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Texture evolution and strengthening mechanism of single crystal copper during ECAP

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ABSTRACT

For finding a process for improving the strength of single crystal Cu while still maintaining good plasticity and electrical conductivity, the die with an internal angle (Φ) = 105° and an outer curvature angle (ψ) = 37°was used to prepare the equal channel angular pressing (ECAP) deformation by route Bc. The deformation behavior of single crystal copper during ECAP was investigated through electron backscatter diffraction (EBSD), optical microscope (OM) and X-ray diffraction (XRD). The mechanical properties and electronic conductivity after deformation were tested. The results show that the texture evolution is {111} < 112 > (intital)→ {111} < 110 > → {111} < 112 > → {112} < 110 > during 4 passes deformation. The tensile strength and the hardness of single crystal copper increased 123% and 115% respectively after four passes, the electrical conductivity has no obvious decrease. This result indicates that ECAP method can significantly improve the strength of single crystal copper, simultaneously, the plasticity and conductivity of the material remains at an excellent level.

1. Introduction

The rapid development of electrical engineering field requires higher performance materials. Single crystal copper attracts much attention due to its excellent electrical conductivity and good plasticity [1–3], but the lack of strength seriously hinders the wide application of materials. Using traditional methods strengthen the material while sacrificing its plasticity and electrical conductivity, such as fine-grains strengthening [4], a large number of defects introduced by refining grains enhance the scattering of moving electrons, which results in a decrease in the conductivity of materials. Finer grains are unable to store more dislocations, leading to the decrease of the plasticity. According to the classical plastic theory, the plasticity of materials depends largely on the ability of work hardening affected by the dislocation storage ability [5]. Therefore, the reasons for the plasticity reduction are as follows: Firstly, the introduction of dislocations at the initial deformation stage leads the materials cannot continue to accumulate dislocations, resulting in the reducing of the plasticity [6]. Secondly, from the macroscopic mechanics of view, the nanocrystalline material undergoes plastic instability at the initial deformation, resulting in a significant decrease in plasticity [5]. Thirdly, the grain refinement greatly increases the strength of materials, but the decrease of the grain size makes the dislocations easily annihilate at the grain boundary, further reducing plasticity [7,8]. Therefore, it is necessary to find a process for improving the strength of single crystal Cu while still maintaining good plasticity and electrical conductivity.

Early studies [9,10] found that nano-sized twins can achieve a high degree of matching between strength and plasticity meanwhile the electrical conductivity is also maintained a high level, and single crystal with low stacking fault energy and special orientation can introduce directional twinning via controlling strictly loading mode under low temperature by equal channel angular pressing (ECAP). Purcek et al. [11] studied the properties of Cu–Cr–Zr alloy after 8 passes ECAP deformation and following aging, and found that the tensile strength increased from 189 MPa to 688 MPa and the conductivity unchanged. In addition, Wang et al. [12] improved the strength and plasticity of the pure cooper simultaneously via introduced a bimodal grain size distribution including nano-grains and micro-sized grains by ECAP + cold rolling, wherein high strength is derived from nano-grains and larger grains are provided good plasticity.

The materials mechanical and physical properties are obviously affected by the microstructure, and the microstructure evolution in deformation can be reflected by texture [13,14]. Therefore, it is necessary to study the influence of texture evolution for materials

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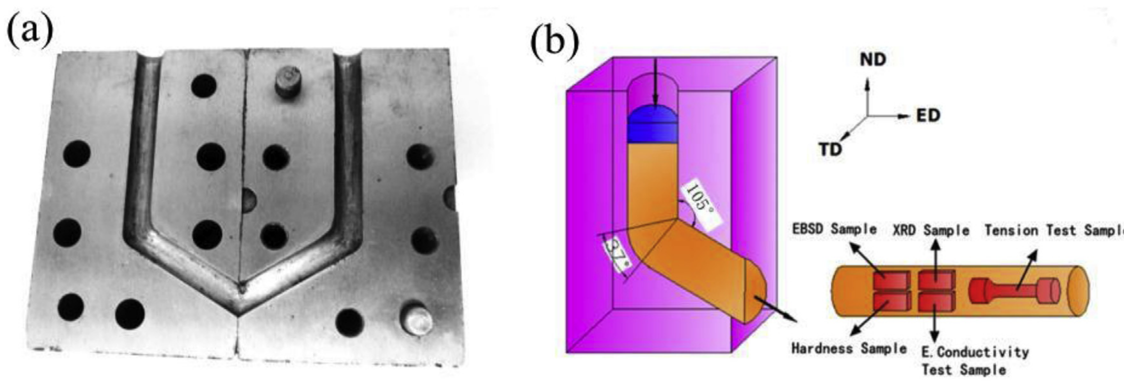

Fig. 1. (a) Schematic figure of the tie (b) Principle of equal-channel angular pressing.

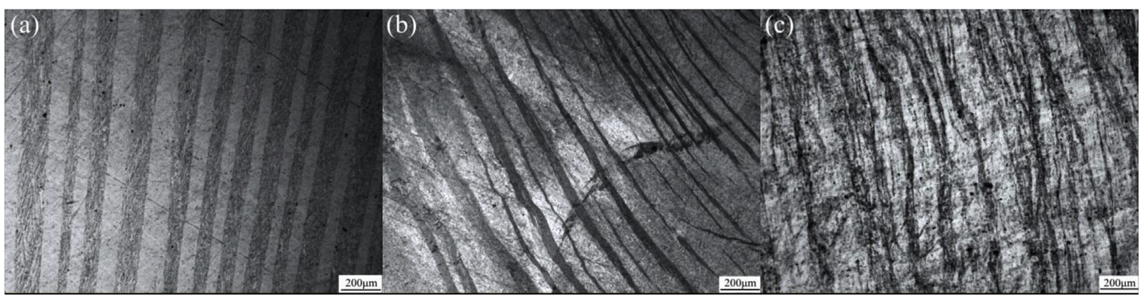

Fig. 2. Optical micrographs of single crystal Cu by (a) 1pass, (b) 2 passes, (c) 4 passes ECAP.

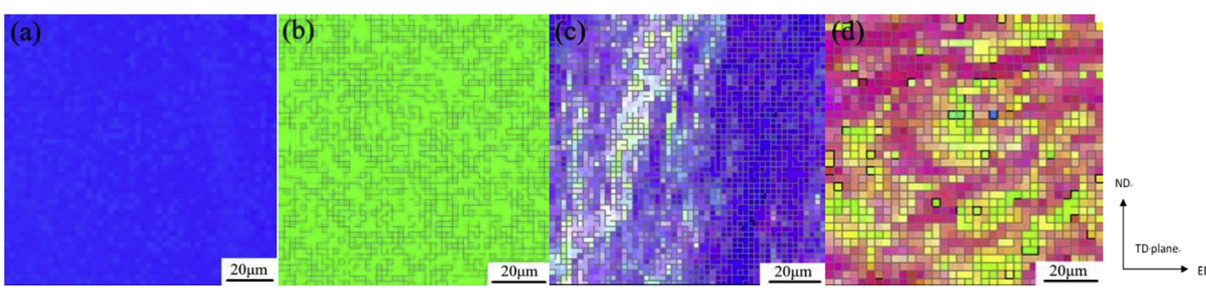

Fig. 3. Orientation image microscopy (OIM) images of single crystal coppersamples ECAP after different passes by route Bc: (a)0 pass(b)1 passes(c)2 passes(d)4 passes.

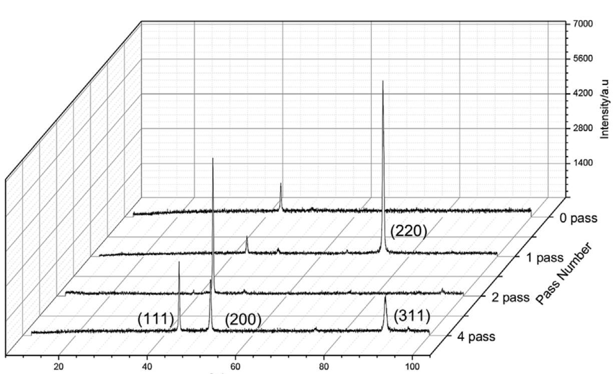

Fig. 4. XRD patterns of single crystal copper after ECAP by route Bc.

properties during the ECAP process. Early study [15] found that loading mode (die angle and deformation route), deformation mechanism and initial orientation significantly affect the evolution of texture. Katayama et al. [16] extruded four differently oriented single crystal Cu samples by one pass ECAP deformation and found the initial crystal orientation, the slip surface and the slip direction significantly affect the formation of shear bands. The loading method mainly affects a change in the ideal positions of the components [17–20]. Study [21] showed that texture evolution is closely related to material properties, but studies are mostly directed at the morphology of texture and little at the influence of texture evolution on material properties. Therefore, it is significant that studying the relationship between them.

2. Experimental materials and procedure

Single crystal rods with high-purity (99.999%) copper was prepared by Ohno Continuous Casting (OCC) method. The rods were cut in a diameter of 16 mm and length of 80 mm. The extrusion was conducted at room temperature using a die with angle of 105°and outer arc of 37°up to 4 passes by route Bc(as shown in Fig. 1). The MoS2 lubricant was used in order to decrease the friction between the die and the sample during the ECAP processing. Following the ECAP processing, the specimens were sliced perpendicular to the pressing direction as shown in Fig. 1(b). EBSD was conducted by Quanta FEG-450 thermal field emission scanning electron microscope to obtain the single crystal copper's microstructures and texture which after ECAP passes. Samples were prepared in the conventional procedure, which start from mechanical grinding and ending up with electrolytic polishing (500 mL distilled water; 250 mL CH3CH2OH; 250 mL H3PO4, 5 g $CO(NH2)_2$ and 50 mL $(CH3)_2CHOH$ mixed solution, polishing voltage 4 V, polishing

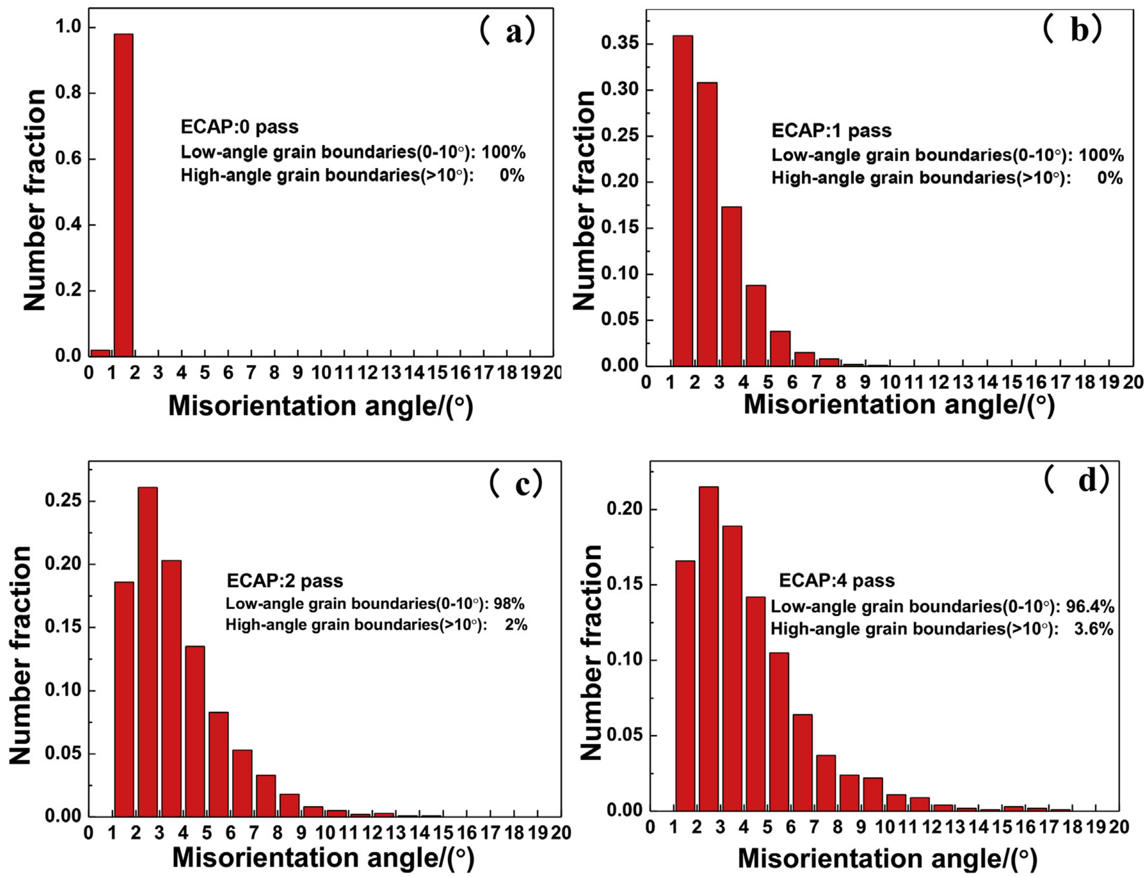

Fig. 5. Misorientation angle distribution of single crystal copper ECAP after different passes by route Bc: (a)origin (b)2 passes (c)4passes.

time was 6min). D8ADVANCE X-ray diffractometer were used to characterize the macroscopic orientation evolution. Using WDW-300D microcomputer controlled electronic universal testing machine to finish the tensile tests.

3. Results

3.1. Microstructure characterization

3.1.1. OM characteristic

The optical microstructures of single crystal Cu after ECAP are shown in Fig. 1. As shown in Fig. 2(a), it can be seen that there are large numbers of parallel-arranged shear bands about 75 μm in the matrix after one pass deformation, and the distance between each shear band is almost equal. Inspection of Fig. 2(b) shows that the shear bands about 60° from the horizontal direction appear after 2 passes, they are thinner and the spacing is no longer equal compared with the previous. After 4 passes, as shown in Fig. 2(c), the shear band further refine and the distance becomes smaller. It is easily seen that the shear bands produced at post can cut and cover the previous ones as the extrusion pass increases, and the shear bands are continuously refined with the increase of the strain.

3.1.2. OIM (Orientation image microscopy)

Fig. 3 shows an microstructure of single crystal Cu after 1, 2, and 4 passes of ECAP. Inspection of Fig. 3 (a) indicates that the original single crystal Cu has a highly uniform (111) orientation. After 1 pass ECAP, the orientation changes from (111) to (101) direction, and large numbers of low angle grain boundaries (LAGBs) appear at the same time as shown in Fig. 3(b). There are shear bands oriented at (111) (211) introduced in the grain after 2 passes, which are about 15° and 40° respectively with the ND axis (Fig. 3(c)). After 4 passes, the shear bands broken and formed island structure meanwhile the orientation of the materials is segregated in (100) and (210) directions. It can be seen from the OIM that no clear boundaries appeared in the grains and the misorientation of each region is smaller after 2 passes, which means no high angle grain boundaries (HAGBs) appears. But after four passes, the orientation of the material has significant change and clear boundaries had been appeared, which shows that the grains tend to be broken and a few HAGBs appeared with the increase of extrusion pass.

3.2. X-ray diffraction (XRD) analysis

XRD diffraction patterns of initial single crystal copper and processed by 1, 2 and 4 ECAP passes on route Bc are shown in Fig. 4. From the figure, the undeformed sample has only a (111)diffraction peak. After single pass, (111) peak intensity decreased obviously, and the (220) diffraction peak that the intensity is much higher than (111) peak appears. After 2 passes, (220) diffraction peak disappears and (111) peak intensity increased significantly. After 4 passes deformation, (111) peak intensity decreases meanwhile (200) and (311) diffraction peaks appear, which means there is no obvious macro orientation. It can be seen that the main diffraction peak of each pass will change during the deformation process, it shows that the deformation has obvious cross slip characteristics, which leads the proliferation of dislocations.

In the process of ECAP deformation, different diffraction peaks increase alternately, and finally multi diffraction peaks is formed, which is due to the increased lattice strain and decreased crystallite size. Furthermore, it is because the shear surface and the orientation factor of each pass change constantly in route Bc. The fluctuation of the diffraction peaks is attribute to the different slip systems start in turn. It is easily seen from the OIM and XRD pattern that the material macro-orientation has been disordered after 4 passes extrusion, but the texture transition needs further study.

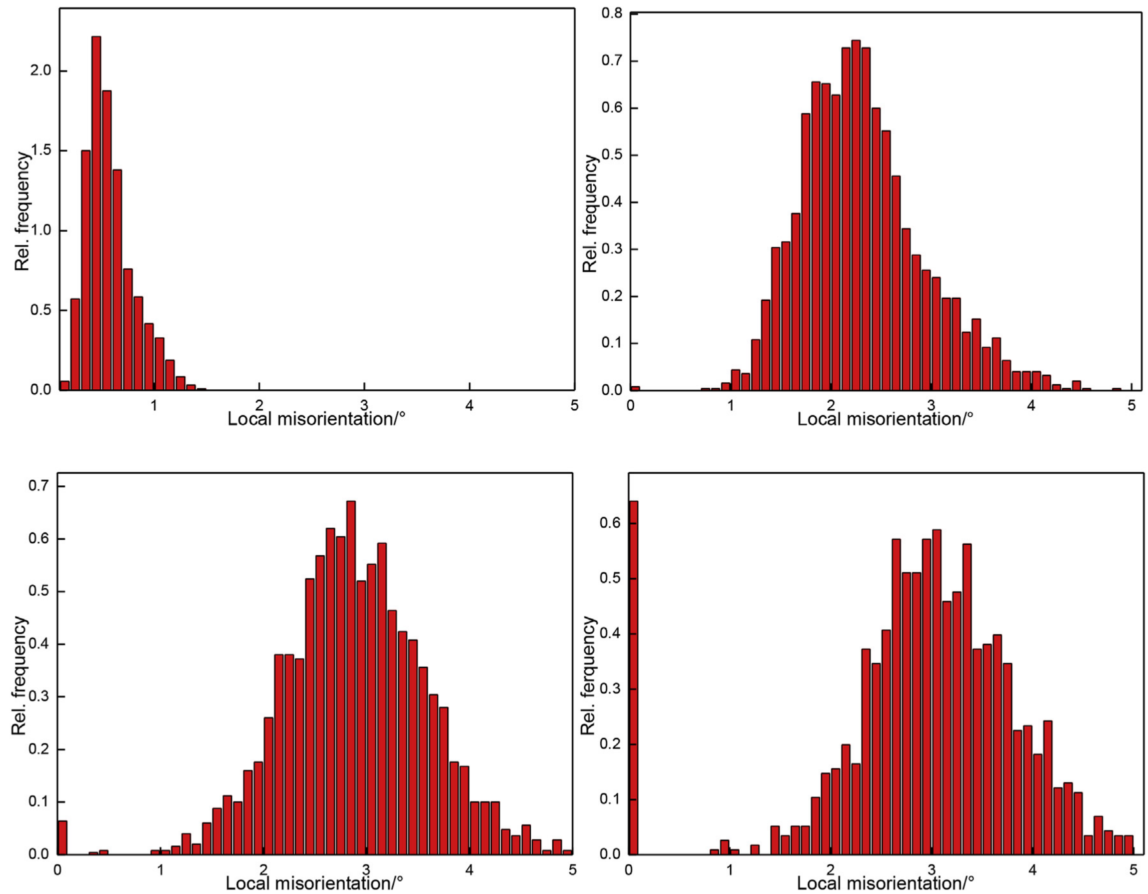

Fig. 6. Local misorientation of single crystal copper ECAP after different passes by route Bc: (a) original state (b) 1 passes (c) 2 passes (d)4 passes.

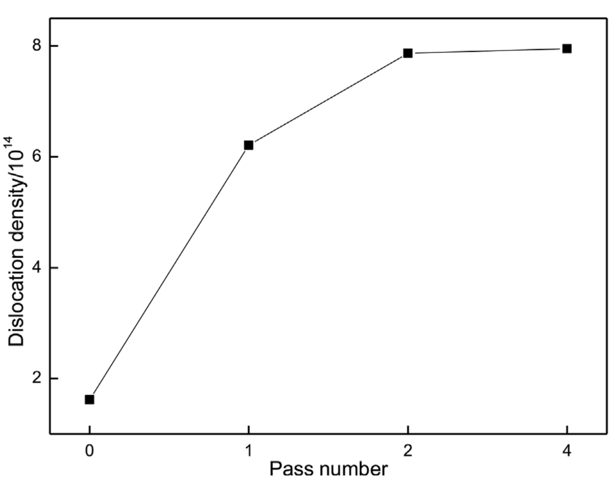

Fig. 7. The GND density of single crystal copper ECAP after different passes by route Bc.

3.3. Grain boundary characteristics

3.3.1. Grain boundary angle

Fig. 5 shows the histograms of grain boundary angle distribution of single crystal copper after 1, 2 and 4 passes deformation. It can be seen that the misorientation of the GBs in original single crystal copper is under 2°, which is a dislocation interface formed by the tangle of dislocations generated during solidification of single crystal copper. After single ECAP pass, a large number of LAGBs exist and a strong maximum is between 1 and 2° as shown in Fig. 5 (b), which shows that the new GBs appeared first in forms of sub-grain boundaries or LAGBs. After 2 ECAP passes, the misorientation of the GBs increases gradually meanwhile the peak value of GB angle changes from 1-2° to 2–3°, and a small number (2%) of HAGBs appear. Inspection of Fig. 5 (d) indicates that more than 10° of GBs further increases to 3.6% after 4 ECAP passes, but LAGBs increases little. From the change of the GBs angle in the ECAP deformation process, the grains are not broken after 4 ECAP passes. The GBs angle raise with the increase of ECAP passes due to the dislocations stacking. After ECAP, a large number of dislocations appear and intertwine tangle to form dislocation cells. With the increase of ECAP passes, dislocation continue proliferate and form sub-grain boundary and eventually developed into HAGBs.

3.3.1. Dislocation density

Fig. 6 shows the histograms of local misorientation of single crystal copper in original state and after 1, 2 and 4 passes deformation. Local misorientation refers to the difference in orientation between any data point and adjacent data point within each grain of EBSD surface scan data. And the geometrically necessary dislocation (GND) density in the crystal can be calculated from Kubin and Mortensen method by using local misorientation of EBSD data [22]:

$$\rho_{GND} = \frac{2\nu}{bu}$$

Where b is the magnitude of Burgers vector, u is the unit length and ν is kernel average misorientation (KAM) that is obtained directly from EBSD data [23]. The calculated GND density of the single crystal copper by ECAP is shown in Fig. 7. It is easy to see that the dislocation density in the crystal gradually increases as the amount of strain increases. The dislocation density of original single crystal Cu reaches 1.62×10^{14}m-2. After 1 pass deformation, the dislocation density sharply increases to 6.21×10^{14}m-2. During the subsequent deformation process, the

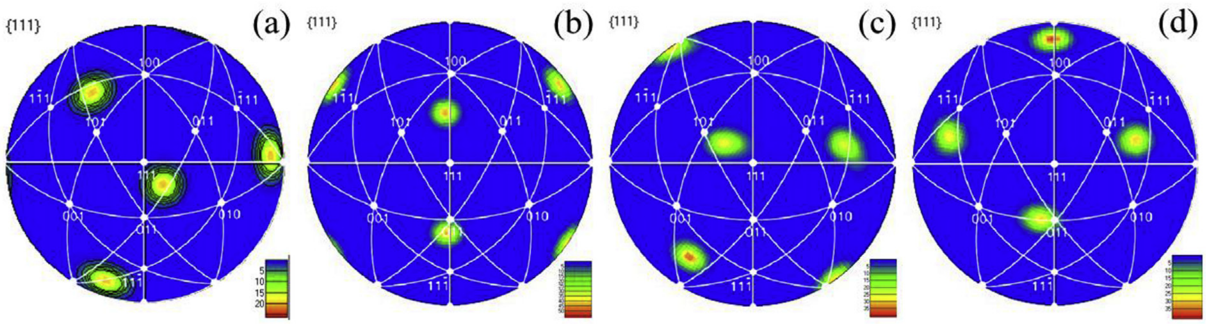

Fig. 8. {111} pole figures of samples ECAP after different passes: (a) Initial condition; (b) 1 pass; (c) 2 passes; (d) 4 passes.

dislocation density is further increased and it reaches 7.95 × 10^{14}m-2 after 4 passes deformation.

3.4. Texture evolution

3.4.1. Pole figure analysis

Fig. 8 shows that the {111} polar figure of single crystal copper in initial and after 1, 2 and 4 passes. As can be seen from the {111} polar figure in Fig. 8(a), the pole density in initial state is 26.44 and the orientation is particularly concentrated. After single pass, the orientation rotates 30° counterclockwise around the < 111 > axis and gradually changes from {111} to {101} and {100} due to the spreading of the shear plane, the pole density changes into 56.91. After 2 ECAP passes, as shown in Fig. 8(c), the orientation rotates counterclockwise continuously and concentrates on {101} direction, the orientations produce slight dispersion, in the same time the pole density declines slightly to 41.24. Inspection of Fig. 8(d) shows that orientation is segregated on {100} plane and pole density increased slightly to 41.94. It can be seen that the crystal orientation is almost not dispersed, the deflection of the grain orientation occurs instead after 4 passes, which shows that the material still maintains the characteristics of single crystal orientation throughout the deformation process.

3.4.2. Analysis of orientation distribution function(ODF)

Fig. 9 shows the orientation distribution function of single crystal copper in initial and after 1, 2 and 4passes. From Fig. 8(a) (φ2 = 45°), it can be seen that the original single crystal copper has very strong {111} < 112 > texture. After single pass, the initial orientation disappears and transforms into {111} < 110 > texture. After 2 passes, it tends to {111} < 112 > texture. After 4 passes deformation, it is {112} < 110 > texture. The shear surface of each pass is greatly changed due to using the route Bc, so the texture also changes after each pass. But the change of texture is not remarkable before 2 passes. With the increase of the deformation to 4 passes, the texture changes obviously but is single still. The single texture indicated that the grains are not fragmented, and it is confirmed that the idea of single crystal strengthening is feasible via ECAP.

3.5. Mechanical properties and conductivity

Fig. 10 shows the mechanical properties and conductivity of single crystal copper in initial and after 1, 2, 3 and 4 passes. As can be seen from Fig. 10(a), the tensile strength of single crystal copper increases from 168 MPa to 290 MPa after one pass, and finally reaches 375 MPa after 4 ECAP passes with the increases of extrusion passes. The elongation of single crystal copper drops from 63% to 32% after single deformation of ECAP, but after 2 ECAP passes, the reducing amplitude also decreases gradually, and it finally reaches 24.5%. The hardness increases to 129.5 Hv at the first two passes, then there is small fluctuations in the later deformation process.

With the increase of ECAP passes, the conductivity decreases gradually. After single pass, the conductivity changed to 99.2% IACS. After 2 ECAP passes, the conductivity is decreased to 99% IACS. After 4 ECAP passes, the conductivity continues to decrease to 98.3% IACS, but the decreasing trend of conductivity gradually slows down. After 16 passes, it is 97.6% IACS, and the conductivity loss rate is less than 3%.

4. Discussion and summary

It can be seen from Fig. 7(b) that the conductivity decreases slightly and fluctuates under high strain, and the conductivity is still 97.6% IACS after 16 passes. In fact, there are many factors that affect the conductivity, such as defect density, orientation angles of grains, texture and stress distribution. The decrease in conductivity is mainly due to the increase of defect density during the deformation, especially the vacancy density. The lattice distortion caused by it will greatly increase the scattering effect on free electrons, which is much higher than other defects. Secondly, the effect of grain orientation angle on conductivity depends on the value of its angle. The conductivity of single crystal copper is higher than polycrystalline copper because single crystal copper eliminates the scattering effect of grain boundaries on electrons. However, it will enhance the scattering of electrons when sub-GBs or GBs appear during the deformation, and the value of orientation angle is larger, the scattering effect is stronger. Thirdly, the influence of texture on electrical conductivity is not clear, early research [1] proves that stable < 110 > fiber texture can reduce the electron scattering of the GBs, resulting in a slight increase on the conductivity of the material. Actually, the influence of texture on electrical conductivity depends on the texture orientation. When the texture orientation is just the atomic close-packed or close-packed direction, the number of free electrons is the most, and the conductivity of the material is also the highest. Therefore, {111} < 110 > texture is beneficial to the conductivity of face centered cubic (fcc) crystal. Moreover, the tensile stress increases the atomic spacing, which leads to an increase in lattice distortion, resulting in a decrease in electrical conductivity; On the contrary, the compressive stress increases the electrical conductivity. It is not difficult to see that the change in conductivity is the result of a combination of several factors, some of which can improve the electrical conductivity, and the others can decrease it. So the electrical conductivity fluctuates under high strain.

The transformation of macro texture affects the mechanical properties of the material, which is reflected in the resolved shear stresses of the corresponding slip system. When the Schmid factor (SF) of the slip system corresponding to the texture formed after deformation, the material is in a soft orientation. It is advantageous for the slip. And the material tends to be hard oriented when the SF is small or tends to zero, and it is difficult to the slip. In the initial deformation stage, the single crystal Cu is oriented at the (111) atomic close-packed plane. The material is in a soft orientation, and the work hardening level is low. After 4 passes, a stable {hkl} < 110 > texture orientation is formed, and the SF of the slip system corresponding to the {hkl} < 110 > texture is reduced at this time, which indicates that the crystal

ECAP.

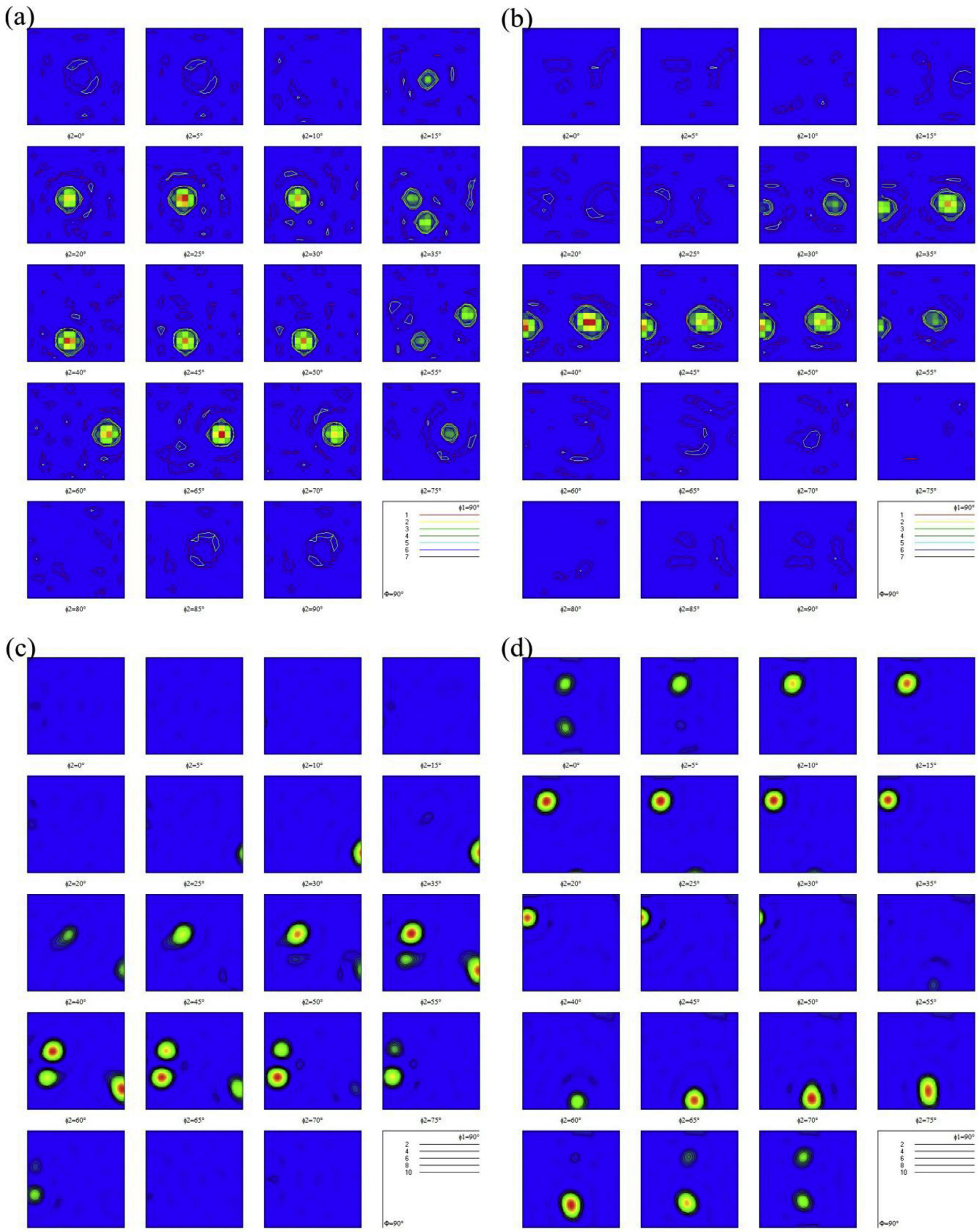

Fig. 9. Orientation distribution function (ODF) of samples ECAP after different passes by route Bc: (a) Initial condition, (b) 1 pass, (c) 2 passes, (d) 4 passes.

orientation turns to the direction that is not conducive for the activation of the slip system during extrusion. Therefore, the stressmust be increased for maintaining the dislocation motion and the secondary slip system is activated to coordinate the deformation simultaneously, which leads to the work hardening and the hardness and strength are sharply enhanced. Zheng et al. [24] also found it tends to a stable

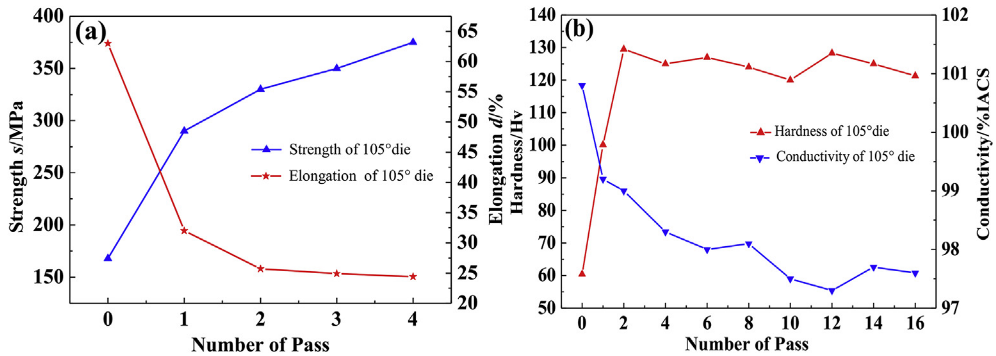

Fig. 10. (a)Mechanical properties of different extrusion pass(b)Relationship between extrusion pass and hardness, conductivity.

orientation of < 110 > after stretching of the single crystals, which is beneficial to improve the work hardening rate of the material. The recrystallized core is formed by the combination of subgrain boundaries produced under high strain during the deformation process. As the deformation increases, the recrystallized core gradually grows and dynamic recrystallization occurs, which lead to a large number of dislocations disappearance and the deformation resistance of the material decreases. Therefore, the hardness of the material shows a fluctuating trend finally.

Studies [25–27] have shown that the dislocation density inside the crystal is positively correlated with the strength of the material, and it is consistent with our experimental results. It can be seen from Fig. 9 that the dislocation density raise when the material strength increases after deformation. In fact, the rise in material strength is due to the dislocation entanglement between each other after the proliferation of dislocations, which hindering the start of slip. The dislocation propagation mode of single crystal copper is cross-slip proliferation mechanism, which is different from other FCC metals such as Al, Al–Cu alloy. It is known from Fig. 4 that there exists obvious cross-slip in crystal during the deformation, and screw dislocations form a edge jog after cross slipping, and the jog is not on the slip surface of the primary dislocation, so it cannot move together with the primary dislocation, which leads that the primary dislocation is pinned, and make the primary dislocation become a Frank-Read dislocation source to produce the new dislocation.

In addition, the strengthening method of single crystal copper is work hardening, but compared with polycrystalline materials, there is a certain difference of the method between two materials. It is known from Figs. 3(a) and 5(a) that there exist a small number of low-angle dislocation interfaces in a single crystal generated in the solidification process. Since the misorientation of these interface are low and the quantity is small, thus, in the early stage of deformation, the main obstacles to dislocation motion are vacancies and other dislocations. As the strain increases, the jog caused by the dislocation proliferation pinches the dislocation, simultaneously, a large number of dislocations entangle each other to form dislocation interfaces and LAGBs to hinder the dislocation motion. However, the obstruction of LAGBs on dislocations is weaker than that of HAGBs with high misorientation, so the work hardening of single crystal copper is weaker than polycrystalline material under the same strain.

5. Conclusion

(1) The strength of single crystal Cu increased from 168 MPa to 375 MPa by ECAP after 4 passes and keeps rising, simultaneously, the plasticity and conductivity of the material remained at an excellent level.
(2) The grain retains the characteristics of single crystal material throughout during the deformation processes, which is one of the reasons why single crystal Cu still has good plasticity and excellent conductivity after ECAP deformation.
(3) The change in conductivity is the result of a combination of several factors such as defect density, orientation angles of grains, texture and stress distribution, so the electrical conductivity fluctuates under high strain.
(4) The dislocation propagation mode of single crystal copper is cross-slip proliferation mechanism, and there is a certain difference of the strengthening method between single crystal copper and polycrystalline material, which leads that the strengthening of single crystal copper is weaker than polycrystalline material under the same strain.

Author contributions

Tingbiao Guo, Shiru Wei, Chen Wang, Qi Li, and Zhi Jia conceived and designed; Tingbiao Guo, Shiru Wei, Chen Wang, and Qi Li carried out the experiments and data collection; Tingbiao Guo, Shiru Wei, and Zhi Jia analyzed the data; Tingbiao Guo contributed reagents/materials/analysis tools; Shiru Wei wrote the paper.

Data availability

Data used in this study is the results of observations or experimentation that validate research findings. The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.msea.2019.05.042.

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