Evolution of microstructure and properties of Cu-4.5 wt.% Ag alloy prepared by vacuum horizontal continuous casting in solid solution and aging treatment

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Abstract
The vacuum horizontal continuous casting method was used for preparing Cu-4.5 wt.% Ag alloy rod containing few oxygen. The evolution of microstructure was observed by metallographic microscope (OM), scanning electron microscope (SEM) and transmission electron microscope (TEM). The results showed that the hardness and electrical conductivity of Cu-4.5 wt.% Ag alloy aged at 450 °C for 12 h were increased by 60 HV and 12 %IACS than solution treated alloy. TEM observation showed that the continuous precipitates of Ag are uniformly distributed in matrix with the form of particles and strips. Through calculation, the strength increment of peak aged Cu-4.5 wt.%Ag alloy from solid solution hardening and precipitation hardening are 86 MPa and 136 MPa, respectively.

1. Introduction
Cu alloys are widely used in rail transportation, aerospace, electronic components and other fields due to its good mechanical properties and electrical conductivity. Among them, Cu-Ag alloy has the best strength and conductivity matching, which can simultaneously possess high strength and high conductivity [1, 2].

When the content of Ag is more than 6 wt.%, the strength of Cu-Ag alloy can reach more than 1 GPa, meanwhile, the conductivity can be maintained above 80 %IACS. It can be applied to pulse magnet of high magnetic field equipment [3, 4]. When the content of Ag is less than 6 wt.%, the electrical conductivity and plasticity of Cu-Ag alloy are only slightly lower than that of pure copper, but the strength and softening temperature are greatly improved. It can be used to prepare ultra-fine wires, which are important sources of materials for cable coils used in medical equipment, robots and drones [5].

Cu-Ag alloy with high Ag content (> 6 wt.%) can obtain high strength, however, its preparation cost is high and its application field is limited. The Cu-Ag alloy with low Ag content (< 6 wt.%) has lower cost and wider application range, but its strength is much lower which can not meet the requirements of ultra-high strength materials.

In these research [6, 7], the third component (Zr, Cr, etc.) is added to improve the hardness of Cu-Ag alloy, but most of the third component will increase the difficulty in preparing the ingot and have adverse effects on the electrical conductivity of the alloy. The strength of Cu-Ag alloy prepared by severe plastic deformation process can also improve the strength of the alloy [8–10], whereas the process is complex and requires high quality ingot, not to speak of the low production efficiency.

Transitional horizontal continuous casting method can enhance the efficiency of production, however, due to the exposure of copper liquid under air during its transferring from melting furnace to holding furnace [11], there is a high oxygen content in the ingot. Oxygen will form Cu₂O or react with hydrogen to produce moisture distributed in the ingot, causing the formation of voids and crack, thereby reducing ingot quality and alloy properties [12–14].
Reducing the oxygen content of ingot can improve ingot quality and properties of the alloy. Therefore, a vacuum horizontal continuous casting equipment is designed by our team. It can guarantee the whole smelting and casting process in an oxygen free environment through drawing vacuum and filling protective gas subsequently, realizing continuous preparation of high-quality alloy ingots with extremely low oxygen content.

In this experiment, oxygen-free copper (OFC) rods and Cu-4.5 wt.% Ag alloy rod containing extremely low oxygen content were prepared by vacuum horizontal continuous casting equipment. Microstructure and performance changes of low-oxygen content Cu-Ag alloy during solid solution and aging process was studied, and the strengthening effect of 4.5 wt.% Ag was obtained through calculation.

2. Material and methods

The raw materials of the alloy casting rod are 99.99 wt.% high-purity cathode copper and 99.99 wt.% pure silver particles. Alloy prepared by vacuum horizontal continuous casting equipment (SYJ300-2R, Shanghai Xiangxue) under argon atmosphere, the actual alloy composition is Cu-4.5 wt.% Ag, seen in figure 1 red point. The Cu-Ag alloy rod was solution treated at 720 °C, which is slightly lower than the eutectic temperature (780 °C) to avoid over-burning and guarantee all Ag atom dissolved into matrix, holding for 5 h, followed by water quench. Using electrical discharge wire-cutting machine, the solution treated alloy rods were cut into thin pieces with a thickness of 3 mm, and then received aging at a temperature of 450 °C for 0 ∼ 36 h, at which aged Cu-Ag alloy have improved properties [15, 16].

The silver and oxygen content of the head and tail part of the ingot was analyzed by inductively coupled plasma mass spectrometry (ICP, IRIS Intrepid II, Thermo Fisher Scientific) and nitrogen and oxygen analyzer (ON736, LECO, America), results are shown in table 1. The hardness values of the alloy were taken from arithmetical mean of the measurements obtained by Vickers hardness tester (200HV5-5, Laizhou Huayin) from more than 15 indentations. The electronic universal testing machine (UTM5105X, Shenzhen Sansi) and SB-2230 DC digital resistance tester (SB-2230, Shanghai Jingmi) were used to measure ultimate tensile strength and electrical conductivity of the Cu-Ag alloy at room temperature. Information on the crystal structure was collected by x-ray diffractometry (XRD, Xpert powder, PANalytical B.V) with Cu-Kα radiation, tube voltage of 40 kV and current of 250 mA. The continuous scanning mode ranged from 20 to 100 at 1° min⁻¹. Optical microscopy (OM, BMM90AE, Shanghai Bimu), scanning electron microscopy (SEM, MLA650 F, FEI) and transmission electron microscopy (TEM, Tecnai, G2-F20, FEI) were utilized to observe the microstructure of the alloy in various states. The sample for OM and SEM observation was mechanically ground and polished, and then corroded with a solution of 5 ml FeCl₃, 20 ml HCl and 100 ml H₂O. The TEM sample was first made into a thin sheet with a thickness of 300 μm and a diameter of 3 mm, and a 1:4 nitric acid methanol solution was

![Figure 1. Phase diagram of Cu-Ag alloy: (a) 0 ∼ 100 wt.% Ag component in alloy; (b) 0 ∼ 10 wt.% Ag component in alloy [17].](image-url)

| Alloy   | Ag (wt.%) | Oxygen (ppm) |
|--------|-----------|--------------|
| OFC    | —         | 2.8          |
| Cu-Ag  | 4.52      | 2.4          |

Table 1. Silver and oxygen content of alloy.
prepared for electrolytic double spraying at about $-40\,^\circ\text{C}$ and 10 V. The dendrite spacing, size of eutectic and precipitation were estimated from several images obtained by OM, SEM and TEM via computer software.

3. Results

3.1. Properties

Figure 2 show the properties of as-cast OFC and Cu-4.5 wt.% Ag prepared by same method, the conductivity of OFC reaches 102 %IACS, but the estimated tensile strength is 87.2 MPa. The addition of Ag improves the strength of Cu alloy, while the conductivity only decreases slightly to 96.83 %IACS, the conductivity and elongation remain high.

After solution treated, aging at 450 $^\circ\text{C}$, the evolution of hardness of the alloy is shown in figure 3. It is clear that the Cu-4.5 wt.% Ag alloy has obvious aging hardening. The hardness increases continuously with aging time in earlier stage. The maximum hardness of 120 HV was reached at 12 h, and then basically remained steady. According to the phase analysis of solution treated ($720\,^\circ\text{C} \times 5\,\text{h}, \text{ST}$) and peak aged ($450\,^\circ\text{C} \times 12\,\text{h}, \text{PA}$) Cu-4.5 wt.% Ag alloy XRD pattern in figure 4, there is only Cu peak in ST Cu-4.5 wt.% Ag XRD pattern, and Ag peak appears in PA state, suggesting the existence of Ag phase after aging.
Figure 5 shows the comparison of the tensile strength and electrical conductivity between the as-cast, ST and PA Cu-4.5 wt.% Ag alloy. The strength of the ST Cu-4.5 wt.%Ag is 224.28 MPa, 10 Mpa higher than that of the as-cast, the peak aged increased by 38 MPa reach a value of 262.38 MPa. And the electrical conductivity after solution treated decreased from 96.83 %IACS to 86.25 %IACS, peak aged subsequently, the electrical conductivity increased to 98.38 %IACS.

3.2. Microstructure

The microstructure of the as-cast Cu-4.5 wt.% Ag alloy rod produced by the vacuum horizontal continuous casting method is shown in figure 6(a) and figure 7(a), the as-cast structure of Cu-4.5 wt.%Ag alloy is primarily composed of $\alpha$-Cu matrix, with a typical dendrite structure. Judging from the growth direction of the dendrite, the primary dendrite grows along the longitudinal section direction of the ingot, which is the cooling direction. And the secondary dendrite extends in the lateral direction of the primary dendrite, with a dendrite spacing about 12 $\mu$m. There is also a spot of non-equilibrium eutectic with a measured size of 2 ~ 8 $\mu$m dispersed in the Cu dendrite arms. According to the phase diagram of Cu- Ag alloy, theoretically, there should be no eutectic structure in alloy if the Ag content is less than 7.9 wt.%. However, during the casting process, the cooling rate is too fast, causing the residual liquid phase unable to solidify in time when the temperature reaches the solidus
As the temperature continues to drop, the eutectic composition is reached, and the eutectic transformation occurs, resulting in the generation of non-equilibrium eutectic structure [18, 19].

Figures 6(b) and 7(b) show that, after solution treated, most of the non-equilibrium eutectic structure disappear. Since the non-equilibrium eutectic is thermodynamically unstable, eutectic will decompose and dissolve into the matrix during the heat treatment process, ensuring there is only solid solution in alloy after solid solution treatment.

There are discontinuous precipitation and continuous precipitation occur in Cu-Ag annealing process. Figures 7(c) and 7(d) show morphology of discontinuous precipitation of Cu-4.5 wt.% Ag peak aged at 450 °C, it is revealed that Ag discontinuous precipitation occur at grain boundary, forming discontinuous precipitation cells. Discontinuous precipitation has the lamella structure as seen in figure 7(d), with a lamella spacing about 260 nm. Continuous precipitations are observed in figure 8, Ag precipitation show a shape of particles and strips, the size of precipitates is 8 ~ 26 nm, with a spacing of 40 nm, which is consistent with other research [20].

4. Discussion

4.1. Strength
It can be seen from figures 2 and 5, the tensile strength are 224.28 MPa and 262.38 MPa for ST sample and PA sample, while the tensile strength of OFC is only 87.2 MPa. According to figures 7 and 8, since eutectic was dissolved into matrix, Ag was present in Cu matrix as solute atom in ST sample, consequently, the strength increment (137.08 MPa) of ST Cu-4.5 wt.% Ag alloy only came from solid solution hardening. And after peak
aged, part of Ag precipitate continuously and discontinuously from matrix. Therefore, the strength increment of 175.18 MPa for PA Cu-4.5 wt.% Ag alloy originated from solid solution hardening and precipitation hardening, and precipitation hardening is more effective than solid solution hardening, which is proved by the calculated hardening value in subsequent discussion.

4.1.1. Solid solution hardening
The atomic radius of Cu is 0.128 nm, and the atomic radius of Ag is 0.144 nm. Since the atomic size discrepancy of Cu and Ag atom is large, the dissolution of Ag atom into the copper matrix will cause lattice distortion, forming a stress field that hinders the movement of dislocations, thereby increasing the strength of the alloy. The solid solution strengthening effect of Ag atoms can be calculated by Labusch-type solid solution strengthening model:

$$\Delta \tau_{ss} = f G \left( \frac{\eta}{1 + \frac{1}{2} \alpha^2 \delta^2} \right) \sqrt{\frac{X_a}{3}}$$

(1)

Where $f$ is a dimensionless constant, $\alpha$ is a constant related to the type of dislocation, values 3 to 16. $\delta$ is the atomic size mismatch factor, and $\eta$ is the elastic mismatch factor, respectively calculated by the following formulas:

$$\delta = \frac{d \ln a}{dX} = \frac{1}{a} \frac{da}{dX}$$

(2)

$$\eta = \frac{d \ln G}{dX} = \frac{1}{G} \frac{dG}{dX}$$

(3)

Where in formula (2) and (3), $a$ and $G$ are lattice constant (examined from XRD diffraction pattern) and shear modulus of alloy. X is the solute Ag concentrations in solid solution, calculated through Vegard Law according to lattice constant [22, 23].

It is known that the strength increment value of ST Cu-4.5 wt.% Ag (137.08 MPa) only originated from strengthening of solute Ag atom. Therefore, the constant $f$ can be obtained by bringing the strength increment value and other parameters into formula 1. Then the increment of the solid solution strengthening of the PA Cu-4.5 wt.% Ag alloy can be calculated according to corresponding parameters of PA Cu-4.5 wt.% Ag alloy, the specific values are shown in table 2.

4.1.2. Precipitation hardening
The TEM observation in figure 8 shows Moiré fringes on Ag precipitates. On account of the Cube on cube orientation relationship between Cu matrix and Ag precipitates [28], the slight difference between the Cu and Ag crystal interplanar spacing of [111] plane will cause interference of the transmission beam and the second diffraction beam, resulting in formation of moiré fringes on the precipitates. The moiré fringe spacing $d$ can be calculated by formula (4) [19]. In formula (4), $d_{Ag}$ and $d_{Cu}$ are spacing of [111] plain in Ag and Cu respectively. The specific parameters and result are shown in table 3, which is close to the spacing detected from the TEM image.
Semi-coherent interfaces exist between the Ag continuous precipitates and the Cu matrix because of the lattice parameter mismatch (about 13% between Cu and Ag) \[^{[30]}\], and the average particle diameter is relatively large, therefore, the Ag precipitates contribution to the strength of PA Cu-4.5 wt.% Ag can be calculated by the Orowan-Ashby equation \[^{[31]}\]:

\[
\tau_p = \frac{G b \sqrt{r f}}{\delta}
\]

Where $G$ and $b$ are shear modulus and Burgers vector of Cu, $r$ and $f$ are the particle radius and volume fraction of the Ag precipitates. The above parameter values and calculation results are shown in Table 4.

According to the calculation results, the solid solution hardening is 86.44 MPa and the precipitation hardening is 136.02 MPa. Adding the strength of OFC 87.2 MPa, the strength of PA Cu-4.5 wt.% Ag should be 309.66 MPa. The calculation value is greater than the actual measured tensile strength of the alloy, which is 262.38 MPa. This is consistent with the experimental results of Li \[^{[31]}\]. They explained that the calculation results is larger because that the Orowan-Ashby equation used for precipitation hardening is suitable for the cases where the separation phase spacing is far larger than the diameter of precipitates, while the actual size of Ag

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**Table 2.** Solid solution strengthening of PA Cu-4.5 wt.% Ag.

| Parameter | Value |
|-----------|-------|
| Solid solution hardening of ST Cu-4.5wt.% Ag, MPa | 137.08 |
| Standard lattice parameter of Ag \[^{[24]}\], nm | 0.408600 |
| Standard lattice parameter of Cu \[^{[25]}\], nm | 0.361500 |
| Lattice parameter of ST Cu-4.5 wt.% Ag, nm | 0.363322 |
| Lattice parameter of PA Cu-4.5 wt.% Ag, nm | 0.362890 |
| Solute Ag concentrations in the ST Cu-4.5 wt.% Ag, at.% | 2.60 |
| Solute Ag concentrations in the in the PA Cu-4.5 wt.% Ag, at.% | 1.28 |
| Shear modulus of pure Cu \[^{[26]}\], GPa | 46 |
| Shear modulus of pure Ag \[^{[24]}\], GPa | 30 |
| Shear modulus of Cu-4.5 wt.% Ag, GPa | 45.77 |
| Atomic size mismatch factor, $\delta$ | 0.012824 |
| Elastic mismatch factor, $\eta$ | 0.349593 |
| Constant, $\alpha$ \[^{[27]}\] | 3 |
| Constant, $f$ | 0.167062 |
| Solid solution hardening of PA Cu-4.5 wt.% Ag, MPa | 86.44 |

**Table 3.** Spacing of moiré fringe on the precipitates.

| Parameter | Value |
|-----------|-------|
| Ag\(_{111}\) interplane spacing \[^{[29]}\], nm | 0.236 |
| Cu\(_{111}\) interplane spacing \[^{[29]}\], nm | 0.209 |
| Calculated spacing of moiré fringe, nm | 1.82 |
| Detected spacing of moiré fringe, nm | 1.86 |

**Table 4.** Precipitation hardening of PA Cu-4.5 wt.% Ag.

| Parameter | Value |
|-----------|-------|
| Shear modulus of Cu \[^{[27]}\], GPa | 46 |
| Burgers vector of Cu \[^{[32]}\], nm | 0.2556 |
| Average Ag precipitation radius, nm | 10.3 |
| Ag precipitation volume fraction, % | 1.42 |
| Precipitation hardening, MPa | 136.02 |

\[
d = \frac{d_{Ag}d_{Cu}}{d_{Ag} - d_{Cu}}
\]  

(4)

Semi-coherent interfaces exist between the Ag continuous precipitates and the Cu matrix because of the lattice parameter mismatch (about 13% between Cu and Ag) \[^{[30]}\].
precipitates is 20 nm. The precipitates spacing is about 40 nm, which is not much larger compared with the size, thereby causing deviation of calculation. In addition, according to previous researches [33], the discontinuous precipitation completed in 2 ~ 4 h. As the aging time increase, discontinuous precipitation will coarse, reducing the performance of alloy and resulting in a larger calculation result in comparison with the actual measured strength.

4.2. Electrical conductivity

Figure 4 shows the variation of electrical conductivity of Cu-4.5 wt.% Ag in different state. Electrical conductivity of ST and PA Cu-4.5 wt.% Ag alloy are very high with a value of 96.83 %IACS and 98.38 %IACS. The vacuum-melted Cu-3 wt.% Ag alloy casting in iron mold under atmospheric environment with electrical conductivity of 92.5 %IACS and 95 %IACS in as-cast and aged state respectively, which is lower than that of the same state of vacuum horizontal continuous casting Cu-4.5 wt.% Ag alloy by about 3 %IACS [5]. According to table 1, the oxygen content of Cu-4.5 wt.% Ag alloy ingot is 2.4 ppm. With such a low oxygen content, Cu₂O impurities and voids caused by moisture were eliminated, which is proved by figure 6. The disappearance of impurities and defects reduce the scattering of electrons, accounting for a higher electrical conductivity. The evolution of oxygen content and electrical conductivity indicates that the vacuum horizontal continuous casting method can reduce the oxygen content of the alloy ingot, thus effectively improving the electrical properties of the alloy.

5. Conclusions

(1) Cu-4.5 wt.% Ag alloy prepared by vacuum horizontal continuous casting method has an oxygen content of 2.4 ppm, which can achieve the strength of 214.34 MPa and conductivity of 96.83 %IACS. Reducing the oxygen content significantly improves the electrical property of Cu-Ag alloy.

(2) After peak aged, the tensile strength and electrical conductivity increase to 262.38 MPa and 98.38 %IACS. Strengthening mainly comes from solid solution hardening and precipitation hardening. Through calculation, the contribution to strength from solid solution and precipitation are 86 MPa and 136 MPa, respectively.

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