Effect of high temperature-high pressure treatment on microstructure and mechanical properties of Cu-Cr alloy

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Abstract

The microstructure of the Cu-Cr alloy prepared by infiltration before and after different pressures treatment at 900 °C for 10 min was observed by metallographic microscope, scanning electron microscope (SEM), and transmission electron microscopy (TEM). And the mechanical properties were measured by nanoindenter, hardness tester and tensile testing machine. According to the experimental results, the effects of high pressure treatment on the mechanical properties were discussed. The results showed that the hardness of Cu matrix and Cr phase were 114Hv and 194Hv under the pressure of 3 GPa, which were 18.75% and 10.23% higher than those of the infiltrated alloy. Also, the hardness and compression yield strength of Cu-Cr alloy were 134 HB and 241 MPa after 3 GPa pressure treatment, respectively, 11.67%, 19.31% higher than those of the infiltrated alloy.

1. Introduction

Cu–Cr alloys have been widely used in electronic and electrical industries due to their high voltage resistance, excellent welding resistance and current carrying capacity [1–4]. With the development of electronic industry, Cu–Cr alloys have been considered as the promising candidate for obtaining the excellent balance between the good electrical conductivity and considerable strength. However, the aggregation of Cr particle formed during the traditional process is harmful for their strength and electrical conductivity [5]. Thus, the approach for improving their properties by controlling the microstructure of the Cu–Cr alloy is always a hot topic.

At present, there are several methods to improve the properties of Cu–Zr alloys, such as alloying design, deep cooling treatment, thermal mechanical treatment and so on [6–9]. Infiltration method is a common choice, but it makes the alloy poor compactness [10], eventually limiting the application of Cu–Cr alloys. In recent years, it is reported that high temperature and high pressure can facilitate the compactness of copper alloys with considerable properties [11–14]. Wu et al [15] has applied high pressure treatment on Cu-Al alloys and the results showed that the hardness and the elastic recovery rate of Cu-Al alloy increase by 13.14% and 71.84%, respectively. Wang et al [16] has studied the high pressure heat treatment on the Cu-Zn alloys and found the high pressure heat treatment contributes to the grain refinement. However, there is less research on the effects of high pressure treatment on Cu-Cr alloy. In Cu-Cr alloys are firstly prepared by infiltration, then the effects of high temperature and high pressure treatment on microstructure and properties of the alloys are investigated. The
results of this work could provide a new method to tailor the microstructure and to improve the properties of Cu–Cr alloys.

2. Experiment

The Cu51.76Cr48.24 alloy (mass fraction, %) was prepared by infiltration. The Cu–Cr alloy was prepared using infiltration method with Oxygen-free copper powder (99.97% purity) and chromium powder (99.97% purity). Oxygen-free copper powder (average particle diameter of 50 mm) of 5 wt% were firstly mixed with 95 wt% chromium powder (average particle diameter of 75 mm). Then the mixture was sintered into a Cr skeleton ingot with a small percentage of copper acting as binder at 1080 °C in a vacuum hot extrusion furnace. The ingot was subsequently immersed into molten Cu at 1200 °C for 2 h. The samples with the size of 98 mm × 10 mm of Cu–Cr alloy were sealed into graphite heaters, and then were pressurized to 1 GPa, 3 GPa, 5 GPa, 6 GPa respectively and heated to 700 °C for 10 min with heat treatment with CS-IB type six-anvil high pressure equipment and heating with electrical resistance. After that, shutting off power and cooling to room temperature, the hardness of the Cu–Cr alloy samples before and after high pressure treatment were tested by FM-ARS-9000 microhardness tester and HB-3000B Brinell hardness tester. And the hardness, elastic modulus, elastic recovery coefficient and hard-to-elastic ratio of Cu phase and Cr phase were tested using Triboindenter Nano mechanics test system with Berkovich indenter. The Berkovich indenter with curvature radius of 150 nm, load resolution of 10 nN and displacement resolution of 0.04 nm exerted the maximum load of 1000 μN and kept at this load for 10 s, using the loading and unloading rate of 100 μN/s. The compression strength was tested by WDW3100 electronic universal testing machine. The experimental results were taken as the average of three test results. Besides, after grinding and polishing, the metallographic samples were corroded by 4gFeCl₃ + 10 mlHCl + 100 mlH₂O solution and the microstructure of the samples were observed by Axiovert200MAT metallographic microscope, S-3400N SEM-BSE and Jeol-2010 TEM.

3. Result and discussion

3.1. Microstructures

Figure 1 shows the microstructure of the Cu–Cr alloy before and after high pressure treatment. It can be seen that the microstructure of the samples are mainly composed of Cu matrix and irregular granular Cr phase. Compared with infiltrated Cu–Cr alloy, the edge of Cr phase is smoother in Cu–Cr alloy microstructure after high pressure treatment., but the morphology size and distribution of Cr phase are not much changed. SEM-BSE images are shown in figure 2, compared with infiltrated alloy, the Cu matrix in Cu–Cr alloy after high pressure heat treatment is more compact, and porosity in Cr phase is smaller. It means that high pressure treatment can improve the compactness of Cu–Cr alloy. As observed in figure 3, the dislocation density increases in the Cu–Cr alloy after high pressure treatment, and a large number of nanometer Cr particles precipitate in Cu matrix and the size is about 2 to 5 nm (figure 4). This is because that high pressure can generate high strain and induce distortion of lattice, leading to the increase of dislocations [17]. Meanwhile, the micro-pores in Cu matrix and Cr phase can be compressed and closed, resulting in improvement of the compactness of the Cu–Cr alloy. It generally believed that the solid solubility of Cr in Cu matrix decreases gradually with the decrease of
temperature. In 1070 °C, the maximum solid solubility of Cr in Cu matrix is 0.65%, but it is near zero at room temperature [18]. Therefore, the supersaturated solution of Cr in Cu matrix of infiltrated Cu-Cr alloy will precipitate in the form of particles in the subsequent high-pressure treatment process. It was also confirmed that the fine Cr phase can be precipitated in the microstructure of Cu-Cr alloy after high pressure treatment in literature [19]. In addition, high pressure can generate more dislocations in the alloy, which provides more
nucleation sites for Cr particles and increases the number of Cr particles, resulting in more dispersed and fine Cr particles precipitated in Cu matrix after high pressure treatment \[20\].

3.2. Micromechanical properties

Figure 5 shows that the load-displacement curves by nano-indentation tests of Cu matrix and Cr phase in Cu-Cr alloy before and after 3 GPa and 5 GPa pressure treatment. It can be seen that the loading and unloading curves of Cu matrix and Cr phase are not overlap, which indicates that both of Cu matrix and Cr phase occurred plastic deformation. According to mechanics theory, the elastic recovery coefficient \(R\) is used to measure the plastic of materials and it can be calculated by the following equation

\[
R = \left( \frac{h_{\text{max}} - h_f}{h_f} \right) \times 100\%
\]  

where, \(h_{\text{max}}\) and \(h_f\) are the maximum displacement and residual displacement after unloading under 1000 μN load in the process of nano-indentation.

The nano-indentation test results of Cu matrix and Cr phase in Cu-Cr alloy before and after 3 GPa pressure treatment are shown in Table 1. It can be seen that 3 GPa pressure treatment can increase the nano indentation hardness, hard elastic ratio, elastic modulus and coefficient recovery of Cu matrix and Cr phase in Cu-Cr alloy. Compared with the infiltration, the hardness, hard elastic ratio, elastic modulus and coefficient recovery of Cu matrix were 17.33%, 8.33%, 6.25% and 7.05% relatively higher than those before 3 GPa pressure treatment, and the hardness, hard elastic ratio, elastic modulus and coefficient recovery of Cr phase are increased by 11.23%, 4.00%, 7.07% and 120.65%, respectively.

![Figure 5. Load-displacement curves for nano-indentation tests of Cu matrix and Cr phase in CuCr50 alloy.](image)

**Table 1.** Nanoindentation results of Cu-Cr alloy.

| Composition phase | Treatment condition | \(H/\text{GPa}\) | \(E_r/\text{GPa}\) | \(H/E_r\) | \(h_{\text{max}}/\text{nm}\) | \(h_f/\text{nm}\) | \(R/\%\) |
|-------------------|---------------------|-----------------|-----------------|---------|-----------------|----------------|-------|
| Cu matrix         | infiltrated         | 2.02            | 84.37           | 0.024   | 131.28          | 96.52          | 36.01 |
|                   | 3 GPa              | 2.37            | 89.64           | 0.026   | 112.74          | 81.37          | 38.55 |
| Cr phase          | infiltrated         | 5.43            | 219.65          | 0.025   | 46.23           | 28.31          | 63.30 |
|                   | 3 GPa              | 6.07            | 235.18          | 0.026   | 37.34           | 15.58          | 139.67 |

The relation between the hardness of the material and the internal dislocation density is as follows \[21\]:

\[
H = H_0 + aG\beta \sqrt{\rho}
\]

where, \(H_0\) is hardness of non-defective material, \(\rho\) is dislocation density, \(G, a\) and \(b\) are material constant. It can be seen from the above formula that the higher dislocation density the materials contain, the higher the hardness of the materials. High pressure can improve the compactness of Cu matrix and Cr phase in the alloy, and make
the Cu matrix and Cr phase induce distortion of lattice, which lead to the number of dislocation increase, resulting in increment for hardness of Cu matrix and Cr phase. The strength of Cr phase is higher than that of Cu matrix, so when subjected to the pressure, Cu matrix is easier to be compressed and deformed relative to Cr phase, resulting in Cu matrix being compressed under 1 GPa pressure, while Cr phase can be compressed under 3 GPa pressure. The compactness of Cu matrix and Cr phase increases with the increase of the pressure. Finally, it tends to be gentle. Therefore, when the pressure is over 1 GPa, the compactness of Cu matrix does not change much, and when the pressure is over 3 GPa, the compactness of Cr phase does not change much as well. As a result, when the pressure is over 1 GPa or 3 GPa, the hardness of Cu matrix and Cr phase does not increase much. The relation between elastic modulus and material porosity can be calculated by equation (3) [22]:

\[ E = E_0(1.9P + 0.9P^2) \]  

(3)

where \( P \) is porosity \((0 < P < 1)\), \( E_0 \) is elastic modulus of no pore materials, \( E \) is elastic modulus of porous materials. From the above equation, it can be seen that the more internal porosity of the material is, the smaller the elastic modulus. High pressure treatment can reduce the porosity in Cu matrix and Cr phase, thus increasing the elastic modulus of Cu matrix and Cr phase. In general, the larger the hard elastic ratio is, the better the wear resistance of the material is, while the larger the elastic recovery coefficient is, the smaller the plastic deformation and the greater the indentation deformation resistance. High temperature and high pressure treatment can improve the hard elastic ratio and elastic recovery coefficient of Cu-Cr alloy. It can be concluded that high temperature and high pressure treatment can improve the wear resistance and compression deformation resistance of the alloy.

3.3. Macroscopic mechanical properties

Figure 7 shows the relationship between hardness, compression yield strength and pressure in Cu-Cr alloy. As can be seen, high pressure can increase the hardness and compression yield strength, and the hardness and compression yield strength of the alloy increase with the increase of pressure in the range of 1 ∼ 6 GPa. When the pressure is over 1 GPa, the hardness and compression yield strength increase slowly. The test results show that after 3 GPa pressure treatment, the hardness and compression yield strength are 134 HB and 241 MPa, respectively, 11.67%, 19.31% higher than those of the infiltrated alloy.

This is because high pressure treatment can improve the compactness of the Cu-Cr alloy, which lead to improving the deformation resistance of alloys, and also can increase the dislocation density of the alloys, resulting in dislocation entanglement in the process of moving, so the resistance of dislocation motion and the slip critical shear stress improve, achieving the dislocation enhancement effect. In addition, there are a large number of superfine Cr particles formed within the Cu matrix. These superfine Cr particles hinder the dislocation movement and produce strengthening effect [23], which leads to the increase of hardness of the alloy. For metal materials, it is generally believed that the higher the hardness of the materials is, the stronger the deformation resistance, and the greater the compression yield strength. High pressure treatment can improve the hardness of Cu-Cr alloys, so the compression yield strength of Cu-Cr alloys also increases after high pressure treatment.
4. Conclusion

(1) High temperature and high pressure treatment can increase compactness of Cu-Cr alloy and dislocation density in the alloy, and promote the precipitation of nanometer Cr particles.

(2) High temperature and high pressure treatment can increase the hardness, elastic modulus, hard elastic ratio and elastic recovery coefficient of Cu matrix and Cr phase in Cu-Cr alloy, effectively improving the hardness and the compressive yield strength of the Cu-Cr alloy.

(3) The hardness and the compressive yield strength of the Cu-Cr alloy increase with the increase of the pressure. When the pressure exceeds 1 GPa, the hardness and the compressive yield strength increases slowly. The hardness and compressive yield strength of Cu-Cr alloy are 134 HB and 241 MPa, respectively, after 3 GPa pressure treatment, which are 11.67% and 19.31% higher than those of the infiltrated alloy.

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