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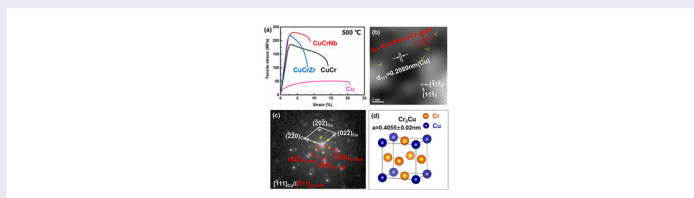
Stabilization of $L1_2$ structured Cr_3Cu precipitates in a Cu-4.06Cr-1.25Nb alloy with high high-temperature strength

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ABSTRACT

Stable $L1_2$ structured Cr_3Cu precipitates were determined in a Cu-4.06Cr-1.25Nb alloy rather than b.c.c. structured Cr precipitates by transmission electron microscope (TEM) and three-dimension atom probe tomography (3DAP) at 450 °C. The precipitation sequence of the studied alloy aged at 400 °C was supersaturated solid solution $\rightarrow$ f.c.c. Cr phase $\rightarrow$ Cr_3Cu phase ($L1_2$). Nb atoms would segregate in the interface between the Cu and Cr phase, reduce the Cu/Cr interfacial energy, affect the precipitation, growth, phase transformation, and coarsening of the precipitate. These Cr_3Cu precipitates significantly improved the high-temperature strength of the Cu-Cr-Nb alloy (223 ± 5 MPa at 500 °C).



IMPACT STATEMENT

TEM images and calculations revealed that Nb can stabilize metastable Cr_3Cu precipitates ($L1_2$) by reducing the Cu/Cr interfacial energy, and provide a higher high-temperature strengthening effect than b.c.c. Cr.

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KEYWORDS

Copper alloys; precipitation; coarsening; first-principles calculation

1. Introduction

Cu-Cr alloys were reported from the 1970s and serviced as lead-frame materials since the 1990s. Nano-scale Cr precipitates formed in the ageing process made the Cu-Cr-based alloy a high strength and conductivity material [1,2]. However, Cr precipitates' coarsening at high temperatures led to a sudden decline in the high-temperature mechanical properties of the alloy [3,4]. Cu-Cr alloys required high heat stability as a key heat sink material in service [5,6]. Therefore, studying the precipitate evolution of Cu-Cr alloy was of great significance to increase the high-temperature mechanical properties of the alloy. The typical precipitation sequence of Cu-Cr alloys during ageing was supersaturated solid solution $\rightarrow$ GP zones $\rightarrow$ f.c.c. Cr precipitate $\rightarrow$ ordered f.c.c. Cr-rich precipitate $\rightarrow$ b.c.c. Cr precipitate [7]. F.c.c. Cr precipitate was a meta-stable phase that was coherent with the matrix [8]. B.c.c. Cr precipitate was an equilibrium phase with

an N-W relationship and K-S relationship [9]. The possible mechanism of transformation from f.c.c. Cr phase to b.c.c. Cr phase was: the dissolution of f.c.c. phase led to an increase in the supersaturation and subsequent homogeneous nucleation of the b.c.c. phase [10]. The reported Cu-Cr alloy showed a significantly coarsening behavior as service at high temperatures. Therefore, high thermal stability phases were required in Cu-Cr system alloys, just as the stability of the austenite phase has an important effect on the strength in steel [11–13]. Researchers had proved that additions of Mg [8], Ti [14], and Zr [15] could affect the precipitation and suppress Cr precipitates' growth. Glenn Research Center had reported a Cu-8Cr-4Nb alloy with excellent high-temperature creep resistance [16–18], which was mainly strengthened by Cr_2Nb particles, but the mechanical properties of the alloy were poor. In recent years, Dierk and Li developed Cu-3.4Cr-0.6Nb and Cu-0.47Cr-0.16Nb alloys with high

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room mechanical properties and emphasized the main strengthening effect was from nano-scale Cr precipitates [19,20]. However, the effect of Nb on the thermal stability of the Cr precipitate and nano-scale Cr precipitates on the high mechanical properties of Cu-Cr-Nb alloys were still unclear.

In this work, $L1_2$ structured Cr_3Cu precipitates were obtained in a Cu-4.06Cr-1.25Nb alloy. The effects of Nb on the precipitation, growth, phase transformation and coarsening of the precipitate were analysed and the effects of the precipitate crystal structure on the high-temperature strength of the alloy were discussed. The purpose of this work is to propose a new stable phase to improve the high-temperature mechanical of Cu-Cr system alloys.

2. Materials and methods

The Cu-Cr-Nb alloy was fabricated by an atmospheric melting. The actual composition of the alloy, Cu-4.06Cr-1.25Nb (mass%), was measured by an inductively coupled plasma optical emission spectrometer (ICP-OES). Cu, Cu-Cr (C18200, USA) and Cu-Cr-Zr (C18150, USA) alloys compared in the present study were commercial alloys received in the cast state. Then, these as-cast ingots were homogenized at 950 °C for 4 h, hot rolled by 80% at 900 °C, solid solution treated at 940 °C for 4 h, cold-rolled by 50%, and ageing treated at 400, 450 and 500 °C for a specific time. Constant strain rate (0.001 s^{-1}) tensile tests were carried out on a UTM5105 electronic universal testing machine with a holding furnace. Before testing at high-temperature, samples were heated to the specified temperature and maintained for 10 min in a furnace. The room temperature tensile samples were processed according to GB/T 228 1-2010, and the high-temperature tensile samples were processed according to GB/T 4338-2006. Repeat 3 times for each condition of testing. Microstructures were characterized by a Talos F200X TEM with an operating voltage of 200 kV. TEM specimens were grounded to 50 μm in thickness and then milled by a PI 691 ion-beam milling. 3DAP was conducted on a CAMECA LEAP-4000X HR APT system. The 3DAP samples with a needle-like shape of $0.5 \times 0.5 \times 20\text{ mm}^3$ were prepared by the standard two-step electro-polishing procedure. The first-principles calculations were performed using the Vienna Ab-initio Simulation Package (VASP) [21]. The generalized gradient approximation (GGA) and the projector augmented wave (PAW) method were used for the exchange–correlation functional [22]. The plane wave truncation energy was 520 eV. According to the orientation relationship (OR) between f.c.c. Cr phase and Cu matrix during the ageing process in the studied

Cu-Cr-Nb alloy, interface models with 15 Å vacuum layer were constructed by the Material Studio software, and the Brillouin zone was divided by $4 \times 4 \times 1$ k-point grids.

3. Results and discussion

3.1. Mechanical properties

Figure 1 shows the tensile strength of Cu, Cu-Cr, Cu-Cr-Zr, and Cu-Cr-Nb alloys tested at 20°C, 400°C, 450°C, and 500°C. The ultimate tensile strength (UTS) of the studied Cu-Cr-Nb alloy at room temperature was $438 \pm 3\text{ MPa}$, which was lower than that of the Cu-Cr-Zr alloy ($484 \pm 4\text{ MPa}$) but was close to that of Cu-Cr alloy ($435 \pm 5\text{ MPa}$). As the tensile test temperature exceeded 450 °C, the UTS of Cu-Cr-Nb alloy at 450 °C and 500 °C were $262 \pm 3\text{ MPa}$ and $223 \pm 5\text{ MPa}$, respectively, which were higher than Cu-Cr alloys and Cu-Cr-Zr alloys. Tensile test results were mentioned in Figure 1e with the standard deviation.

3.2. Precipitation

Figure 2 shows the TEM images of the studied Cu-Cr-Nb alloy after ageing treated at 400 °C for 2 h and 20 h. Fine Cr precipitates were detected in the matrix as aged at 400 °C for 2 h (Figure 2a). The high-resolution TEM (HRTEM) image (Figure 2b) and its fast Fourier transform (FFT) image (Figure 2c) showed that Cr precipitates had an f.c.c. structure and a cube-on-cube coherent OR with the Cu matrix. After ageing for 20 h, many Cr precipitates still maintained a fine size of smaller than 10 nm (Figure 2d). After carefully examining more than 10 HRTEM images (aged at 400 °C for 20 h), only f.c.c. Cr precipitates (Figure 2e and Figure 2f) and ordered f.c.c. Cr-rich precipitates were detected which surprised us (Figure 2g and Figure 2h). The OR between the f.c.c. Cr/ ordered f.c.c. Cr-rich precipitates and the matrix were still cube-on-cube OR. To verify the stability of ordered f.c.c. Cr-rich precipitates, we increased the ageing temperatures.

Figure 3a–h show TEM images of the Cu-Cr-Nb alloy after ageing at 450 °C for 2 h, 20 h, and 100 h. After ageing for 2 h, diffraction spots of f.c.c. structured Cr precipitates were detected, additional diffraction spots (with weak diffraction intensity) were also found at the mid-points between the (000) and Cr' diffraction spots (Figure 3c), which indicated that Cr particles had been ordered. With the increase of ageing time to 20 h and 100 h, these additional diffraction spots were still visible (Figure 3f and h). The ordered f.c.c. Cr-rich phase was stable at 450 °C and maintained a cube-on-cube coherent OR with the matrix (Figure 3h). Cr precipitates in Cu-Cr, Cu-Cr-Mg,

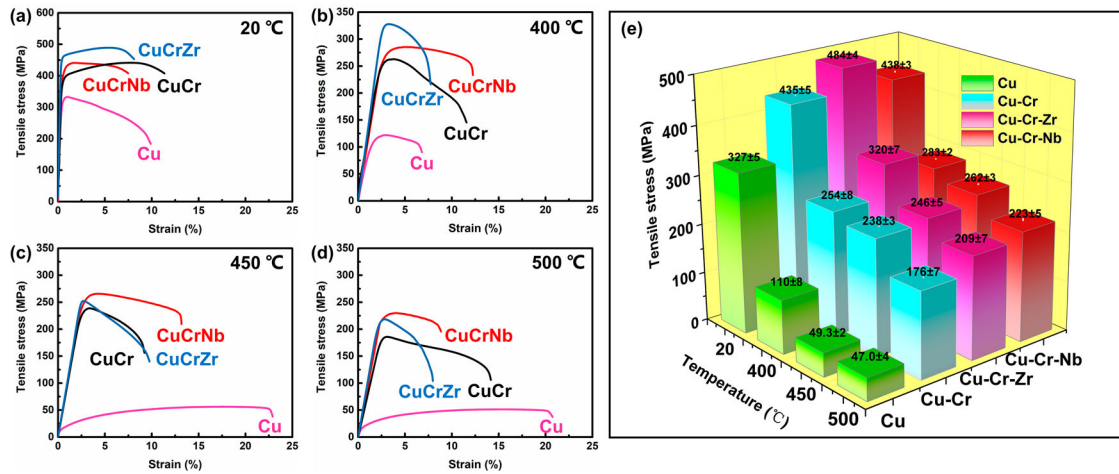


Figure 1. Engineering stress-strain curves ((a)-(d)) and tensile test results with the standard deviation (e) of Cu, Cu-Cr, Cu-Cr-Zr and Cu-Cr-Nb alloys at 20 °C, 400, 450 and 500 °C.

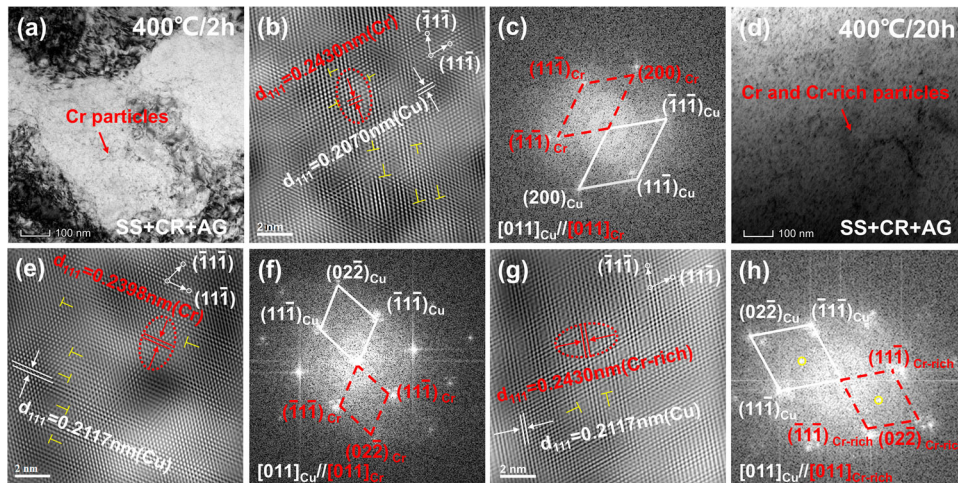


Figure 2. The TEM image of the Cu-Cr-Nb alloy aged treated at 400 °C for different time. 2 h: (a) Bright-field image (BF image), (b) HRTEM image, (c) FFT image; 20 h: (d) Bright-field image, (e) HRTEM image, (f) FFT image, (g) HRTEM image, (h) FFT image. $\circ$ marked spots were superlattice spots from the order f.c.c. structure.

Cu-Cr-Zr, Cu-Cr-Zr-Ti alloys usually exist in the form of stable b.c.c. structure after a long-time ageing treatment [7,8,10,14]. However, in the studied alloy, even ageing at 500 °C, 550 °C, 600 °C for 1 h, Cr-rich precipitates still maintained an ordered f.c.c. structure in the Cu-Cr-Nb alloy (which will be reported in future articles).

Figure 4 shows 3DAP results of the Cu-Cr-Nb alloy aged at 400 °C for 2 h and 20 h. In the early ageing stage at 400 °C for 2 h, coffee-bean-like precipitates were pure Cr phase with an f.c.c. structure. After ageing for 20 h, precipitates coarsened, and Cu atoms were enriched in some precipitates. The average diameters of precipitates were 3.4 ± 0.2 nm and 7.7 ± 0.1 nm as aged at 400 °C for 2 h and 20 h. The element percentage of Nb on the Cu/Cr interface was 2.7% after ageing for 2 h, while that was 3.0% as aged for 20 h. Combined with the symmetry of crystal structure and the enrichment of Cu atoms

in precipitates (Figure 4b), it could be inferred that the ordered f.c.c. Cr-rich phase was $L1_2$ structured Cr_3Cu (Figure 3i). The precipitation sequence of the studied Cu-Cr-Nb alloy during ageing at 400 °C was supersaturated solid solution $\rightarrow$ f.c.c. Cr phase $\rightarrow$ $L1_2$ structured Cr_3Cu phase.

3.3. Effect of Nb on the precipitate structure and mechanical properties

Figure 5 shows the first-principles calculation results, indicating that the Nb atom was easier to replace the Cu side atom rather than the Cr side on the Cu/Cr interface. The interfacial energy of the Cu/Cr interface was 107.1 mJ/m², however, that of Cu/Cr interface with Nb atoms segregation was 78.2 mJ/m². The segregation of 2-3% of Nb atoms was difficult to cause the Cu/Cr interfacial

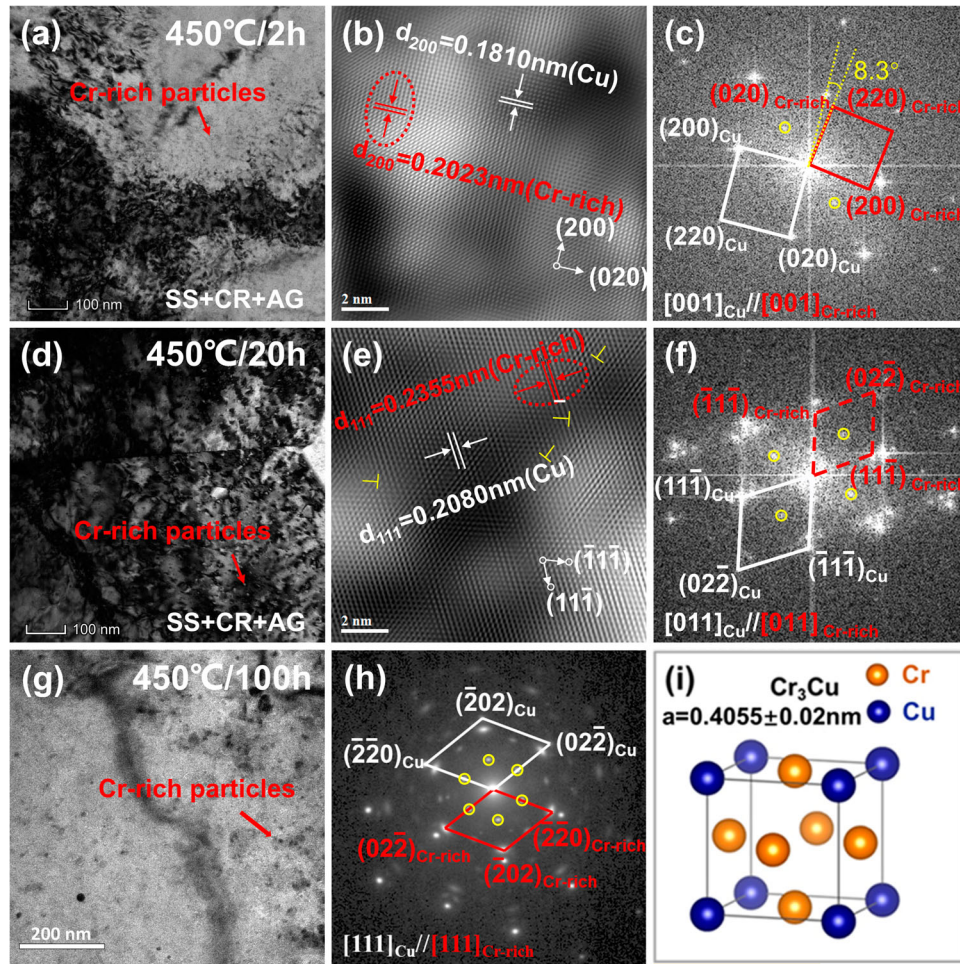


Figure 3. The TEM image of Cu-Cr-Nb alloy aged at 450 °C for different time. 2 h: (a) BF image, (b) HRTEM image, (c) FFT image; 20 h: (d) BF image, (e) HRTEM image, (f) FFT image; 100 h: (g) BF image, (h) SAED image; (i) Crystal structure of L₁₂ ordered Cr₃Cu. The ordered f.c.c. Cr-rich particles in Figure 3a–h and Figure 2g, h were finally determined to be L₁₂ ordered Cr₃Cu particles.

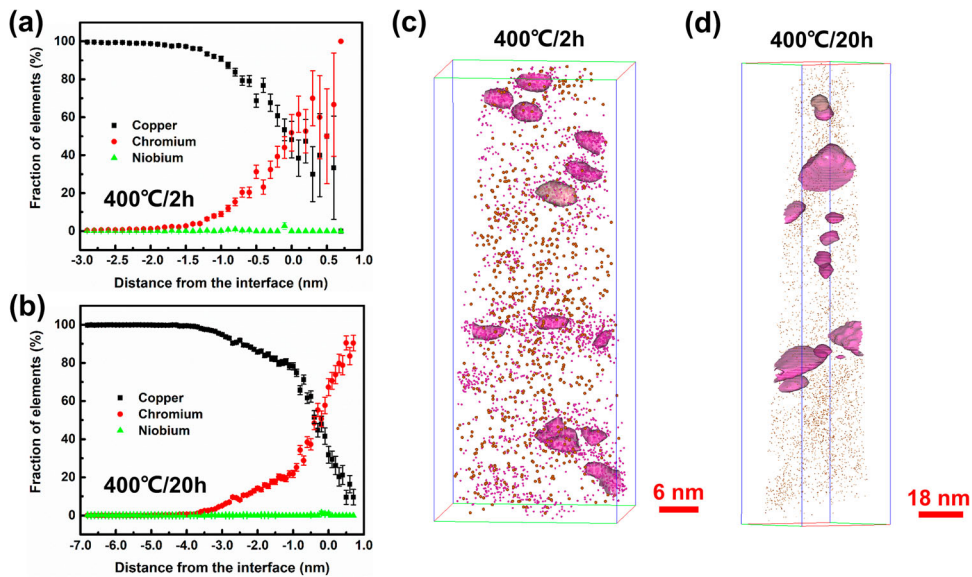


Figure 4. 3DAP data of Cr density maps and corresponding tested concentration of Cr and Nb in Cu-Cr-Nb alloy aged at 400 °C. (a) and (b) show the element concentration in the sample for 2 and 20 h, respectively. (c) and (d) show the distribution of Cr and Nb in the sample aged for 2 and 20 h, respectively.

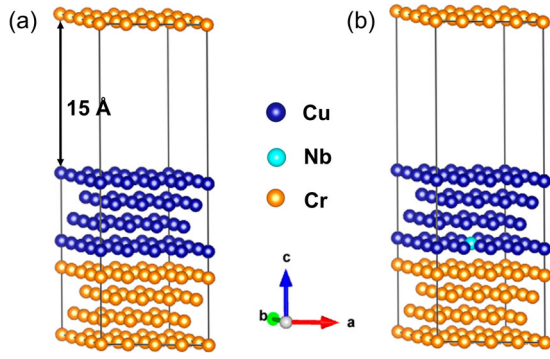


Figure 5. Cu(111)/Cr(111) interface structure model. (a) Cu/Cr interface; (b) Cu/Cr interface with Nb atom segregation.

energy to drop by about 27%. To verify the rationality of the first-principles calculation results, we used a formula to recalculate the influence of Nb on the interfacial energy [10].

$$\gamma = \varepsilon((\chi_p - \chi_m) / a)^2 \quad (1)$$

where a is the lattice parameter (without Nb: $a = (0.368 + 0.3615)/2$ nm [10,23]. With Nb: $a = (0.4108 + 0.3615)/2$ nm) and ε is the ordering effective energy (1.14×10^{-20} J [10]). χ_p and χ_m are solute atom concentrations in the precipitate (100%) and the matrix (0%). For Cr nuclei, $\gamma = 86$ mJ/m² (without Nb) and $\gamma = 77$ mJ/m² (with Nb). The calculation results of the two methods both indicated that Nb segregation on the interface can reduce the interfacial energy, but the latter is more reasonable (drop by 10.5%).

The nucleation of Cr precipitates from the perspective of thermodynamics and kinetics was discussed to analyse the effect of Nb segregation on precipitates. The nucleation barrier (ΔG^*) for f.c.c. Cr precipitates could be computed as follows [10,24]:

$$\Delta G^* = \frac{16\pi}{3} \frac{\gamma^3}{(\Delta G_n + \Delta G_e)^2} \quad (2)$$

where γ is the interfacial energy between precipitate and matrix. ΔG_n is the driving force. ΔG_e is the elastic energy. The driving force could be computed by [14]:

$$\Delta G_n = \frac{G_A - (G_m + G_p)}{V_{mol}} \quad (3)$$

where G_A , G_m and G_p are the Gibbs free energies of the alloy, matrix and precipitates, respectively. The thermodynamic database was derived from references [25]. V_{mol} is the molar volume of the precipitates (7.2×10^{-6} m³/mol for Cr). The elastic energy could be computed

by [26]:

$$\Delta G_e = \frac{E}{1 - \nu} \delta^2 (\chi_p - \chi_m)^2 \quad (4)$$

where E , ν and δ are the elastic modulus (130 GPa), the Poisson ratio (0.34) for Cu matrix, and the misfit between matrix and precipitate, respectively. For Cr nuclei, without Nb atoms segregated on the interface, the misfit between matrix and precipitate is 1.9% [10]. For that with Nb atoms segregated on the interface, it could be computed by:

$$\delta = 2 \frac{a_p - a_m}{a_p + a_m} \quad (5)$$

where a_m (0.3615 nm) and a_p (0.4108 ± 0.0053 nm) are the lattice parameter of matrix and precipitate. When Nb atoms segregated on the interface, the misfit between matrix and precipitate is $11.5 \pm 0.6\%$, which is higher than that without Nb. This is caused by the difference between the lattice constant of the Cr particle and Cu matrix in the Cu-Cr-Nb alloy, but the lattice constant of the two are similar in the Cu-Cr alloy.

For Cr nuclei with Nb atoms segregated on the interface, the nucleation barrier is 2.9×10^{-22} J, which are lower than that for Cr nuclei without Nb atoms segregated on the interface (1.6×10^{-21} J). The precipitation kinetic calculation based on the change of alloy electrical conductivity during ageing. The calculated activation energy is 80.93 kJ/mol, which is lower than 85.36 kJ/mol reported in the Cu-Cr-Zr alloy [27], and 93.10 kJ/mol reported in the Cu-Cr binary alloy [28]. Therefore, Nb could increase the nuclei probability and accelerate f.c.c. Cr's precipitation in the early ageing process. Finally, these led to a rapid decrease of solute atom concentration in the matrix and the formation of Cr-poor zones around the f.c.c. Cr phase, thereby suppressing the growth and phase transformation of Cr precipitates.

From the TEM images of Figure 2 and Figure 3, we could certify that nano-scale Cr and stable Cr₃Cu precipitates kept nano-scale size even exposed at high temperatures for a long time. According to the particle coarsening model proposed by Lifshitz Slyozov [29] and Wagner (LSW) [30], the relation between the particle's average radius ($\bar{r}$) and time (t) in the coarsening process could be expressed as:

$$\bar{r}^3 - \bar{r}_0^3 = \frac{8}{9} \frac{DM\gamma C_\alpha(\infty)}{RT\rho} \quad (6)$$

where $\bar{r}_0$ is the particle's average radius at t_0 . D is the diffusion coefficient of solute atoms in the matrix ($D = 1.2 \times 10^{-5} \exp(-\frac{80930}{RT})$ [31]). M is the molecular weight of Cr phase (52 g/mol). γ is the interface energy between matrix and precipitate. $C_\alpha(\infty)$ is the

equilibrium solubility of the precipitate phase ($C_\alpha(\infty) = 24840 \exp(-\frac{114880}{RT})$) [32]. R is the ideal gas constant (8.314 J/(mol·K)). T is the Kelvin temperature (673 K). ρ is the Cr precipitate's density (7.19 g/cm³).

The calculation result shows that f.c.c. Cr particles in Cu-Cr-Nb alloy grew to 11.2 nm after ageing at 400 °C for 20 h. We also considered that f.c.c. Cr particles had been ordered before ageing at 400 °C for 20 h (Figure 2g, h and Figure 4b). The interface energy between matrix and L1₂ ordered Cr₃Cu was $0.75^2 = 9/16$ times lower than that of matrix and f.c.c. Cr [10]. L1₂ ordered Cr₃Cu particles would grow to 9.4 nm after aged at 400 °C for 20 h. Suppose Cr particles were b.c.c. structure, the interfacial energy of Cr_{bcc}/Cu_{fcc} was 625 mJ/m² (non-coherent interface) [33], particles would grow to 22.3 nm after aged at 400 °C for 20 h. The L1₂ ordered Cr₃Cu had stronger resistance to coarsening than the b.c.c. Cr. Therefore, the main reason for the Cu-Cr-Nb alloy had higher mechanical properties than Cu-Cr alloy at high temperature was that L1₂ ordered Cr₃Cu particles had higher resistance to coarsening than the b.c.c. Cr.

4. Conclusions

In summary, a Cu-4.06Cr-1.25Nb alloy was fabricated by an atmospheric melting, showing a high tensile strength at high temperature (223 ± 5 MPa at 500 °C). Stable L1₂ structured Cr₃Cu precipitates were detected in the studied alloy with high thermal stability. The precipitation sequence during ageing at 400 °C was supersaturated solid solution → f.c.c. pure Cr phase → L1₂ structured Cr₃Cu phase. Nb atoms segregated on the interface between the Cu matrix and Cr precipitates (Cr₃Cu precipitates). The addition of Nb reduced the interfacial energy, promoted the f.c.c. structured Cr's precipitation, and inhibited precipitates' growth, phase transformation and coarsening. Compared with the b.c.c. structured Cr precipitate, the f.c.c. Cr precipitate and the L1₂ structured Cr₃Cu precipitate coarsened much slower at high temperatures. These led to the studied Cu-Cr-Nb alloy having a higher high-temperature mechanical property than Cu-Cr alloy.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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