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RESEARCH PAPER

SIMULTANEOUSLY ENHANCING MECHANICAL PROPERTIES AND ELECTRICAL CONDUCTIVITY OF Cu-0.5%Cr ALLOY PROCESSED BY ECAP AND DCT

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ABSTRACT

Mechanical properties and electrical conductivity of Cu-0.5%Cr alloy were simultaneously enhanced by combing the equal channel angular pressing (ECAP) and deep cryogenic treatment (DCT). The effect of DCT on the microstructure and properties of Cu-0.5%Cr alloy prepared by ECAP was investigated. The results show that the grains were elongated and refined along the deformation shear direction, and the dislocation density increased significantly by ECAP deformation. After the subsequent DCT, the grains were further refined, and at the same time, the dislocation density was further increased. With the increase of passes of ECAP, the microhardness and tensile strength of Cu-0.5%Cr alloy increased significantly, but the elongation to failure and electrical conductivity decreased slightly. After the DCT, the microhardness, electrical conductivity, tensile strength and elongation to failure of the Cu-0.5%Cr alloy were improved. After the ECAP (four passes) and DCT (12 h), the tensile strength, elongation to failure and electrical conductivity reached 483 MPa, 17.6% and 29%IACS respectively. The improvement of tensile properties could be attributed to the increase of dislocation density and grain refinement. The electrical conductivity was improved by the DCT due to the decrease of vacancy concentration.

Keywords: Cu-0.5%Cr alloy; ECAP; DCT; Microstructure; Mechanical properties; Electrical conductivity

INTRODUCTION

With the rapid development of electronics industry, it is essential to fulfil the growing performance demands of Cu-Cr alloys. More attentions have been paid to equal channel angular pressing (ECAP) in order to improve the microhardness and tensile strength of Cu-Cr alloys [1-3]. Grains were significantly refined after the ECAP, the dislocation density was also increased, thus resulting in the increase of microhardness and tensile strength of Cu-Cr alloys [4]. However, the electrical conductivity and elongation to failure were degraded. The microhardness and electrical conductivity of the alloys could be improved by the following aging treatment [5-6].

Deep cryogenic treatment (DCT) is known as an extension of traditional heat treatment process. The process of cooling materials at cryogenic temperature using the liquid nitrogen is utilized to enhance the mechanical and physical properties of materials [7-11]. After the DCT, The retained austenite of die steel was transformed into the martensite with the ultrafine carbide precipitation and residual stress elimination [12-13]. DCT could accelerate the dispersion and precipitation of the second phase particles in the aluminum alloy [14]. For copper alloys, the activation energy of the phase transition could be increased, and the duration of the phase transition can be shortened, thereby fine grains were formed [15-16]. The electrical conductivity of Cu-Be and Cu-Cr-Zr alloys increased significantly with the decrease of solute atomic concentration in the matrix by DCT [15, 17].

In the present, the effect of DCT on microstructure and properties of the ECAPed Cu-0.5%Cr alloy was investigated to simultaneously enhance mechanical properties and electrical conductivity of Cu-0.5%Cr alloy.

MATERIAL AND METHODS

Cu-0.5%Cr alloy were supplied by CHINALCO Luoyang Copper Co. Ltd. The samples with a size of 12×12×80 mm^3 were solid-solution treated at 1273 K for 0.5 hour in the NBD-

1200 tube furnace. The ECAP was performed at room temperature using a die with a channel angle 110°. The sample were pressed for totals of 4 passes and without rotation between each pass in the processing route designated A. The equivalent strain for each pass is 0.8 [18].

After the ECAP processing, the samples were treated with different time of cryogenic treatment. After precooling for 5 minutes, the timer is started, and the sample is taken out after the time is up. The time was 3h, 6h, 12h, 18h, 24h, 36h and 48h, respectively.

Microstructures of the material were examined on optical microscope and JEOL JEM2100 high-resolution transmission electron microscope (TEM). The samples for TEM were thinned by the Tenupol-5 twin-jet electro-polishing device in a solution of 33% nitric acid and 67% methanol at -25 ℃ with a current of 5-15V. X-ray diffraction (XRD) measurements were performed on a D/max-2500 pc X-ray with a Cu target operating at 40 kV/100 mA. A series of θ-2θ scans were performed to record the XRD patterns ranged from 30° to 120°. Microhardness was conducted on the samples with a load of 200 g for 15 s. Tensile tests were performed using a Shimadzu AGS-10kND machine with a cross-head speed of 1mm min^{-1}. The electrical conductivity was measured with a 7501-A eddy current conductor with an accuracy ± 2%.

RESULTS AND DISCUSSION

3.1. Microstructure characterization

3.1.1. Optical observations

The optical microstructures of Cu-0.5%Cr alloy are revealed after the DCT as shown in Figure 1. It can be observed that the grains are relatively large, and the grain boundaries are clear after solid solution treatment, meanwhile, twins are formed. The average grain size is about 70 μm in Fig.1 (a). After DCT for 12 hours, the grains are obviously refined, and the distribution is relatively more uniform, as shown in Fig.1 (b).

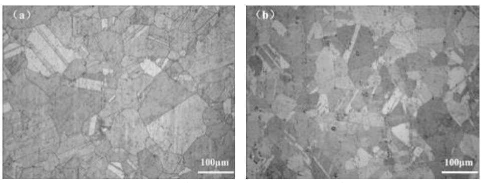

Fig. 1 Optical images of Cu-0.5%Cr alloy after DCT: (a) solid solution, (b) t =12h

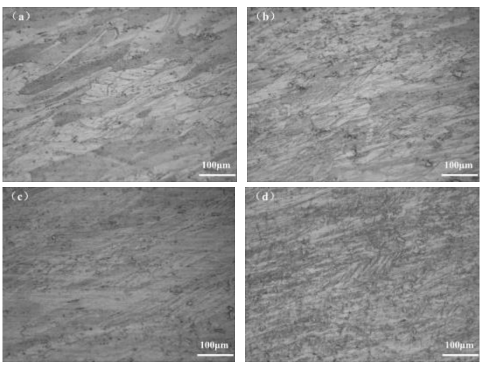

Fig. 2 Optical images of Cu-0.5%Cr alloy after the ECAP and DCT: (a) 1P, (b) 1P + DCT (12h), (c) 2P, (d) 2P + DCT (12h)

After one ECAP pass, the coarse grains of the solid solution state are elongated and refined along the direction of deformation and shear, and the grain boundaries are relatively clear (shown in Fig.2 (a)). With the increase of ECAP passes, the grains are significantly refined along the shear direction in Fig.2 (c). After the subsequent DCT for 12h, it can be seen that the grains are further refined by comparing Fig.2 (b) and Fig.2 (d).

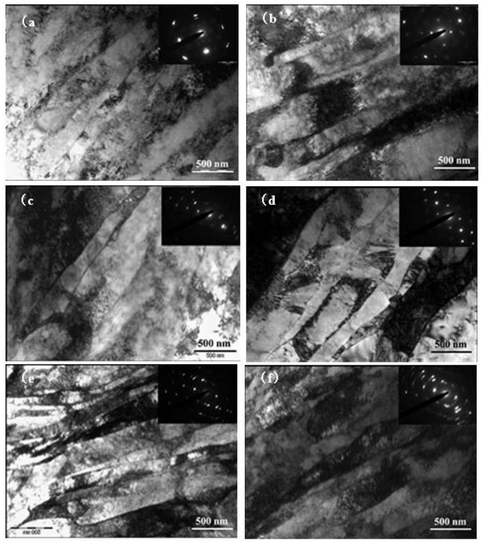

Fig. 3 TEM micrographs of Cu-0.5%Cr alloy after ECAP and DCT for 12h: (a) 1P, (b) 1P + DCT, (c) 2P, (d) 2P + DCT, (e) 4P, (f) 4P + DCT

3.1.2. TEM microstructure

From TEM micrographs, it can be observed that the disordered dislocations spread along the elongated boundaries and low-angle grain boundaries were formatted by one pass of ECAP, which can be speculated from the scattered spots in the selected area electron diffraction (SAD) pattern in Fig.3 (a). As the number of ECAP passes increases, grains are further refined, and dislocation density increases. The SAD in Fig.3 (e) with a continuous ring indicates the formation of large-angle grain boundaries.

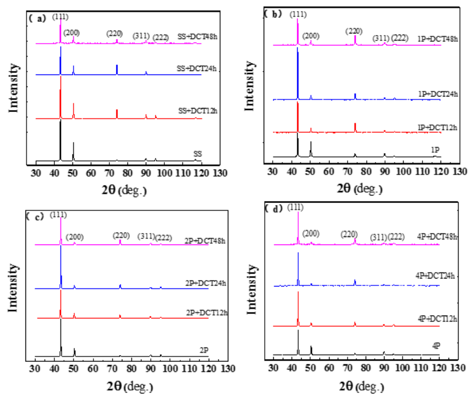

Fig. 4 X-ray diffraction pattern of Cu-0.5%Cr alloy after ECAP and DCT for different time

The dislocation density increases significantly along the grain boundaries and inside the grains by DCT treatment. After 4 passes of ECAP + DCT (12h), the banded grains with thickness of 200 ~ 300nm are obtained in Fig.3 (f). The almost continuous diffraction ring in the corresponding SAD pattern indicates that a nano-scale grain structure with a large-angle grain boundary is formed. Therefore, after DCT, the microstructure of Cu-0.5%Cr alloy is refined, while the dislocation density is significantly increased.

3.2 XRD

As shown in Figure 4. With the extension of the DCT time, the intensity of the diffraction peaks of {111}, {220} and {311} increased significantly in Fig.4 (a), while the intensity of the diffraction peaks of the {200} plane shows a downward trend. The results show that DCT causes slight changes in lattice constant. The intensity of the diffraction peak changes significantly through ECAP deformation and DCT treatment in Fig.4 (b-d). This is because the deformation process causes the microstructure to deform and thus results in the occurrence of a new preferred orientation.

Table 1 The average crystallite size and dislocation density of Cu-0.5%Cr alloy after ECAP and DCT

Samples		$<\varepsilon^2>^{1/2}$/%	d/μm	ρ/ 10^{-15} m^{-2}
ECAP	0P	0.080	65	0.1
	1P	0.120	0.418	1.62
	2P	0.140	0.288	2.55
	4P	0.150	0.224	2.83
ECAP+ DCT(12h)	0P+DCT	0.089	51	1.15
	1P+DCT	0.268	0.338	1.89
	2P+DCT	0.321	0.289	2.46
	4P+DCT	0.352	0.242	2.77
ECAP+ DCT(48h)	0P+DCT	0.092	47	1.26
	1P+DCT	0.273	0.326	1.95
	2P+DCT	0.327	0.279	2.54
	4P+DCT	0.358	0.237	2.81

Table 2 The lattice constants（Å）of Cu-0.5%Cr alloy after ECAP and DCT

Samples	0P	1P	2P	4P
DCT （0h）	3.62284	3.62316	3.62018	3.62046
DCT（12h）	3.61860	3.62054	3.62014	3.62016
DCT（24h）	3.62288	3.62104	3.62000	3.62053
DCT（48h）	3.62310	3.62300	3.62148	3.62170

The dislocation density was calculated by $\rho = \frac{2\sqrt{3}\langle\varepsilon^2\rangle^{1/2}}{d \cdot b}$ [19-20], where b is the Burgers vector and equal to $\sqrt{2}a/2$ for Cu (0.256 nm), which was listed in Table 1. It can be found that the grain size decreases and the dislocation density increases after the DCT treatment. During the rapid cooling process, Cu-0.5% Cr alloy generates a large amount of residual stress due to volume shrinkage, resulting in increased dislocations [16]. The lattice constants（Å）of Cu-0.5%Cr alloy after the ECAP and DCT are calculated by XRD analysis (Table 2).

It can be observed that the grain size decreases significantly after DCT (12h) treatment. As the number of ECAP passes increases, the effect of DCT treatment on grain refinement also gradually weakens, which is consistent with the results of the previous microstructure. DCT can increase the activation energy of phase transformation and shorten the phase transformation duration, thus fine grains are introduced to copper alloys [15, 16]. There are a large number of black flocculent distributions in the grain boundary and in the crystal by TEM, which is consistent with the results of XRD calculations, the dislocation density increases through DCT treatment. For pure copper, the DCT can increase the dislocation density, resulting in the formation of fine grains [21].

3.3. Mirohardness and electrical conductivity

With the increase of the ECAP passes, the microhardness value increases obviously in Fig.5 (a). After one pass of ECAP, the microhardness reaches 144HV. As the number of deformation passes increases, the upward trend of microhardness gradually decreases, and basically reaches saturation in four ECAP pass. This is due to the fact that the grain size does not change obviously, and the phase equilibrium of dislocation accumulation and annihilation [22-24].

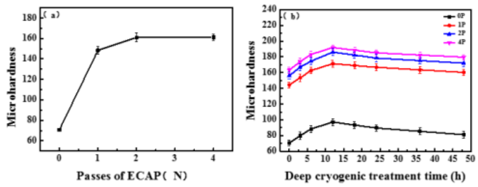

Fig. 5 Microhardness of Cu-0.5%Cr alloy processed by the different passes of ECAP and DCT for different time

Fig.5 (b) shows the microhardness of Cu-0.5%Cr alloy processed by the different passes of ECAP and DCT for different time. The microhardness increases first and then decreases with the increase of the DCT time. After four ECAP passes and 12 hours of DCT, the microhardness reaches the maximum value of 192HV. The DCT treatment is carried out in an environment of liquid nitrogen at -196℃, and the lattice shrinks at an ultra-low temperature [25, 26]. Due to the different shrinkage of each crystal plane, micro-deformation occurred, which causes the increase of dislocations and the formation of sub-crystalline structure, and at the same time produces a certain work hardening. From the microstructure and XRD analysis, it can be seen that the grains are refined after DCT treatment, Cr particles are precipitated in the matrix, and the dislocation density increases. With the extension of cryogenic time, the degree of grain refinement and dislocation density will not be further increased. When the material is taken out from the liquid nitrogen, the temperature environment will be changed significantly, and the recovery occurs at lower temperature environment [27], which may be an explanation of the decrease in microhardness.

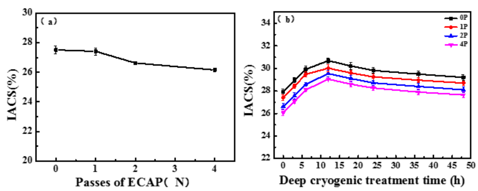

Fig. 6 The curves of the electrical conductivity of Cu-0.5%Cr alloy by the different passes of ECAP and DCT time

Fig.6 (a) is the curve of the electrical conductivity change of ECAP deformed Cu-0.5%Cr alloy. The results show that as the ECAP passes increase, the electrical conductivity decreases slightly. Compared with the solution treatment, the electrical conductivity decreases only about 6.1% after four ECAP deformations passes. From Table 1, it can be seen that after ECAP, crystal defects such as dislocations of Cu-0.5%Cr alloy increase, the electron scattering effect increases, and the electrical conductivity decreases slightly. Similar changes in the electrical conductivity of Cu-0.5%Cr alloy with large strains were confirmed [28, 29].

After the subsequent DCT **(**12h**)**, the change curve of electrical conductivity is shown in Fig.6 (b). It is observed that with the extension of the DCT time, the electrical conductivity tends to increase first and then decrease. After DCT for 12h, the maximum solution solid Cu-0.5%Cr alloy is 30.7% IACS. After four ECAP passes and the DCT for 12 hours, it is improved by about 10.7% compared with no DCT. In other words, the electrical conductivity after DCT is higher than that of the ECAP before the DCT for 12h, which is similar to the change in electrical conductivity of pure copper DCT treatment [21].

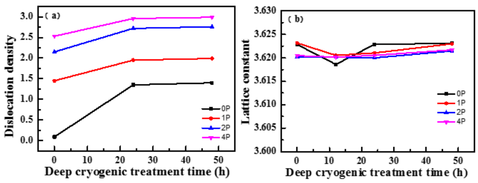

Fig. 7 The curves of the dislocation density and lattice constant of Cu-0.5%Cr alloy by the different passes of ECAP and DCT time

The lattice distortion leads to the increase of subgrain boundaries and grain boundaries, and increases the scattering effect on electrons, resulting in a slight decrease in the electrical conductivity. The similar result was found that the electrical conductivity of Cu decreased after large plastic strains [29]. The electrical conductivity is affected by the vacancy concentration. Some studies have shown that the ECAP process enhances the concentration of vacancy and leads to the decrease of electrical conductivity [30-31]. After the DCT treatment, the volume and lattice shrinkage, the concentration of vacancy decreases, thus improving the electrical conductivity. For the solution treated Cu-0.5%Cr alloy, the lattice constant tends to decrease first and then increase with the increase of DCT treatment time, as shown in Fig.7 (b). Similarly, the lattice constant has the same trend after ECAP + DCT. The lattice distortion makes the electron scattering stronger and reduces the electrical conductivity. The increased electron scattering results in reduced electrical conductivity, which is caused by lattice distortion. The effect of vacancy concentration is greater than the lattice distortion, so before the DCT for 12h, the electrical conductivity shows an upward trend. The former influence factors are opposite, the electrical conductivity has decreased. Therefore, after DCT, the electrical conductivity of Cu-0.5%Cr alloy has a similar trend to that of pure copper [21].

3.3 Tensile properties

In Figure 8 the tensile strength of the solution treated Cu-0.5%Cr alloy is 262MPa. After four ECAP passes, the tensile strength is increased to 451 MPa. After four ECAP passes and the DCT for 12 hours, it arrives at 483 MPa. The increase in the tensile strength of the Cu-0.5%Cr alloy is attributed to grain refinement and increased dislocation density.

Fig.9 (a) shows the tensile strength and elongation to failure of Cu-0.5%Cr alloy processed by the ECAP and DCT for 12h. It can be seen that the tensile strength is significantly improved before two passes of ECAP, and then slightly improved. The elongation to failure decreases significantly after ECAP deformation, as the number of deformation passes further increases**,** it remains basically constant (~16%). After DCT treatment, the tensile strength and elongation at break of Cu-0.5% Cr alloy are improved. After four ECAP passes and the DCT for 12 hours, the tensile strength is 483 MPa, and the elongation to failure is 17.6%.The strength properties of 316l stainless steel rolled at low temperature are significantly improved compared with that rolled at room temperature [32]. Asymmetric rolling at cryogenic temperature provides greater strength tensile properties of pure aluminium than rolling at ambient temperature [33].

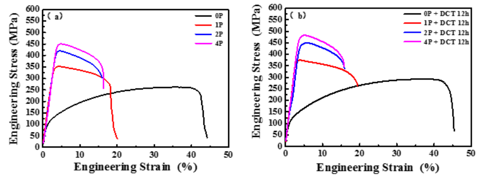

Fig. 8 Curves of tensile engineering stress-strain of Cu-0.5%Cr alloy subjected to ECAP and DCT for 12h: (a) ECAP (b) ECAP and DCT 12h

In general, the tensile strength can be improved by solution strengthening, precipitation, dispersion and work hardening. However, the electrical conductivity will also decrease, and vice versa. Fig. 9 (b) is the variations of the tensile strength and electrical conductivity of Cu-0.5%Cr alloy processed by ECAP and DCT. It can be seen that with the increase of ECAP passes, the tensile strength is inversely related to the electrical conductivity. However, after the DCT treatment, the tensile strength and electrical conductivity of Cu-0.5%Cr alloy is simultaneously improved. Shangina *et al*. [4] showed that the tensile strength of Cu-0.5%Cr after ECAP and aging treatment reached 462 MPa, and the electrical conductivity was 72% IACS. Although the electrical conductivity treated by ECAP and DCT failed to reach a high level (no more than 40% IACS), the combination of ECAP and DCT can balance the relationship between strength and electrical conductivity, which can be further tailored by the aging treatment in future.

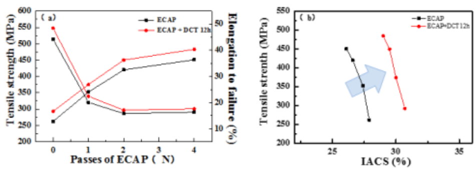

Fig. 9 Tensile strength, electrical conductivity and elongation to failure curves of Cu-0.5%Cr alloy treated with ECAP+DCT for 12 h

CONCLUSION

1) Uniform and fine equiaxed grains were observed in Cu-0.5%Cr alloy with the increase of ECAP passes and the grains were further refined, the dislocation density increased after the subsequent DCT. After 4 passes of ECAP + DCT (12h), the thickness of banded grains were refined to 200 ~ 300nm.
2) After the ECAP and DCT, microhardness and tensile properties were significantly improved due to the grain refinement and accumulated dislocations. The electrical conductivity was enhanced by DCT due to the decrease in lattice constant and the change in vacancy concentration.
3) After 4 passes of ECAP + DCT (12h), the microhardness, tensile strength, elongation to failure and electrical conductivity reached 192HV, 483MPa, 17.6% and 29%IACS, respectively. In comparison to the counterpart, the microhardness, tensile strength, elongation to failure and electrical conductivity are increased by 17.8%, 7%, 8% and 11%, respectively.

4) The process combing ECAP and DCT could simultaneously improve the tensile properties and electrical conductivity of Cu-0.5%Cr alloy.

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