Structural accommodation by twinning of copper during severe plastic deformation

S Rogachev and N Zemlyakova

1The National University of Science and Technology “MISIS”, 119049 Leninsky pr. 4, Moscow, Russian Federation
2MERI RAS, 603024 Belinsky Str. 85, Nizhny Novgorod, Russian Federation

*serti222@yandex.ru

Abstract. The influence of severe plastic deformation on structure and physical and mechanical properties of pure copper was investigated. The copper was in its initial state after 2, 4, and 8 passes of equal channel angular pressing (ECAP). We used electron microscopy, X-ray analysis and electrical conductivity technique. The transformation of macrocrystalline structure under ECAP is a result of deformation twinning under the action of partial dislocations. It was found that the refinement of the initial deformation macro-twin of 200 μm to a deformation micro-twin of 240 nm occurs during ECAP without the development of dynamic recrystallization process and is accompanied by an increase in microhardness.

1. Introduction
Currently the problem of modern materials science is obtaining new alloys with very small size of grain structure such as nanocrystalline (d < 100 nm), sub-nanocrystalline (d ~ 100-300 nm) and ultrafine-grain (300 nm < d < 1000 nm). Until now, there are no clear answers referred to the accommodation mechanisms of grain structure during of severe plastic deformation (SPD) [1]. The study of the structure at the micro level (less than 100 nm) includes the formation of nano-twins, a mosaic block. At the meso-level (less than 1000 nm), this is the formation of fragments, subgrains, micro-twins under the influence of mesodefects in the form of dislocations and disclinations. At the macro level – more, than 1000 nm [2]. The accommodation of the metal structure with SPD on the micro, meso, and macro levels has been studied recently [3-5].

The optimal variant of a homogeneous copper nanostructure consists in obtaining twin grains. At present, a homogeneous structure of copper in the form of twin grains, with a size of 100 nm, is quite problematic to obtain. Currently, a twin copper nanostructure is obtained in small samples: either by SPD by high-pressure torsion on samples 1 mm thick, or after dynamic deformation of Cu single crystal 4.5 mm thick at 90 K [6, 7].

The aim of the work is to describe the possibility of structure accommodation by twinning for copper (99.97%) after deformation treatments at a temperature (T = 20 °C) by the methods of drawing and ECAP (route Bc after 2, 4, and 8 passes).

2. Experimental Procedure
Pure copper (99.97%) rod with a diameter of 20 mm was obtained in the initial large-crystalline deformed state and after 2, 4, and 8 passes of equal-channel angular pressing (ECAP) of Bc route at
room temperature with an angle of channel intersection of 90°. The samples were subjected to ECAP in its deformed state (after drawing). We investigated the microstructure accommodation and microhardness, electrical properties (resistivity), X-ray (coherent scattering regions, lattice parameter) of pure copper with its initial grain size of 200 mm undergone by drawing and severe plastic deformation by ECAP. The microstructure was studied using transmission electron microscope JEM-2100 (JEOL) in a light and dark field. To prepare the foils from the tested material, the plates were cut. Then, they were mechanically grinded to the thickness of 120 - 140 µm and polished. From the received plates, the disks with a diameter of 3 mm were cut and subjected to jet electro-polishing by Struers Lectropol-5 apparatus, using an electrolyte "methanol + nitric acid". The DRON – 3 diffractometer was used to X – ray measurement. The electrical conductivity was measured by multifunctional portable device MECI-2M according to ISO 15549-2009. The electrical conductivity was measured. The electrical resistivity was calculated as the reciprocal of the electrical conductivity. The microhardness was measured in accordance to the standard procedure.

3. Results and Discussion

The Table 1 shows the microhardness values, lattice parameter, electrical resistivity and coherent scattering regions of copper at various states.

Table 1. Results of microhardness, X-ray diffraction analysis and resistivity of the copper in initial state and after 2, 4, and 8 passes of ECAP

| Material state | Microhardness, MPa | Lattice parameter, a, Å | Electrical Resistivity, ρ, Ω mm/m | Coherent Scattering Regions, D, nm |
|----------------|--------------------|-------------------------|-----------------------------------|-----------------------------------|
| Initial state (after drawing) | 1240 | 3.6177 | 0.0228 | 118 ± 14 |
| ECAP, 2 passes | 1390 | 3.6174 | 0.0227 | 177 ± 11 |
| ECAP, 4 passes | 1440 | 3.6161 | 0.0241 | 118 ± 14 |
| ECAP, 8 passes | 1470 | 3.6154 | 0.0215 | 127 ± 11 |

According to the data given in the Table 1, it’s possible to determine what processes take place during severe plastic deformation. It follows from the data of the Table 1 that with an increase in the number of passes a monotonic increase in the microhardness and decrease in the lattice parameter were observed. Non monotonic change is in the sizes of the coherent scattering and electrical resistivity. Known, that the structure is depend from different defects of material. The coherent scatter regions (D) increase after 2 passes of ECAP by 50%. The increase in the coherent scattering can be explained by the formation of a band-like fragmented structure with an increased number of defects along the boundaries of the fragments after two passes of ECAP [9]. During 2 passes of ECAP the lattice parameter and electrical resistivity are constant. But, during 4 passes of ECAP the lattice parameter was decreased, that characteristic of lattice distortion, which leads to increase in electrical resistivity (see Table 1). During 8 passes, the lattice parameter decreases to a minimum, and lattice distortions reach a maximum, but electrical resistivity is minimal. In this case, the microhardness practically does not change, i.e. the number of defects in the structure increases slightly after four passes of ECAP. This can be explained using microstructure analysis.

It is known, that the increase in ρ depends on the number of various defects obtained during the deformation of copper and they can be arranged as the influence decreases: vacancies, interstitial atoms, grain boundaries, dislocations [8].
There is a change in the coarse-grained macrostructure of copper in the initial state after 2 passes (Fig. 1, a), after 4 passes (Fig. 1, b) and after 8 passes (Fig. 1, c) ECAP. The TEM analysis showed: the initial state of the copper represented by the deformation macro-twins grains of 200 µm is changed during ECAP.

![Figure 1](image-url)

**Figure. 1.** The change in the structure of copper from 2 passes (a) and 4 passes (b), and 8 passes of ECAP (c)

The electron microscopy analysis of the copper after 2 passes of ECAP shows one grain with deformation twin of size 125 nm × 1 µm. After four passes of ECAP, one can see one grain of size 280 × 600 nm with many deformation nano-twins of size 10 × 600 nm (Fig. 1, b). After 8 passes of ECAP, one can see 500 nm sub-grains free of defects (in a large oval on Fig. 1, c) and the more of deformation twins, but their size decreases to size of 250 × 150 nm. Since the twins propagated over the entire length of the grain, the degree of structure refinement at this stage of deformation can be estimated from their length, especially at the early stage of ECAP.

It should be noted that deformation twins 200 µm in size take place in the structure of pure copper both after drawing, and after 2, 4, 8 passes of ECAP. Consequently, it can be argued that the mechanism of shear deformation by twinning affects the accommodation of the structure of coarse-crystalline copper during ECAP, which confirms the presence of deformation twins. From the data of the Table 1, it follows that for the first 4 passes of ECAP, the microhardness increased by 200 MPa, and for the next four passes its increase is 7 times less. Since the increase in microhardness is proportional to the number of dislocations, it is important to know that during the first 2 passes of
ECAP, the number of lattice dislocations (source of Frank-Read) reach saturation and form partial dislocations (Shockley), which are involved in the creation of deformation micro-twins. Thus, the decrease in electrical resistivity in the ECAP process occurs during the transition from a fragmented to submicrocrystalline structure and the formation of high-angle boundaries of the subgrains and micro-twins deformation in subgrains [8, 9].

4. Conclusions
The accommodation of twinning in the ECAP process is the result of shear deformation, which leads to the formation of micro and nano-twins of deformation in an already formed submicrocrystalline structure of copper. The formation of micro-twins of deformation occurs due to Shoely partial dislocations.

The transformation of initial state structure of the copper with deformation twins size of 200 µm under 8 passes of ECAP to deformation twins of size 250 × 150 nm is a result of local shear deformation.

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