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ARTICLE

Texture Evolution and Strengthening Behavior of Single Crystal Copper During Equal Channel Angular Pressing

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Abstract: ECAP was adopted in this study to reinforce the single crystal copper by route A, Bc and C, and the effect of different routes was studied. The texture of single crystal copper during ECAP with a die channel angle Φ=120° and ψ =37° was investigated by electron backscatter diffraction (EBSD). At the same time, the elongation and strength of the material were tested. The results show that after 5 passes, the tensile strength and elongation of route A, Bc and C are 405 MPa and 30%, 395 MPa and 26.7%, and 385 MPa and 27.9%, respectively. After 6 passes, the textures, which are formed using the route A to complete the extrusion, are translated from the initial {111}<112> texture to {112}<110> and weaker {110}<112> textures. The texture of route Bc is {001}<110>, while the texture of route C is dispersed and the coexistence of many kinds of textures appears. Meanwhile, the conductivity of the materials decreases little. It can be seen that ECAP can significantly reinforce the material without obviously decreasing the conductivity. Different extrusion routes can remarkably influence the properties of the material.

Key words: equal channel angular pressing; electron backscattered diffraction; texture; single crystal copper

With the rapid development of the modern industry, the demand for excellent electrical conductivity and strength of materials is quickly increasing. Single crystal copper has been widely used and involved in various fields due to its excellent electrical conductivity[1-3]. However, the tensile strength of it is substantially lower than that of the polycrystalline copper, which seriously restricts the application of the material. Therefore, the reinforcement and toughening of single crystal copper become an urgent problem to be solved. For metallic materials, high strength and high electrical conductivity are mutually exclusive in nature[4]. The traditional strengthening and toughening methods, such as grain refinement and alloying [5-7], are unable to reach the expected requirements, for the reason that alloying may increase the resistance of single crystal copper while decrease the conductivity of the materials[8]. Li[4] et al reported that the tensile strength and conductivity of Cu-Zr alloys can reach 602.04 MPa and 81% IACS by ultra-low temperature rolling, respectively. Ko[9] et al obtained a Cu-3%Ag alloy with a tensile strength of 765 MPa and a conductivity of 86% IACS by the combination of ECAP and low temperature cold rolling. Through grain refinement, a large number of grain boundaries (GBs) perpendicular to the direction of the electron transport were produced, which may result in the electron scattering and decrease the conductivity of the material. From previous studies, it can be found that the conductivity of the materials prepared by these methods is still greatly improved. Therefore, the reinforcement of single crystal copper through severe plastic deformation (SPD) is a feasible technique. Equal channel angular pressing (ECAP) as a SPD method[10,11] has been successfully applied to many materials[12,13]. Controlling the ECAP deformation mode aims at ensuring that the material always maintains the monocrystal characteristics. At the same time, controlling the amount of deformation can effectively control the number of lateral grain boundaries, and the lower lateral GBs will enable the materials to obtain more excellent electrical conductivity and higher

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strength simultaneously.

Previous studies have showed that different deformation modes can have different effects on the microstructure and strengthening in material[14,15]. During ECAP, high density shear bands will cause the materials to form a special orientation by the pressure of route A. On route Bc, microstructure with the cellular structures will be formed, and grains are divided into a large number of dislocation sub-grains. The deformation on route C will make the edge deformation smaller, so the organization still tends to be unevenly distributed. From the Furukawal's studies, we can conclude that the route Bc has a more powerful strengthening effect than the route A and C.

The texture of materials can reflect the microstructure and its evolution[16,17]. Therefore, the texture study of the material is very important. So, EBSD was used to test micro texture features in the present paper. Simultaneously, the mechanical properties and electrical conductivity of single crystal copper were also investigated.

1 Experiment

The experiments were conducted using single crystal rods of high-purity (99.999%) copper with a diameter of 16 mm and a length of 80 mm, which were processed using YT071-100 A hydraulic machine with different modes: route A, Bc and C. The structures are depicted schematically in Fig.1, showing a vertical section through the ECAP die and sampling place, containing a channel bent through an abrupt angle of Φ=120° and ψ=37°. The EBSD sample, hardness sample, electrical conductivity sample and tension test sample are shown in Fig.1a. In order to reduce the friction between the contact surface of the die and the samples, the crystals were pressed using a MoS2 lubricant at a pressing speed of 20 mm·s^{-1}. The die structure and assembly drawing are shown in Fig.1b and 1c.

Tensile tests were carried out on the WDW-300D microcomputer controlled electronic universal testing machine at a tensile rate of 1 mm/min until the samples were fractured. The tension samples with the dimensions of Φ5 mm×20 mm were machined from the ECAP-processed billets along the extrusion direction (ED).

The microstructures of the single crystal copper after several passes through three routes of ECAP were revealed by electron backscatter diffraction (EBSD). The surface of the detector is the transverse direction plane of the material. The EBSD measurements were carried out using HKL system in Quanta FEG-450 scanning electron microscope (SEM), which has a field emission gun. Samples were prepared following the conventional procedure, starting from mechanical grinding (using 800#~7000# metallographic sandpaper and 0.5 μm diamond abrasion paste to complete the polish) and ending up with electrolytic polishing (500 mL distilled water, 250 mL CH_3CH_2OH, 250 mL H_3PO_4, 5 g $CO(NH_2)_2$ and 50 mL $(CH_3)_2CHOH$ mixed solution, polishing voltage 4 V and polishing time 6 min).

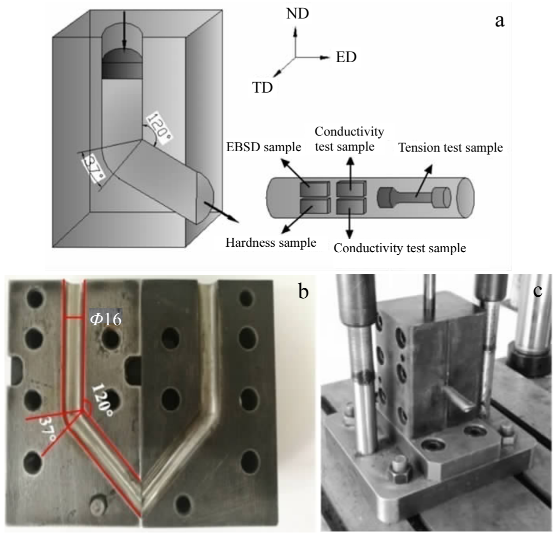

Fig.1 Principle of equal-channel angular pressing (a), die structure (b), and actual assembly drawing (c)

2 Results and Discussion

2.1 Microstructures after ECAP processing

In order to obtain the microstructure of single crystal copper during ECAP, the samples were tested by EBSD. Orientation images were gained simultaneously. Fig.2 shows the OIM images of single crystal copper in initial un-pressed condition and ECAPed by 1, 2, 4 and 6 passes through route A, Bc and C. Fig.2a shows the OIM image in un-pressed condition. It clearly shows that the color-coded image is blue based on the color code triangle shown in Fig.2, which corresponds to a <111> direction. Besides, there is no grain boundary in the image.

After single ECAP pass，the orientation changes slightly and turns into <112> direction, but there is no obvious deformation structure. For the testing location is in the middle of the sample, the structure is reasonably similar to the initial orientation[18]. There are a few different orientation areas in the Fig.2b, which are unit cell rod-like microstructures.

After two ECAP passes, the images of route A (Fig.2c) and route C (Fig.2i) are similar in microstructure, and a few different orientation structures with direction of <110> appear, while other regional orientations also change significantly to the<100>direction, but no deformation band appears simultaneously. The single crystal copper was not broken. Fig.2f shows that the deformation bands appear and have a deflection of 30° compared to the ND direction. This is due to a change in the shear plane, resulting in deflection of the deformation band. Compared with the single ECAP pass, after 2 passes ECAP by route Bc, in the Fig.2f, most areas'

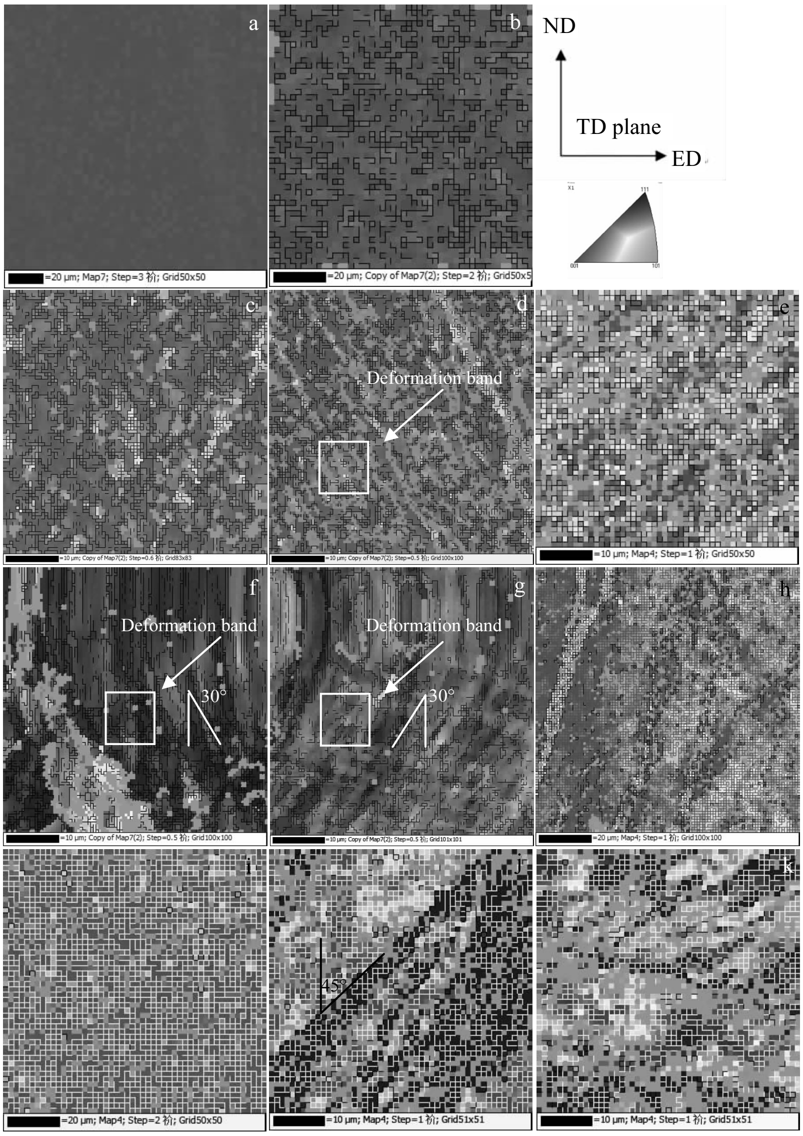

Fig.2 Orientation image microscopy (OIM) images of single crystal copper samples after ECAP for several passes through different routes: (a) 0 pass, (b) 1 pass, (c) 2 passes of route A, (d) 4 passes of route A, (e) 6 passes of route A, (f) 2 passes of route Bc, (g) 4 passes of route Bc, (h) 6 passes of route Bc, (i) 2 passes of route C, (j) 4 passes of route C, and (k) 6 passes of route C

orientation changed little. At the same time, there are island regions which have completely different orientations from other areas.

After 4 passes of ECAP by route A, deformation band also appears in Fig.2d. In Fig.2g, the direction of deformation band has an opposite deflection to the 2 ECAP passes, but the angle is still 30°. Compared with the orientation of two ECAP passes, microstructure orientations of 4 passes on route A and route Bc do not change much. The 30 μm wide deformation band appears in the microstructure on route C and the angle between it and ND direction is 45°. Most of the deformation bands are oriented in the original direction of <111>, also including island structures with different orientations. Other regions which do not belong to the deformation band in Fig.2i tend to <210> directions.

After 6 ECAP passes, the orientations of microstructure on route A and route C tend to disperse. The orientation of microstructure on route Bc is still strong, tending to the <100> direction. Besides, some clear bands of deformation still can be seen, and have the <110> direction.

Combined with the OIM images, it can be found that the original orientation of the material is changed least and it is easier to form the deformation band using the route Bc for ECAP, while the orientation is changed more obvious by route A and C. But more specific differences need further study.

There are no clear regional boundaries after 4 passes of ECAP through route A and Bc, and the disorientation of each region is smaller. There is no large angle grain boundary whereas sub-grain and small angle grain boundary appear. In Fig.2i, there are great differences in the orientation of the two regions, and a clear dividing line between them appears. Thus, a large angle grain boundary may appear. At the same time, it is proved that when the route C is used for pressing, the dislocation is more easily accumulated to form sub-crystal. After 6 passes, the orientation of route A is completely dispersed, which results in large angle grain boundaries. There are still some orientation trends in Fig.2h and 2k, which are pressed by route Bc and C.

2.2 Mechanical properties and conductivity

The mechanical properties of the sample after ECAP can accurately reflect the strengthening degree of ECAP process for single crystal copper. Therefore, the tensile and hardness tests were carried out. Fig.3a shows the mechanical properties and elongation for the sample in initial un-pressed condition and after 1, 2, 3, 4, and 5 ECAP passes on route A, Bc and C. As can be seen from the image, the conductivity gradually decreases and the strength increases after ECAP. After two ECAP passes, the sample which adopts the route C has the highest tensile strength of 340 MPa, and the next is the route A with 330 MPa. The sample of route Bc has the lowest strength of 320 MPa. After three ECAP passes, the tensile strength of route A is much higher than that of the other two ECAP passes, reaching 405 MPa. Before 4 ECAP passes, the best elongation belongs to the sample using route C, followed by the route A, while the elongation of the sample on Bc route is the worst. After five ECAP passes, the strength of route A is still the best one, reaching 415 MPa, and the next is the route Bc of 395 MPa. The lowest strength is 385 MPa of route C. The elongation of route A suddenly rises, so route A has the highest elongation.

During ECAP, dislocation movement and increment are the main deformation modes. After pressing, the dislocation density increases obviously, and a large number of cellular structures and sub-grain boundaries are formed[19].

As can be seen from Fig.3b, the hardness of the samples through route C is the highest after the 2 ECAP passes, followed by the route A, and the lowest is route Bc, which coincides with the strength law (Fig.3a). But after the four ECAP passes, the highest hardness of the samples is using route A. After 10 passes of ECAP, the hardness of the three routes fluctuates little.

From the overall trends, the conductivity of the samples after ECAP decreased, especially after the 1 and 2 passes. The conductivity of the material is the highest under the route Bc before 8 passes. In the subsequent extrusion, the conductivity of the route C slightly fluctuates, and the conductivity of the route Bc continues to decrease, resulting in the conductivity of route Bc being less than that of route C. But these comparisons were carried out in a small range for electrical conductivity. So, it is considered that ECAP has almost no effect on the electric conductivity of single crystal copper.

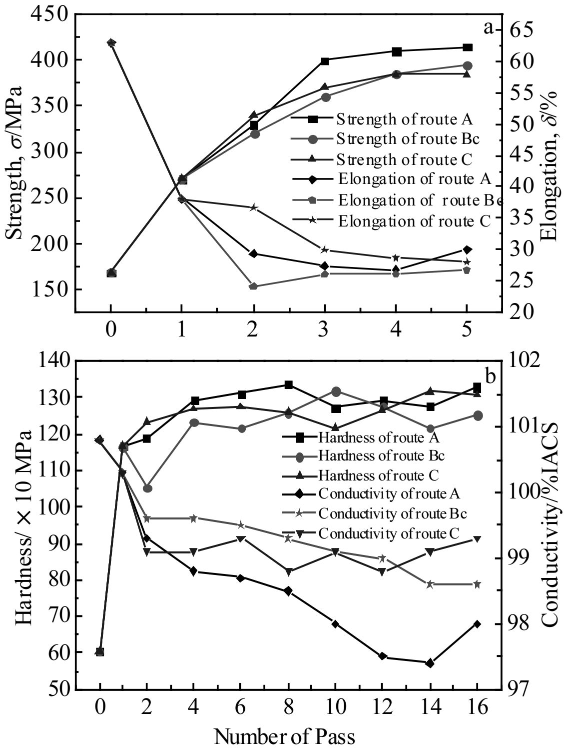

Fig.3 Mechanical properties of different ECAP passes by route A, Bc and C (a); relationship between ECAP pass, hardness and conductivity (b)

In the first few passes, the sample adopting route A has the best strength and elongation, but its conductivity is the worst. The sample of route Bc has the best conductivity, while the tensile strength of it is 5% less than that of route A. The strength of route C is 7.2% less than that of the route A, and the conductivity of it is slightly lower than that of the route Bc. In comprehensive consideration, the strength of the sample using route C is too low to strengthen materials. The route A is selected as the strength priority, while the route Bc is selected as the conductivity priority.

2.3 Grain boundary distribution

In order to understand the fracture of grain in the pressure process and research the influence of grain boundaries on the conductivity of the material, the grain boundary angle distribution was analyzed. Fig.4 shows the histograms of the fractions of grain boundaries as a function of the boundary misorientation angle for the sample in the original condition and after 1, 2, 4, 6, ECAP passes processed on route A, 4 and 6 passes on route Bc and C. As can be seen from the graph, large angle GBs hardly exist before four ECAP passes. All of them are dominated by small angle GBs and sub-grain boundaries. It is shown that the new GBs which are produced in the deformation process will first appear in sub-grain boundaries or small angle GBs with smaller misorientation[20]. This is due to a large number of dislocations within the grains

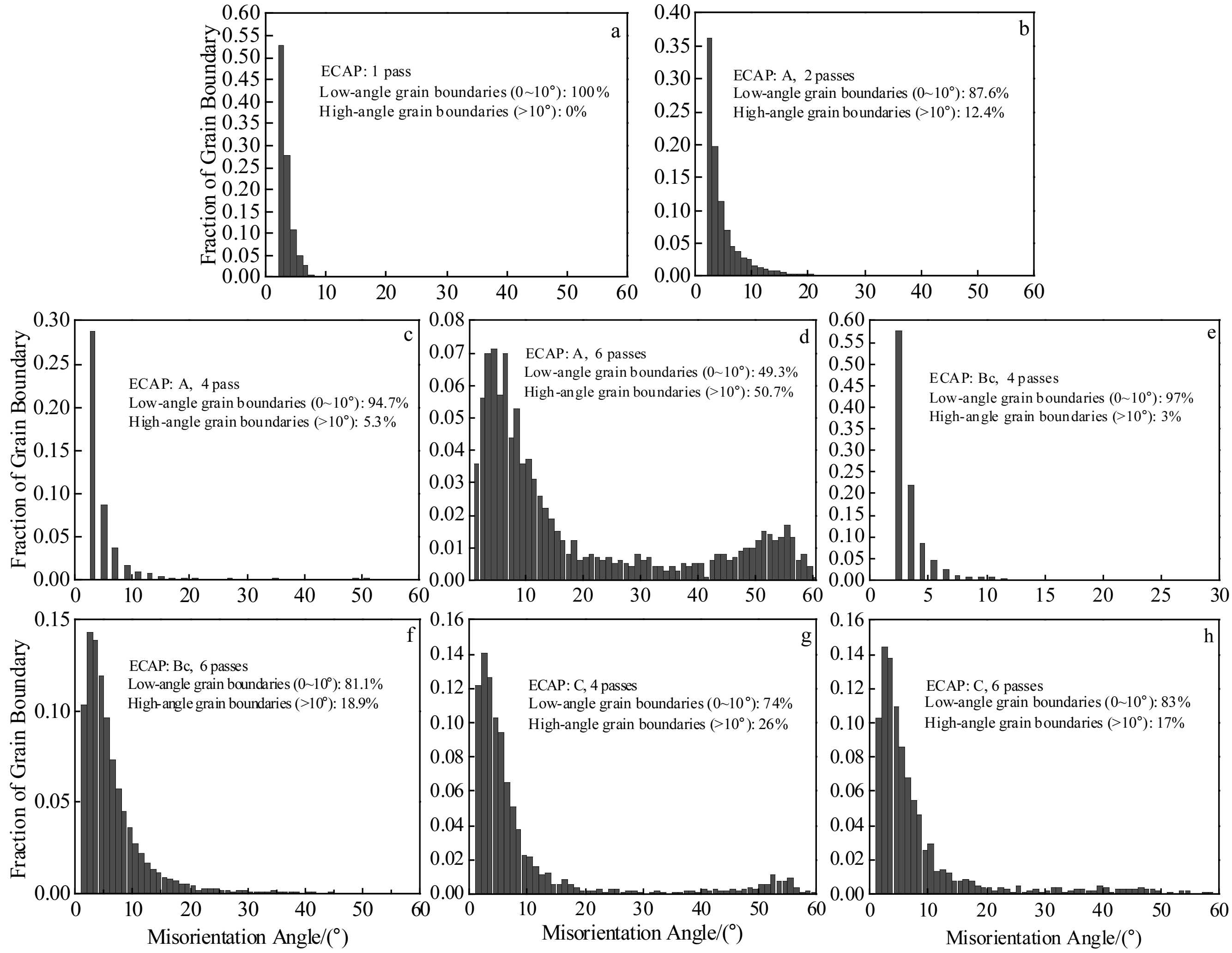

Fig.4 Misorientation angle distribution of Cu samples after several ECAP passes by route A, Bc and C: (a) 1 pass; (b) 2 passes of route A; (c) 4 passes of route A; (d) 6 passes of route A; (e) 4 passes of route Bc; (f) 6 passes of route Bc; (g) 4 passes of route C; (h) 6 passes of route C

starting from a smaller strain, and a large-scale slip occurs. Therefore, the small angle GBs increase greatly after single pass extrusion. Before 4 passes ECAP, there is merely large angle GBs in Fig.4g, but the peak is very low. This also confirms the speculations of the large angle grain boundary in the OIM image. Since the GB angle becomes larger after 6 ECAP passes, the large angle GBs begin to appear gradually. The large angle GB peak of 55° angle appears in Fig.4g, proving that it has the greater degree of fragmentation. Although the angle of GBs is increased, from the overall trend, the various passes do not strictly follow this trend. Such as the graph of 2 passes in Fig.4b and 4 passes in Fig.4c by route A, HAGB is increased and LAGB is decreased simultaneously. This is due to different extrusion methods.

In the previous study, we think that the bigger the GB angle, the greater the number of transverse grain boundaries, and the worse the conductivity of the material [21]. However, through the comparison of this study, we find that it is wrong that the larger the angle of the GBs, the worse the conductivity of the material. For example, after 4 passes of extrusion, the GB angle of the route C is obviously larger than that of the route A, but the result is that the conductivity of the route C is higher. After 6 passes, the GB angle of the route Bc is larger than that of the route C. However, the conductivity of the route Bc is higher. Therefore, in our opinions, the number of transverse grain boundaries does affect the conductivity of the material, and the number of transverse grain boundaries is closely related to the extrusion routes and the amount of deformation.

2.4 Pole figure analysis

The orientation distribution can be roughly observed from region OIM images. Therefore, it is necessary to observe the pole figure carefully. In order to understand the orientation transition of single crystal copper in ECAP process, the samples using three routes for different ECAP passes are detected by EBSD. Finally, the pole figures are obtained by software analysis. Fig.5 shows the {111} pole figure of

ECAPed samples through three routes. It is found that the initial sample pole figure has very typical single crystal characteristics, and the orientations closely surrounding the poles are very focused, and no other orientation exists in Fig.5a. After single ECAP pass, pole density is decreased, and the orientation rotates but is still the single orientation in Fig.5b. In the pressure of the route A, there is no orientation dispersion before four passes, until the 6 passes were performed (Fig.5i) other orientations appear. In the pressure of the route Bc, there are no other orientations until the 6 passes were performed, while the concentration of its single orientation becomes smaller. When the route C is used for pressure, the single-oriented failure of the sample is the most serious. In the 4 ECAP passes, orientation dispersion and other orientations appeared, and further expanded after 6 ECAP passes. In the 6 passes pressure, the preferred orientation of the route Bc is the best, and the route C is the worst. Because of the deformation mode for route C, the material slips in the opposite direction when pressure progresses. This leads to the formation of band structures which have different orientations. And then the multi textures appear. Therefore, combining the previous OIM images and the grain boundary distribution, the route Bc is the best, the route A is the second, and the C path is the worst from the point of maintaining the single orientation.

2.5 Analysis of orientation distribution function

It is difficult to obtain specific texture types of single crystal through pole figures, while the orientation distribution function can accurately obtain the texture component of the material. The deformation process and fracture of single crystal materials are determined by analyzing the evolution mechanism of texture in ECAP process. Therefore, ODF analysis of initial single crystal copper which was processed by 1, 2, 4, and 6 passes of ECAP through route A, Bc and C was conducted. And ODF is compared with the pole figure and is analyzed to reduce the experimental error. Fig.6 shows the orientation distribution function (φ_2=45°, 0°) for the sample in un-pressed condition and after 1, 2, 4, and 6 passes of ECAP through route A, Bc and C. After 1~4 passes of ECAP processes, there is no texture in the φ_2=0° ODF. Therefore, only the images of 6 ECAP processing are placed. It can be clearly seen that the initial single crystal copper has a very strong {111}<112> texture, and almost no other texture appears in Fig.6a. After single ECAP pass, the orientation gathered near {111}<112> disappears and transforms to {112}<110> texture (Fig.6b). After two ECAP passes, {112}<110> texture gradually moves to {001}<110> texture on route A (Fig.6c). On route Bc, the texture changes to {111}<112>, but a small amount of {111}<110> texture still exists (Fig.6f). The textures of route C also migrate to {001}<110> (Fig.6i). After four ECAP passes, texture of route A has no obvious change (Fig.6d). The {111}<112> texture of route Bc gradually changes to {111}<110> texture (Fig.6g). But the textures of route C are composed of {111}<110> and {112}<110> (Fig.6j). After six ECAP passes, a small amount of {110}<112> texture appears on the route A. At the same time, {112}<110> texture still exists, but shows almost no change (Fig.6e). Meanwhile, the texture around the {112}<110> is obtained from the φ_2=0° ODF (Fig.6n). The

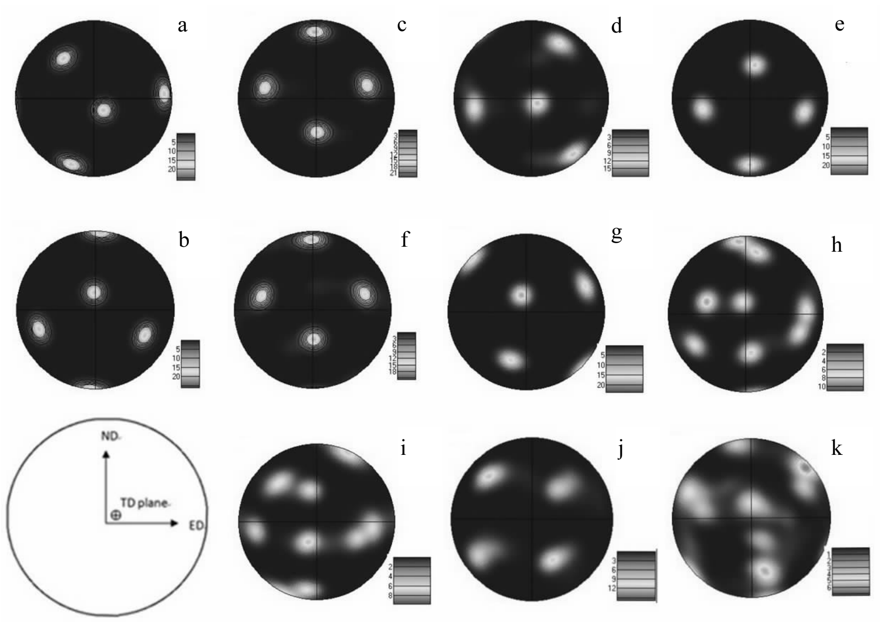

Fig.5 {111} pole figures of samples after ECAP for several passes by route A, Bc and C: (a) initial condition; (b) 1 pass; (c) 2 passes of route A; (d) 2 passes of route Bc; (e) 2 passes of route C; (f) 4 passes of route A; (g) 4 passes of route Bc; (h) 4 passes of route C; (i) 6 passes of route A; (j) 6 passes of route Bc; (k) 6 passes of route C

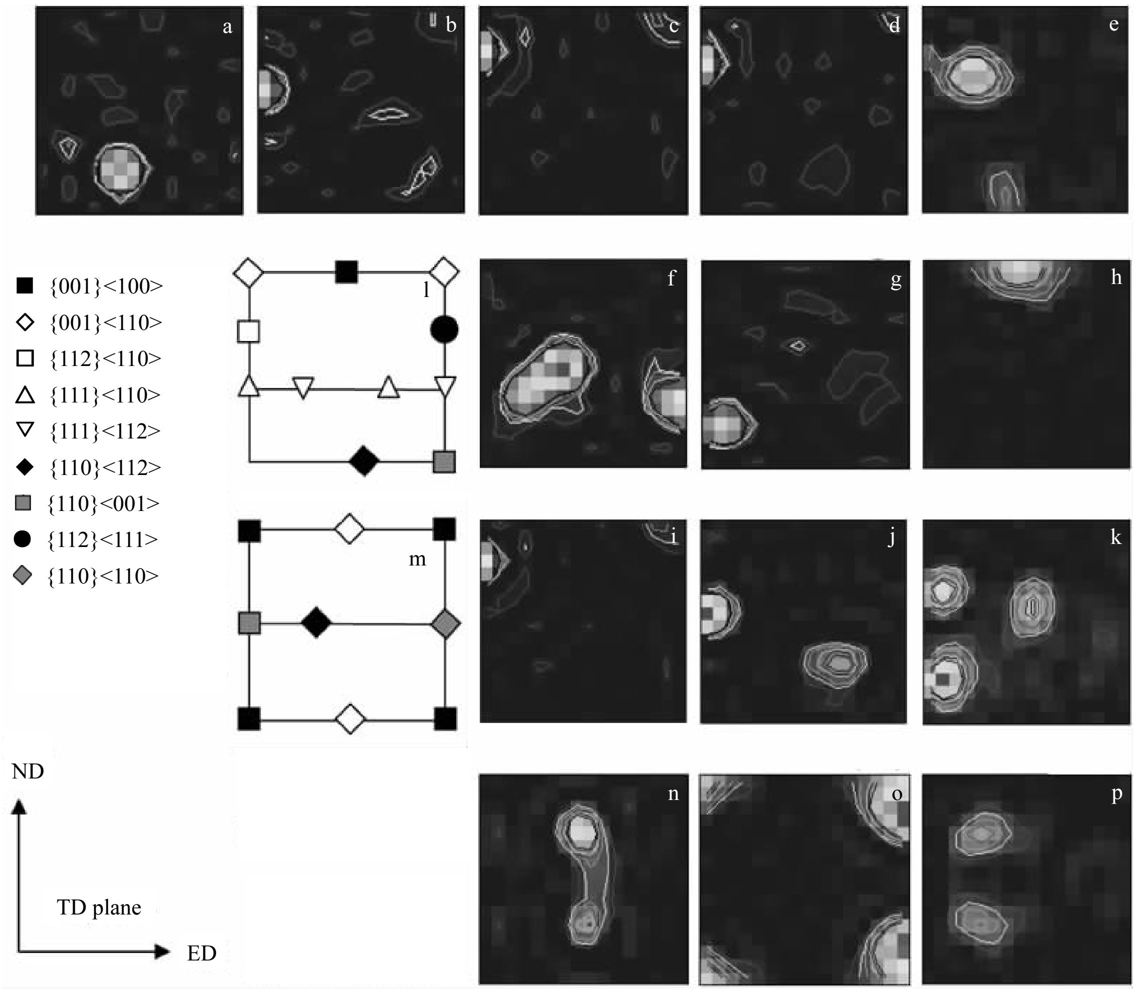

Fig.6 Orientation distribution function (ODF) of samples after ECAP for several passes through routes A, Bc and C: (a) initial condition; (b) 1 pass of φ_2=45°; (c) 2 A passes; (d) 4 A passes; (e) 6 A passes; (f) 2 Bc passes of φ_2=45°; (g) 4 Bc passes; (h) 6 Bc passes; (i) 2 C passes of φ_2=45°; (j) 4 C passes; (k) 6 C passes; (l) φ_2=45° section, relative positions of important orientations; (m) φ_2=0° section, relative positions of important orientations; (n) 6 A passes of φ_2=0°; (o) 6 Bc passes of φ_2=0°; (p) 6 C passes of φ_2=0°

texture of route Bc turns into {001}<110> rotational cube texture. This can also be observed in the φ_2=0° ODF (Fig.6o). The texture of route C has undergone complex changes, which is composed of many different textures, and single direction disappears.

The initial un-pressed single crystal copper has a crystalline orientation of {111}, which is consistent with the results of OIM images. After a single ECAP pass, the grain rotates and the crystal orientation turns to {112}, which is still consistent with the result obtained from the OIM. After two ECAP passes, the textures of route A and C change little. Meanwhile, the texture is almost identical to each other. However, the shear surface of route Bc varies greatly in each pass, resulting in a large difference between the textures formed by the other two routes. This also confirms the result of the previous pole figure.

2.6 Effect of deformation routes on material properties

In this paper, we hope to strengthen the single crystal copper and obtain higher electrical conductivity by ECAP, and try not to break the single crystal, that is to say, maintaining the single crystal characteristics. Because the single crystal characteristics are reflected in many aspects, such as orientation, grain boundary distribution and textures, a series of experiments and tests were conducted.

Through former researches[21], the dislocation density of single crystal copper increases rapidly after ECAP, and dislocation tangles to form dislocation cells and dislocation walls. With the multiplication and movement of dislocations, sub-crystals begin to appear in large numbers, and develops into a small angle grain boundary (SAGB). And then the SAGB develops into a large angle grain boundary (LAGB). As the deformation continues, the number of dislocation cells will not increase because of the dislocation annihilation[22,23].

When route A is used for pressing, the two shear planes intersect with each other almost perpendicularly. For one cycle of every two passes, the dislocations accumulate continuously on the shear plane without mutual offsetting. A large number of sub-grain boundaries appear in a direction perpendicular to the axial direction, so the increase of cumulative strain leads to the highest strength. But the conductivity decreases obviously because a large number of transverse sub-grains appear. The deformation uniformity is poor, so the orientation tends to be varied at the high strain and a variety of textures are produced. With the increase of the extrusion passes, the dislocations continue to multiply, accumulating and forming sub-crystals, and the sub-grain forms a small angle grain boundary. A large number of large angle grain boundaries are formed after the 6 pass deformation.

Using the route Bc extrusion, there are also two shear planes, but 4 passes complete a cycle, and dislocations are difficult to continuously accumulate into a large number of transverse sub-grain boundaries on one plane, so it has high conductivity. But the cumulative strain becomes smaller because dislocations offset each other when dislocation moves, which causes that the strength is lower than that using route A. It is the same reason that single crystals are the least likely to break up. At the same time, the deformation is more uniform, so that the orientation is not easy to disperse to form a variety of textures. With the increase of the extrusion passes, the angle of grain boundary changes very little.

When route C is used for pressing, the shear plane is always the same surface, but the shear direction of each pass is changed. In this way, repeat cutting on one surface causes a material to be extremely easy to form a transversal sub-grain boundary, which leads to a decrease in electrical conductivity. At the same time, the difference of orientation between the two sides of the shear plane is very large, and a variety of textures will be produced under the lower strain. It is easiest for the grain to break up. The grain boundary angle has a low grain boundary peak in 4 passes. The peak, however, disappears after 6 passes. It is obvious that this is caused by its unique single shear surface.

From the comparison of the results, different routes and shear ways will affect the speed of dislocation multiplication and the efficiency and direction of the stacking. Then it affects the number of large angle grain boundary and transverse grain boundary. Therefore, it has a great influence on the physical properties and electrical conductivity of the reinforced material.

3 Conclusions

1) During ECAP, the different routes and number of passes have different reinforcement effect on the material. After 5 passes by route A, the tensile strength of single crystal copper increases from 168 MPa to 415 MPa, and the elongation decreases from 63% to 30%. And next is the route Bc (27.9%). The elongation of route C is the worst (26.7%). But the sample using route Bc has the best conductivity, and next is the route C and route A.

2) After 6 passes of ECAP, the initial {111}<112> texture transforms into the intense {112}<110> texture and weaker {110}<112> texture by route A, and into {001}<110> texture by route Bc, and the original texture is dispersed by route C.

3) ECAP has a significant reinforcement effect on single crystal copper and does not obviously decrease the electrical conductivity. High strength and high conductivity materials can be obtained by an appropriate ECAP deformation mode.

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单晶铜在 ECAP 过程中的织构转变和强化行为

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摘　要：由于优异的导电和导热性能，单晶铜在各个关键领域得到了广泛的应用。然而，较低的强度严重限制了其在更广阔领域的发展和使用。利用 ECAP 对单晶铜进行强韧化调控，同时探索变形路径对材料性能强化机理的影响。采用电子背散射衍射来对 ECAP 变形过程中单晶铜的织构进行检测。模具的内角 Φ=120°，外角 ψ=37°。同时检测强化后材料的强度和延伸率。结果表明：5 道次挤压后，A 路径，Bc 路径和 C 路径的强度分别为 405、395、385 MPa，延伸率分别为 30%、30%、27.9%。6 道次挤压后，A 路径的织构从原始的{111}<112>变为{112}<110>以及较弱的{110}<112>两种织构；Bc 路径为{001}<110>织构；而 C 路径的织构发生了明显分散，出现多种织构并存的情况。在挤压过程中材料的导电性只有轻微的降低。可以看出，在合适的应变量下，ECAP 可以使单晶铜强度明显提高而且导电性损失很小。同时，采用不同的挤压路径可以显著影响材料的性能。

关键词：等通道转角挤压；电子背散射衍射；织构；单晶铜

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