

## Article

# Obtaining Symmetrical Gradient Structure in Copper Wire by Combined Processing

Andrey Volokitin <sup>1,\*</sup>, Irina Volokitina <sup>2,\*</sup> , Mehmet Seref Sonmez <sup>3</sup> , Anastasiya Denissova <sup>1,\*</sup> and Zoya Gelmanova <sup>4</sup>

<sup>1</sup> Department of Metal Forming, Karaganda Industrial University, Temirtau 101400, Kazakhstan

<sup>2</sup> Department of Metallurgy and Material Science, Karaganda Industrial University, Temirtau 101400, Kazakhstan

<sup>3</sup> Department of Metallurgical and Materials Engineering, Faculty of Chemistry and Metallurgy, Istanbul Technical University, Sariyer, Istanbul 34469, Turkey; ssonmez@itu.edu.tr

<sup>4</sup> Department of Management and Business, Karaganda Industrial University, Temirtau 101400, Kazakhstan; zoyakgiu@mail.ru

\* Correspondence: a.volokitin@tttu.edu.kz (A.V.); i.volokitina@tttu.edu.kz (I.V.); knastyai@mail.ru (A.D.); Tel.: +7-701-250-93-53 (I.V.)

**Abstract:** Traditionally, structural wire is characterized by a homogeneous microstructure, where the average grain size in different parts of the wire is uniform. According to the classical Hall–Petch relationship, a homogeneous polycrystalline metal can be strengthened by decreasing the average grain size since an increase in the volume fraction of grain boundaries will further impede the motion of dislocations. However, a decrease in the grain size inevitably leads to a decrease in the ductility and deformability of the material due to limited dislocation mobility. Putting a gradient microstructure into the wire has promising potential for overcoming the compromise between strength and ductility. This is proposed a new combined technology in this paper in order to obtain a gradient microstructure. This technology consists of deforming the wire in a rotating equal-channel step die and subsequent traditional drawing. Deformation of copper wire with a diameter of 6.5 mm to a diameter of 5.0 mm was carried out in three passes at room temperature. As a result of such processing, a gradient microstructure with a surface nanostructured layer (grain size ~400 nm) with a gradual increase in grain size towards the center of the wire was obtained. As a result, the microhardness in the surface zone was 1150 MPa, 770 Mpa in the neutral zone, and 685 MPa in the central zone of the wire. Such a symmetrical spread of microhardness, observed over the entire cross-section of the rod, is a direct confirmation of the presence of a gradient microstructure in deformed materials. The strength characteristics of the wire were doubled: the tensile strength increased from 335 MPa to 675 MPa, and the yield strength from 230 MPa to 445 MPa. At the same time, the relative elongation decreased from 20% to 16%, and the relative contraction from 28% to 23%. Despite the fact that the ductility of copper is decreased after cyclic deformation, its values remain at a fairly high level. The validity of all results is confirmed by numerous experiments using a complex of traditional and modern research methods, which include optical, scanning, and transmission microscopy; determination of mechanical properties under tension; and measurement of hardness and electrical resistance. These methods allow reliable interpretation of the fine microstructure of the wire and provide information on its strength, plastic, and electrical properties.

**Keywords:** drawing; microstructure; copper; severe plastic deformation; wire



**Citation:** Volokitin, A.; Volokitina, I.; Sonmez, M.S.; Denissova, A.; Gelmanova, Z. Obtaining Symmetrical Gradient Structure in Copper Wire by Combined Processing. *Symmetry* **2024**, *16*, 1515. <https://doi.org/10.3390/sym16111515>

Academic Editors: Vasilis K. Oikonomou and Sergei Odintsov

Received: 10 September 2024

Revised: 28 October 2024

Accepted: 1 November 2024

Published: 12 November 2024



**Copyright:** © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

## 1. Introduction

Metallic materials are characterized by special properties determined by their structure. The mechanical and exploitational characteristics of these materials are critical for their practical application. It is quite obvious that the properties of materials are based on

structure and can be significantly affected by the manufacturing process. Modern production methods give an opportunity to create specialized materials based on both steels and non-ferrous metals (for example, gradient or composite structures [1–5]), which are able to satisfy the individual and ever-increasing demands driven by the requirements of modern technologies. Nevertheless, the production of such materials necessitates adding expensive alloying or the introduction of energy and cost-intensive types of heat treatment in order to enhance their mechanical properties (especially specific strength). One promising option is to improve the performance characteristics of metallic materials by reducing the grain size in their structure. The most recently used method is severe plastic deformation (SPD), which allows ultrafine-grained structures and nanostructures to be obtained [6–10].

Two factors that are critical in the preparation of such materials are processing temperature and shear stress. Shear stress, among other things, is a factor that influences the closure of pores, thereby increasing the density of the prepared materials. Therefore, deformation processes in which the shear stress component predominates are most suitable for producing very fine-grained structures. The relationship between mechanical properties and grain size is usually described by the Hall–Petch relationship, which determines the increase in strength characteristics as grain size decreases. However, this dependence is not entirely unambiguous, since it is based on the interaction between dislocations and sub-grain boundaries.

The formation of metallic materials by severe plastic deformation is characterized by a rather complex distribution of the supplied mechanical energy. Depending on the deformation conditions, the vast majority of this energy (in some cases up to 98%) can be dissipated as heat of deformation. The remaining part of this energy is used to effect the desired change in shape, i.e., plastic deformation [11].

The heat of deformation released during the process of deformation is not distributed uniformly throughout the volume of metal being formed, but only in precisely localized areas, so-called heat lines. These thermal lines have been confirmed to correspond to areas of shear strain concentration (very often called shear bands) [12]. A smaller part of mechanical energy is stored in the material in the form of emerging and then moving dislocations. An increase in dislocation density gradually complicates the process of plastic deformation and thus becomes the main factor causing an increase in deformation stresses and strain hardening. Thus, in these cases, the stable continuous plastic flow of the material is replaced by intermittent plastic flow localized in shear bands [13]. As a result, SPD is accompanied by two characteristic features of structure evolution: the continuous evolution of dislocation substructure due to crystallographic slip in the subregions of split grain, and the highly localized flow of material in connection with the formation of shear bands. Therefore, factors such as the deformation mode and loading history are decisive for the specific implementation and sequence of the evolution of these characteristic phenomena [14].

The original goal of traditional forming methods was to achieve the desired shape of a molded workpiece. However, over time it became obvious that it was necessary to influence its final properties. Increasing demands to reduce weight and simultaneously increase the depth and strength of some materials have led to the optimization of all production processes and various modifications of the original deformation technologies. Therefore, recently they began to use combinations of SPD technologies with traditional deformation methods. Among the early methods based on a combination of deformation technologies, the following processes, consisting of individual technological solutions, can be especially noted. Thus, for example, products from Cu, Al, Ti, or Mg alloys have been prepared under laboratory conditions using a combination of equal-channel angular pressing (ECAP) with rolling [15], extrusion [16], drawing [17], extrusion and drawing [18], forging and drawing [19], and rotary forging [20], followed by thermal [21] or thermo-mechanical treatment [22]. One example is the use of nonmonotonic shear deformation when drawing steel rods, which forms a gradient microstructure. A nanostructure with

ultra-high microhardness (HV-7 GPa) is created in the surface layers, which significantly increases rod wear resistance.

The technology developed in this work is based on the well-known ECAP method [23–25]. Based on ECAP, several modified methods that are aimed at partially eliminating the disadvantages of the classical ECAP method have been developed. For processing long workpieces by simple shear using the ECAP method in continuous mode, a device based on the “Conform” scheme has been developed [26]. The disadvantages of this method include the need to create special equipment; the fact that the geometry of the tool does not allow the workpiece to be processed according to the “circle-circle” scheme; and that tribological difficulties arise during deformation using this method since the deformation force is formed due to active friction forces. To eliminate these disadvantages, the Multi-ECAP–Conform method was developed [27]. The main feature of this method is the combination of the ECAP method in parallel channels and the continuous ECAP–Conform process. However, this method also has disadvantages, which include the need to prepare special, complex equipment; the need to comply with ideal tribological conditions of the process; and low process productivity. Another new method of SPD for processing and improving the properties of wire is the ECA drawing method [28]. The method is implemented as follows: carbon wire is pulled through a special profile assembly tool. Continuity of the deformation process is ensured by combining the ECA drawing with the traditional method of wire drawing. This method’s disadvantages include the extremely asymmetric deformation scheme, which causes non-uniform tribological conditions as well as contact forces between wire and tool, and, as a result, leads to the formation of a non-uniform structure and properties along the cross-section of wire.

Recently, there has been a tendency to combine traditional metal deformation methods to obtain new processing technologies. One of these methods for producing wire with an ultrafine-grained structure is drawing with torsion and alternating bending [29], as well as ECAP in parallel channels that are rotating [30–32]. However, an important difference between our technology and other known SPD methods is that in an equal-channel stepped die, the wire is twisted due to the die rotation around the axis of the wire, which allows twisting throughout the entire volume of the workpiece with subsequent calibration of the cross-section due to the passage of the die channel. The choice of drawing method after SPD in a rotating die is based on the process simplicity and high tensile stresses that arise directly in the deformation zone. As a result, the wire cross-section not only takes on a round and symmetrical shape but also causes the annihilation of irregularities, which makes it possible to give the wire a marketable appearance without the need for additional straightening operations.

The novelty of this work is the study of gradient microstructure formation and increased mechanical properties in copper wire subjected to deformation using a newly developed technology, which consists of deforming the wire in a rotating equal-channel step die and subsequent traditional drawing. The economic effect of this research is achieved by obtaining the high-strength copper wire with a symmetrical gradient structure. By obtaining a symmetrically distributed microstructure from the center to the periphery of the workpiece, there is an opportunity to predict the finished product’s physical and mechanical properties, which will affect its performance properties.

## 2. Materials and Methods

Microcomposite copper alloys are one of the promising classes of metals that make it possible to create high-strength and electrical conductivity combinations. Systems with limited solubility of components, such as Cu-Ag, Cu-Nb, and Cu-Fe, are the basis of these alloys. Their fairly high electrical conductivity is ensured by a copper matrix, which contains virtually no impurities. Simultaneously, extremely high strength can be achieved due to the second phase’s sufficient dispersion. Highly dispersed structures in such material can be created using large plastic deformation. Typically, microcomposite alloys are processed by drawing. However, in some cases, when the requirements for the mechanical

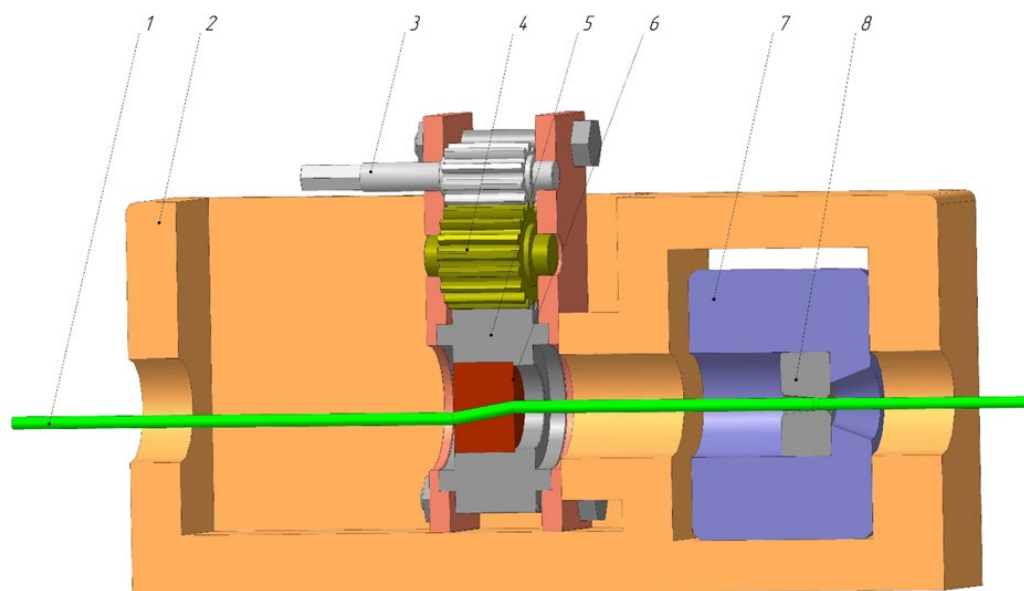
properties of semi-finished products such as wire increase, the number of drawing cycles can be reduced by changing the usual deformation pattern. For this reason, wires produced from low-alloyed copper alloys are certainly more costly than copper and are still in limited supply. However, their benefits can compensate for this disadvantage and effectively resolve a number of technical issues related to copper saving and the development of modern technologies.

Alloys of the Cu-Fe system are attractive because of their relative cheapness. Therefore, in this work, the Cu-14Fe alloy (86 mass% Cu) was used. The properties of the initial copper alloy are presented in Table 1.

**Table 1.** Properties of initial copper alloy.

| Tensile Strength,<br>$R_m$ , MPa | Yield Strength,<br>$R_{p0.2}$ , MPa | Relative<br>Elongation, $E$ , % | Relative<br>Contraction, $\psi$ , % | Microhardness,<br>MPa |
|----------------------------------|-------------------------------------|---------------------------------|-------------------------------------|-----------------------|
| 335                              | 230                                 | 20                              | 28                                  | 415                   |

The idea suggested in this paper is to deform copper wire in a rotating equal-channel stepped die, and subsequently use traditional drawing. The die is rotated about the copper wire axis, creating tension through equal-channel angular drawing and twisting in the die. The die rotation is performed in a specially developed device, which is mounted in the drawing mill equipment line in the process lubrication unit, allowing the lubricant to be supplied to the die and the wire in the drawing unit (Figure 1). The design of the rotating module is a series of alternating gears mounted in a vertical plane and connected by two frames. The special design of a rotating die consists of two separate elements, due to which it is possible to insert a pointed wire into the die and secure the front end of the wire with pull-out pliers. Two die channels (input and output) are located parallel to each other and have a conical shape to facilitate the removal of wire and the introduction of lubricant into the die. The central channel is located at an angle of  $145^\circ$  to the inlet and outlet channels. The rotating bandage has the same geometry, which allows us to connect two segments without additional fasteners according to the type of wedging.



**Figure 1.** Scheme of the proposed wire deformation technology: 1—wire; 2—setup housing; 3—drive gear; 4—intermediate gear; 5—rotating die case; 6—rotating die; 7—cold drawing die case; 8—drawing die.

To conduct the physical experiment on the copper wire deformation, an industrial drum drawing machine B-1/550M (Chelyabinsk, Russia) was used. The deformation was

performed at room temperature, and the number of passes was three. The original size of the copper wire was 6.5 mm. After 3 deformation cycles, the wire diameter was 5.0 mm. The deformation parameters are given in Table 2.

**Table 2.** Parameters of deformation process.

| Passage No.              | ECA Twisting |             |                  |                | Drawing    |            |           |                 |             |                   |                               |                    |
|--------------------------|--------------|-------------|------------------|----------------|------------|------------|-----------|-----------------|-------------|-------------------|-------------------------------|--------------------|
|                          | $D_0$ , mm   | $V_0$ , m/s | $\omega$ , rev/s | $\sigma$ , MPa | $D_1$ , mm | $D_2$ , mm | $\lambda$ | $V_{0-1}$ , m/s | $V_1$ , m/s | $\varepsilon$ , % | $\sigma$ , MPa                | $\varepsilon_{ln}$ |
| 1 pass                   | 6.5          | 0.43        | 18               | 65.00          | 6.5        | 6.0        | 1.17      | 0.43            | 0.50        | 14.79             | 144.00                        | 0.16               |
| 2nd pass                 | 6.0          | 0.42        | 18               | 68.00          | 6.0        | 5.5        | 1.19      | 0.42            | 0.50        | 15.97             | 147.00                        | 0.17               |
| 3rd pass                 | 5.5          | 0.41        | 18               | 71.00          | 5.5        | 5.0        | 1.21      | 0.41            | 0.50        | 17.36             | 149.00                        | 0.19               |
| $\varepsilon$ (sum), 41% |              |             |                  |                |            |            |           |                 |             |                   | $\varepsilon_{ln}$ (sum) 0.52 |                    |

Where  $D_0$ —initial wire diameter before entering the die;  $V_0$ —drawing speed in the die;  $\omega$ —angular velocity of the die rotation;  $\sigma$ —deformation stress;  $D_1$ —wire diameter before entering the die;  $D_2$ —wire diameter after exiting the die;  $\lambda$ —drawing ratio;  $V_{0-1}$ —initial drawing speed;  $V_1$ —speed at the die exit;  $\varepsilon$ —deformation degree;  $\varepsilon_{ln}$ —logarithmic degree of deformation;  $\varepsilon$  (sum)—total degree of deformation.

The microstructure of the deformed copper samples was studied using optical and transmission microscopy. The optical microscopy was conducted on a Leica microscope. A more detailed analysis was performed on a JEM2100 transmission microscope (Jeol Ltd., Tokyo, Japan) at an acquiring voltage of 160–200 kV. Samples were produced from the flats which were perpendicular and parallel to the direction of the pattern. Electropolishing was conducted in a solution of 250 mL of  $H_3PO_4$ , 500 mL of distilled water, 50 mL of propanol, 250 mL of ethanol, and 5 g urea at 5 °C and 5 V.

Diffraction techniques for measuring crystal orientation in localized areas, such as EBSD, now have a great interest in characterizing small-scale microstructural features. EBSD analysis was carried out on a Philips XL-30 REM (MEMS and Nanotechnology Exchange, Arlington, VA, USA) instrument with a field cathode. Statistical analysis was performed using a critical misorientation angle of 15° to distinguish between low-angle and high-angle grain boundaries.

In [33], it was established that when obtaining a pronounced gradient microstructure, it is possible to split the sample cross-section into three zones: surface (0.75 radius), intermediate (0.5 radius), and central. Therefore, in our work, we will adhere to the same division.

A LEICA DM IRM HC (Germany) optical microscope equipped with an attachment for determining the microhardness of individual phases was used to study gradient microstructure. Microhardness was measured using the Vickers method on smooth etched samples after EBSD. The method of indenting a diamond pyramid with an angle between opposite faces of 136° under a load of P 0.5 N was used. The measurement step is shown in Figure 2a, and the sample size depends on the resulting wire diameter. Each point on the microhardness graphs is the average of five measurements. The method was chosen by the existence of a gradient microstructure in the wire.

Mechanical tensile testing was executed in compliance with GOST 11701-84 on the Instron 5882 (Instron, Norwood, MA, USA) universal testing machine at room temperature with a traverse movement speed of 2 mm/min. The dimensions of the test specimens are shown in Figure 2b.

To measure conductivity, the AUTOSIGMA 2000 (Hyoshin Mechatronics Co., Ltd., Fuchuan, Republic of Korea) instrument was used, which can measure both in % IACS and MS/m and operate at frequencies in the range of 60–500 kHz with measurement accuracy of  $\pm 0.1$  at room temperature.

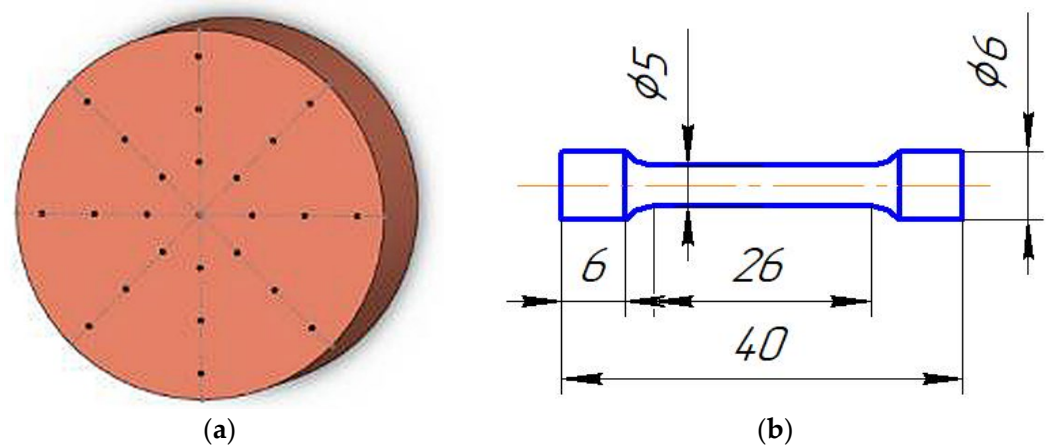


Figure 2. Samples for testing: (a)—determination of microhardness; (b)—tension.

### 3. Results and Discussion

For a general observation of the gradient microstructure in the wire, the first studies were carried out using an optical microscope (Figure 3). The aim of these studies was to obtain a general idea of how the wire microstructure changes during multiple deformation cycles. Analysis of the microstructure showed that with each subsequent deformation cycle, the gradient of the microstructure in the cross-section of the wire becomes more pronounced. This phenomenon is due to the fact that the deformation of the wire does not occur uniformly across its entire cross-section.

In the undeformed (annealed) state, the wire structure exhibits a microstructure with an average grain of  $67\ \mu\text{m}$  and a large number of twins (Figure 3a).

The described microstructure gradient of the wire is a consequence of uneven stress distribution during the deformation process. Surface layers experience more intense impact, while the central zone is under less stress. This results in the formation of specific structures characteristic of each zone. Next, EBSD analysis, which allows one to determine misorientation angles between grains and identify grain boundaries and sub-boundaries, as well as many other types of useful information, was performed (Figure 4).

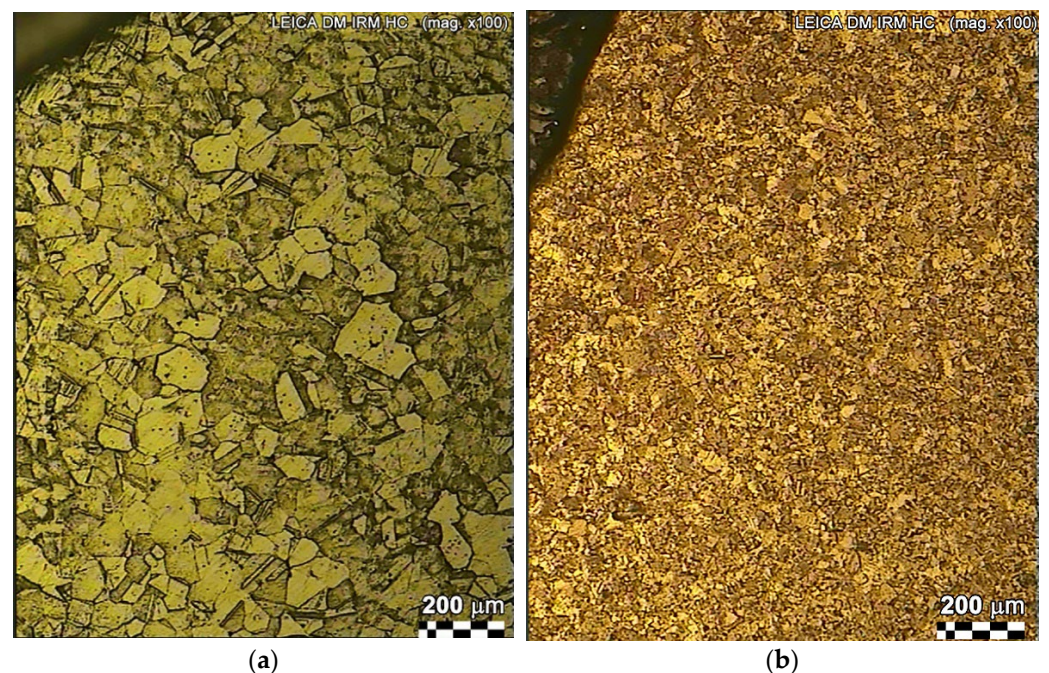
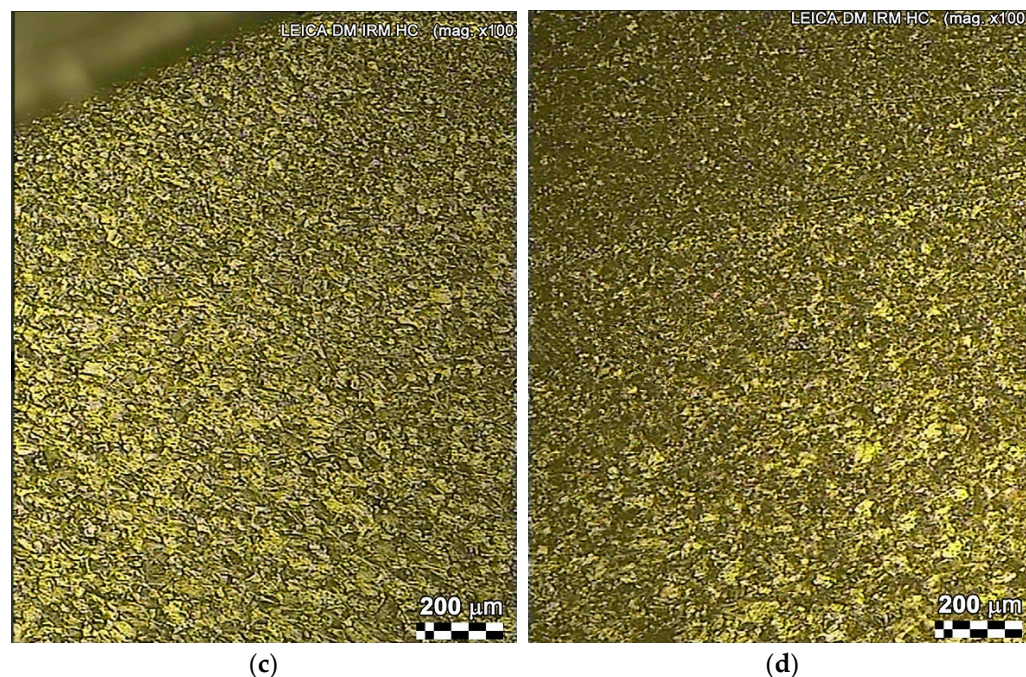
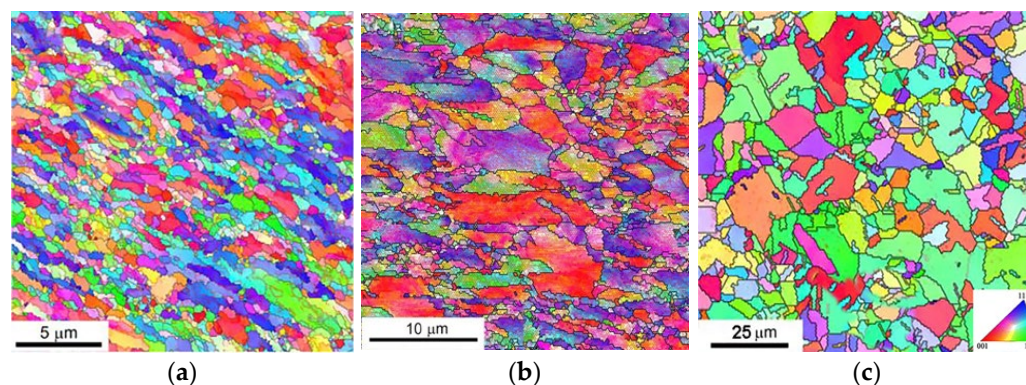


Figure 3. Cont.



**Figure 3.** Optical micrographs of copper wire (transverse direction): (a)—initial; (b)—1 deformation cycle; (c)—2 deformation cycles; (d)—4 deformation cycles.

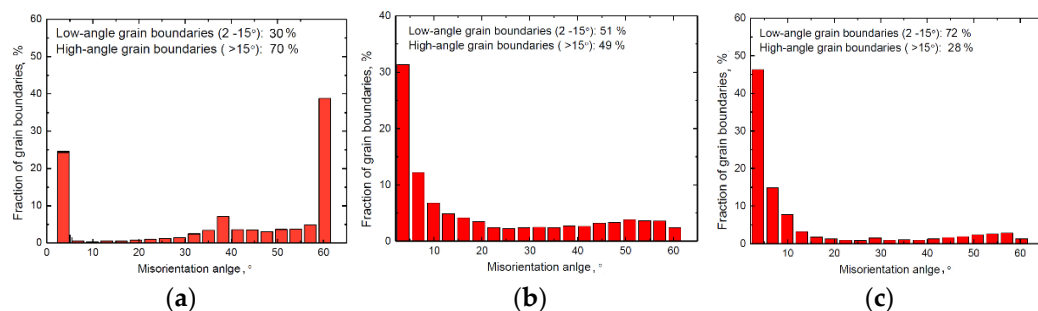


**Figure 4.** EBSD analysis after 3 passes of deformation (transverse direction): (a)—surface zone; (b)—intermediate zone; (c)—central zone.

The deformation texture of the copper wire after drawing was examined both in the center and at the periphery of the sample. The obtained data allowed us to conclude that in the central region of the copper wire sample, there is a clearly expressed complex axial texture consisting of two components:  $\langle 100 \rangle$  and  $\langle 111 \rangle$ . The texture after three deformation cycles has 65% of crystallites with  $\langle 111 \rangle$  orientation and 35% of crystallites with  $\langle 100 \rangle$  orientation. Orientation  $\langle 111 \rangle$  is more consistent and plausible, as this texture requires sliding in three out of six possible directions  $\langle 110 \rangle$ , while to create a  $\langle 100 \rangle$  texture, sliding must occur in four directions  $\langle 110 \rangle$ . Thus, the  $\langle 111 \rangle$  texture dominates both at the surface and in the intermediate zone (Figure 4a,b). The texture  $\langle 100 \rangle$  is seen to a larger degree in the central zone (Figure 4c). The appearance of the  $\langle 112 \rangle$  texture is observed in the surface zone, which can be treated as a recrystallization effect caused by heating the wire surface during the friction process of twisting the wire in an equal-channel stepped die and drawing. This can be interpreted also by the effect of three systems of deformation in the surface zone: shear, compression, and tension, whereas only two systems—tension and compression—act in the central zone.

Based on the obtained results, it is expected that the surface and intermediate zones of the wire will have a high elastic modulus due to the presence of a large number of  $\langle 111 \rangle$  orientations in the structure, as a result of which high strength and plastic properties are expected to be obtained. Since the texture with the  $\langle 111 \rangle$  orientation is preferable for metals with an FCC lattice, the yield strength and tensile strength values are the lowest in the  $\langle 001 \rangle$  direction.

The histogram of the misorientation angles shown in Figure 5 additionally demonstrates boundary distribution information on disorientation angles in different zones of the copper wire.



**Figure 5.** Histogram of misorientation angles obtained after 3 deformation passes: (a)—surface zone; (b)—intermediate zone; (c)—central zone.

A study of the deformed wire microstructure revealed a clear relationship between the distribution of high-angle grain boundaries (HAGBs) and low-angle grain boundaries (LAGBs) in different zones of the sample.

The surface zone of the wire, subject to the greatest stress and shear deformation, is characterized by the largest number of HAGBs. This is related to the fact that cyclic deformations, especially in the surface layer, encourage the rapid generation of new grains, which are different from each other in their orientation. Over three deformation cycles, about 70% of the grains formed during deformation exhibit an orientation angle of  $30^\circ$  or more, which is typical for HAGBs (Figure 5a).

The neutral zone of the wire shows a more uniform distribution of grain boundaries. Here, elongated regions are observed, separated by both LAGBs and HAGBs. In contrast to the surface zone, in the neutral zone, the intensity of new grain formation is much lower, therefore the proportion of HAGBs in it is smaller and amounts to 49% (Figure 5b).

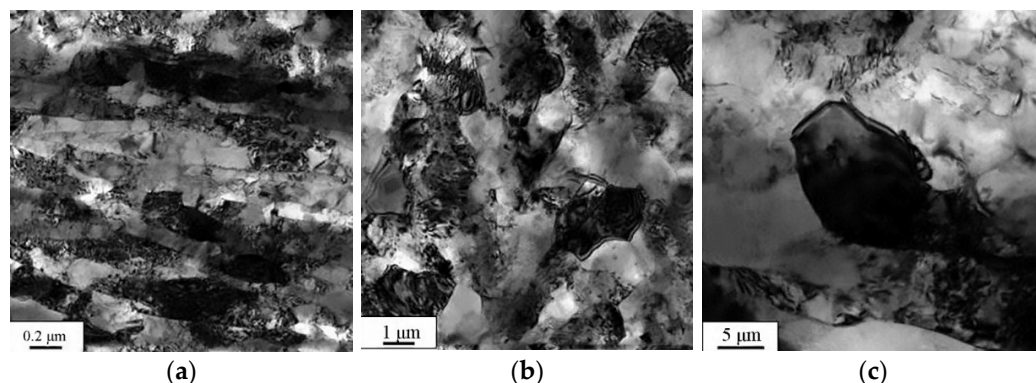
The central zone of the wire, subject to the least shear deformation, is therefore characterized by a predominance of LAGBs  $\approx 72\%$ . In this zone, where deformation is minimal, the formation of new grains practically does not occur (Figure 5c).

High-angle boundaries increasing in copper wire are due to the occurrence of dynamic recovery and recrystallization during twisting in an equal-channel stepped die. Drawing after twisting leads to a more active occurrence of these processes.

The studies have shown that depending on the deformation depth, the number of twin boundaries varies significantly. For example, in the surface zone of the deformed wire after three deformation passes, the percentage of  $\Sigma 3$  boundaries is 28%, in the intermediate zone—42%, and in the central zone—15%. Data obtained from various studies [34,35] confirm that twin formation requires a higher voltage compared to the voltage required to activate slip. In this case, the smaller the grain size, the faster the voltage required for the formation of twins increases. This explains the observed distribution of twin boundaries in the deformed wire. In the middle layer, where the grain size is smaller than in the surface and central layers, the stress for twin formation is achieved at a lower total stress, which leads to more active twinning.

For wire formed during deformation, a gradient microstructure detailed study was carried out using a transmission microscope (Figure 6). TEM examination of deformed material after three deformation passes revealed a complex evolution of microstructure

depending on the sample depth. Clear zoning is observed, reflecting the different effects of deformation on various material areas.



**Figure 6.** Microphotographs obtained after 3 passes of deformation (transverse direction): (a)—surface zone; (b)—intermediate zone; (c)—central zone.

The surface zone is characterized by the formation of submicron-elongated grains with a size of about 400 nm (Figure 6a). These grains have a thin and clear boundary, which indicates their high degree of purity. This structure arises as a result of intense sliding of dislocations, which form new grains during deformation. This zone is characterized by a lamellar structure—an alternation of elongated grains and thin layers formed by an accumulation of dislocations.

The intermediate zone demonstrates the development of a cellular structure with a grain size of 5  $\mu\text{m}$  (Figure 6b). It is formed by a grid of dislocation walls that separate grains into subgrains (subgrain boundaries). This structure is formed as a result of dislocation interaction, which, unable to move freely, forms clusters called dislocation walls.

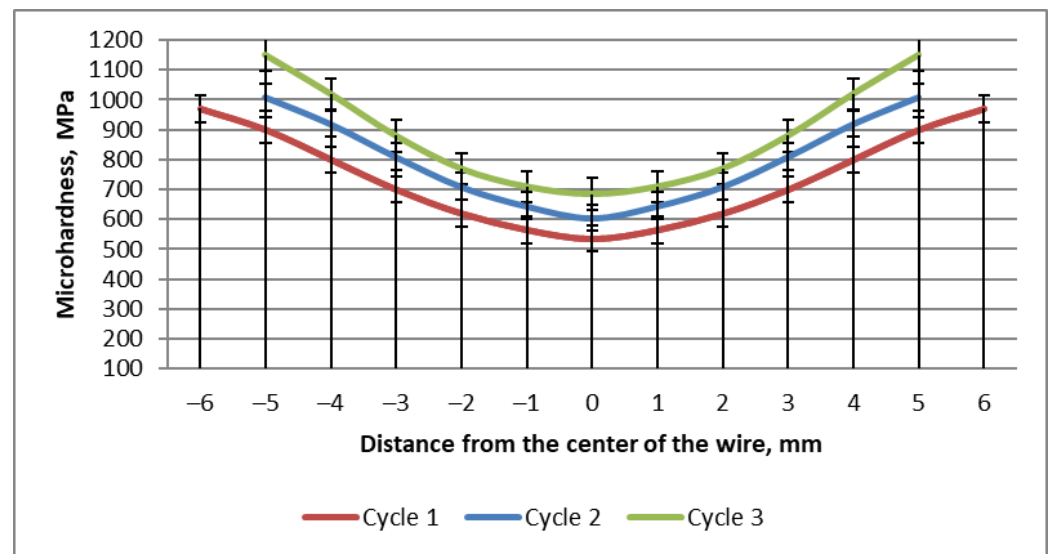
The central zone is presented by a large number of annealing twins in the grains, which are a legacy of the original microstructure (Figure 6c). They grow from grain boundaries, which often have an uneven, blurred structure, indicating their complex history of formation. The annealing twins that broke up the grains indicate that the deformation in the central zone was less intense than at the surface and affected the already existing structure. Most grain boundaries are thin and blurred.

To check the presence of a gradient microstructure in the wire, tests to determine the microhardness of the cross-section were conducted. Figure 7 shows the change in microhardness value on the copper wire cross-section after each deformation cycle. The center of the wire cross-section is marked with the number 0 on the horizontal line of the graph. Microhardness measurements were then taken every 1 mm to the left (negative numbers on the horizontal line) and to the right (positive numbers on the horizontal line) from the center of the wire. Analysis of these graphs allows us to trace in detail the change in microhardness depending on the distance from the wire surface and the number of deformation cycles.

The initial microhardness of the copper wire, measured along the entire perimeter, was 415 MPa. This suggests that the wire before deformation was homogeneous and had standard mechanical properties. However, after three passes of deformation of copper wire by twisting in a die and drawing it between 6.5 mm and 6.0 mm diameters, the microhardness increased significantly and acquired a pronounced gradient character. According to the measurement results, it was shown that the microhardness of the surface zone was 1150 MPa, the neutral zone was 770 MPa, and the central zone of the wire was 685 MPa. Such a symmetrical spread of microhardness, observed over the entire cross-section of the rod, is a direct confirmation of the presence of a gradient microstructure.

In this case, due to plastic deformation, the formation of dislocations, atomic defects, and subgrains occurs in the surface zone of the wire, which increases the strength and hardness of the material. With distance from the surface of the rod, the concentration of

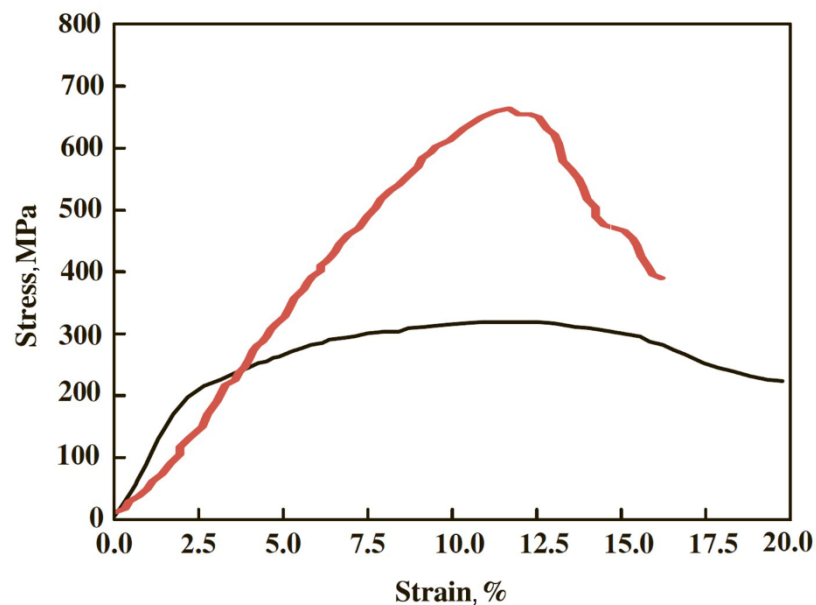
these structural elements gradually decreases, which leads to a decrease in microhardness in the neutral and center zones of the wire.



**Figure 7.** Microhardness distribution over the wire cross-section after each deformation pass.

These results are of great practical importance for optimizing metal processing processes, as they allow one to determine the optimal deformation modes to achieve the required mechanical properties of materials and their symmetry relative to the center of the wire.

Figure 8 shows the stress–strain curves for copper wire samples subjected to tensile tests. These curves reflect the behavior of materials under load and allow us to determine key mechanical characteristics after each deformation pass.

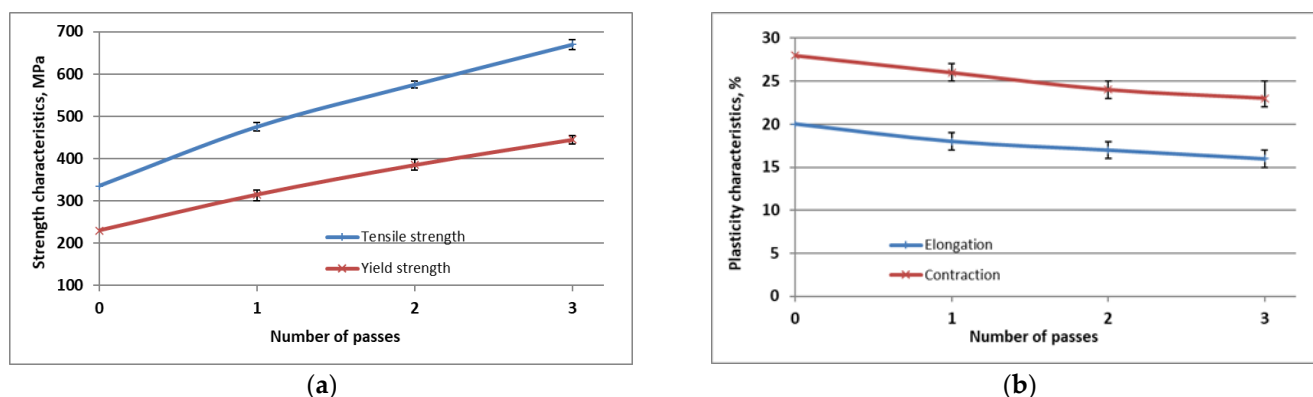


**Figure 8.** Tensile diagrams before (black line) and after (red line) deformation.

Analysis of the curves shows a significant influence of deformation on the copper wire's strength properties.

The tensile strength of the strained wire is doubled after three passes in comparison to the unstrained wire, rising from 335 MPa to 675 MPa. This indicates that deformation

improves the tensile strength of the material. Flow strength, which defines the initiation of plastic deformation, was also increased after deformation in three passes from 230 MPa to 445 MPa, which is a 93% improvement (Figure 9a).



**Figure 9.** Changes in mechanical properties graphs after each deformation pass: (a)—characteristics of strength; (b)—characteristics of plastic.

At the same time, relative elongation ( $E$ ), which characterizes the ability of a material to stretch before failure, decreases from 20% to 16%. The relative contraction ( $\psi$ ) decreased from 28% to 23%. Despite the fact that the ductility of copper decreases after cyclic deformation, its parameters stay at a rather high rate. This is caused by the material gradient microstructure formation, which is an irregular spreading of structural elements in the metal volume (Figure 9b).

Cyclic deformation, i.e., a load repeatedly applied and removed, causes the formation of dislocations—the linear defects in the crystal lattice of copper. As these dislocations slide over each other, they interact to form a complex spatial grid. As a result of this interaction, zones with an increased dislocation density are formed, alternating with zones where their concentration is lower. This is how a gradient microstructure is formed. This uneven microstructure gives copper increased strength. Copper becomes more resistant to deformation because dislocations have difficulty moving through the material due to their interaction with other dislocations and obstacles they form. However, it is important to understand that in this case, the increase in strength does not occur at the expense of a decrease in ductility. In the case of a gradient microstructure, a sufficient level of ductility is maintained, which makes copper more flexible and resistant to brittle fracture.

In addition to hardness, strength, and ductility, electrical conductivity should be considered an important parameter for copper wire. Therefore, improvements in hardness and strength must be achieved without excessive reduction in electrical conductivity. Therefore, in this study, changes in wire conductivity values were also determined. Electrical conductivity, which was 78% IACS in the initial state, decreased to 65% IACS after deformation, but the obtained values are higher than those obtained in other works for this alloy [36].

For visual interpretation, all the data obtained as a result of this study are summarized in Table 3, ordered by hump and vertical.

**Table 3.** Wire parameters after 3 cycles of deformation in different wire sections.

| Wire Cross-Sectional Areas | Grain Size, $\mu\text{m}$ | HAGBs, % | $\Sigma 3$ , % | Microhardness, Mpa |
|----------------------------|---------------------------|----------|----------------|--------------------|
| surface                    | 0.4                       | 70       | 28             | 1150               |
| intermediate               | 5.0                       | 49       | 42             | 770                |
| central                    | 17.0                      | 28       | 15             | 685                |

The combination of different material processing technologies, especially during cyclic deformation, leads to complex deformation schemes that profoundly affect material microstructure. As a result of such deformation, a gradient microstructure, which is characterized by differences in size, shape, and orientation of grains in different zones of the wire cross-section, is formed. The gradient occurs due to the non-uniform distribution of deformation throughout the volume of the wire. In the surface zone of the wire, deformation is more intense than in deeper layers, which is explained by friction during processing and the peculiarities of load application. This difference in deformation leads to various changes in the microstructure of different zones of the wire. The fine-grained structure typical of the surface zone has increased strength due to an increase in the area of grain boundary, which prevents the movement of dislocations. Microhardness is directly related to the number and movement of dislocations in the material. Therefore, in the surface zone, where deformation is more active, dislocations move more intensively, resulting in the highest microhardness. At the same time, the coarse-grained structure of the central part provides higher plasticity. This is due to the fact that larger grains have greater freedom of dislocation movement, which allows the material to deform without cracking. Thus, the gradient microstructure of deformed copper provides a unique balance between strength and plasticity. Such a material can withstand significant loads while maintaining sufficient flexibility. Therefore, understanding the patterns of microstructure change and its effect on the mechanical characteristics of the material is a key factor in the development of new materials and the optimization of existing technological processes.

#### 4. Conclusions

It is unlikely that at present there are specialists or companies in the fields of mechanical engineering and metallurgy, aviation, and space technology who are not convinced of the advantages of low-alloy copper alloys over unalloyed copper. Therefore, in this paper, a study of Cu-14Fe copper wire deformed using a new technological scheme of plastic structure formation based on a combination of twisting in an equal-channel step die and traditional drawing was conducted. As a result of the deformation, copper wire with a gradient microstructure was obtained. Gradient microstructure is an interesting phenomenon and involves the presence of an ultrafine grain structure, predominant in the surface zone (grain size 400 nm), and a relatively coarse grain structure, localized in the central zone (grain size 17  $\mu\text{m}$ ). This non-uniform distribution of microstructure in the cross-section of the wire is a consequence of the peculiarities of the deformation process. The quantitative ratio of high-angle boundaries in the surface zone is significantly higher than in the central zone.

To understand the relationship between strength and microstructure, the microhardness of the copper wire was determined. Thus, for three deformation cycles, the microhardness was 1150 MPa in the surface zone, 770 MPa in the neutral zone, and 685 MPa in the central zone of the wire. Such a symmetrical spread of microhardness, observed throughout the cross-section of the rod, is a direct confirmation of the presence of a gradient microstructure. It is evident that the microhardness decreases as it moves from the surface zone to the central zone, which can be explained in terms of dislocation and boundary hardening. The strength characteristics of the wire have doubled: tensile strength has increased from 335 MPa to 675 MPa, and yield strength from 230 MPa to 445 MPa. At the same time, the relative elongation decreased from 20% to 16%, and relative narrowing decreased from 28% to 23%, respectively. The electrical conductivity is 65% IACS with a high rate of strength and plastic characteristics.

Studying the gradient microstructure and its effect on the properties of deformed copper is important for new materials with improved properties development. For example, a deep understanding of this phenomenon allows the creation of wire with increased strength in areas of high load and increased ductility in areas of low load. This may be especially important in areas such as the production of cables, tools, and machine parts. Therefore, further testing of the developed technology on factory equipment is planned.

**Author Contributions:** Conceptualization, I.V. and A.V.; methodology, I.V. and A.D.; investigation, I.V. and A.V.; data curation M.S.S. and Z.G.; writing—original draft preparation, I.V., A.V. and A.D.; drafting the work or reviewing it critically for important intellectual content, I.V. and M.S.S.; writing—review and editing, I.V., A.V. and Z.G.; supervision, I.V., M.S.S. and A.V.; project administration, M.S.S.; funding acquisition, Z.G. and M.S.S. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research has been funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP19676903).

**Data Availability Statement:** Data are contained within the article.

**Conflicts of Interest:** The authors declare no conflicts of interest.

## References

- Volokitina, I.; Volokitin, A.; Panin, E. Gradient microstructure formation in carbon steel bars. *J. Mater. Res. Technol.* **2024**, *31*, 2985–2993. [\[CrossRef\]](#)
- Latypova, M.A.; Chigirinsky, V.V.; Kolesnikov, A.S. Effect of Controlled Rolling on the Structural and Phase Transformations. *Prog. Phys. Met.* **2023**, *24*, 132–156. [\[CrossRef\]](#)
- Sapargaliyeva, B.; Agabekova, A.; Syrlybekkyzy, S.; Ulyeva, G.; Yerzhanov, A.; Kozlov, P. Study of changes in microstructure and metal interface Cu/Al during bimetallic construction wire straining. *Case Stud. Constr. Mater.* **2023**, *18*, e02162. [\[CrossRef\]](#)
- Nurumgaliyev, A.; Zhuniskaliyev, T.; Shevko, V.; Yerekeyeva, G. Modeling and development of technology for smelting a complex alloy (ligature) Fe-Si-Mn-Al from manganese-containing briquettes and high-ash coals. *Sci. Rep.* **2024**, *14*, 7456. [\[CrossRef\]](#)
- Volokitina, I.; Volokitin, A.; Makhmutov, B. Formation of Symmetric Gradient Microstructure in Carbon Steel Bars. *Symmetry* **2024**, *16*, 997. [\[CrossRef\]](#)
- Smirnov, A.; Smirnova, E.; Alexandrov, S. A New Experimental Method for Determining the Thickness of Thin Surface Layers of Intensive Plastic Deformation Using Electron Backscatter Diffraction Data. *Symmetry* **2020**, *12*, 677. [\[CrossRef\]](#)
- Snopiński, P. Electron Microscopy Study of Structural Defects Formed in Additively Manufactured AlSi10Mg Alloy Processed by Equal Channel Angular Pressing. *Symmetry* **2023**, *15*, 860. [\[CrossRef\]](#)
- Volokitina, I. Effect of Preliminary Heat Treatment on Deformation of Brass by the Method of ECAP. *Met. Sci. Heat Treat.* **2021**, *63*, 163–167. [\[CrossRef\]](#)
- Zhangabay, N.; Baidilla, I.; Tagybayev, A.; Anarbayev, Y.; Kozlov, P. Thermophysical indicators of elaborated sandwich cladding constructions with heat-reflective coverings and air gaps. *Case Stud. Constr. Mater.* **2023**, *18*, e02161. [\[CrossRef\]](#)
- Bychkov, A.; Kolesnikov, A. Natural Aging of Aluminum Alloy 2024 After Severe Plastic Deformation. *Metall. Microstruct. Anal.* **2023**, *12*, 564–566. [\[CrossRef\]](#)
- Humphreys, F.J.; Hetherly, M.; Rollett, A.; Rohrer, G.; Humphreys, J. *Recrystallization and Related Annealing Phenomena*, 2nd ed.; Elsevier Ltd.: Oxford, UK, 2004. [\[CrossRef\]](#)
- Rosakis, P.; Rosakis, A.J.; Ravichandran, G.; Hodowany, J. A thermodynamic internal variable model for the partition of plastic work into heat and stored energy in metals. *J. Mech. Phys. Solids* **2000**, *48*, 581–607. [\[CrossRef\]](#)
- Hirsch, J.; Lücke, K.; Hatherly, M. Overview No. 76: Mechanism of deformation and development of rolling textures in polycrystalline f.c.c. Metals—III. The influence of slip inhomogeneities and twinning. *Acta Metall.* **1988**, *36*, 2905–2927. [\[CrossRef\]](#)
- Hughes, D.A.; Hansen, N. High angle boundaries formed by grain subdivision mechanisms. *Acta Mater.* **1997**, *45*, 3871–3886. [\[CrossRef\]](#)
- Immanue, R.J.; Panigrahi, S.K.; Malas, J.C. Chapter 5—Materials development for sustainable manufacturing. In *Sustainable Manufacturing Processes*; Academic Press: Cambridge, MA, USA, 2023; pp. 155–194. [\[CrossRef\]](#)
- Knapiński, M.; Bajor, T.; Kawalek, A.; Banaszek, G. Analysis of Copper Welding Parameters during the Manufacture of Tubular Profiles Using Unconventional Extrusion Processes. *Materials* **2024**, *17*, 4737. [\[CrossRef\]](#) [\[PubMed\]](#)
- Volokitina, I.E.; Volokitin, A.V. Changes in Microstructure and Mechanical Properties of Steel-Copper Wire During Deformation. *Metallurgist* **2023**, *67*, 232–239. [\[CrossRef\]](#)
- Spuskanyuk, V.; Davydenko, O.; Berezina, A.; Gangalo, O.; Sennikova, L.; Tikhonovsky, M.; Spiridonov, D. Effect of combining the equal-channel angular hydroextrusion, direct hydroextrusion and drawing on properties of copper wire. *J. Mater. Process. Technol.* **2010**, *210*, 1709–1715. [\[CrossRef\]](#)
- Markov, O.E.; Perig, A.V.; Zlygoriev, V.N. Development of forging processes using intermediate workpiece profiling before drawing: Research into strained state. *J. Braz. Soc. Mech. Sci. Eng.* **2017**, *39*, 4649–4665. [\[CrossRef\]](#)
- Kunčická, L.; Kocich, R. Optimizing electric conductivity of innovative Al-Cu laminated composites via thermomechanical treatment. *Mater. Des.* **2022**, *215*, 110441. [\[CrossRef\]](#)
- Bachmaier, A.; Grosdidier, T.; Ivanisenko, Y. Severe Plastic Deformation and Thermomechanical Processing: Nanostructuring and Properties. *Metals* **2020**, *10*, 1306. [\[CrossRef\]](#)
- Volokitina, I.; Volokitin, A.; Denissova, A.; Kuatbay, Y.; Liseitsev, Y. Effect of thermomechanical processing of building stainless wire to increase its durability. *Case Stud. Constr. Mater.* **2023**, *19*, e02346. [\[CrossRef\]](#)

23. Mythili, R.; Kirana, R.; Herojit Singh, L.; Govindaraj, R.; Sinha, A.; Singh, M.; Saroja, S.; Vijayalakshmi, M.; Deb, S. Identification of Retained Austenite in 9Cr-1.4W-0.06Ta-0.12C Reduced Activation Ferritic Martensitic Steel. *Symmetry* **2022**, *14*, 196. [CrossRef]
24. Panin, E.; Fedorova, T.; Lawrinuk, D.; Kolesnikov, A.; Yerzhanov, A.; Gelmanova, Z.; Liseitsev, Y. Improvement of strength and performance properties of copper wire during severe plastic deformation and drawing process. *Case Stud. Constr. Mater.* **2023**, *19*, e02609. [CrossRef]
25. Sapargaliyeva, B.; Agabekova, A.; Syrlybekkyzy, S.; Nurshakhanova, L.; Nurbaeva, F.; Kolesnikov, A.; Sabyrbayeva, G.; Izbassar, A.; Kolesnikova, O.; Liseitsev, Y. Increasing strength and performance properties of bimetallic rods during severe plastic deformation. *Case Stud. Constr. Mater.* **2023**, *19*, e02256. [CrossRef]
26. Moradia, S.; Gerdooei, M.; Ghaforian Nosrati, H.; Varedi-Koulaeid, S.M. Optimization and Investigation of Effective Parameters in ECAP-Conform Process Using Response Surface Method, Case Study: AA7075. *Rod. Modares Mech. Eng.* **2022**, *22*, 57–70. Available online: <https://www.researchgate.net/publication/356666364> (accessed on 1 November 2021).
27. Raab, G.I.; Fakhretdinova, E.I.; Valiev, R.Z. Computer Study of the Effect of Tooling Geometry on Deformation Parameters in the Plastic Shaping of Aluminum Wire Rod by Multi-ECAP-Conform. *Metallurgist* **2016**, *59*, 1007–1014. [CrossRef]
28. Rubanik, V.V.; Tsarenko, Y.V. Ultrasound influence on the physical and mechanical properties of wire at equal-channel angular broaching. *Dokl. Natl. Acad. Sci. Belarus* **2020**, *64*, 94–102. [CrossRef]
29. Chukin, M.V.; Polyakova, M.A.; Golubchik, E.M.; Rudakov, V.P.; Noskov, S.E.; Gulin, A.E. Method of obtaining ultrafine-grained semi-finished products by drawing with torsion. Patent No. 2467816 Russian Federation, MPK B21C 1/00, B21J 5/06, C21D 7/00, 28 February 2011.
30. Liu, Z.; Liang, G.; Wang, E.; Wang, Z. The effect of cumulative large plastic strain on the structure and properties of a Cu–Zn alloy. *Mater. Sci. Eng. A* **1998**, *242*, 137–140. [CrossRef]
31. Liu, Z.; Wang, Z. Finite-element analysis of the load of equal-cross-section lateral extrusion. *J. Mater. Process. Technol.* **1999**, *94*, 193–196.
32. Raab, G. Plastic flow at equal channel angular processing in parallel channels. *Mater. Sci. Eng. A* **2005**, *410*, 230–233. [CrossRef]
33. Galkin, S.P. Method of helical rolling. NITU MISIS—No. 2006110612/02, avt. 04.04.2006; Bulletin of inventions, 2007, No. 5. Patent No. 2293619 Russian Federation, MPK B21B 19/00, 20 February 2007.
34. Wang, K.; Tao, N.R.; Liu, G.; Lu, J.; Lu, K. Plastic strain-induced grain refinement at the nanometer scale in copper. *Acta Mater.* **2006**, *54*, 5281–5291. [CrossRef]
35. Li, W.L.; Tao, N.R.; Lu, K. Fabrication of a gradient nano-micro-structured surface layer on bulk copper by means of a surface mechanical grinding treatment. *Scripta Mater.* **2008**, *59*, 546–549. [CrossRef]
36. Stepanov, N.D.; Kuznetsov, A.V.; Salishchev, G.A.; Khlebova, N.E.; Pantsyrniy, V.I. Microstructure and mechanical properties of Cu-14%Fe alloy subjected to severe cold rolling. *Mater. Sci. Eng. A* **2012**, *564*, 264–272. [CrossRef]

**Disclaimer/Publisher’s Note:** The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.