutectic structure grew up dependently, as shown in figure 1(b). For Y = 0.5%, 1.0% (figures 1(c) and (d), respectively), the morphology changed from a eutectic to a banded structure, and then to a dendrite structure. Meanwhile, the dendrite became coarse and the inter-dendrite became nodular, and the content of the (Al-Ni)-rich phase decreased. The morphology is related to the temperature gradient and growth rate. Dendrite structures formed when the temperature of the dendrite tip was higher than that of the eutectic alloy. The dendrite eutectic interface of (Al-Ni)-rich phase and (Fe-Co-Cr)-rich phase was in the low super cooling state of the eutectic couple zone. When the Y content was 1.0%, it resulted in a necking phenomenon at the second dendrite root as shown in figure 1(d), which indicated that Y enriched the solidification front of the eutectic structure and inhibited the growth of the inter-dendrite phase. Table 1 showed the EDS results of the
AlCoCrFeNi$_{2.1}$Y$_{1.0\%}$ alloy. It could be seen that the microstructure of AlCoCrFeNi$_{2.1}$Y$_{1.0\%}$ was eutectic structure composed of (Al-Ni)-rich phase and (Fe-Co-Cr)-rich phase. A new type of (Al-Ni-Y)-rich phase (marked as C) formed with the increase of Y content. The content of Y in the (Al-Ni)-rich (marked with B) phase was higher than that in the (Fe-Co-Cr)-rich (marked with A) phase.

Figure 1(e) displayed the XRD patterns of the AlCoCrFeNi$_{2.1}$Y$_{x}$ (x = 0, 0.1%, 0.5%, and 1.0%) alloys. The alloys were mainly composed of BCC (B2) and FCC (L1$_2$) solid solutions. In all samples, Bragg peaks of FCC (FeCoNi-M) and BCC (AlNi-M) phase could be observed. The intensity of the FCC (L1$_2$) peaks in the (1 1 1) plane decreased with the increase of Y content, which suggested a change in the volume fraction or in the crystal

### Table 1. EDS results of the AlCoCrFeNi$_{2.1}$Y$_{1.0\%}$ alloy (at.%).

|   | Ni   | Fe   | Co   | Cr   | Al   | Y   |
|---|------|------|------|------|------|-----|
| A | 30.03| 20.22| 19.44| 19.00| 11.32| —   |
| B | 44.95| 12.98| 14.06| 11.58| 14.37| 2.05|
| C | 32.90| 16.68| 16.80| 17.16| 14.79| 1.68|

Figure 1. Microstructures of the AlCoCrFeNi$_{2.1}$Y$_{x}$ for (a) Y$_{0.0\%}$, (b) Y$_{0.1\%}$, (c) Y$_{0.5\%}$, and (d) Y$_{1.0\%}$; (e) XRD results; (f) DSC curves.
When Y ≥ 0.5%, the diffraction peaks at lower angle regions became weaker, probably indicating that Y atoms in the lattice skeleton caused orientation effect. When the Y content was 1.0%, the diffraction peak of the FCC phase in the (111) plane was weak and the peak in the (200) plane was strong.

The AlCoCrFeNi$_2.1Y_x$ ($x=0, 0.1\%, 0.5\%$, and $1.0\%$) samples were measured with DSC at a heating rate of 30 K min$^{-1}$ and a maximum heating temperature of 1350 $^\circ$C, as shown in figure 1(f). When Y ≥ 0.1%, an endothermic peak occurred near 1210 $^\circ$C, indicating the formation of a new phase. The curves showed an obvious endothermic peak near 1210 $^\circ$C indicating the increase of the new phase when Y ≥ 0.5%. The mixing enthalpies Y-Al, and Y-Ni were calculated based on the mixing enthalpy equation (equation (1))\[26\], and were determined to be −38 kJ mol$^{-1}$ and −31 kJ mol$^{-1}$, respectively. A typical compound was formed by two or more elements of Y, Al and Ni for a high negative mixing enthalpy.

$$\Delta H_{\text{mix}} = \sum_{i=1}^{n} 4\Delta H_{ij}^\text{mix} c_i c_j$$

3.2. Precipitation of Y-rich phases

Figure 2 showed the TEM images and selected area electron diffraction (SAED) patterns of the as-cast AlCoCrFeNi$_2.1Y_x$ ($x=0, 0.1\%, 0.5\%$, and $1.0\%$) HEAs. The microstructure was composed of FCC (L1$_2$) (Fe-Co-Cr)-rich phase (grey region), BCC (B2) (Ni-Al)-rich phase (black dendrites), and precipitates with several nanometers in size appeared in the FCC (L1$_2$) phase (figure 2(a)), which was similar with the results reported by Lu et al\[27\]. Figure 2(b) showed a TEM image of AlCoCrFeNi$_2.1Y_{0.5\%}$. As could be seen in figure 2(b), a number of Y-rich nano phase appeared in the FCC (L1$_2$) phase marked by black arrows. The SAED patterns of the FCC phase (marked as A) and the BCC phase (marked as B) along [011] zone axis were presented in figures 2(a) and (b), respectively. The EDS results of the AlCoCrFeNi$_2.1Y_x$ ($x=0, 0.5\%$) alloys for the FCC and the BCC phases were shown in table 2, which indicated that the microstructure of AlCoCrFeNi$_2.1Y_{0.5\%}$ was a eutectic structure composed of (Al-Ni)-rich B2 phase and (Fe-Co-Cr)-rich FCC (L1$_2$) phase. The solubility of Y into the BCC phase was higher than that of the FCC phase, and the Y content in the BCC phase was 1.0% in AlCoCrFeNi$_2.1Y_{0.5\%}$.

Table 2. EDS results of the alloys with 0.0 and 0.5% Y content (at.%).

|       | Ni  | Fe  | Co  | Cr  | Al  | Y   |
|-------|-----|-----|-----|-----|-----|-----|
| $Y_0$ | A 33.4 | 18.9 | 17.1 | 20.6 | 10.0 | —   |
|       | B 42.3 | 11.2 | 13.3 | 7.6  | 25.7 | —   |
| $Y_{0.5\%}$ | A 32.1 | 18.5 | 19.7 | 19.9 | 9.7  | 0.1 |
|       | B 38.9 | 11.1 | 12.7 | 11.9 | 24.4 | 1.00|

Figure 3 illustrated the TEM images and SAED of AlCoCrFeNi$_2.1Y_{1.0\%}$ before and after compressive. The growth of B2 phase was changed and the lamellar structure changed to be bamboo-like structures. Both the amount and size of nanoparticles increased when Y content increased to 1.0%. The HRTEM of the bamboo-like structure of the B2 phase (marked by the bright yellow circle) was analyzed in figure 3(a). The new phase was a BCC structure (zone axis: [0 1 2]) and formed due to super saturation precipitation and segregation of Y at the solidification front of the B2 phase. A lot of dislocations piled up at grain boundary as well as within grains.
deformation ability of the new BCC phase in bamboo joint was different from that of the B2 phase as shown in figure 3(b). The lamellar B2 phase separated from the bamboo-like BCC phase due to the segregation of Y, which might be the key factor for the decrease of mechanical properties of the alloy with the further increase of Y content.

3.3. Double yielding phenomenon

Figure 4 illustrated the results of compressive test of AlCoCrFeNi$_2.1$Y$_x$ specimens for as-cast specimens of Y$_x$ ($x = 0, 0.3\%, 0.5\%, 1.0\%$). The compression strength and strain first increased and then decreased with increasing of Y content as shown in figure 4(a). The yield strength increased with the increase of Y content under the same strain. Double yielding behavior could be observed when Y = 0.5\% and Y = 1.0\% (figure 4(b)). The first and second yielding of Y$_{0.5\%}$ were marked and analyzed in figure 4(c). The true strain (Ln (1 + $\varepsilon$)) and the true stress ($\sigma$ (1 + $\varepsilon$)) were calculated based on engineering strain ($\varepsilon$) and engineering stress ($\sigma$). The working hardening rate of Y$_{0.5\%}$ was the derivative of the
true stress. The curve was shown in figure 4(d). It included three stages: (1) Stage I: the working hardening rate was increased with a high speed after elastically deformed. Beyond the elastic limit of the alloy (2.2 ± 0.2%), the first yielding occurred at 600 ± 20 MPa (marked in figure 4(c)). A slight increase in the elastic limit of the alloy could be observed due to the presence of the Y element; (2) Stage II: after the first yielding, the slope of the stress–strain curve in the strain range between 2.0 ± 0.2 and 5.0 ± 0.3%, deviated markedly from that of Stage I. The working hardening rate was grown at low speed. The second yielding occurred at 850 ± 20 MPa (marked in figure 4(c)), which caused an increase in yielding strength by 40%. (3) Stage III, the growth of working hardening rate was small and the curve was instability gradually.

3.4. Interaction of nano-precipitates and dislocations

Figure 5 and 6 show the TEM images and SAED patterns of AlCoCrFeNi_{2.1}Y_{0.5%} after compression deformation. After the first yielding, the microstructure was composed of FCC (L12) (marked as A) and BCC (B2) phase (marked as B) as shown in figure 5. Particles with a few nanometers in size, and a large number of nano-precipitates appeared in the BCC phase. A super-lattice was formed in the SAED pattern of the B2 phase. The nano-particles were (Al-Ni-Y)-rich phase and Single Cubic (SC) structure incoherent with the B2 phase as shown in figure 5(b). The SAED pattern of the nano particle along [1 2 4] zone axis was presented in figure 5(c) based on the fast fourier transformation (FFT) result. While the nano-precipitates were Y-rich phase and FCC structure coherent/semicoherent with the FCC (L12) phase. The SAED pattern of the nano-precipitate along [1 1 1] zone axis was shown in figure 5(e). For Y ≥ 0.1%, the nano-precipitates formed which agreed with the DSC results as shown in figure 1(d). B2 is (Al-Ni)-riched phase. The mixing enthalpy of Al-Ni, Y-Al, and Y-Ni was −22 kJ mol⁻¹, −38 kJ mol⁻¹ and −31 kJ mol⁻¹, respectively. The HRTEM images in figure 5 and the EDS results in figure 1(d) demonstrated dispersed precipitations caused by Y. First, the solute effect [28] of Y existed in the form of atom could cause the constituent super cooling at the interface of solid-liquid, which promoted the formation of Y-rich nano-precipitates. Second, the high negative mixing enthalpies of Y-Al and Y-Ni indicated that two or more elements of Y, Al, and Ni could form compounds with low interfacial energy, and Y promoted the formation of dispersed nano-phases under the deformation stress. Dislocations (marked with black arrows in figure 5(a)) distributed near the nano-precipitates. It indicated that there was interaction between nano-precipitates and dislocations.
The TEM image of the specimen after the second yielding, the nano-precipitates and dislocations were presented in Figure 6. The nano-precipitates were incoherent with the B2 phase and dislocations distributed near the nano phase. The number of nano-precipitates and dislocations (marked with black arrows) density increased for the further deformation as shown in Figure 6(a). It indicated that Yttrium further promoted the aggregation of Al-Ni-Y for the large negative enthalpy of mixing and decreased the nucleation energy. Besides, deformation induced precipitation of the (Al-Ni-Y)-rich nano phase. At the same time, serious lattice distortion occurred in the nano particle which was shown in the inverse FFT in Figures 6(b) and (c). It could be deduced that the second yielding phenomenon was mainly caused by the interaction between nano-precipitates and dislocations. It indicated that the precipitation process of the nano phases could be accelerated by compression and the pinning effect of the precipitates on the dislocations could increase the yielding strength. While, the nano precipitate (marked with gray arrows) were formed around the nano particle in the FCC (L12) phase and lattice distortion expanded (marked with black arrows) into the neighboring matrix with the further deformation as shown in Figure 6(d).

4. Conclusions

The microstructures and mechanical properties of the prepared AlCoCrFeNi2.1Yx (x = 0, 0.1%, 0.3%, 0.5%, and 1.0%) alloys were analyzed. The main conclusions could be drawn as follows.

(1) Fine (Al-Ni-Y)-rich and (Fe-Co-Cr-Y)-rich nano-particles were formed due to the addition of Y and the amount of the nano-phase increased with increasing Y content. The adoption of Y promoted dispersed precipitation of (Al-Ni-Y)-rich nano-phase under deformation.

(2) Double yielding phenomenon was presented when the Y content was 0.5% ≤ Y ≤ 1.0%, which was caused by the interaction between nano-precipitates and dislocations.

(3) Y promoted the preferential growth of the FCC (L12) phase and inhibited the growth of the B2 phase. When the Y content was 1.0%, the complete lamellar structure was transformed into bamboo-like structure. During tensile deformation, high density of dislocations was introduced around the bamboo-like joint.
Further increase of Y content led to lamellar separation for the segregation of Y, which was the main factor that led to mechanical property degradation.

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Conflicts of interest

The authors declare no conflict of interest.

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