Annealing hardening behaviour of cold rolled Al$_{0.5}$CoNiCu high-entropy alloy

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Abstract. The usually, softening occurs when metals are annealed, while for some special metals or alloys, the abnormal phenomenon of annealing hardening will occur. By means of X-ray diffraction (XRD), metallographic (OM) observation, transmission electron microscopy (TEM) and hardness test, the microstructure and hardness of 79% cold rolled Al$_{0.5}$CrFeCoNiCu high entropy alloy was studied after isothermal annealing for 1 hour at different temperatures, which revealed the cause of annealing induced hardening of the alloy. The results show that the homogenized high-entropy alloy is a face centered cubic (FCC) biphasic structure with close lattice constant. After annealing at 300-750°C, the microhardness of the alloy is higher than that of the cold rolled alloy (399hv); With the increase of annealing temperature, the hardness increased first and then decreased. After annealing at 500 °C for 1 hour, the hardness reached the peak (489hv). XRD phase analysis shows that the alloy will precipitate body centered cubic (BCC) phase when annealed at 500-950°C and precipitate σ phase when annealed at 700-850 °C; In addition, when annealed at 650°C and above, the dislocation recovery speed in FCC phase is accelerated and recrystallization will occur, resulting in rapid softening of the alloy. TEM observation showed that there were L1$_2$ nano-precipitates with ordered structure in the FCC phase of the matrix. Therefore, the reason for the hardness peak of the alloy annealed at 500°C is the joint action of dislocation strengthening and precipitation strengthening (L1$_2$ and BCC phases).

1. Introduction

High entropy alloy is a new type of alloy, which is composed of many components with equal molar or close to equimolar ratio. It tends to form solid solution phase and inhibit the formation of intermetallic compounds [1-3]. Because of its high strength, high plasticity and excellent wear resistance, high entropy alloy is expected to become the most competitive candidate material in traditional materials. FCC high entropy alloy represented by CrMnFeCoNi has a series of excellent properties, such as high damage tolerance, good radiation resistance, high wear resistance and corrosion resistance; However,
even though FCC high entropy alloy has excellent plasticity, its strength is relatively low, which is difficult to meet the needs of engineering applications [4]. Similar to the traditional industrial alloys, the comprehensive mechanical properties of FCC high entropy alloys can be improved through plastic deformation and subsequent stabilization annealing.

For most deformed metals, annealing leads to softening due to dislocation annihilation caused by dislocation cross slip or climbing during recrystallization or recovery [5]. However, annealing hardening occurs in some metals subjected to large plastic deformation. This anomaly is attributed to three mechanisms in the annealing process below the recrystallization temperature: (1) usually in nanocrystalline metals, annealing reduces the dislocation source, resulting in higher stress required to activate new dislocation sources [6-8], (2) solute segregation or the formation of a second phase that hinders dislocation movement (e.g. Mo segregation in CoCrFeNiMo) [9,10], and (3) annealing induced twinning or martensitic transformation usually occurs in FCC metals with low to medium stacking fault energy (SFE,<40mJ/m2) [11,12]. Studies have shown that CrMnFeCoNi alloy will form a second phase after annealing under the following two conditions: (a) annealing for a very long time [13, 14] and (b) severe plastic deformation before annealing [9, 15]. The precipitates induced by prolonging the annealing time usually precipitate along the grain boundary [13, 14]. In addition, precipitates were also observed in CrMnFeCoNi alloy after severe plastic deformation and annealing [9]. Annealing induced hardening occurs at relatively low temperature, and its hardening behavior is mainly due to the formation of multiphase nanostructures in the high entropy alloy matrix. In addition, abnormal hardening caused by annealing was observed in cold-rolled CrMnFeCoNi alloy after annealing at 400℃. The abnormal hardening phenomenon is due to the formation of long-range ordered structure in FCC matrix. By increasing the annealing time at 400 ℃, the nano grains with sharp grain boundaries also contribute to hardening [16]. The same annealing induced hardening phenomenon is also reported in TiZrNbHfTa single-phase high entropy alloy. Due to the phase decomposition in the initial solid solution state, the second phase is formed after long-time annealing [17]. Due to the changes of dislocation density and grain boundary relaxation, annealing induced hardening is reported in CoCrNi medium entropy alloy without the formation of a second phase [18]. In conclusion, the mechanism of annealing and hardening of metals in different systems is not exactly the same; There are few reports on the annealing behavior of metals with multiphase structure.

At present, the research on the annealing behavior of high entropy alloys mainly focuses on FCC or BCC high entropy alloys with single-phase structure, while the high entropy alloys with multiphase structure have broad application prospects because of their better comprehensive properties [19]. Therefore, the annealing behavior, microstructure evolution and internal mechanism of multiphase high entropy alloys are worth studying. In this paper, the vacuum arc melting of Al0.5CrFeCoNiCu high entropy alloy was homogenized annealing, cold rolling and subsequent annealing at 300-1000℃. The annealing induced hardening behavior of this biphasic FCC high entropy alloy was studied. Combined with TEM microstructure observation and XRD phase analysis, the evolution of crystal defect substructure, precipitation behavior and phase separation mechanism of the alloy during annealing were revealed. This paper can provide a new understanding for the annealing hardening behavior of high entropy alloys.

2. Experiment

The raw materials are high-purity aluminum (99.999%), high-purity iron (99.99%), high-purity chromium (99.99%), high-purity cobalt (99.95%), high-purity nickel (99.99%), high-purity copper (99.99%). According to the proportioning of 0.5:1:1:1:1 molar ratio, 150g samples are melted in vacuum in an electric arc furnace under the protection of high-purity argon and melted repeatedly for 5 times to ensure uniform ingot composition. Then, the as-cast alloy was homogenized and annealed at 1150℃ for 4 hours, and the oil was taken out and cooled to room temperature. After cutting a piece of square sample of 40mm×35mm×7.5mm by wire-cut, use sandpaper to polish the processing trace until the ingot is bright, and then conduct multi pass cold rolling (CR) deformation. The total reduction is 79%, and the final plate thickness is about 1.6mm. Cut the cold-rolled sheet into hardness test blocks of 12mm
Homogenization heat treatment and isothermal annealing treatment are carried out in muffle furnace, and the temperature is controlled at the set temperature±3℃. After taking out, it is quickly water quenched. After mechanical polishing of high entropy alloys in different treatment states, hardness test and microstructure characterization were carried out. The model of automatic microhardness tester is Q10A+, the loading force is 4.9N and the loading time is 30s. XRD phase analysis was tested on D/Max2500 diffractometer with scanning angle 2θ of 30-100° and scanning speed of 1°/min. The data was analyzed by MDI Jade 6.0 software. Mechanically grind and throw the samples after different treatments to 60μm. After that, a circular sheet with a diameter of 3mm was prepared and thinned with Tenupol-5 chemical double jet thinner. The corrosive solution was 10vol% perchloric acid and 90vol% alcohol. The prepared transmission samples were metallographically observed under the laser confocal microscope, and then the microstructure was observed and analyzed by energy dispersive spectroscopy (EDS) using FEI Tecnai F20 transmission electron microscope.

### 3. Results and analysis

#### 3.1. hardness curve

Fig 1 shows the influence of different oil antiwear agents on the engine friction coefficient[5]. Fig. 1 is the microhardness curve of cold rolled high-entropy alloy after isothermal annealing at different temperatures for 1 hour. After cold rolling, the hardness of the alloy is 399HV, and the microhardness increases first and then decreases with the increase of annealing temperature. When the annealing temperature is lower than 500℃, the hardness value gradually increases to the peak value with the increase of annealing temperature, 489HV. When the annealing temperature is higher than 500℃, the hardness decreases gradually. Moreover, when the annealing temperature is higher than 750℃, the alloy softens rapidly (from 408HV to 234HV). At this time, the hardness is lower than that of the cold-rolled alloy. The abnormal increase of hardness of cold rolled high-entropy alloy after isothermal annealing at 750℃.

![Hardness curve](image)

**Fig. 1. Hardness curve of high-entropy alloy after cold rolled and isothermally annealed at 300-1000℃ for 1 hour**

#### 3.2. XRD phase analysis

Fig. 2 shows the XRD patterns and phase identification results of high-entropy alloys in different treatment states. The cold rolled alloy has a single FCC1 structure (lattice constant a=3.598Å); According to literature [20], this kind of high-entropy alloy with high Cu content should be the phase structure of two double FCC phases with similar lattice constants (FCC2, a=3.615Å). According to the analysis of references [20, 21] and TEM results (see 2.4), the alloy is a high-entropy alloy with two-
state FCC phase structure. XRD detected that CR alloy is single-phase FCC1, which is due to the large dislocation density in the alloy after severe cold rolling, resulting in the broadening of the diffraction peak of the alloy, and the diffraction peaks close to the peak position are combined.

The phase structure of the alloy annealed at 300-450°C is consistent with that of CR alloy, indicating that there is no obvious new phase precipitation and dislocation recovery and annihilation. The alloy annealed at 500-600°C is composed of FCC1+body centered cubic (BCC) phase. The lattice constant of this kind of BCC phase is 2.876Å, which is generally a brittle hard phase, and its precipitation can increase the hardness of the alloy. At 650°C/1h, the sample is composed of FCC1+BCC+FCC2 phase. At this temperature, the dislocation recovery is obvious, the diffraction peak becomes narrower, and the widened FCC diffraction peak gradually becomes a double diffraction peak of FCC1 and FCC2 with close peak positions. When annealed at 700-850°C, it has obvious tetragonal structureσPhase (a=8.85Å, C=4.59Å) precipitates, whose melting point is 875°C, and the main precipitation temperature is 700-850°C, which is consistent with our research report [22, 23]. Generally speaking, hardσPhase energy can greatly improve the strength and strain hardening ability [24]. However, there was no phase when annealing temperature is higher than 900-950°C, the phase composition is still FCC1+BCC+FCC2. However, it should be noted that the sample is the initial FCC1+FCC2 structure at 1000°C/h, indicating that there is no precipitate after annealing above 1000°C. The phase composition observed in the sample at 1000°C/1h is very similar to that observed in the original alloy, which indicates that the solidified phase is a thermodynamically stable high-temperature phase, not just the product of micro segregation caused by solidification.

Based on the analysis of the above results, it can be seen that there is obvious precipitation of BCC phase after annealing between 500-950°C; 700-850°C σphase precipitation temperature. The dislocation recovers rapidly after 650°C, and the dislocation density decreases rapidly, showing the original structure of FCC1+FCC2. Therefore, annealing from 300°C to 500°C is due to the decomposition of total dislocations into two incomplete dislocations, and the incomplete dislocations formed react with each other to form L-C dislocation lock, resulting in hardening [25]. However, the author believes that it should also be partly due to the precipitation of nano-sized BCC, which leads to hardening. Although the precipitation of BCC phase is not detected by XRD in this temperature range (300-450°C), it may be due to the small size and small quantity of this phase. Therefore, the peak hardness of 489HV can be obtained at 500°C/1h, which is the joint action of BCC phase precipitation strengthening and dislocation strengthening. The rapid decrease of microhardness at 650°C is due to the decrease of dislocation density, and (partial) recrystallization will occur after annealing at higher temperature, resulting in alloy softening.

Fig. 2 XRD patterns of high-entropy alloys with different annealing treatments
3.3. OM observation

Typical metallographic structures of alloys in different treatment states are shown in Fig. 3. The grains of CR alloy are seriously distorted due to severe cold rolling, and the clear grain boundaries cannot be corroded due to high dislocation density (Fig.3 (a1)), but it can be seen that it is a two-phase structure, and a small amount of FCC2 phase is distributed at the grain boundaries of the dominant phase FCC1 (Fig.3 (a2)). Large stress still exists in the samples annealed at 400℃/1h and 550℃/1h, and the interface is very fuzzy. Only the main FCC1 phase can be observed (Fig.3 (b-c)). No obvious grain growth was observed during low temperature annealing. However, many slip bands can be seen in 650℃/1h and 750℃/1h samples (Fig.3 (d2-e2)), and the interface between FCC1 and FCC2 becomes obvious, but the two-phase ratio does not change significantly. The annealed BCC and σphase cannot be distinguished from the metallographic picture for the time being. It is worth noting that the alloy recrystallized obviously after annealing at 1000℃ (Fig.3 (f2)).

![Metallographic pictures of high-entropy alloys in different processing states](image)

3.4. TEM observation

In order to reveal the microstructure of the alloy more microscopically, the homogenized annealed alloy and 750℃/1h alloy were observed by TEM. Fig. 4 is the TEM microstructure of the alloy after homogenization treatment. Fig. 4 (a) is the dark field image of FCC2 phase, showing the alternating...
distribution of double FCC. Combined with the analysis of the corresponding selected area electron diffraction picture (SAED, fig. 4 (b)), it can be seen that many nanoparticles with L12 structure are distributed in FCC2. Fig. 4 (c) is a representative high angle annular dark field image (HAADF) picture. EDS surface analysis of the two FCC phases shows that FCC1 phase is a phase poor in Cu and rich in Co, Cr, Fe and Ni, while FCC2 phase with fine nano precipitates is a phase rich in Cu, Al and Ni. The authors carried out a lot of TEM characterization and only observed the phase of FCC1 and FCC2 (containing L12 particles).

![Fig. 4 TEM characterization of high-entropy alloy after homogenization](image)

(a) A dark field image of FCC2 and (b) corresponding SEAD pattern, and (c) a representative HAADF image and corresponding EDS mapping.

Figure 5 shows the TEM microstructure of cold rolled high-entropy alloy. 79% cold rolling deformation introduces a large number of dislocations. Due to the existence of internal stress and dislocations, the grain boundaries of the alloy are difficult to be clearly identified. High density dislocation entanglement will strengthen the alloy, so the microhardness of the cold-rolled alloy is as high as 399HV (see Figure 1).

Fig. 6 shows the TEM microstructure of the alloy after treatment at 500°C/1h. Figure 6 (a) shows that the dislocation density of the alloy is very high, which shows that after annealing at 500°C for 1 hour, the dislocation will not recover and annihilate rapidly. In addition, in the FCC1 phase, a small number of stacking faults (Fig. 6 (b)) and incomplete dislocations at both ends of the stacking fault react with each other during annealing at 500°C to form an immovable L-C dislocation lock (Fig. 6(c)). This kind of dislocation will hinder the relative stability of the slip effect [25]. In addition, a small amount of nano precipitates were observed in the FCC1 phase. Combined with the axial SAED picture (Fig. 6 (d)) at the corresponding position [112], it can be seen that this precipitate is an L12 nanoparticles with ordered FCC structure. Fig. 6 (e) is a high-resolution TEM image of L12 phase observed along the [001] band axis in the FCC1 matrix, combined with its Fourier transform (FFT) image (Fig. 6(f)). Such precipitates have complete coherent relationship with FCC1 matrix and have good ordered coherent strengthening effect [26]. However, only a small amount of ordered precipitates can be detected by SAED technology, which shows that L12 phase in FCC1 matrix is not fully precipitated after annealing at 500°C. XRD and TEM characterization of the alloy at 500°C/1h proved the precipitation of L12 phase.
in BCC and FCC matrix, both of which can strengthen the alloy and cause annealing hardening. In addition, the stable L-C lock formed by the interaction of dislocation and stacking fault excited by annealing is also the cause of alloy hardening.

Fig. 7 shows the TEM microstructure of the sample at 750°C/1h. In addition to FCC1 and FCC2, there are also BCC phase of equiaxed grains and submicron phase in the alloy (Fig. 7a-b). The illustration in Figure 7 (a) is a SAED picture of BCC phase [111] with axis. The illustration in Figure 7 (b) shows the square structure of FCC phase [001] SAED picture with axis. In addition, ordered FCC nano precipitates with L12 structure were also found in FCC1 phase, but the distribution density may not be high, so it is difficult to be detected by XRD. Fig. 7 (c) is an axial SAED picture of FCC1 phase [110], with superlattice diffraction spots caused by L12 phase. Figure 7 (d) shows the high-resolution image of the corresponding FCC1 phase and L12 structure nano phase, which shows that L12 phase and FCC1 have very good coherence and good enhancement effect [26]. However, the precipitation temperature range of the precipitated phase of this L12 structure is not clear.

Fig. 5. TEM microstructure of cold-rolled high-entropy alloy.

Fig. 6. Microstructure of 500°C/1h treated high-entropy alloy.
(a-b) Bright field-TEM image of dislocation and stacking fault, respectively; (c) High-resolution TEM image for L-C locks; (d) bright field-TEM image of nano-precipitates in FCC1 matrix and corresponding SAED pattern taken along [112] axis zone; (e-f) High-resolution TEM image for L12 phase in FCC1 matrix and corresponding FFT pattern.
3.5. The analysis of phase separation

When the mixing entropy is high in the high-entropy alloy system, the high-entropy alloy will form a stable solid solution phase. However, intermetallic compounds often appear in the microstructure of high-entropy alloys, because the mixing entropy is not the decisive factor of the microstructure of high-entropy alloys. The parameters such as mixing entropy ($\Delta S_{\text{mix}}$), mixing enthalpy ($\Delta H_{\text{mix}}$), atomic size difference ($\delta$) and valence electron concentration (VEC) will affect the microstructure of the alloy [27].

The enthalpies of formation of Cu with Ni, Co, Cr and Fe are 4, 6, 12 and 13kJ/mol respectively, which are positive [21]; This shows that Cu is incompatible with these four elements and is prone to phase separation. Therefore, FCC1 phase poor in Cu and FCC2 phase rich in Cu are observed in the original alloy. The atomic radius of Al element is 1.43Å, while the atomic radius of the other five elements is 1.25-1.28Å. Therefore, for the displacement solid solution containing more Al elements, the lattice distortion is large, the lattice is easy to collapse, and a more stable BCC phase is formed. Therefore, obvious BCC phase precipitation is observed when the alloy is annealed at 500-950°C. In addition, when a small amount of Al element is dissolved in FCC matrix, L12 precipitates with "Ni3Al" structure are easy to precipitate after annealing [26].

Literature [28] shows that at temperatures between 700 and 850°C, BCC phase can accommodate appropriate concentrations of Fe, CO and Cu elements, up to ~8, ~10 and ~10 at% respectively, which is much higher than that of Cr element (~3 at%). Therefore, the formation of BCC phase will lead to the enrichment of Cr in the surrounding matrix, which will make FCC1 phase unstable in the formation of intermetallic compounds. This situation is exacerbated in the FCC2 matrix rich in Cu, because Cr, CO and Fe are discharged as the precipitates transition from their metastable state to their equilibrium state, in which only Ni and Al maintain a certain concentration in the solid solution [28]. Therefore, $\sigma$Phase rich in Cr is usually observed near the BCC phase (Fig. 5 (b)). In addition, in Al0.5CrFeCoNiCu high-entropy alloy, the $\sigma$ phase with complex structure forms more slowly compared with BCC phase and has a lower solid solution line temperature between 850°C and 900°C. Generally speaking, the occurrence of phase transition requires the cooperative movement of different kinds of atoms over a relatively large distance. The rapid formation of BCC phase after isothermal annealing shows that the nucleation kinetics and diffusion growth of the phase in Al0.5CrFeCoNiCu is not slow. The formation of $\sigma$ phase...
does require a higher temperature (above 700℃). The precipitation of σ phase requires greater diffusion driving force; However, the annealing time does not need to be too long, and obvious σ phase is detected one hour after annealing (Figure 2). Generally speaking, when annealing at lower than 700℃, the holding time will be prolonged, which will also occur σ phase precipitation. For example, in duplex stainless steel with relatively simple composition, Cr concentration is close to Al0.5CrFeCoNiCu, according to reports, σ phase is quickly formed at a temperature between 700 and 850℃[29]. In contrast, in nickel base superalloys with more complex components, they also have similar Cr content and need to be kept at 700 to 850℃ for hundreds of hours to form σ phase [30]. Therefore, although the slow diffusion in high-entropy alloys is widely reported, the results of this study clearly prove that the diffusion in Al0.5CrFeCoNiCu was not abnormally slow.

4. Conclusion

Based on the results and discussions presented above, the conclusions are obtained as below:

(1) The hardness of cold rolled 79% Al0.5CrFeCoNiCu high-entropy alloy is 399HV; The hardness value will increase significantly at first and then decrease with the increase of annealing temperature. The highest hardness value of annealing at 500℃ is 489HV, which is the effect of dislocation strengthening and second phase strengthening (L12 and BCC phases). The main reason for the abnormal hardening phenomenon of annealing in this alloy is that annealing activates dislocation, stacking fault, L-C locking reaction and annealing induced precipitation strengthening. Annealed at higher temperatures, although some hard σ phase precipitates, the softening caused by dislocation recovery and recrystallized in FCC is too obvious, so the hardness decreases significantly.

(2) The homogenized high-entropy alloy is composed of FCC1 phase poor in Cu (rich in Fe, Co, Cr, Ni) and FCC2 phase rich in Cu (contains L12 ordered phase). The alloy has obvious BCC phase precipitation annealed at 500-950℃, and σ phase precipitation annealed at 700-850℃. In addition, ordered L12 nano precipitates were observed in the FCC1 phase of the alloy at 500℃/1h and 750℃/1h, indicating that both FCC phases in the alloy can be strengthened by ordered L12 phase.

(3) The structure of L12 nano precipitates in FCC phase is as same as they ordered precipitate in nickel base superalloy, but the precipitation temperature is not clear. In addition, the annealing precipitation behavior of BCC phase and σ phase shows that the element diffusion in Al0.5CrFeCoNiCu high-entropy alloy is not abnormally slow.

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