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EVOLUTION OF MICROSTRUCTURE OF COPPER AND ITS ALLOYS DURING SEVERE PLASTIC DEFORMATION PROCESS

The changes in the properties of copper and its alloys as a result of severe plastic deformation (SPD) are reviewed. The literature review shows that contemporary materials science is aimed at solving the problem of producing ultrafine-grained materials with high-angle grain boundaries. With using of the methods of severe plastic deformation, it is possible to obtain so-called bulk nanostructured materials with grain sizes of 0.1–0.2 microns and specific substructures containing the lattice and grain-boundary dislocations. Such structures are characterised by large elastic distortions of the crystal lattice. It is believed that such fine-grained structures should simultaneously provide a high level of plastic and strength characteristics due to special stressed high-angle grains. The article discusses SPD methods such as torsion with shear, equal-channel angular pressing, screw extrusion, all-round forging, rolling with shear, *etc.* Among the most significant results of SPD, it is worth highlighting the increase in strength, fatigue resistance and elastic properties of the material. These changes make copper and its alloys subjected to SPD very attractive for use in many industries, including electrical engineering, aviation, medicine, *etc*.

Keywords: copper, copper alloys, severe plastic deformation, fine-grained microstructure, mechanical properties.

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1. Introduction

Numerous experimental studies conducted in recent years indicate that the method of severe plastic deformation (SPD), that is, the use of large plastic deformations realized under high applied pressure, is very promising for producing bulk nanostructured workpieces from various metals and alloys [1–15]. Bulk nanostructured materials are of great interest due to their specific structure with a developed network of grain boundaries, new attractive properties and high potential for industrial application. SPD materials demonstrate very high strength combined with plasticity, cyclic deformation without softening, and low-temperature or high-speed superplasticity [16].

As known, the chemical element Cu belongs to group 11 with atomic number 29 in the periodic table and shares some properties with elements in this group: they have one *s*-orbital electron on top of a filled *d*-electron, making copper a high plasticity and electrical conductivity. Due to its properties, copper is used as a conductor of heat and electricity, building material and a component of various metal alloys. Copper is not an allotropic species. It has a stable structure at room, high, and low temperatures. Cu has a structure formed by grains in a regular system known as a face-centred cubic (f.c.c.) lattice. This means that atoms are arranged as the vertices of a cube and at the centres of cube faces. Other materials that have the same lattice are aluminium, silver or gold. Materials with an f.c.c. structure tend to increase sensitivity to strain rate due to a decrease in the activation volume in the nanocrystalline system. The sliding direction of copper is ⟨110⟩ and the {111} sliding planes provide minimum shear strength [17, 18].

Copper has been used for thousands of years and its mechanical properties are well known. Its use in many industries is intensive and has been steadily increasing over the past decades. The main applications are wires, industrial equipment, copper products, solar collectors, integrated circuits and general electronics. Copper can also be recycled very efficiently. It is often used as a model material for fundamental studies of metal damage mechanisms [19, 20]. Research is carried out on both polycrystals and single crystals to obtain basic knowledge about fatigue damage mechanisms, changes in dislocation of structures, localization of cyclic plasticity, and initiation and propagation of fatigue cracks. Attempts to improve the mechanical properties of structural materials in recent decades have led to the use of SPD, resulting in fine-grained structures with improved mechanical properties [21–25]. Fatigue strength is an ongoing research problem [16]. This article briefly summarizes the main fatigue properties of conventionally grained (CG) materials, particularly copper. However, the main objective is to present and discuss the mechanical behaviour of ultrafine-grained (UFG) copper produced by various SPD methods. Cur-

rently, emphasis is placed on fatigue properties and their relationships with the UFG microstructure: relationship of cyclic softening/hardening with the stability of UFG structure, influence of fatigue loading mode on the grains dynamic coarsening, associated fatigue life, mechanism of cyclic sliding localization and fatigue cracks occurrence [16, 18, 26].

The structural strength of materials plays an important role in ensuring the reliable and durable operation of machine parts and units. The creation of new types of equipment in aviation, mechanical engineering, oil and gas production and other industries places more stringent and increased demands on the performance of structures. This necessitates the use of materials with a higher complex of physical and mechanical properties. For metallic materials, this problem is solved either by creating new alloy compositions or by developing new highly effective thermomechanical methods for directed influence on the structure of serial industrial alloys [27].

Despite great prospects, until recently the issue of using nanostructured (NS) metals and alloys as structural and functional materials of a new generation remained controversial. Production of materials with an ultradispersed structure today is a complex technological problem. The most promising method for producing semi-finished products with submicrocrystalline (SMC) and nanostructure is deformation–heat treatment, including SPD, achieved by conventional methods of metal forming [28].

Possibilities of alloying have now largely been exhausted. In addition, the development of completely new alloys requires large material costs for the creation of new compositions, their certification and implementation. Meanwhile, in recent decades, new directions in materials science and materials processing have been intensively developing, which consists in the formation of ultradispersed structural states in metals and alloys. This makes it possible to increase sharply specific strength in the range of operating temperatures, while in the range of pressure treatment temperatures technological plasticity significantly increases. Based on this direction, it is possible to create a fundamentally new set of physicochemical and mechanical properties in conventional industrial materials. This applies to SMC and NC metals and alloys, with grain sizes up to 0.1 μm or less [29].

2. Severe Plastic Deformation for Improving Desired Characteristics

It is known that accumulation of defects in the crystal structure primarily dislocations occurs as a result of cold plastic deformation. It causes appearing of a subgrain structure inside the primary initial grains. In addition, during unidirectional deformation, primary grains acquire an elongated shape, *i.e.*, their size decreases in one or two directions. Plastic deformation leads to refinement of structure, but with traditional types of processing, the material is simultaneously hardened, its plasticity decreas-

es, and further deformation becomes impossible. Therefore, with using of the conventional deformation methods, it is impossible to obtain nanosize grains. To form nanostructures, it is necessary to implement special methods of plastic deformation, where the material is subjected to simultaneous compression along at least two axes. In this case, initiation of cracks in the material becomes more difficult and it can continue to be deformed to refine the structure [30–32].

A feature of the stress state during severe plastic deformation is a high proportion of tangential stresses. With an increase in tangential stresses exceeding the elastic limit, plastic deformation of the material occurs. The type of stress state and the ratio of stress components in the deformation zone have a significant impact on material behaviour in the deformation region, as well as on its final structure and properties. To ensure a change in properties in the desired direction, deformation conditions must be changed, varying the degree of deformation and temperature. Correction of thermal deformation parameters is usually used when using many technological processes, such as various extrusion schemes, screw rolling, drawing with superimposed rotation of the workpiece and others [33].

A review of the world literature shows that modern materials science is aimed at solving the problem of producing UFG materials with high-angle grain boundaries. With using of the methods of severe plastic deformation, it is possible to obtain so-called bulk nanostructured materials with a grain size of 0.1–0.2 μm and a specific substructure containing lattice and grain-boundary dislocations and disclinations. This structure is characterised by large elastic distortions of a crystal lattice. It is believed that such fine-grained structures should simultaneously provide a high level of plasticity and strength characteristics due to special stressed high-angle grains, which can be obtained if multidirectional deformation occurs during thermomechanical processing [34–36].

3. SPD-Induced Changes in the Cu Microstructure

Severe plastic deformation is a process when a material is subjected to high deformations without significant change in its bulk. In recent decades, this method has attracted the attention of many researchers due to its ability to improve significantly the mechanical properties of various metals and alloys, including copper [37].

The evolution of copper microstructure and its alloys during SPD is an important object of study in materials science. This process can occur under various influences, such as twisting, rolling, extrusion, *etc.* As a result of severe plastic deformation, changes occur in the structure of a material, which significantly affects its mechanical properties. We can observe:

• increasing strength (decreasing the size of grains and increasing their number in crystal lattice leads to an increase in copper strength);

• improvement of fatigue strength (fine-grained structure formed as a result of SPD reduces the likelihood of cracks and, therefore, increases fatigue strength);

• ductility increasing (recrystallization and crystal growth help to improve the ductility of copper) [38].

Changes in copper microstructure during SPD occur due to several mechanisms. They are as follows:

• dislocation sliding — under the influence of external loads, dislocations, which are defects in the crystal lattice, begin to slide relative to each other; this process results in a change in the shape of crystals;

• recrystallization — high deformations are usually accompanied by the formation of new crystals through recrystallization; this process helps reduce microstresses and improve material characteristics;

• phase transformations — SPD can cause phase transformations in the copper crystal structure, which leads to a change in its properties [39].

The creation of such structures can be carried out by methods of combined plastic deformation with shear, allowing one to achieve relatively large deformations at relatively low temperatures under conditions of high applied pressures. In materials science, the term 'severe plastic deformation' is more often used, meaning the use of simple shear combined with compression (tension) in the deformation scheme [40].

Torsion with shear is historically the first scheme of severe plastic deformation. In this method, a disk-shaped sample is clamped between two hammers, one of which is stationary and the other is rotating. With simultaneous compression and torsion, the material is subject to compressive normal stresses and tangential stresses generated by the rotation of the upper anvil. The main disadvantage of the scheme is obtaining a sample of low height [40, 41].

The use of an equal-channel angular pressing (ECAP) scheme makes it possible to obtain larger samples. During this process, the workpiece is repeatedly pressed in special equipment through two channels with identical cross sections intersecting at a certain angle. If necessary, in the case of difficult-to-deform materials, deformation is carried out at elevated temperatures or increased channel intersection angles. In this case, special requirements are placed on the heat resistance and strength of the equipment. Shear deformation occurs when the workpiece passes through its intersection zone [40, 42].

During severe plastic deformation, using the screw extrusion method, the sample is forced through a channel that has a screw cross-section. The angle of helix inclination to the direction of the extrusion axis varies along the height of the matrix, and at its initial and final sections, it is equal to zero. Features of matrix channel geometry led to the fact that when extruded through it, the identity of processed workpiece initial and final shapes and sizes is preserved, and this, in turn, allows for the repeated extrusion of it to accumulate large degrees of deformation [43].

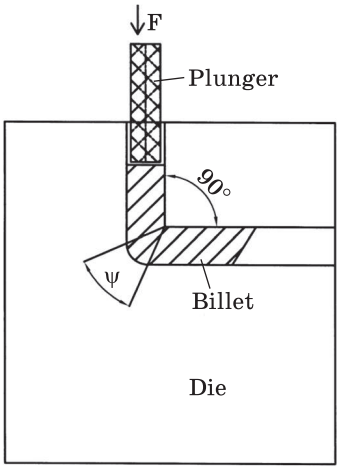

Fig. 1. Schematic diagram of an equal-channel angular pressing method [46]

All of the above methods have a common drawback, namely, a sample is shaped in a stamping tool of complex shape. In this case, the tool is subjected to very high loads and wears out quickly. Firstly, this affects the cost of material obtained by such methods, and secondly, it limits the size of the samples obtained in this way [39].

All-round forging is a cheaper and very promising method. The essence of this method is to use all-round isothermal forging with a gradual decrease in the deformation temperature. Refinement of microstructure occurs due to the development of dynamic/post-dynamic recrystallization processes. The all-round forging scheme is based on the use of multiple repetitions of free forging operations: upsetting broaching with a change in the axis of the applied deforming force. The uniformity of deformation in this technological scheme is lower compared to ECA pressing or torsion. However, this method makes it possible to obtain an NC state in fairly brittle materials, since processing begins at elevated temperatures and small specific loads on the tool are provided [44].

Shear rolling is a very promising method. Its peculiarity is that the impact is simultaneously carried out not on the entire volume of metal, but only on part of it. This makes it possible to increase the size of manufactured samples [45].

The author of Ref. [46] studied changes in the mechanical properties of copper subjected to SPD using the ECAP method. The principle of this method is shown in Fig. 1. It consists of pressing a rod blank through a matrix with an angular channel having an angle ψ, often equal to 90°. During the pressing of the workpiece with a plunger, shear deformation is introduced through the channel elbow. Since the cross-sectional dimensions of the workpiece remain constant after passing through the channel, the procedure can be repeated. The result is very high plastic deformation of processed material. Most laboratory ECAP dies have a channel with a quadratic cross-section. Workpieces of appropriate sizes can be turned in increments of 90° between individual passes. Indeed, a rotating procedure is also possible for dies and samples with a circular cross-section. There are four different ECAP routes. Route *A* means repeating the pressing without the workpiece rotating. Route B_A represents a 90° rotation of the workpiece in the opposite direction; route B_C represents a 90° rotation in the same direction after each subsequent push. Route *C* represents a 180°

turn after each pass. At the current level of knowledge, local grain coarsening is not a necessary condition for the formation of cyclic slip bands and the initiation of fatigue cracks. The slip bands, their shape and main features resemble those formed in CG Cu. At the same time, grain coarsening is certainly an important effect that occurs in UMG Cu under special conditions. The role of roughened structure in the crack initiation process and specific initiation mechanisms are not well understood. This is in contrast to CG Cu, which describes specific dislocation structures associated with cyclic slip bands. In general, there are no similar and convincing observations on UFG copper. SPD can significantly improve the fatigue performance of copper under stress-controlled conditions. Fatigue strength at 108 cycles can reach 150 and 120 MPa at 10^{10} cycles, provided that the stability of the UFG microstructure is maintained. It depends both on the material; that is, the details of microstructure obtained during SPD, as well as the type of cyclic loading [47–49]. During the plastic deformation tests, the UFG structure is more prone to grain coarsening, and fatigue life at the same plastic deformation amplitude is significantly shorter than computer graphics materials. The amplitude of plastic deformation appears to be the unifying parameter for predicting lifespan. Fatigue cracks initiate in cyclic slip bands, which are observed under all types of loads. As the severity of cyclic loads decreases, their density decreases, and their appearance changes slightly [43, 46].

Authors of Ref. [50] studied accumulative roller bonding (ARB), which is an SPD process that is applied to 99.97% commercial grade copper for up to 7 cycles at room temperature without lubrication, cryogenic temperature (CT) and high temperature (HT) conditions respectively. Microstructural characteristics were studied using optical microscopy (OM) and scanning electron microscopy (SEM). Observations showed that refined grains in Cu were obtained after ARB, and continuous recrystallization microstructure was observed. Vickers hardness (*VH*) measurements were carried out on deformed specimens, showing a gradual increase in hardness with increasing strain/initially a decrease in grain size, but then a decrease in hardness with further increases in strain [50]. Figure 2 shows the microstructure of pure copper after processing by ARB welding in one-step.

The main visible feature of OM is that in pure copper the structure is granular, with equiaxed grains in random orientation. The thickness of grains is about 25 microns. However, in the first ARB cycle, there is a mixture of grains, which were not deformed, without guidance, with other grains having a thickness reduction of up to 15 μm, and having an elongated morphology oriented in the direction of force. Twins within the grains can also be identified, which tells us that the stacking sequence within the grain has changed. Twinning is a material strengthening mechanism in addition to sliding. Twins were formed as a result of a transverse force applied parallel to one plane and in one direction. The twin prevents move-

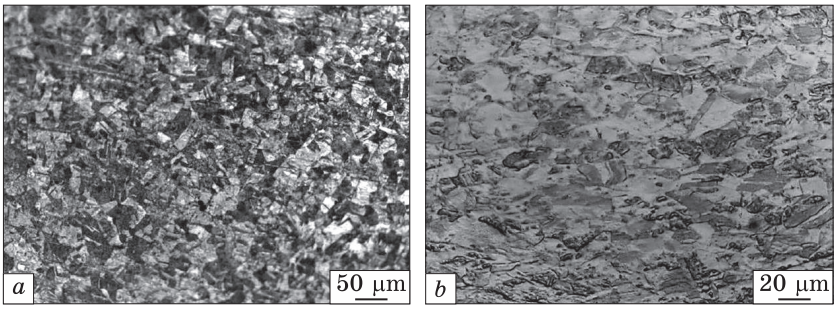

Fig. 2. Samples of (*a*) pure copper without accumulative roller bonding and (*b*) copper with 1-stroke of accumulative roller bonding [50]

ment of dislocations, complicating the sliding process, maintaining the strengthening mechanism and creating a separation of grains by size [50].

By interpreting and analysing the results of experiments, the following conclusions can be drawn [50]:

• proposed accumulating roller process contributes to the refinement of copper grains;

• procedure causes greater deformation of grains, starting from the third cycle; grain size can be controlled by heat treatment;

• to obtain the greatest hardness of the material, it is necessary not to exceed three ARB cycles, since after this a reverse Hall–Petch is formed and the material loses strength; in fact, only three cycles need to be completed, reducing time and energy in production; as an added benefit, this process also has high productivity and production viability; for large materials, working with wide strips in coils is not difficult;

• the effect of increasing body conductivity turned out to be insignificant.

Authors of Ref. [51] studied the microstructure of pure copper using two severe plastic deformation methods: equal-channel angular pressing and hard cyclic viscoplastic deformation (HCVD). At the first stage, metal was severely deformed up to 10 BC ECAP routes and ultrafine-grained microstructure was obtained. The elongated laminar substructure has small angles and blurred grain boundaries, but a high dislocation density. The metal has high hardness and strength, but low tensile ductility. In the second stage, deformation amplitude of tension–compression cycles increased stepwise from 0.2 to 2.5% over 30 cycles and 5 series. The results show that HCVD deformation produces an ultrafine-grained microstructure with high-angle grain boundaries. The mechanism of microstructure evolution includes the destruction of elongated (ECAP) subgrains under the action of shear stresses by atomic layers of crystals and the formation of a new microstructure with high-angle grain boundaries. In this case, the density of dislocation bands decreased (from $\approx 4.3 \cdot 10^{14}$ m^{-2} to

$\approx 2.1 \cdot 10^{14}$ m^{-2}) mainly inside the grains, and the density of dislocations increased near new high-angle grain boundaries. Specimens with this microstructure exhibit relatively stable viscoplastic behaviour, high levels of tensile-compressive stresses, and high uniform tensile elongation.

Another group of scientists [52] studied annealed pure copper subjected to ECAP to obtain NC structure. Structural changes and properties caused by heat treatment and various loading methods of copper were analysed. The results show that heat treatment and severe cyclic deformation in the viscoplastic regime affect the properties of copper to a significant extent. The tensile strength and microhardness of NC copper are decreased during heat treatment. Copper acquires increased true tensile properties after severe cyclic deformation in the viscoplastic regime. In various processing modes, the microstructure of pure copper develops, which affects the change in its mechanical properties. During the first pass of ECAP, large equiaxed grains are strongly deformed in the direction of metal flow. Subgrains with gently sloping boundaries are formed in-group slip. Reducing the grain size leads to a significant increase in hardness and mechanical properties. The increases in tensile stress and hardness are maximal during the first ECAP pass. As the number of presses increases, there is an improvement in properties compared to the first ECAP and the percentage of stress relaxation as the number of passes is increased. Mechanical properties, grain growth and relaxation processes can be stabilized by appropriate heat treatment. The results of this study confirm that low-temperature SPD annealing of copper results in increased ductility of ultrafine-grained materials. HCVD deformation affects the growth of nanograins by coagulation and orientation of grains in the metal along the direction of the applied load. HCVD deformation improves the ductility of NC copper, while the ductility of annealed copper is reduced.

The microstructure evolution and mechanical changes of copper subjected to SPD using ECAP have been studied in Ref. [53]. The samples were subjected to ECAP using three different processing routes: B_C, A and C. Microstructure refinement was dependent on processing, with route B_C being the most efficient. Mechanical response is modelled by an equation containing two dislocation evolution terms: one for the interior of cells/subgrains and one for the walls of cells/subgrains. Deformation structure develops from elongated dislocation cells to subgrains and equiaxed grains with a diameter of 200–500 nm. Disorientation between adjacent regions, measured by electron backscatter diffraction, gradually increases. Mechanical response is represented by the Voss equation with a saturation stress of 450 MPa. Microstructures resulting from adiabatic shear as well as the localization during high strain rate deformation and ECAP are very similar, resulting in similar grain sizes. It is shown that both processes have very similar Zener–Hollomon parameters ($\ln Z$ 25). Calculations show

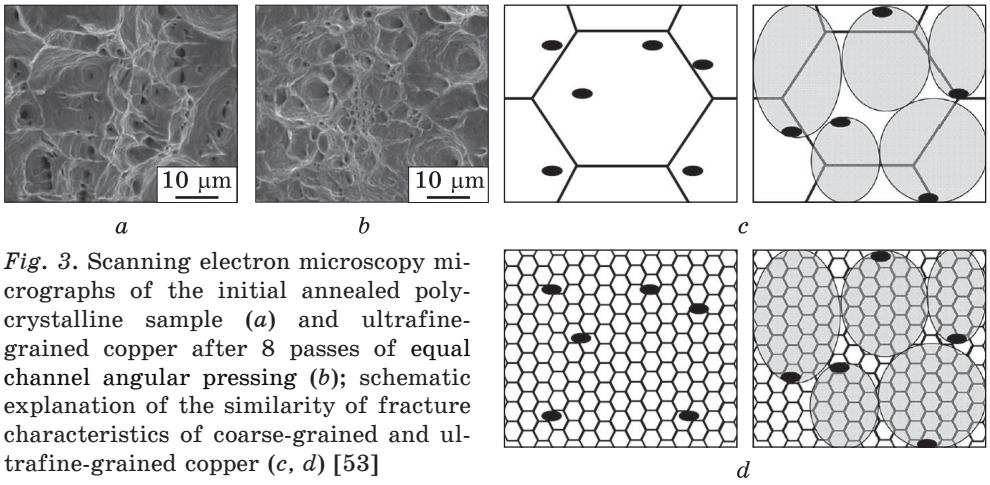

Fig. 3. Scanning electron microscopy micrographs of the initial annealed polycrystalline sample (*a*) and ultrafine-grained copper after 8 passes of equal channel angular pressing (*b*); schematic explanation of the similarity of fracture characteristics of coarse-grained and ultrafine-grained copper (*c*, *d*) [53]

that grain boundaries with a size of 200 nm can rotate by 300 during ECAP, thereby creating and maintaining a stable equiaxed structure. This is confirmed by the calculation of grain boundary mobility, which shows that their speed is 40 nm/s for a grain size of 200 nm at 350 K, which is typical for the ECAP process. This leads to the movement of grain boundaries necessary to maintain an equiaxed structure. Figures 3, *a*, and *b* show the fracture surface of the original and eight-pass ECAP material, respectively (tensile specimens). In the fractograms in the lower part of dimples, you can see holes, which are centres of destruction. There is no significant difference in dimple size between the two fractograms, despite the two orders of magnitude difference in grain size. The interpretation of similarity in fracture morphology is that the nucleation sites (most likely inclusions in this case) have a similar distribution for two conditions because the material composition is the same. Therefore, knowing that the fracture morphology is dictated by the inclusions, it is reasonable to assume that the dimple size will be the same. Figures 3, *c*, and *d* show the distribution of inclusions resulting in dimples independent of grain size. However, dimples appear smaller for ultra-fine-grained material. This is a direct consequence of decreased ductility [54–56].

The microstructure of copper chips formed as a result of plane strain machining at ambient temperature has been studied in Ref. [57]. The analysis was carried out using transmission electron microscopy (TEM) and orientation imaging microscopy (OIM). The strain applied to the chips varied depending on the change in the rake angle of the tool (Fig. 4). Characterization of orthogonal chip faces showed that the microstructure was essentially uniform across the chip volume, also indicating uniform deformation. TEM analysis shows that the increase in strain generated during chip formation leads to grain refinement and the creation of nanocrystal-

line materials. OIM observations on mutually orthogonal chip faces indicate that the microstructure and hence deformation is essentially uniform throughout the chip volume. Corresponding pole figures show the presence of texture shear and the persistence of these texture-induced deformations even before shear deformation. The OIM data is also consistent with TEM observations for the specific orientation in which the samples were examined under TEM. These preliminary observations regarding the uniformity of chip microstructure have particularly stimulated the development of various geometrically constrained machining processes, *e.g.*, large-strain-extrusion machining (LSEM), as a means of producing bulk nanostructured materials. In LSEM, the geometry and size of a chip are directly controlled by the point of its formation under large plastic deformations, simultaneously imposed to produce microstructure refinement. Consequently, bulk nanostructured materials in the form of sheets, rods and foil can be produced using this controlled chip formation process from various material systems [58]. Observed homogeneity of microstructure (and deformation) throughout the sample volume suggests that these processing processes are very likely to be scaled up to produce volumetric shapes using UFG and nanocrystalline microstructures.

The evolution of the structure and properties of copper during the process of hot cumulative rolling (HCR) has been studied in works [59, 60]. This process is shown schematically in Fig. 5. To implement this SPD method, two plates of equal thickness are taken from the metal being processed. One surface of each plate is polished, and then the plates are folded with polished sides and rolled at elevated temperatures with a reduction ratio of 2. In this case, the length of the resulting assembly becomes twice the initial length of the plates, and the thickness is equal to the thickness of the original plate. After this, the resulting plate is cut into two equal parts, one side is pre-

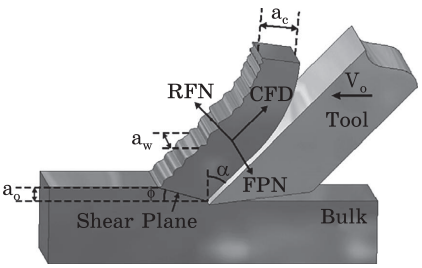

Fig. 4. Scheme of chip formation during machining with a flat deformation [57]

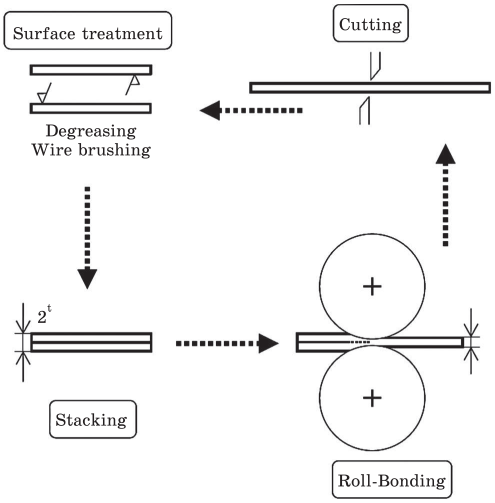

Fig. 5. Schematic representation of the cumulative rolling process [60]

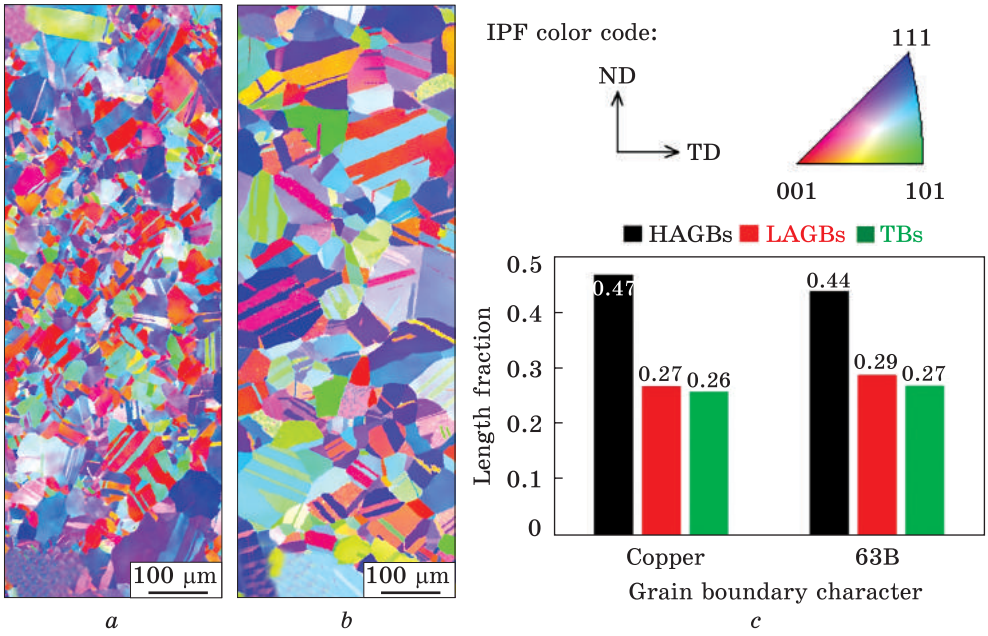

Fig. 6. Initial microstructure map of (*a*) copper, (*b*) brass, (*c*) proportion of large-angle, small-angle, and twin boundaries in each material [61]

pared on each, and they are folded and rolled. Repeating this procedure many cycles will allow you to obtain a homogeneous fine-grained structure in the material.

As in the case of conventional rolling, the microstructure of the surface layer and internal layers of material is deformed unevenly; so, a large number of deformation cycles are required to form a homogeneous structure. In this work, copper grade 4N and brass grade 63B (containing 37% Zn) were studied. Figure 6 shows a map of each material structure in its original state [61].

Authors of Ref. [62] studied new technologies for combined metal pressing processes aimed at eliminating restrictions on the length of the workpiece and including two or more methods of traditional deformation. The main feature of such combined processes is that they either reduce or eliminate the disadvantages of individual metal extrusion processes involved in the combined processes. In this work, authors analyse both complex processing using traditional techniques and equal-channel angular pressing (ECAP), as well as the influence of individual processes on the formation of the ultrafine-grained structure of copper and its ability to achieve a high-strength state due to each of the processes used. Using the example of copper wire, features of thinning the components of the structure and change in mechanical properties during the combined method of pressing and equal-channel drawing are illustrated. Stepped die and tradi-

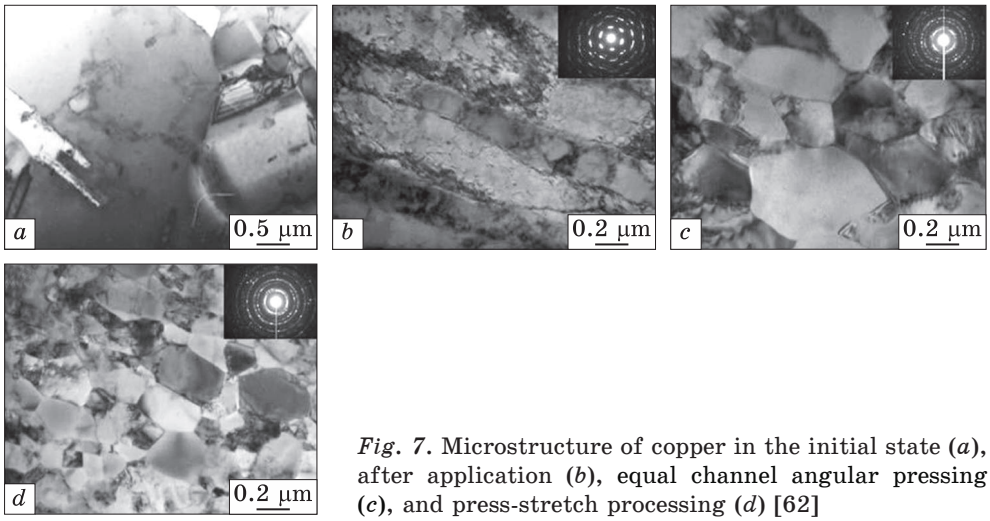

Fig. 7. Microstructure of copper in the initial state (*a*), after application (*b*), equal channel angular pressing (*c*), and press-stretch processing (*d*) [62]

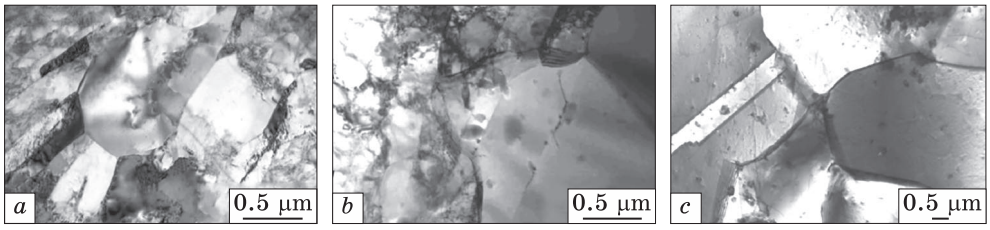

Fig. 8. The M1 copper structure after rolling, equal channel angular pressing, and heating at 150 °C (*a*), 200 °C (*b*), and 270 °C (*c*) [63]

tional drawing techniques are used. The results show an increase in the thinning of copper structure both on the surface and in the central regions of the wire. This indicates the uniformity of cross-sectional structure and, therefore, stability of resulting wire properties with ultrafine-grained structure. Figure 7 shows TEM images of the sample structure. Figure 8, *a* shows the microstructure of copper in its initial state, after annealing at 700 °C and cooling in water. The structure consists of polyhedral grains with twins (Fig. 8, *a*). To assess the effectiveness of plastic deformations, it is necessary to compare the microstructure of alloy before and after deformation. Images of the microstructure after four deformation passes are shown in Fig. 7, *b*–*d*.

The article [63] describes an investigation of a new combined method of plastic deformation, rolling-pressing influence, on the structure and mechanical properties of copper. Deformation was carried out at room temperature and consisted of three cycles. Scientists have concluded that the rolling-pressing method significantly improves the structure of industrial-grade copper. The strength of copper blanks doubled after three pass-

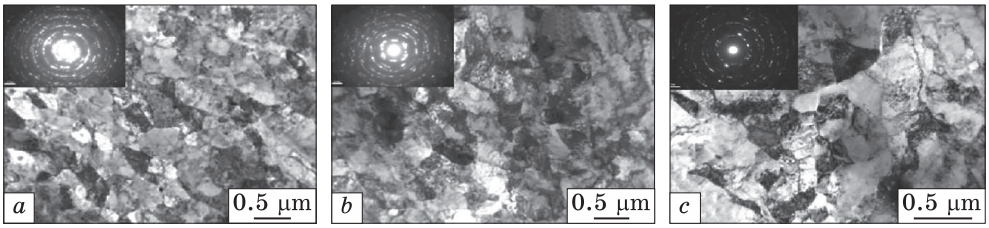

Fig. 9. Microstructure of copper after 6 equal channel angular pressing cycles in a stepped matrix, observed by transmission electron microscope at 25 °C (*a*), 90 °C (*b*), and 180 °C (*c*) [64]

es. Tensile strength and yield strength increased from 235 to 482 MPa and from 198 to 405 MPa, respectively. Relative elongation and relative contraction decreased by 8% and 7%, respectively. The ultrafine-grained structure with an average grain size of 2 μm turned out to be resistant to subsequent annealing at temperatures up to 150 °C. At a temperature of 150 °C, there are grains with a banded contrast at the boundaries, individual recrystallization nuclei of 0.15–0.20 μm that appeared at the boundaries of structurally deformed grains (Fig. 8, *a*). The initial stage of recrystallization is observed at 150 °C. Significant changes occurred in the structure of copper when the annealing temperature was increased to 200 °C (Fig. 8, *b*). The sample had a mixed structure, consisting of areas with deformed grains and new recrystallized grains 1–5 μm in size. The fully recrystallized structure formed at 270 °C in the copper sample is shown in Fig. 8, *c*. Grain size 10–20 μm. There, the annealing of twins occurs inside some grains. The grain boundaries have a typical striped contrast. Subsequent heating does not stimulate grain growth.

Article [64] is scoped on studying the effect of equal-channel angular pressing in a stepped matrix on the microstructure and properties of preheated copper products. It has been established that the grain size after ECAP slightly depends on preliminary heat treatment, but applied to harden reduces the hardness of copper by 15%, which, in turn, makes it possible to reduce increasing in pressing during the first pass from 620 to 510 kN. The minimum average grain diameter achieved during quenching of the M1 alloy at 700 °C by ECAP in a stepped matrix at room temperature and 6 deformation cycles is 0.6 μm. Figure 9 shows changes in copper microstructure as a result of ECAP. Studies were carried out using the TEM method. For comparison, photographs were taken after the 6th ECAP cycle at different pressing temperatures. Most highly deformable metals and alloys have insufficient ductility, which leads to significant brittleness. Plastic properties of metal can be improved by reducing stress. This is usually achieved by annealing, holding and tempering the metal. However, the desired final heat treatment also depends on the recrystallization temperature. The structure formed during ECAP at room temperature

determines the behaviour of copper during subsequent heating. It was found that samples subjected to quenching and 6 pressing cycles at 25 °C have the most fine-grained structure.

4. Effect of SPD on the Microstructure of Cu Alloys

Copper alloys are materials in which copper is mixed with other metals and non-metallic elements to improve their properties. These alloys are used in many applications because of the combination of copper's strength, electrical conductivity, and other beneficial properties with those of the other alloy components. Some of the most common copper alloys are as follow:

• bronze — an alloy of copper and tin, which usually also contains other metals such as Al, Ni, and Mn; bronze has good corrosion resistance and wear resistance, which makes it suitable for the manufacture of bearings, screws, coins and other products;

• brass (an alloy of Cu and Zn); brass is characterized by good machinability and excellent corrosion resistance; it is widely used in areas where aesthetics and anticorrosion properties are important, such as in the production of jewellery, musical instruments, and architectural details;

• beryllium bronze — a Cu–Be alloy that is highly strength, hard, and corrosion resistant; it finds use in springs, bearings, electrical contacts, and other critical applications;

• phosphor bronze — an alloy of Cu with P, which has good corrosion resistance, suitable for the production of wires, contact parts and ship equipment;

• nickel–silver bronze — an alloy of Cu with Ni and other elements has high strength, anti-corrosion properties and good resistance to high temperatures; it is used in the aerospace and marine industries.

Copper and copper alloys are among the most versatile materials used in industry as structural materials due to their unique properties such as strength and machinability, corrosion resistance and wear resistance. Among the numerous industrial copper materials, brasses and aluminium bronzes, which include alloys of the Cu–Zn and Cu–Al systems, have a combination of enhanced mechanical, functional and physicochemical properties, are unmatched in any other alloy system, and therefore have found wide application in electronic, automotive, mechanical engineering, shipbuilding, chemical and petrochemical industries.

It is worth noting that single-phase hard alloys based on copper Cu–Zn, Cu–Al and Cu–Si attract the attention of researchers. These materials are classified as model systems of binary alloys, but they are also the basis for the creation of industrial alloys and are of great interest as objects of research for understanding the influence of the content of alloying elements on the 'microstructure–properties' relationship.

The study of copper and Cu-based alloy microstructure evolution under severe plastic deformation has important practical applications, especially in the development of new materials with improved mechanical properties for use in various industries.

The main aspects of copper alloy evolution during SPD include:

• granulometric changes (SPD can lead to a decrease in grain sizes in the crystal structure of the material); this occurs due to the destruction of existing grain boundaries and the formation of new ones;

• increased formation of structural defects (as a result of deformation, various structural defects can form, such as dislocations, grain boundaries, density of grain growth nuclei, *etc.*); these defects have a significant impact on the mechanical properties of material;

• change in texture (under the influence of severe deformation, a change in the orientational structure of the crystals occurs, which results in the formation of material new texture);

• formation of nanostructures (in some cases, severe plastic deformation can lead to the formation of nanostructures in the material); these nanostructures have dimensions on a nanoscale and may have unique properties;

• improvement of mechanical properties (as a result of the evolution of microstructure under the influence of severe deformation, the material can acquire improved mechanical characteristics, such as increased strength, hardness, fatigue resistance, *etc.*).

It is important to emphasize that to achieve optimal results with SPD, it is necessary to control carefully process parameters such as temperature, strain rate, pressure, *etc*. Incorrect adjustment of these parameters can lead to undesirable effects or even degradation of material properties.

The effect of SPD on the final grain size of material has been studied in Ref. [65]. The authors of Ref. [65] concluded that a decrease in the grain size of a material is only a consequence of many different processes that accompany the plastic deformation of metals. The authors [65] carried out constitutive experimental, finite element method and discrete complex studies for two Cu–Cr–Zr copper alloys subjected to ECAP and medium-density fibreboard deformation processes. This combination of methods makes it possible to identify additional microstructural effects, such as micro- and macroscopic localization phenomena, combined dislocation phenomena, and associated evolution of dislocation cells and grains, inhomogeneities of the triple junction network and appearance of ultrafine grains, inhomogeneities of the triple junction network and appearance of ultrafine grains. In many cases, resulting deformation inhomogeneities play a significant role in both macro- and microscale deformation. Heterogeneity of the network of grain boundary transitions may be of decisive importance for the creation of nanostructured copper-based alloys suitable for use in electrical engineering. Deformation microstructures in

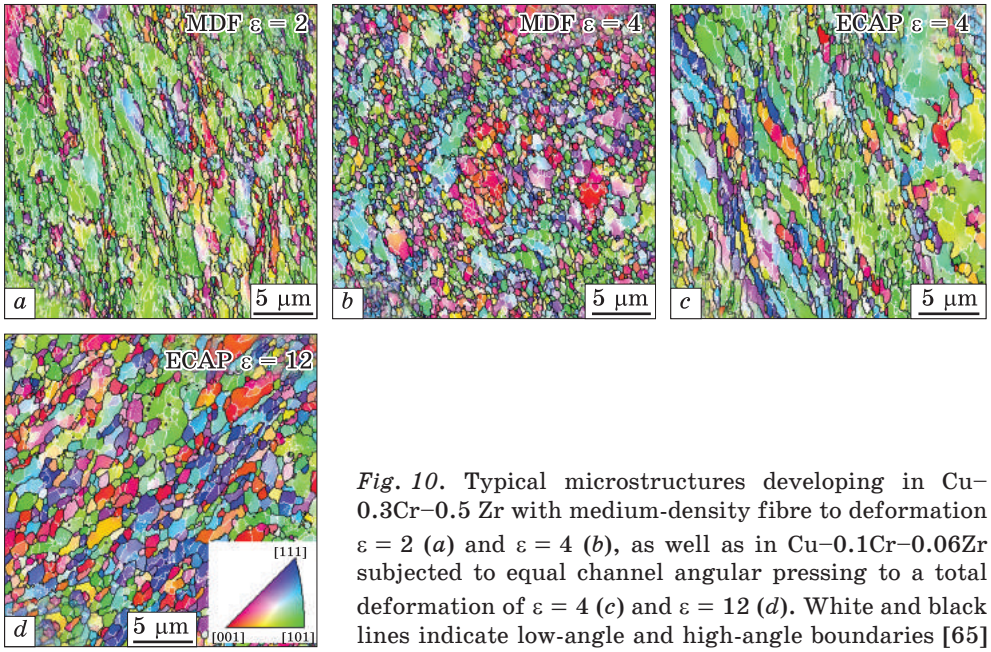

Fig. 10. Typical microstructures developing in Cu–0.3Cr–0.5 Zr with medium-density fibre to deformation ε = 2 (*a*) and ε = 4 (*b*), as well as in Cu–0.1Cr–0.06Zr subjected to equal channel angular pressing to a total deformation of ε = 4 (*c*) and ε = 12 (*d*). White and black lines indicate low-angle and high-angle boundaries [65]

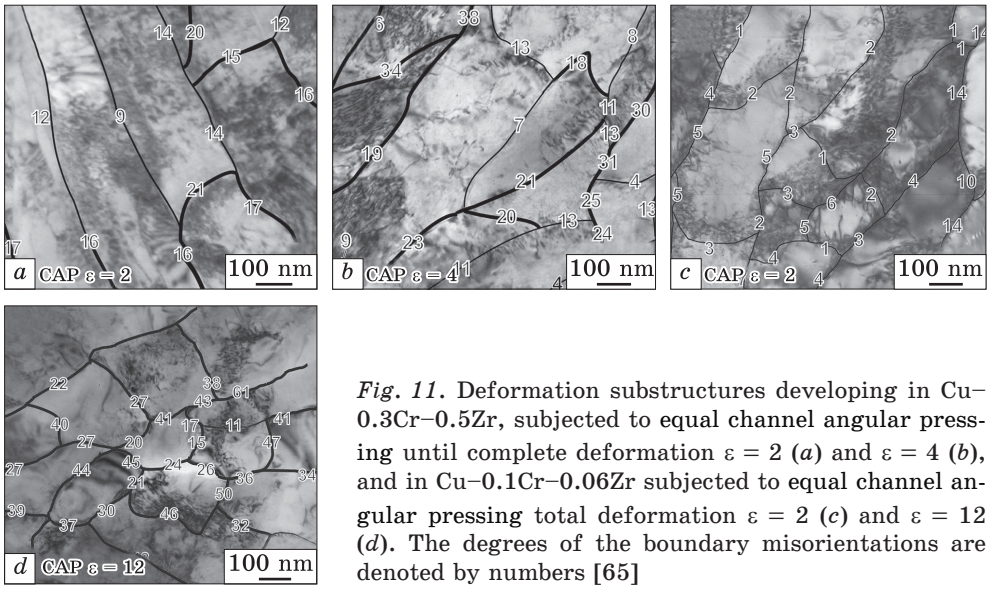

Fig. 11. Deformation substructures developing in Cu–0.3Cr–0.5Zr, subjected to equal channel angular pressing until complete deformation ε = 2 (*a*) and ε = 4 (*b*), and in Cu–0.1Cr–0.06Zr subjected to equal channel angular pressing total deformation ε = 2 (*c*) and ε = 12 (*d*). The degrees of the boundary misorientations are denoted by numbers [65]

copper alloys Cu–Cr–Zr subjected to ECAP and medium-density fibreboard are shown in detail in Fig. 10. Figure 11 shows in more detail the fine structures developing in the microshear deformation bands. Figure 11, *a* shows clearly that the microshear band developed in the aged Cu–0.3Cr–0.5Zr alloy at deformation 2 includes a frequent network of (sub)bounda-

ries from small angles to large ones. Therefore, the microshear band is a grain–subgrain mixture and can be considered a preferred nucleation site for UFG development. Ultrafine grains easily transform into microshear bands during severe plastic deformation (Fig. 11, *b*). In contrast to the dispersion-hardened Cu–0.3Cr–0.5Zr alloy, Cu–0.1Cr–0.06Zr alloy treated with a solution is characterized by a lower dislocation density, which leads to smaller, simultaneously developed misorientation angles of dislocation subboundaries strain levels (Fig. 11, *a* and *c*). In addition, the Cu–0.1Cr–0.06Zr alloy demonstrates sluggish kinetics of UFG development. The microshear band that appears at a total ECAP strain 2 in this alloy consists of predominantly flat dislocation subboundaries (Fig. 11, *c*). However, an increase in total strain leads to the development of UFG along the microshear band in a low-alloy solution of Cu–0.1Cr–0.06Zr (Fig. 11, *d*) [65].

The work [66] is devoted to the influence of the ECAP process on the deformability, microstructure and conductivity of the Cu–Co–Ni alloy during SPD. The evolution of microstructure was studied using microscopic observations and electron backscatter diffraction (EBSD) in a scanning electron microscope. The Vickers method was used to test the microhardness of samples after different variants of the ECAP process. Conductivity was measured using an eddy current conductivity meter based on the complex impedance of the measuring probe. The results showed the possibility of deforming Cu–Co–Ni alloys during the pressing process of ECAP corner channels and developing their microstructure and properties. The method is an effective means of strengthening the test copper alloy by thinning the microstructure. After the first pass, the grain size decreased by 80%. An increase in the plastic deformation temperature did not significantly affect the resulting level of microstructure fragmentation — the average grain size was 1.4–1.5 μm. Fragmentation of microstructure had a negligible effect on the conductivity, which after the ECAP process underwent fluctuations of ≈13 mS/m.

Authors of Ref. [67] studied the influence of ageing and ECAP on the evolution of Cu–Cr–Zr–Sc alloy microstructure and properties. Precipitation and SPD have been proven as effective methods for separately improving the properties of copper alloys. After 12 passes of combined thermomechanical processing, the high-angle distance between alloy grains was reduced to 300 nm, and many low-angle grain boundaries appeared. When the ECAP deformation reached 8 passes, stacking faults appeared in the grains due to severe shear deformation. After 12 passes, lamellar twins were discovered in the sample. Pre-ageing and pre-ECAP treatments can improve the microstructure of Cu–Cr–Zr–Sc alloy and significantly improve the mechanical properties and electrical conductivity of the alloy. After 12 passes of deformation and ageing at 400 °C for 15 min, the studied Cu–Cr–Zr–Sc alloy had an elongation of 17.5% and an electrical conductivity of 80.1%. Electron backscatter diffraction and Kikuchi trans-

mission diffraction methods were used to analyse the evolution of sample microstructure with different ECAP passes.

In the work [68], authors investigated the SPD methods of copper and Al–Cu alloy using multiple compression tests in a channel head. The materials were tested under plane strain conditions at a constant strain rate of 0.001 s^{-1}. Intensive grain refinement was observed, leading to the formation of nanosized grains after SPD with a concomitant increase in flow stress and hardness. The microstructure of highly deformed materials was studied using optical and scanning electron microscopes. Shear band formation has been identified as a failure mechanism in two-phase Al–Cu alloy. Results indicate the difficulty of obtaining strong plastic deformation for alloys having a two-phase microstructure.

Authors of Ref. [69] studied the tensile deformation behaviour of high-strength nanostructured Cu–Si solid solution alloys processed by high-pressure torsion (HPT) with 5 turns at room and low temperatures. With increasing Si concentration, the strength of nanostructured Cu–Si solid solution alloys increased significantly. Maximum tensile strength was 980 MPa at room temperature and 1350 MPa at 77 K in the Cu–2.04 Si [wt.%] alloy. This significant strengthening was achieved by grain refinement and increasing dislocation density through severe plastic deformation with the effect of Si addition in reducing the stacking fault energy of the Cu–Si alloy. With increasing Si concentration, the rate sensitivity of nanostructured Cu–Si solid solution alloys decreased due to an increase in dislocation density, which led to an acceleration of plastic instability of the samples under tension, caused by a decrease in the ability to rate hardening after necking.

The influence of various types of SPD (ECAP and quasi-hydrostatic extrusion at 77 and 300 K) on the structure formation of a dispersion-strengthened Cu–Cr–Zr alloy is studied in Ref. [70]. A combination of experimental methods was used. Sputtering with deuterium ions was used as a tool for layer-by-layer study of the alloy structure. The difference in the sputtering coefficients of the matrix (Cu) and precipitates (Cr + Zr) made it possible to visualize the structure of the alloy to a total depth of 0.5–1 μm. The influence of severe plastic deformation on the distribution of precipitates is considered. It is shown that the main feature of microstructure is associated with the high density of Cr-enriched precipitates, which completely determine the surface roughness. Their distribution is not related to grain size. It has been shown that the combination of equal-channel angular pressing and quasi-hydrostatic extrusion leads to an increase in the microhardness of the Cu–Cr–Zr alloy up to 2300 MPa in the case of low-temperature quasi-hydrostatic extrusion (at 77 K) and maintaining high conductivity. It has been proven that the high anisotropy of the shape of precipitates, microhardness and sputtering coefficient of the Cu–Cr–Zr alloy is due to ECAP.

5. Applications of Cu and Cu-Based Alloys after SPD

The areas of copper application are determined by its high electrical and thermal conductivity, ductility, and corrosion resistance. Copper is used for the manufacture of chemical equipment (heat exchangers, refrigerators, plasma torch parts, *etc*.), high-voltage power line wires, trolley wires, collector busbars of electric machines, furnaces for arc melting of active metals, water-cooled moulds, crystallisers, *etc*. More than 30% of copper is used in the form of copper alloys — brasses and bronzes [71–73].

High-tech brasses are used to produce products that require deep drawing, such as radiator and condenser tubes, bellows, flexible hoses, pipes, and tapes. Multicomponent brasses, which have fairly high strength and corrosion resistance, are used in shipbuilding, electrical engineering, and heating engineering.

The bronzes, aluminium bronzes are the most widely used. They are used in marine shipbuilding, general mechanical engineering, locomotive and carriage building, automobile and aircraft manufacturing for the manufacture of critical parts: gears, bushings, valve seats, pressure screw nuts, springs and spring-loaded products [74, 75].

Tin bronzes with phosphorus, which have high antifriction and anticorrosion properties, are used in mechanical engineering for the manufacture of thrust bearings for heavy cranes, lead screw nuts, gears, worm wheels and other parts that operate under high friction. Some alloys are used for parts of water, steam and gas fittings. A group of bronzes alloyed with phosphorus, with high elastic properties, is used for the manufacture of round and flat springs.

Beryllium bronze is used for the most critical products — flat springs, membranes, precision instrument-making parts, spring elements of electronic devices, and electrodes of welding machines. Since beryllium bronzes do not produce sparks upon impact, they are used to make tools for working in hazardous areas.

Copper alloys with improved strength and fatigue resistance resulting from SPD can be used in bearings, bushings, gears and other mechanical components. In addition, due to their improved strength, lightness and resistance to high temperatures, they can be used in the production of aerospace components. In the medical field, copper alloys can be used to make medical instruments, prosthetics and other medical devices. Brass and other copper alloys after SPD can be used in the jewellery industry to create jewellery, coins, and other decorative items. Bronze and other copper alloys with good corrosion resistance can be used in the production of marine components and equipment.

It is important to emphasize that the selection of specific materials including bulk copper alloys (as well as a variety of other substitutional–interstitial alloys [76, 77]) and Cu nanoparticles [78] for the application of

them depends on the requirements of specific application and characteristics, which are required for a particular component or product [35].

6. Conclusions

In conclusion, SPD is a powerful technique that can significantly modify the properties of copper and its alloys. This process leads to significant changes in the microstructure of the material, which in turn affects its mechanical, electrical and other characteristics.

Among the most significant results of SPD, it is worth highlighting the increase in strength, fatigue resistance and elastic properties of the material. These changes make copper and its alloys subjected to SPD very attractive for use in many industries, including electrical engineering, aviation, medicine and others.

However, despite the obvious advantages, it should be noted that the successful implementation of SPD requires careful control of process parameters, such as temperature, strain rate, and pressure, which allows achieving the desired material characteristics.

Generally, results of studies in the field of changes in the properties of copper and its alloys after SPD highlight the potential of this method for creating materials with improved characteristics, which opens new prospects in various fields of technology and industry.

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ЕВОЛЮЦІЯ МІКРОСТРУКТУРИ МІДІ ТА ЇЇ СПЛАВІВ У ПРОЦЕСІ ІНТЕНСИВНОЇ ПЛАСТИЧНОЇ ДЕФОРМАЦІЇ

Досліджено зміни властивостей міді та її сплавів у результаті інтенсивної пластичної деформації (ІПД). Аналіз даних літератури свідчить про те, що сучасне матеріалознавство спрямовано на вирішення задачі одержання ультрадрібнозернистих

матеріалів з великокутовими межами зерен. Методами ІПД вдається одержати так звані об'ємні наноструктурні матеріали з розміром зерен у 0,1–0,2 мкм і специфічними субструктурами, що містять ґратчасті й зерномежові дислокації та дисклінації. Такі структури характеризуються великими пружними спотвореннями кристалічної ґратки. Вважається, що подібні дрібнозернисті структури мають забезпечити одночасно високий рівень характеристик пластичності та міцності за рахунок особливих напружених висококутових зерен. Розглянуто методи ІПД, такі як кручення зі зсувом, рівноканальне кутове пресування, гвинтова екструзія, всебічне кування, вальцювання зі зсувом тощо. Серед найістотніших результатів ІПД варто виділити підвищення міцності, втомної стійкості та пружних властивостей матеріалу. Ці зміни роблять мідь та її сплави, піддані ІПД, дуже привабливими для використання у багатьох галузях промисловості, зокрема електротехніці, авіації, медицині.

Ключові слова: мідь, мідні сплави, інтенсивна пластична деформація, дрібнозерниста мікроструктура, механічні властивості.