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ARTICLE

Effects of Different Preparation Techniques on Mechanical Property and Electrical Conductivity of Cu-8wt% Ag Alloy by Continuous Casting

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Abstract: Cu-8wt%Ag alloy was prepared by two kinds of preparation techniques, namely continuous casting + cold wire-drawing deformation preparation (CC + CD) and continuous casting + equal channel angular pressing + cold wire-drawing deformation preparation (CC + ECAP + CD), and subsequently the aging treatment was performed. We measured the mechanical property and the electrical property of the Cu-8wt%Ag alloy with different strains, explored the changing rules of the microstructure, the mechanical property and the electrical property by different preparation techniques. Then we analyzed the changing reasons of performance and discussed whether the better comprehensive performance could be acquired by combining the ECAP with the traditional cold-processing. The results show that the comprehensive performance of the Cu-8wt%Ag alloy prepared by CC + ECAP + CD is a little better than that by CC + CD. It is significant and valuable to solve the waxing and waning problem between the high strength and high conductivity of the Cu-Ag alloy.

Key words: Cu-8wt%Ag alloy; continuous casting (CC); equal channel angular pressing (ECAP); cold wire-drawing (CD)

With the rapid development in the electronic industries, the Cu-Ag alloy has been applied to many fields such as microelectronics, transportation, aerospace and mechanical manufacturing, and because of its excellent strength and electrical conductivity, has been the preferred conductor material in products such as the medical equipment, the contact wire of the high-speed train, the strong magnetic field system and the lead frame of the large scale integrated circuit [1-3]. But there is a waxing and waning relationship between strength and conductivity of the Cu-Ag alloy; therefore it is a key research and development direction for researchers to solve the contradictory relationship and to find a kind of material with both of high strength and high conductivity and a preparation technique [3].

In the past forty years, the size of the Cu-Ag alloy in-situ composites with the fibrous microstructure has been down to nanometer through cold-working such as rolling, drawing and forging, and owing to the great compatibility between Cu phase and Ag phase, the strength of mixture is usually more than the theoretical estimate value [2,4]. In addition, severe plastic deformation (SPD), such as equal channel angular pressing (ECAP), high-pressure torsion (HPT), and accumulative roll bonding (ARB), due to the simple process and low cost, has been recognized as one of the most promising technology by international researchers[5-7]. At present, many researchers have focused on the combination between the severe plastic deformation and the traditional cold working or the heat treatment process to obtain better comprehensive performance[8-10]. For example, Young et al.[11] have got a nano structural Cu-3wt%Ag alloy by

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combining the ECAP with the traditional cold working technique, the strength and the conductivity of which are 765 MPa and 86%IACS, respectively. But there are a few literatures about how this combinative technique improves the comprehensive performance of the alloys and the improvement is not clear. Therefore, in the present paper, the Cu-8wt%Ag alloy is prepared by two kinds of preparation techniques, namely CC + CD and CC + ECAP + CD. This paper analyzed its microstructural transformation by the different preparation techniques, researched the effects of its strength, micro hardness and conductivity on the mechanical property and the electrical property and discussed whether the better comprehensive performance could be obtained by combining the ECAP with the traditional cold working technique.

1 Experiment

Bars with Φ8 mm were prepared by the continuous casting, where Ag materials with the purity of 99.5% and 99.99% (Ag content of 8wt%) were continuously cast in a high speed continuous casting furnace of the set temperature 1180 ℃, the vacuum degree 1.1×10^{-2} Pa, pressure 8.6×10^{6} Pa, the cooling water temperature 27.4 ℃ and the speed 130 mm/min. By chemical analysis, Ag content of the alloys was 7.94 wt%.

There were two kinds of preparation techniques designed: CC + CD and CC + ECAP + CD. In the former, bars having been continuously cast were drawn at room temperature to wires with Φ1.078, Φ0.398 and Φ0.148 mm (corresponding with the true strain η = 4.01, 6.02, 7.95). The drawing reduction was expressed by the tensile ratio:

$$\eta = \ln(A_0/A) \tag{1}$$

where, A_0 and A are the initial and the final cross-sectional area of samples, respectively.

In the latter, bars having been continuously cast were processed by a wire cutting machine to cylindrical samples with the diameter Φ8 mm and the length 50 mm. Samples were processed by two passes of ECAP at speed of 5 mm/min and the C path[12] with the best grain refinement effect and the final equivalent strain was 2. The die of the ECAP was made of 9SiCr and its internal angle was 90°. The mixture of MoS_2 and engine oil worked as a lubricant. And then the samples after two passes of the ECAP were drawn at room temperature by the above-mentioned same cold-drawing method followed by the aging treatment at 350 ℃, 15 min to obtain the fine wires with Φ0.148 mm.

The longitudinal section of bars, ECAP samples and drawn-wires was observed by scanning electron microscopy. The corrosive liquid used to be polishing samples was made of 2.0 g $FeCl_3$, 100 mL C_2H_5OH and 5.0 mL HCl. According to the national standard of metallic materials tensile testing at ambient temperature GB-T228-2002, drawn-wires were processed to tensile samples with the scale distance 100 mm. At room temperature, the tensile experiment was carried out at speed of 1 mm/min by an electronic universal testing machine (SHIMADZU AG-X, Japan) to test the mechanical property of samples. The micro hardness of the alloys with the different diameter was measured by intelligent digital micro hardness tester (HXS-1000A). The electrical resistance of the alloys was measured by the DC digital resistance tester (SB2230) and the conductivity can be calculated by the following equations. At 20 ℃, the conductivity (IACS) of the copper wires annealed is 0.017241 Ω mm^2 m^{-1}, as follows:

$$(\%\mathrm{IACS}) = \frac{0.017241}{\rho}\times 100\% \tag{2}$$

$$R = \frac{\rho l}{S} = \frac{4\rho l}{\pi D^2} \Rightarrow \rho = \frac{\pi D^2 R}{4l} \tag{3}$$

where, ρ is the electrical resistivity, Ω mm^2 m^{-1}; R is the electrical resistance, mΩ; l is the length of wires, m, and the testing value is 1 m; D is the diameter, mm.

Eq.(3) into Eq.(2) we got

$$(\%\mathrm{IACS}) = \frac{0.017\ 241\times 1000\times 4}{\pi D^2 R}\times 100\% \tag{4}$$

2 Results and Discussion

2.1 Microstructure

Fig.1 shows the SEM images of the Cu-8wt%Ag alloy under different states. We find through the microstructural evolution of the alloys under different processing states that under the continuous casting state the eutectic phase and the secondary Ag phase are arrayed along the casting direction and form a certain orientation, as seen in Fig.1a. After one pass of the ECAP grains are continuously crushed, slid and rotated to make the Cu matrix, the eutectic phase and the secondary Ag phase elongated along the direction of 45° with the extrusion direction are refined to a large extent, we observe the apparent slip band, as seen the black arrow in Fig.1b.

In the drawing deformation process, with the increase of the true strain, the secondary Ag phase with dot distribution gradually appears in the matrix, and tissue in radial direction is gradually refined while it in longitudinal direction gradually forms fiber along the drawing direction. For the alloy prepared by CC + CD after the aging treatment, the fine secondary Ag phase is precipitated around the fibrous phase, part of which is distorted because the phase boundary partly migrates, as seen in Fig.1c. It demonstrates that recrystallization occurs in tissue, but migration of the grain boundary is never across the initial position of the phase boundary, limiting the size of most of the recrystallization grains within the fibrous dimension, which is called the in-situ recrystallization. The in-situ recrystallization would occur because the pinning effect of the fine relative dislocation precipitated during the aging treatment seriously hinders the nucleation and growth of recrystallization and the geometrical softening of the Cu alloys caused by recrystallization[13]. In the alloy prepared by CC + ECAP + CD followed by the aging treatment, a small amount of larger grains are obviously observed (as seen in Fig.1d). Through the microscope, we observe that the fine recrystallization grains are clearly distinguished, part of the grain boundaries begin to migrate across the phase boundary of the fibrous phase resulting in the phase boundary apparently bending and that some fibrous tissue is partially coarsened and disconnected to form the discontinuous globular grains but the genetic form of the initial fibrous tissue is still foiled [14,15]. Finally, comparing the alloys prepared by two kinds of different preparation techniques after the aging treatment, we find that the fibrous tissue of the alloys prepared by CC + ECAP + CD is obviously finer than that by CC + CD and the secondary Ag phase is diffusely precipitated more obviously.

2.2 Mechanical property

Fig.2 shows the relationship between the ultimate tensile strength & the micro hardness and the wire-diameter of the Cu-8wt%Ag by the different preparation techniques. The alloys which are deformed with the true strain of 2 by the ECAP process are cold-drawn to be different diameter wires with Φ1.078, Φ0.398 and Φ0.148 mm corresponding to the true strain of CD η=4.01, 6.02, 7.95. The alloys without ECAP are cold-drawn to be the same three kinds of diameter wires by the same way. These wires are employed in tests of ultimate tensile strength and HV. As seen in Fig.2, it is apparent that both of the ultimate tensile strength and the micro hardness increase to some extent and the secondary Ag phase gradually transforms to the Ag fibrous bundle with the gradual decrease of the wire-diameter (namely the gradual increase of the true strain). The diameter and the gap between the Ag fibrous bundles in the eutectic fiber gradually decrease with the increase of the strain. Besides owing to the gradual redution of the gap between boundaries the dislocation structure can not exist stably and is absorbed by the boundaries, hindering the proliferation of the dislocation, which is difficult to move to improve the capacity of the grain resistance to plastic deformation. Meanwhile, the strengthening of the alloys are mainly composed of boundary strengthening, Ag fibrous strengthening in eutectic tissue and secondary Ag fibrous strengthening [16,17].

As seen in Fig.2a, it is apparent that the ultimate tensile strength of the alloys prepared by CC + ECAP + CD is obvious higher than that by CC + CD. Specifically, when the wire-diameter is Φ1.078, Φ0.398 and Φ0.148 mm the ultimate tensile strength increases by 22.93%, 15.95% and 9.61%, respectively. After the severe deformation the ultimate tensile strength of the alloys is improved greatly because of the eutectic phase and the Ag precipitation phase in the alloys transforming to the fibrous tissue reinforcement phase, the solid-solution strengthening and the deformation strengthening[11]. Meanwhile, as seen in Fig.2b, it is apparent that the microhardness of the alloys prepared by CC + ECAP + CD is obvious higher than that by CC + CD specifically, when the wire-diameter is Φ1.078, Φ0.398 and Φ0.148 mm the hardness increases by 16.08%, 14.77% and 5.62%, respectively. Consequently, it is an effective criterion for the

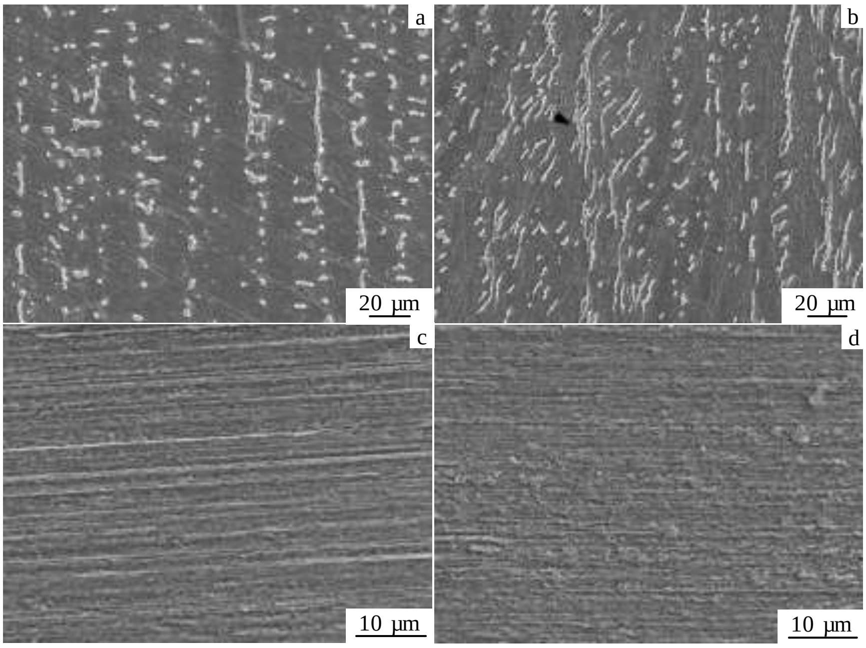

Fig.1 SEM images of Cu-8wt%Ag alloy: (a) continuous casting state, (b) one pass of ECAP, (c) CC + CD + aging treatment, and (d) CC + ECAP + CD + aging treatment

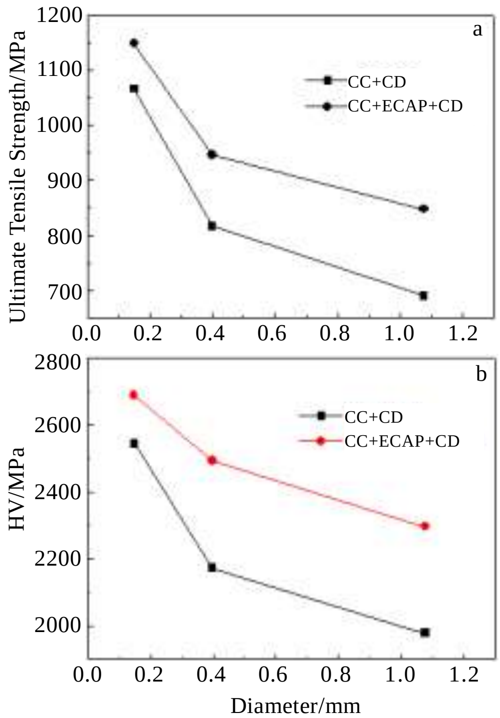

Fig.2 Ultimate tensile strength-wire diameter curves (a) and curves of hardness-wire diameter (b) of Cu-8wt%Ag alloy by different processing techniques

ECAP extrusion technology to improve the mechanical property of the material. Because the grains in the alloys after the ECAP experience the severe shear deformation, the dislocation is continuously rotated and the grains are continuously crushed and refined to reduce the proportion of the grain boundary. Meanwhile, the density is higher with the continuous proliferation of the dislocation, which offers the good initial tissue for the subsequent cold-drawing deformation and also works as the foundation of the final performance of the alloys.

2.3 Electrical property

Fig.3 shows the conductivity calculated by Eq.(3), of the Cu-8wt%Ag alloy by different preparation techniques. It declines with the decrease of the wire-diameter (namely the increase of the cold-drawing true strain η). With the decrease of the wire-diameter, namely the increase of the true strain, the fibration of the tissue in the alloys is more and more apparent, especially during the high strain stage ($\eta > 6$). When the diameter of the fibrous bundles is less than the mean free distance with the continuous decrease of the gap between the fibrous bundles, the dislocation substructure can not exist stably and is absorbed by the boundaries gradually to reduce seriously the dislocation density in the Cu matrix, weakening gradually the scattering effect of the dislocation on the conductive electrons. It is the main factor to the conductivity of the alloys that the scattering effect is strengthened on the interface between the eutectic fiber and the Cu matrix fiber, the interface between the secondary Ag fiber and the Cu matrix and the Cu matrix grain boundary to decrease the electrical resistance of the alloys[18,19].

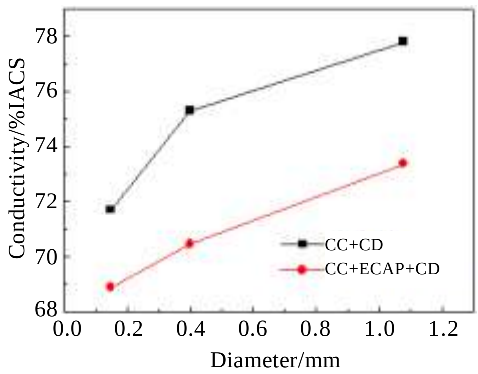

Fig.3 Curves of conductivity-wire diameter of Cu-8wt%Ag alloy with different processing techniques

Comparing the two kinds of preparation techniques, it is found that the conductivity of the alloys prepared by CC + CD is higher than that by CC + ECAP + CD. Specifically, when the wire-diameter is Φ1.078, Φ0.398 and Φ0.148 mm the ultimate tensile strength increases by 6.02%, 6.88% and 4.09%, respectively, as seen in Fig.2. It is a possible reason that the grains of the alloys processed by ECAP before cold-drawing are deformed, rotated and crushed by the shearing force to refine continuously the grains to increase the proportion of the grain boundaries, resulting in strengthening the scattering effect of the surface defects (the grain boundaries and the phase boundaries, etc.). Meanwhile, with the increase of the shear strain, the dislocation of the alloys is also more and more to strengthen the scattering effect of the line defect (dislocation) on electrons. Therefore the conductivity of the alloys is improved by the increase of the scattering value (ρ_{dis}) of the line defect on electrons and the scattering value (ρ_{int}) of the face defect on electrons. Consequently, the alloys processed by ECAP before cold-drawing obtain the microstructure with the high line defect and the high face defect.

2.4 Comprehensive performance

For the general conductive alloys, there is the waxing and waning relationship between strength and conductivity. In the present paper, the mechanical property of the Cu-8wt%Ag alloy prepared by two kinds of preparation techniques improves while the conductivity declines, as seen in Fig.2 and Fig.3. Both of the tensile strength and the micro hardness are improved by the aging treatment. The initial tissue form influences directly the final fiber distribution. Through the aging treatment the strengthening phase in the initial tissue is distributed reasonably, the secondary Ag phase appears significantly near the large angle, the dislocation density gradually declines and the rate of the flow stress and the work

Table 1 Ultimate tensile strength (UTS), microhardness (HV) and conductivity of Cu-8wt%Ag alloy by different preparation techniques before and after the aging treatment

Processing condition	Ultimate tensile strength/MPa		Microhardness, HV/MPa		Conductivity/%IACS	
	Processing state	Aging treatment	Processing state	Aging treatment	Processing state	Aging treatment
CC + CD	1066.333	689.671	2544	1718	71.70	88.50
CC + ECAP + CD	1168.820	708.327	2687	2015	68.88	90.05

hardening are continuously reduced. When the desolvation of the solid-solution is basically completed, the conductivity tends to be constant[20]. As a result, the conductivity of the alloys is greatly improved while the strength is decreased.

Table 1 shows the performance comparison between the Cu-8wt%Ag alloys prepared by different preparation techniques before and after the aging treatment when the wire-diameter is Φ0.148 mm. By investigating the effect of two kinds of different preparation techniques on the mechanical property and the electrical property, we find that the improvement of the mechanical property of the alloys prepared by CC + ECAP + CD is more than that by CC + CD while the conductivity declines. But finally after the aging treatment the conductivity of the alloys by the two preparation techniques is both increased while the mechanical property declines slightly. In addition, the comprehensive performance of the alloys prepared by CC + ECAP + CD is a little better than that by CC + CD. Specifically, the tensile strength, the micro hardness and the conductivity of the alloys processed by ECAP are increased by 2.71%, 17.29% and 1.75%, respectively, as seen in Table 1.

Although the ECAP extrusion process is not easy to, it does improve the comprehensive performance of the alloys from the above data. Therefore, it is a significant and valuable method to the alloys prepared by CC + ECAP + CD.

3 Conclusions

1) After one pass of ECAP the grains are continuously crushed, slid and rotated to elongate every phase along the direction of 45 ° with the extrusion direction and are refined to a large extent. After the aging treatment the fibrous tissue of the alloys prepared by CC + ECAP + CD is apparently finer than that by CC + CD and the secondary Ag phase is diffusely precipitated more obviously.

2) The conductivity of the alloys prepared by CC + ECAP + CD is lower than that by CC + CD while the tensile strength and the micro hardness are much better. It indicates that the ECAP process affects sensitively the mechanical property and the electrical property of the alloys to the ECAP process. The grains in the alloys after the ECAP experience the severe shear deformation and are continuously crushed and refined, which offers the initial microstructure to increase the strength and to decrease the conductivity of the alloys.

3) After the aging treatment, the comprehensive performance of the Cu-8wt%Ag alloy prepared by CC + ECAP + CD is a little better than that by CC + CD. Consequently, the ECAP extrusion process is not easy to industrially produce, but it does improve the comprehensive performance of the alloys. It is significant and valuable to solve the waxing and waning problem between the high strength and high conductivity of the Cu-Ag alloy.

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不同制备工艺对连铸 Cu-8%Ag 合金力学性能及导电率的影响

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摘 要：采用2种制备工艺，即连铸+冷拉丝加工工艺(CC+CD)和连铸+等径角挤压+冷拉丝加工工艺(CC+ECAP+CD)制备Cu-8%Ag合金(质量分数)，随后进行时效处理。分别测试了不同应变量下Cu-8%Ag合金的力学性能和电学性能，探究了不同制备工艺下合金的显微组织、力学性能及电学性能的变化规律，分析了影响其性能变化的原因，并讨论了ECAP技术与传统冷加工技术相结合是否可以获得更好的综合性能。结果表明：采用CC+ECAP+CD制备的Cu-8%Ag合金的综合性能略高于CC+CD制备的合金。这对解决Cu-Ag合金的高强度与高导电率之间此消彼长的问题具有一定的研究意义和应用价值。

关键词：Cu-8wt%Ag 合金；连铸(CC)；等径角挤压变形（ECAP）；冷拉丝(CD)

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