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Article

Grain Refinement and High-Performance of Equal-Channel Angular Pressed Cu-Mg Alloy for Electrical Contact Wire

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Abstract: Multi-pass equal-channel angular pressing (EACP) was applied to produce ultrafine-grained (UFG) Cu-0.2wt%Mg alloy contact wire with high mechanical/electric performance, aim to overcome the catenary barrier of high-speed trains by maximizing the tension and improving the power delivery. Microstructure evolution and overall properties of the Cu-Mg alloy after different severe-plastic-deformation (SPD) routes were investigated by microscopic observation, tensile and electric tests. The results show that the Cu-Mg alloy after multi-pass ECAP at 473 K obtains ultrafine grains, higher strength and desired conductivity. More passes of ECAP leads to finer grains and higher strength, but increasing ECAP temperature significantly lower the strength increment of the UFG alloy. Grain refinement via continuous SPD processing can endow the Cu-Mg alloy superior strength and good conductivity characteristics, which are advantageous to high-speed electrification railway systems.

Keywords: severe-plastic-deformation (SPD); contact wire; Cu-Mg alloy; ultrafine grained (UFG); conductivity; strength

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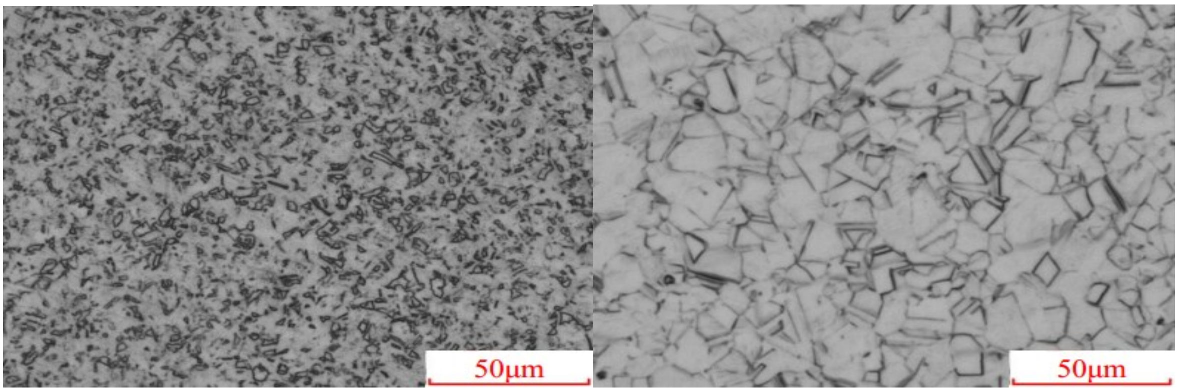
1. Introduction

With the rapid development of high-speed electric railway, mainly, advanced metallic materials used for contact wire are desired. At present, the task of increasing the speed of the trains (≥300 km/h) makes high-performance copper contact wire become a research focus. In this situation, copper alloy are usually required to possess high tensile strength (above 550 MPa) and good conductivity (about 60% IACS), with the aim to overcome the catenary barrier of high-speed trains by maximizing the tension and improving the power delivery. As well known, high strength and good conductivity of metals are often mutually exclusive. It is important to note that some abnormal methods have been reported to achieve a good combination of strength and conductivity [1,2], but those methods are relatively complicated in commercial applications. Most of the reports were focused on adding small amounts of alloy elements (such as Cr, Ag, Zr, Nb, Co, *etc.*) to pure copper [3–6] or applying hardening processes (such as drawing or rolling) [6]. Those conventional strengthening methods induce various kinds of defects (dislocations, reinforcing phases, point defects, grain boundaries), raising electrical resistivity of Cu alloy because of the scattering of conducting electrons [7,8]. For example, a Cu-0.7%Cr-0.3%Fe alloy, after cold working (40% CW), plus aged at 450 °C for 90 min, had the ultimate tensile strength of 460 MPa and an electrical conductivity of about 67% IACS (International Annealed Copper Standard, 17.24×10^{-9} Ωm is defined as 100% IACS) [9].

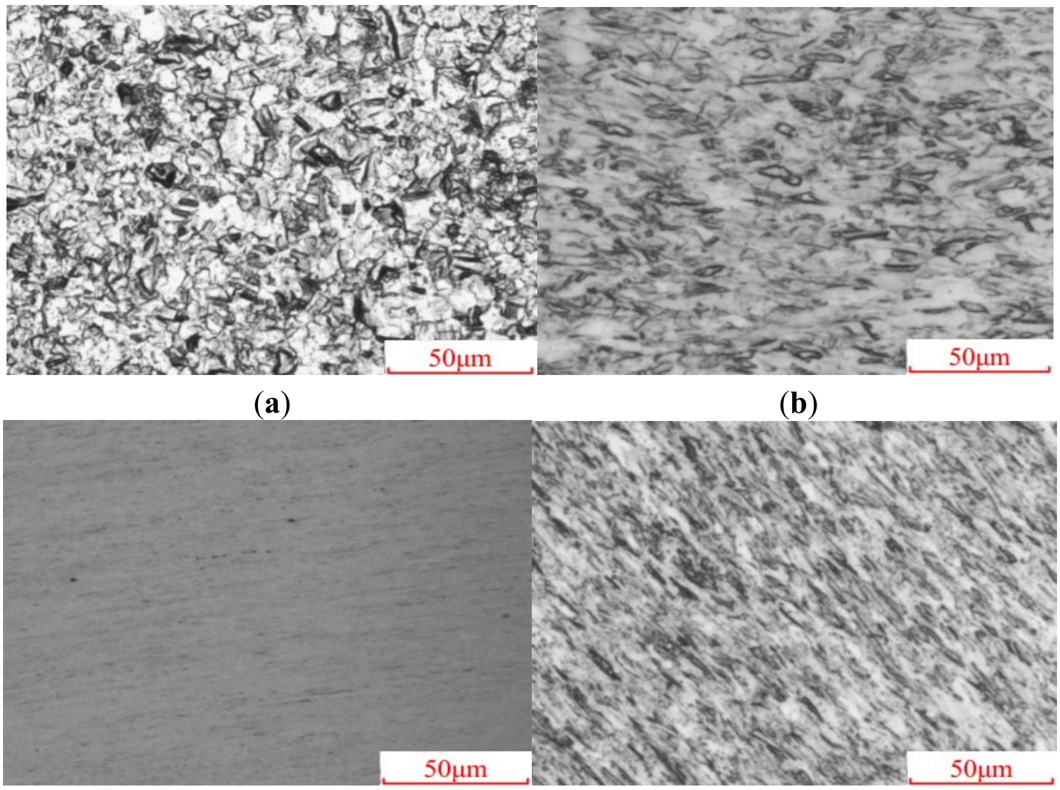
In view of the deteriorative effect on the conductivity of copper, the alloying content and hardening degree should be restricted during the fabrication of high-strength and good-conductivity copper alloy. Grain refinement of single solid-solution copper alloy (e.g., Cu alloy containing a small amount of Mg) could be an effective way to overcome the contradiction. At present, China Railway Construction Electrification Bureau Group (Kang Yuan New Materials Co., Ltd.) has developed fine-grained Cu-0.4wt%Mg contact wire by Conform-process plus cold drawing. Figure 1 presents the optical microstructure of the Cu-0.4wt%Mg alloy after Conform-process and subsequent cold drawing. The product achieved a good combination of ultimate tensile strength (UTS: 522 MPa) and conductivity (68.6% IACS), and was successfully applied in the high-speed railways of Zhengzhou-Xi'an and Korea. Recently, a novel severe-plastic deformation (SPD) procedure, namely equal-channel angular pressing (ECAP), has been applied for obtaining ultrafine-grained (UFG) copper alloy with high strength, large ductility, and good electrical conductivity [10–12]. In the present work, the SPD process was combined with the manufacturing processes of Cu-Mg alloy contact wire with a lower Mg content (about 0.2 wt% Mg), with the aim to achieve further improvement in conductivity and strength. Under the same deformation conditions, the average grain size of the Cu-0.2wt%Mg alloy (in Figure 2a) is obviously finer than that of the Cu-0.4wt%Mg alloy (in Figure 1a). Until now, there have been few reports on mechanical and conductivity properties of UFG Cu-Mg alloys fabricated by SPD methods. Herein, this paper studies the microstructure change, tensile strength, and electrical conductivity of the on-line conformed Cu-Mg binary alloy subjected to experimental multi-pass ECAP, and investigates the origin of the good characteristics.

Figure 1. Optical micrographs of Cu-0.4wt%Mg alloy by Conform-process (**a**) and subsequent cold drawing (**b**) for contact wire products.



(**a**) (**b**)

Figure 2. OM (Optical Micrographs) of Cu-0.2wt%Mg alloy (**a**) in Conform state and after ECAP at 473 K for (**b**) 1 pass, (**c**) 4 passes and (**d**) 16 passes.



(**a**) (**b**)

(**c**) (**d**)

2. Experimental Section

The material used was Cu-0.2wt%Mg (oxygen ≤10 ppm, 1 ppm = 10^{-6}) alloy, which was prepared by the upward-casting and then extruded by Conform-process in China railway construction electrification bureau group (Kang Yuan New Materials Co., Ltd., Jiangyin, China). Mg atoms of the binary alloy mainly exist in the FCC (Face-Center-Cubic)-structured copper crystal. The dimension of the ECAP billet was 19.5 mm × 19.5 mm × 40 mm. The ECAP process was carried out using a rotary die with an intersection angle of 90°, which details are described in previous references [13,14]. The billets were extruded from 1 pass to 16 passes at 473 K and 673 K, respectively. An Olympus BX51M optical microscope (OM, Olympus Corporation, Tokyo, Japan) was used to observe the microstructure at the flow plane of the ECAPed billets. The composition of the etchant was glacial acetic acid 25 mL, phosphoric acid 55 mL, and nitric acid 20 mL. The etching time was 5 s. A JEM-2000EX transmission electron microscope (TEM, JEOL Ltd., Tokyo, Japan) was applied to observe the microstructure and the grain size of the Cu-Mg alloy after ECAP.

Microhardness of the ECAPed sample was measured by HXD-1000TC device (Taiming Optical Instrument Co., Ltd., Shanghai, China) under a load of 100 g for 15 s. Tensile specimen with a dimension of 3 mm × 3 mm in cross-section, and 15 mm in gage length, was cut from the billet along the longitudinal direction. Tensile tests were performed by a RGM-4050 testing machine at an initial strain rate of 1000 μm/s at room temperature, and three samples cut from one billet were tested for each state. After tensile testing, the fracture surfaces were observed by a HITACHI S-3400N scanning electron microscope (SEM, HITACHI Ltd., Tokyo, Japan).

A QJ36S digital apparatus (Shuangte Electrical Instrument Co., Ltd., Shanghai, China) was implemented for the direct current (DC) electrical resistance measurement via a four-point probe method. The test samples with a dimension of 3 mm × 3 mm × 3 mm were cut from the billets along the longitudinal direction, and their surfaces were polished before the electrical resistance measurements.

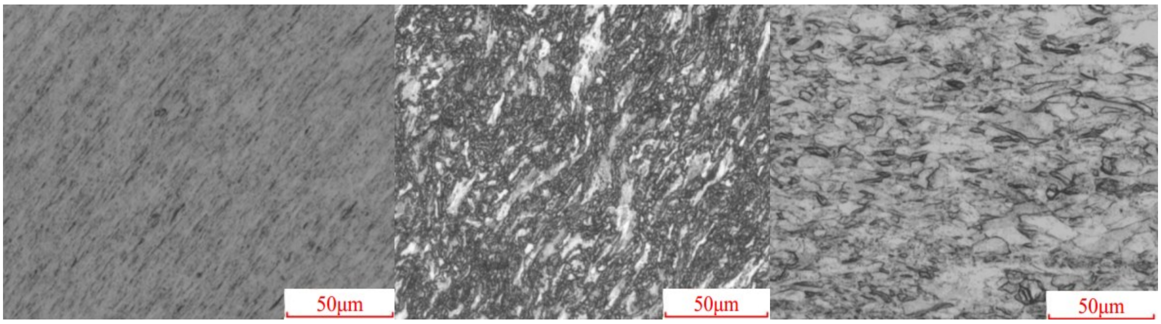
3. Results and Discussion

3.1. Microstructure

Figure 2 presents the optical micrographs of the Conformed (as-achieved) alloy and the ones after ECAP at 473 K for different passes. As shown in Figure 2a, the average grain size of the Conformed sample was about 5–8 μm. There are only equiaxed α-Cu grains in microstructure of the alloy. After continuous ECAP processing, the grains were gradually refined and elongated with increasing the number of ECAP passes. The grain size was hard to measure when the sample was subjected to 16 passes of ECAP. Only fine strain-induced plastic flows can be observed by optical microscopy. The detailed microstructure of the 16-pass ECAPed copper alloy should be observed by TEM.

Figure 3 presents optical micrographs of the Cu-0.2wt%Mg alloy after ECAP at 673 K for one pass, four passes and 16 passes, respectively. Similar to the samples after ECAP at 473 K, the α-Cu grains were fined and elongated, however, the efficiency of grain refinement during ECAP at 673 K was not as good as that at 473 K. This phenomenon can be contributed to the faster grain recovery and recrystallization at 673 K [15]. Compared with the current product of Cu-0.4wt%Mg contact wires processed by Conform plus cold-drawing (in Figure 1), the grains of the Cu-0.2wt%Mg after Conform plus multi-pass ECAP are obviously finer.

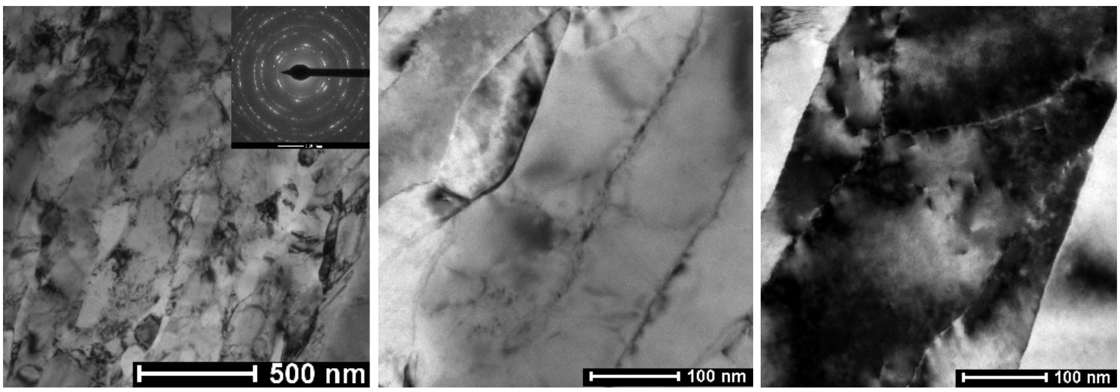
Figure 3. OM micrographs of the Cu-0.2wt%Mg alloy after ECAP at 673 K for (**a**) 1 pass, (**b**) 4 passes and (**c**) 16 passes.



(**a**) (**b**) (**c**)

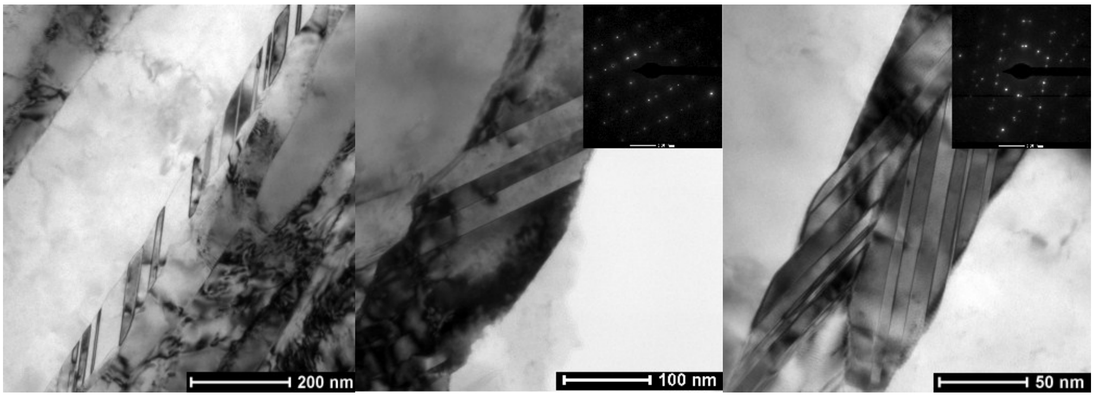
Figure 4 presents TEM micrographs of Cu-0.2wt%Mg alloy after 16 passes of ECAP at 473 K. It can be seen that Mg element is completely dissolved into the Cu matrix and the dislocation density in the ECAPed alloy is pretty low (Figure 4a,b). Meanwhile, the grain size has already been reduced to about 200 nm. The corresponding SAD patterns (upper-right insertion of Figure 4a) are almost continuous diffraction rings, indicating the existence of a majority of high angle grain boundaries. Figure 4c presents dislocation cell structure in the ECAPed alloy. These cells may form individual subgrains upon further plastic straining. As well known, dislocation tangling is frequently observed in the interior of grains, where the grain is heavily strained [16]. There are also some nano-twins in particular grains, as can be seen in Figure 4d–f. Nano-twins were created by the shear stress and severe strain during the ECAP process.

Figure 4. TEM microstructure of the ECAPed Cu-0.2wt%Mg alloy after 16 passes at 473 K. (**a**) Elongated ultra-fine grains observed at low magnification, (**b**) Elongated ultra-fine grains observed at high magnification, (**c**) characteristics of grain boundaries, (**d**) twins and intragranular dislocations, (**e**) characteristics of twins observed at high magnification and (**f**) characteristics of twins observed at the higher magnification.



(**a**) (**b**) (**c**)

Figure 4. *Cont.*

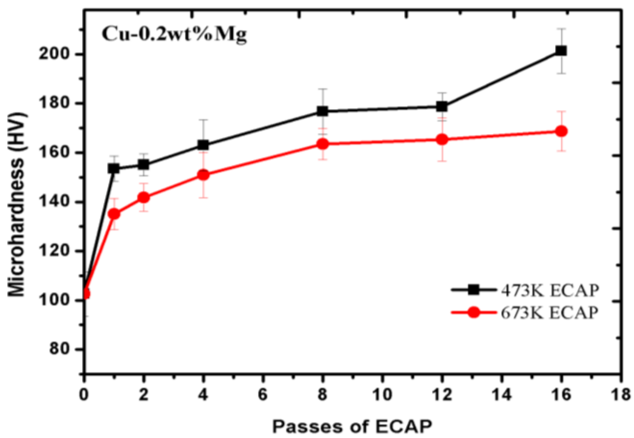


(d) **(e)** **(f)**

3.2. Microhardness

Figure 5 presents the Vickers microhardness of Cu-0.2wt%Mg alloy subjected to different passes of the ECAP processing. Firstly, it can be seen that all the samples ECAPed at 473 K have higher hardness values than those of the samples at 673 K with the same ECAP passes. This is due to more obvious strain hardening and grain refinement at the lower ECAP temperature. Secondly, the results show an obvious increase in hardness from the first pass to four passes. The rapid increase of hardness at initial passes seems to be attributed to strain hardening rather than grain refinement in the initial stage [17]. Thirdly, the hardness value of the sample ECAPed at 673 K has a slight increase with more passes. The reason might be that the materials reached the steady-state density of dislocation and dynamic recovery occurred in the grains of Cu-Mg alloy [18]. While the hardness value of the sample ECAPed at 473 K obviously increase after 16 passes. This could be due to the efficient grain refinement and the slight dynamic recovery happened at 473 K.

Figure 5. Microhardness of Cu-0.2wt%Mg alloy variation with the passes of ECAP at 473 K and 673 K.



3.3. Tensile Properties

Figure 6 shows engineering stress-strain curves of UFG Cu-0.2wt%Mg alloy processed by ECAP at different temperature. The as-achieved sample in Conform state shows high ductility and low strength, while the ECAPed samples exhibit much higher strength with adequate ductility. This might be caused by two reasons. Firstly, the grains in Conform state are relative large and equiaxed. A smaller grain size leads to a higher mechanical strength, which is widely known as the Hall-Petch effect. Secondly, the dislocation was annihilation in the dynamic recovery caused by the high temperature during Conform process. After multi-pass ECAP, the strength of the alloy was improved significantly, but the elongation was obviously decreased. The increase of strength attributes to strain hardening and grain refinement at a few passes. Ultrafine grains with high-angle grain boundaries impeded the motion of dislocations, which is the main reason for strength improvement of the ECAPed Cu-Mg alloy. Simultaneously, the twin lamellas in grains may act as barriers to largely reduce the dislocation mean free path, and, thus, further harden the alloy. Moreover, the strain hardening and the elongated grains caused by multi-pass ECAP reduced the ductility of the Cu-0.2wt%Mg alloy.

Figure 6. Stress-strain tensile curves of the samples subjected to various passes ECAP at 437 K and 673 K.

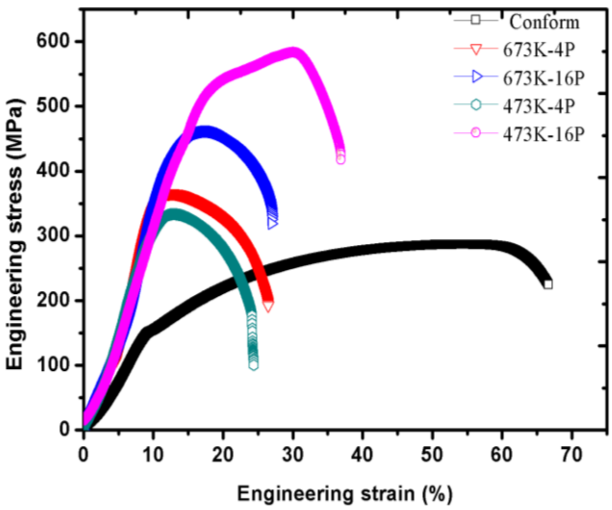


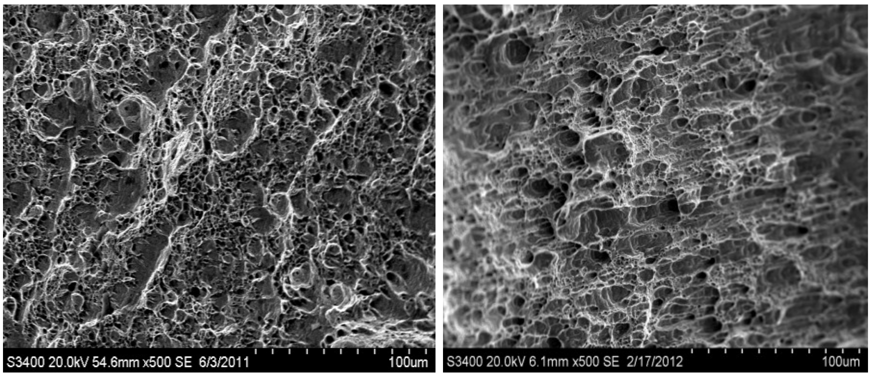
Table 1 lists microhardness and tensile properties of the samples subjected to ECAP at 473 K and 673 K. Firstly, the 16-pass samples exhibit higher strength and better ductility than the four-pass samples ECAPed at the two temperatures. This may be induced by the further refined grains and a more uniform distribution of grains. Secondly, the sample after 16 passes of EACP at 473 K obtained a higher strength and better ductility than that at 673 K. The 16-pass sample at 473 K results in a high tensile strength (583.4 MPa), good total elongation (37.9%), and high microhardness (201.23 HV). The improvement in strength may be caused by the obstruction of the large-angle grain boundaries and twin-grain boundaries to the dislocation movement. The better ductility may be induced by the deformation mechanism change from dislocation slip to grain-boundary sliding (GBS) [15].

Table 1. Microhardness and tensile properties of the samples ECAPed at 473 K or 673 K.

Sample	Microhardness (HV)	Ultimate tensile stress (MPa)	Total elongation (%)
Conform	102.6	286.4	66.7
473 K-4 P	162.9	333.2	24.7
473 K-16 P	201.2	583.4	37.9
673 K-4 P	150.9	365.5	22.5
673 K-16 P	168.6	461.1	26.9

SEM observation was done to clarify the failure mechanisms in the ECAPed Cu-Mg alloy. Figure 7 presents SEM morphologies of the fracture surfaces of the alloy subjected to 16 passes ECAP at two different temperatures. Some round and equiaxed dimples are observed on the fracture surface of the 16-pass sample ECAPed at 637 K (Figure 7a), which is a typical ductile fracture. This kind of fracture occurs due to microvoid formation and coalescence [17]. Some elongated dimples are seen at the fracture surface of the 16-pass sample ECAPed at 437 K (Figure 7b). This result occurs due to internal shearing between voids and seems to be governed by a simple shear deformation. The difference of dimple pattern between the two ECAPed samples may stem from the deformation mechanism change from dislocations slip to GBS. This phenomenon is attributed to the extremely small volume of the ultrafine grains with large angle boundary, and these grains are more inclined to form 45° slip plane under the applied stress (corresponding to Schmid's law). Therefore, GBS is much easier to happen than dislocation slip for the UFG Cu-Mg alloy.

Figure 7. SEM fracture surface of the samples subjected to 16 passes ECAP at (**a**) 637 K and (**b**) 473 K.

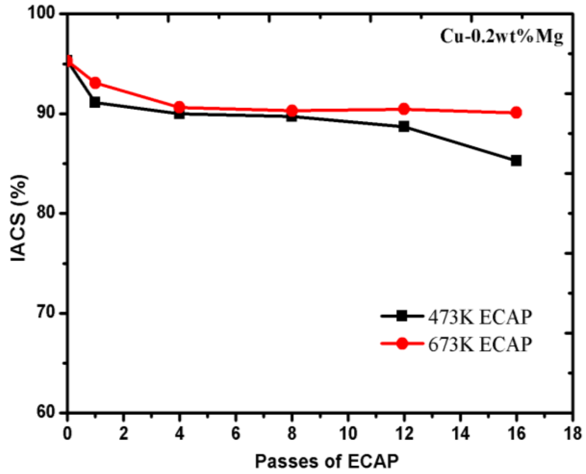


(**a**) (**b**)

3.4. Conductivity

The conductivity of the Conformed alloy after different ECAP passes were evaluated and is shown in Figure 8. It can be found that the conductivity of the alloy decreases when increasing the ECAP passes. The decrease of conductivity is attributed to the increase of grain boundaries, dislocations and large-angle grain boundaries caused by grain refinement. As is well known, grain boundaries and dislocations can increase the scattering of conducting electrons, leading to the increase of the electrical resistivity of the metal [8]. In addition, the large-angle grain boundaries have a large effect on the scattering of conducting electrons, greater than that of the low-angle grain boundaries [19].

Figure 8. Conductivity of Cu-0.2wt%Mg alloy variation with the passes of ECAP at 473 K and 673 K.



Compared with the ECAPed samples at 673 K, the conductivity of the ECAPed samples at 473 K are obviously lower. This phenomenon should be caused by more grain boundaries (including large-angle grain boundaries) and dislocation multiplication during the ECAP process executed in lower temperature. Compared with the product of Cu-0.4wt%Mg contact wires fabricated by Conform + drawing process, the Cu-0.2wt%Mg alloy after Conform + 16-pass ECAP at 473 K has the higher conductivity (84.5% IACS). This good result should be attributed to the lower dislocation density and lower lattice strain after multi-pass ECAP processing.

Thus, it can be seen that grain refinement via multi-pass ECAP processing can endow the Cu-0.2wt%Mg alloy with superior strength and good conductivity characteristics, which are advantageous to high-speed electrification railway systems. This new technique can be easily integrated into the manufacturing processes of Cu-Mg alloy contact wire (*i.e.*, after the Conform process), of which successful application makes the trains safe at higher speeds.

4. Conclusions

(1) Multi-pass ECAP processing, compared with cold drawing, improves grain refinement effect of the Conformed Cu-0.2wt%Mg alloy. More passes of ECAP leads to finer grains, and the grain size of the 16-pass sample ECAPed at 473 K is about 200 nm.

(2) Compared with the as-achieved sample in Conform state, the ones after multi-pass ECAP exhibit much higher strength with adequate ductility. The ECAPed samples for 16 passes have much higher strength and better ductility than those for less passes.

(3) With increasing the ECAP pass, hardness and strengthen of the ECAPed samples increased obviously but the conductivity decreased gradually. However, the conductivity of the Cu-0.2wt%Mg alloy after Conform plus ECAP is still much higher than that of the current Cu-0.4wt%Mg product processed by Conform plus cold drawing.

(4) Conform plus ECAP provides a simple and effective procedure to obtain high strength and good conductivity Cu-Mg alloy, in comparison with current Conform plus cold drawing. After Conform plus ECAP for 16 passes at 473 K, Cu-0.2wt%Mg alloy exhibits superior tensile

strength (583.4 MPa), adequate total elongation (37.9%), good conductivity (84.5% IACS), and high hardness (201.2 HV).

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Author Contributions

The work presented here was carried out in collaboration between all authors. A. Ma, C. Zhu and J. Jiang defined the research theme. A. Ma, C. Zhu and D. Song designed methods and experiments, carried out the laboratory experiments, analyzed the data, interpreted the results and wrote the paper. J. Chen, S. Ni and Q. He co-designed experiments, discussed analyses and interpretation. All authors have contributed to, seen and approved the manuscript.

The author hopes that this paper can make its due contribution to successful application of high-strength and high-conductivity Cu-Mg alloy contact wire.

Conflicts of Interest

The authors declare no conflict of interest.

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