

Article

Evolution of Microstructure and Properties of Oxygen-Free Copper During Severe Drawing Process

Qianqian Wu ^{1,2}, Yuefeng Luo ³, Guoquan Chen ³, Ziqian Zhao ^{1,4,*}, Meng Zhou ⁵ and Zhu Xiao ^{1,2,*}¹ School of Materials Science and Engineering, Central South University, Changsha 410083, China² Key Laboratory of Non-Ferrous Metal Materials Science and Engineering, Ministry of Education, Changsha 410083, China³ Zhejiang Libo Industrial Company Limited, Shaoxing 312050, China⁴ State Key Laboratory for Powder Metallurgy, Central South University, Changsha 410083, China⁵ School of Materials Science and Engineering, Henan University of Science and Technology, Luoyang 471023, China

* Correspondence: ziqian.zhao@csu.edu.cn (Z.Z.); xiaozhumse@163.com (Z.X.); Tel.: +86-15632197993 (Z.Z.); +86-13974910804 (Z.X.)

Abstract

Oxygen-free copper wires are widely required in advanced electronic and electrical systems. However, the relationship between deformation-induced microstructure evolution and property variation during severe cold drawing is not fully understood. In this study, oxygen-free copper rods with a diameter of $\phi 20$ mm were fabricated by upward continuous casting, followed by multi-pass cold drawing with deformation degrees up to 99.75%. The microstructure evolution, texture transformation, and property changes during drawing and annealing were systematically investigated. It found that increasing drawing deformation progressively elongated, fragmented, and refined the grains, reducing the average grain size from 339.18 μm to 2.12 μm . Dense dislocation cells, substructures, and nanoscale twins were generated during severe deformation, while the texture gradually evolved from random orientations to preferred $\langle 111 \rangle$ and $\langle 100 \rangle$ textures. After annealing, fine recrystallized equiaxed grains with abundant annealing twins formed together with recrystallization $\langle 101 \rangle$ texture. At 99.75% deformation, the tensile strength reached 486 MPa, with an electrical conductivity of 96.7% IACS (International Annealed Copper Standard). After annealing, the electrical conductivity recovered to 101.5% IACS, while the tensile strength decreased to 187 MPa. This work establishes the relationship between severe deformation microstructure evolution and property response in oxygen-free copper, providing guidance for the fabrication of high-strength and high-conductivity copper conductors.

Keywords: oxygen-free copper; drawing deformation; texture evolution; electrical conductivity

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1. Introduction

Copper and its alloys are among the most important metallic materials in the era of artificial intelligence and high-performance electronics because of their excellent electrical conductivity, thermal conductivity, and favorable mechanical properties [1–4]. As a representative copper-based material, pure copper has been extensively applied in 5G communication cables, high-performance computing server cooling systems, and high-fidelity electrical conductors. Accordingly, considerable efforts have been devoted to optimiz-

ing the processing and strengthening theories of pure copper in order to achieve a superior balance between mechanical strength and electrical conductivity [5,6]. Han et al. [7] combined high-density electropulsing with cryogenic rolling to tailor the microstructure and refine the grain size of pure copper, thereby simultaneously improving strength and ductility. In addition, microalloying strategies have been employed to enhance the softening resistance of pure copper through the addition of trace alloying elements such as Al and Sc [8]. Fan et al. [9] fabricated pure copper wires under different drawing strains via directional solidification and demonstrated that the change in mechanical strength was closely associated with the evolution of dislocation density in the copper matrix.

However, industrial pure copper generally contains a certain amount of oxygen, which tends to segregate at grain boundaries in the form of CuO or Cu₂O. Such oxygen-containing phases result in microstructural heterogeneity, deteriorated ductility, and reduced electrical conductivity. Moreover, stress concentration caused by non-uniform oxygen distribution can induce local cracking during cold deformation, thereby increasing the risk of wire breakage during severe drawing processes. To overcome these limitations, oxygen-free copper with an extremely low oxygen content has been developed owing to its homogeneous microstructure, superior ductility, and excellent electrical conductivity [10]. In recent years, the processing technology and deformation theory of oxygen-free copper have attracted increasing attention for achieving ultrahigh conductivity approaching 100% IACS together with reliable mechanical performance. Zhang et al. [11] prepared oxygen-free copper with different phosphorus contents by vacuum melting and reported that grain refinement became more pronounced with increasing P addition, accompanied by improved hardness and elevated softening temperature. Dobatkin et al. [12] demonstrated that both the processing route and deformation degree during equal channel angular pressing (ECAP) significantly influenced the microstructure, mechanical properties, and fatigue resistance of oxygen-free copper. Sun et al. [13] further revealed that the electrical conductivity and strength of cold-drawn oxygen-free copper wires were primarily governed by the evolution of dislocations, texture, and grain structure and evaluated the contribution of each factor to the overall properties.

Among various manufacturing technologies, cold drawing remains the most widely used method for producing high-performance copper wires because of its simplicity, efficiency, and ability to continuously refine the microstructure. During the drawing process, severe plastic deformation induces substantial changes in grain morphology, grain boundary characteristics, microstructure texture, and dislocation distribution, which collectively determine the final mechanical and electrical performance of the Cu wires. Nevertheless, the intrinsic correlation between deformation microstructure evolution and property variation in oxygen-free copper during severe cold drawing has not yet been fully established, particularly under ultrahigh deformation conditions.

In this study, oxygen-free copper rods with a diameter of $\phi 20$ mm were fabricated by upward continuous casting, followed by multi-pass cold drawing to obtain wires with different deformation degrees, including a severe deformation level as high as 99.75%. The evolution of microstructure and texture during the drawing process was systematically characterized using electron backscatter diffraction (EBSD). Particular attention was paid to the variation in grain size, grain boundary distribution, and crystallographic texture, as well as their correlations with the mechanical and electrical properties of the oxygen-free copper wires. The present work aims to clarify the deformation-induced microstructural evolution mechanisms in oxygen-free copper during severe cold drawing and to provide theoretical guidance for the design and fabrication of high-strength, high-conductivity copper conductors.

2. Materials and Methods

Electrolytic copper (99.99 wt.%) was used as the raw material. Oxygen-free copper rods with a diameter of $\varnothing 20$ mm were fabricated by the upward continuous casting process. Subsequently, the rods were subjected to multi-pass cold drawing at ambient temperature with different deformation degrees, followed by annealing treatment, to obtain oxygen-free copper wires with various diameters and processing states, as listed in Table 1. The reduction ratio in area and drawing strain were used to characterize the deformation degree of the wires. The reduction ratio in area (ε) and drawing strain (η) were calculated according to Equations (1) and (2):

$$\varepsilon = \frac{A_0 - A_i}{A_0} \times 100\% \quad (1)$$

$$\eta = \ln\left(\frac{A_0}{A_i}\right) = 2\ln\left(\frac{D_0}{D_i}\right) \quad (2)$$

where A_0 and A_i are the initial and final cross-sectional areas of the wires, respectively, while D_0 and D_i represent the initial and final wire diameters. WD and WA denote the cold-drawn and annealed states of the wires, respectively. The annealing treatment was conducted at 400 °C for 0.5 h under a protective Ar atmosphere. After annealing, the specimens were water-quenched.

Table 1. Selected as-drawn samples for testing.

Diameter/mm	Reduction Ratio in Area/%	Drawing Strain
WD 20	0	0
WD 8.0	84	1.83
WD 1.0	99.75	5.99
WA 1.0	99.75	5.99

To study the microstructure and property evolution of oxygen-free copper rods during drawing deformation, radial and axial sections were cut from samples with different drawing strains. The metallographic structures were observed under a Leica EC3 optical microscope (Leica Microsystems, Wetzlar, Germany) at different magnifications after standard grinding, mechanical polishing, and chemical etching.

Electron backscatter diffraction (EBSD) analysis was carried out on the radial sections of the samples using a JSM-7800F field-emission scanning electron microscope (JEOL, Akishima, Tokyo, Japan) equipped with an Oxford Instruments EBSD detector (Oxford Instruments, Abingdon, Oxfordshire, UK). Prior to EBSD characterization, the samples were mechanically ground using SiC abrasive papers with progressively finer grit sizes, followed by polishing with diamond suspensions and final electropolishing using the $\text{H}_3\text{PO}_4\text{:CH}_3\text{OH:H}_2\text{O} = 1\text{:}1\text{:}2$ electrolyte at 5 V for 10–15 s to remove surface deformation layers. EBSD scans were performed at an accelerating voltage of 20.0 kV, a working distance of 15 mm, and a step size of 0.7 μm .

Transmission electron microscopy (TEM) characterization was further conducted using a JEM-2100 transmission electron microscope (JEOL, Akishima, Tokyo, Japan) operated at 200 kV to investigate the dislocation configurations and substructure evolution of the deformed oxygen-free copper wires. Thin foil TEM specimens were prepared by mechanical thinning followed by twin-jet electropolishing in a 33% HNO_3 + 67% CH_3OH solution at -30 °C.

The microhardness was measured at room temperature using an HXD-1000T Vickers microhardness tester (Shanghai Taiming Optical Instrument, Shanghai, China) with a load of 300 g and a dwell time of 10 s. At least 5 parallel measurements were performed for each sample, and the average value was reported.

The mechanical properties were analyzed using an MTS universal testing machine (MTS Systems, Eden Prairie, MN, USA) at a tensile strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. The electrical conductivity was measured using a TX-300 intelligent metal conductor (Tianjin Tianyan Testing Instrument, Tianjin, China) resistivity instrument, and the reported values represented the average of three independent measurements.

3. Results

3.1. Evolution of Microstructure During Cold Drawing

Figure 1 presents the optical microstructures of oxygen-free copper in both the radial and axial directions at different drawing deformations. The as-cast oxygen-free copper rod exhibits coarse columnar grains aligned in the radial direction, as shown in Figure 1a,e. After multi-pass cold drawing to a deformation of 84% (WD 8.0), severe plastic deformation leads to significant grain elongation and fragmentation along the drawing direction. Meanwhile, distinct deformation bands are formed within regions subjected to localized strain concentration, as indicated in Figure 1b,f. With further increasing deformation to 99.75% (WD 1.0), the grains in the radial section are substantially refined, and the original grain boundaries become difficult to resolve under optical microscopy, as shown in Figure 1c. In the axial section, the grains are continuously elongated along the drawing direction, gradually evolving into a typical fibrous microstructure with blurred grain boundaries [14], as shown in Figure 1g. Following annealing treatment after severe deformation (WA 1.0), the stored deformation energy drives recovery and recrystallization, resulting in the formation of a large number of fine equiaxed grains, as shown in Figure 1d,h.

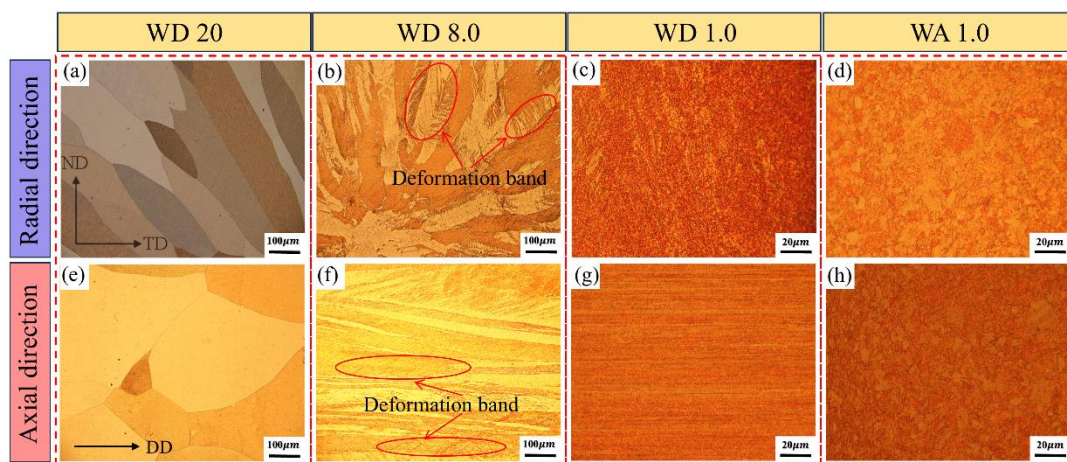


Figure 1. Metallographic structures of oxygen-free copper in radial and axial directions with different drawing deformations: (a,e) WD 20, $\epsilon = 0\%$; (b,f) WD 8.0, $\epsilon = 84\%$; (c,g) WD 1.0, $\epsilon = 99.75\%$; (d,h) WA 1.0, $\epsilon = 99.75\%$.

EBSD analysis is employed to further characterize the microstructural evolution of oxygen-free copper during cold drawing. Figure 2 presents the grain orientation maps and corresponding grain size distributions of oxygen-free copper with different drawing deformations. As shown in Figure 2a,d, the undeformed oxygen-free copper consists of coarse grains with random crystallographic orientations, and the average grain size is approximately $339.18 \mu\text{m}$. After cold drawing to a deformation of 84%, as shown in Figure 2b, the grains become significantly elongated and fragmented along the radial direction and exhibit pronounced preferred orientation. During the drawing process, severe plastic deformation continuously generates and accumulates dislocations within the grains. The interaction and entanglement of dislocations promote the formation of deformation substructures, thereby accelerating grain refinement. Consequently, the average grain size

decreases dramatically from 339.18 μm to 13.08 μm (Figure 2e). Simultaneously, grain rotation and dislocation slip occur during deformation, resulting in the gradual transformation of randomly oriented grains into preferred $\langle 111 \rangle$ and $\langle 100 \rangle$ orientations. With further increasing deformation to 99.75%, as shown in Figure 2c, the grains are refined to the submicron-to-micron scale, with most grain sizes distributed within 1–8 μm and an average grain size of 2.12 μm , as shown in Figure 2f. At this stage, the $\langle 111 \rangle$ and $\langle 100 \rangle$ textures become more prominent and exhibit an interlaced distribution.

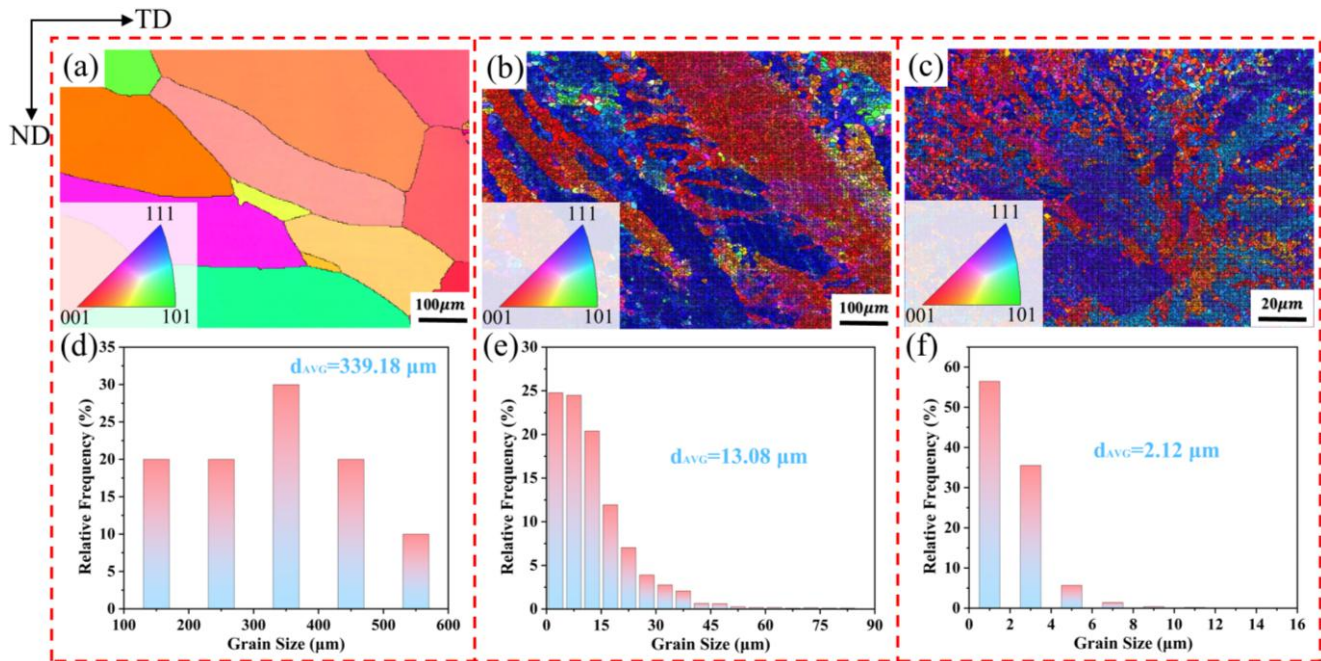


Figure 2. Grain orientation distribution maps and grain size distribution maps of oxygen-free copper in the radial direction with different drawing deformations: (a,d) WD 20, $\varepsilon = 0\%$; (b,e) WD 8.0, $\varepsilon = 84\%$; (c,f) WD 1.0, $\varepsilon = 99.75\%$.

Figure 3 presents the grain boundary distribution maps and corresponding grain boundary misorientation distributions of oxygen-free copper at different drawing deformations. In the grain boundary maps, black lines represent high-angle grain boundaries (HAGBs, $\theta > 15^\circ$), whereas red lines denote low-angle grain boundaries (LAGBs, $2^\circ < \theta < 15^\circ$). As shown in Figure 3a, the as-cast oxygen-free copper is dominated by coarse grains separated mainly by HAGBs, indicating a relatively low internal strain state. The corresponding statistical result in Figure 3d shows that the fraction of LAGBs is only 28.58%. After cold drawing to a deformation of 84%, severe plastic deformation is imposed on the copper under both radial and circumferential compressive stresses and axial tensile stress during drawing. Under such deformation conditions, dislocations are continuously generated, accumulated, and entangled within the grains, leading to the formation of a large number of subgrains with low misorientations, as shown in Figure 3b. Consequently, the fraction of LAGBs increases significantly to 78.21%, as shown in Figure 3e. With further increasing deformation to 99.75%, the fraction of LAGBs decreases to 57.9%, as shown in Figure 3f. This variation is also reflected in Figure 3c, where the density of red lines representing LAGBs is noticeably reduced. The decrease in LAGB fraction is possibly attributed to the continuous accumulation of plastic strain during severe drawing deformation, which progressively increases the misorientation between adjacent deformation substructures. As a result, some LAGBs gradually evolve into HAGBs during the continuous subdivision and refinement of the deformed grains.

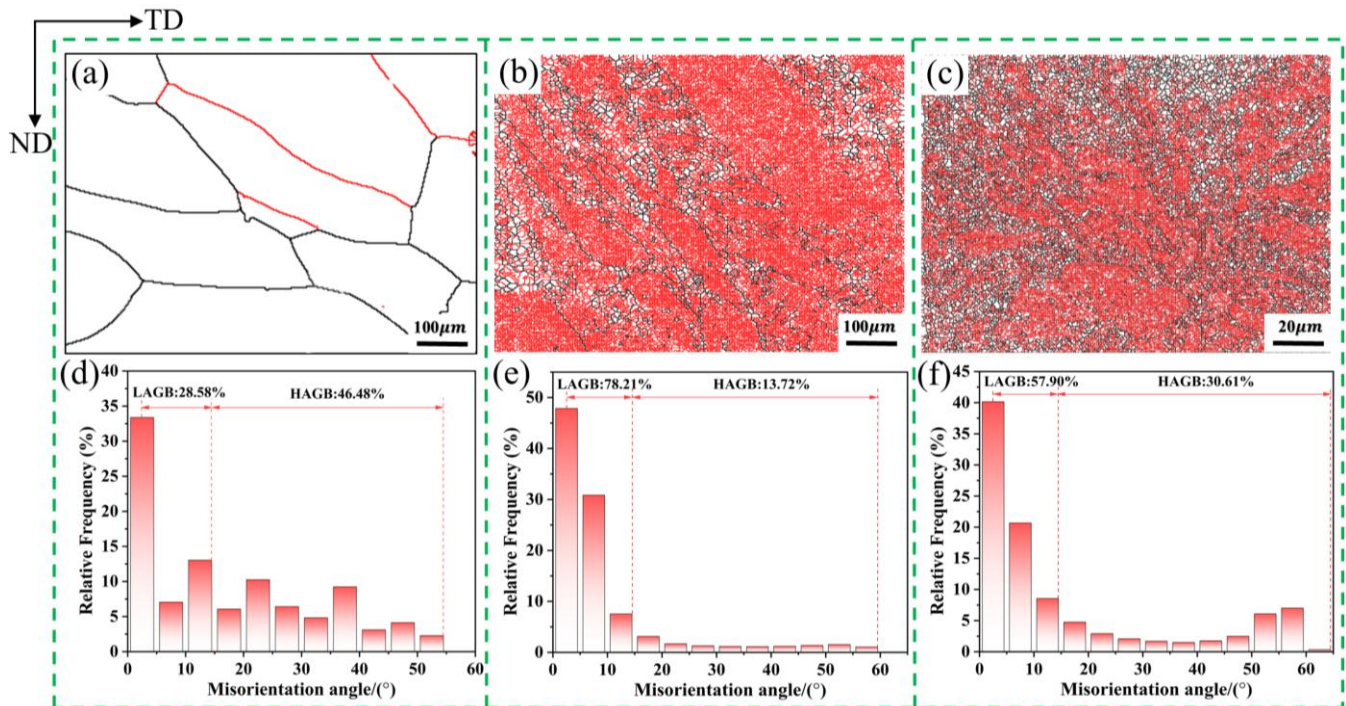


Figure 3. Grain boundary distribution maps and grain boundary misorientation maps of oxygen-free copper in the radial direction with different drawing deformations: (a,d) WD 20, $\varepsilon = 0\%$; (b,e) WD 8.0, $\varepsilon = 84\%$; (c,f) WD 1.0, $\varepsilon = 99.75\%$.

Figure 4 shows the TEM microstructures of oxygen-free copper at different drawing deformations. In the undeformed state, dislocation cells and dislocation tangles are observed inside the grains, as shown in Figure 4a. These substructures are mainly formed due to the thermal stress and traction-induced deformation during the upward continuous casting process. At this stage, the dislocation density remains relatively low and exhibits an inhomogeneous distribution. As shown in Figure 4b, the interiors of the dislocation cells are nearly dislocation-free, whereas highly tangled dislocations accumulate at the cell walls. After cold drawing to a deformation of 84%, the grains are significantly elongated, and the dislocation density increases rapidly. The extensive interaction and entanglement of dislocations promote the formation of dense dislocation cell structures, as shown in Figure 4c. In addition, nanoscale deformation twins are observed in Figure 4d, indicating the activation of twinning deformation under severe plastic deformation conditions. With further increasing deformation to 99.75%, the grains become further elongated and refined. Meanwhile, the dislocation density continuously increases, and the dislocation cell size decreases markedly. The tangled dislocations at the cell walls gradually rearrange into more stable subgrain boundaries, as shown in Figure 4e. In Figure 4f, locally recrystallized grains are observed in regions with high stored deformation energy. This phenomenon is likely associated with localized temperature rise caused by frictional heating during severe cold drawing, which promotes recovery and partial recrystallization in highly strained regions [15,16].

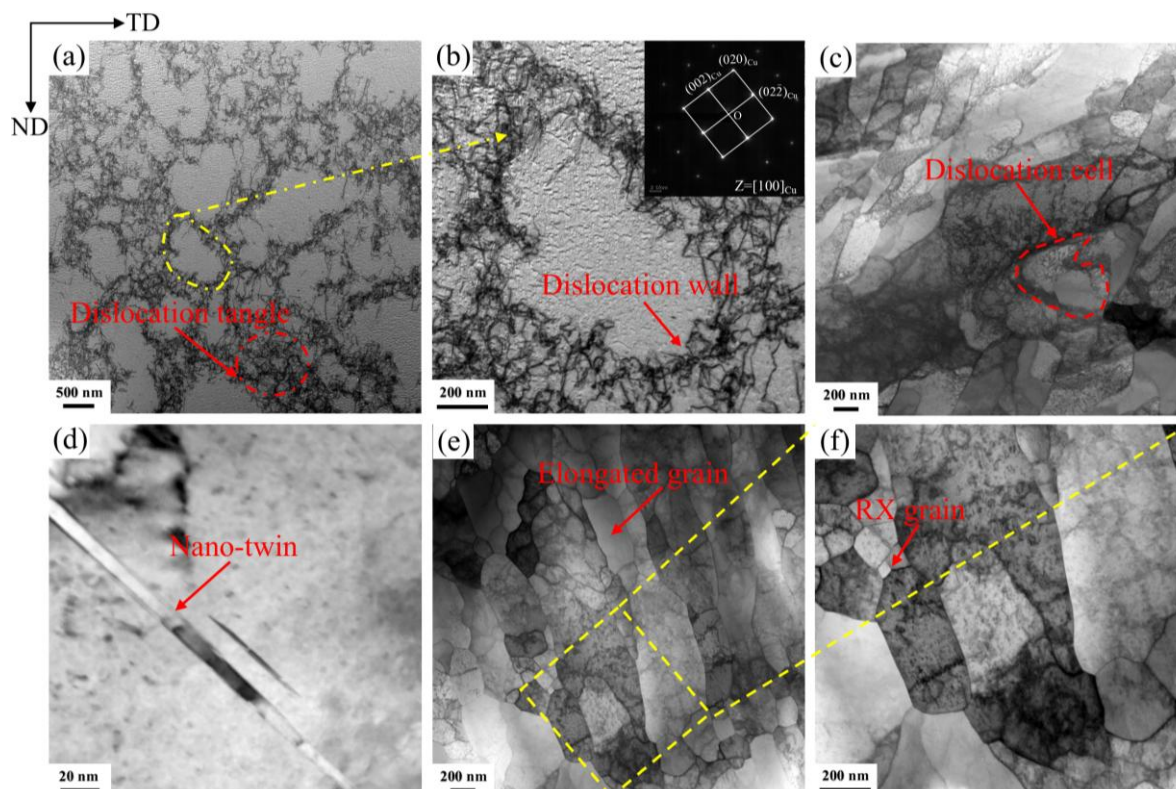


Figure 4. TEM images of oxygen-free copper in the radial direction with different drawing deformations: (a,b) WD 20, $\varepsilon = 0\%$; (c,d) WD 8.0, $\varepsilon = 84\%$; (e,f) WD 1.0, $\varepsilon = 99.75\%$.

3.2. Evolution of Microstructure After Annealing

Figure 5 presents the EBSD characterization of annealed oxygen-free copper after severe cold drawing (WA 1.0). Following subsequent annealing treatment, the heavily deformed microstructure undergoes significant recovery and recrystallization, resulting in the formation of fine equiaxed recrystallized grains accompanied by abundant annealing twins [17], as shown in Figure 5. As shown in Figure 5a, the recrystallized grains exhibit more $\langle 101 \rangle$ orientation (green color) compared with the severely deformed state, indicating the development of recrystallization texture during annealing. Figure 5b shows that the average grain size, including twins, is approximately $3.07 \mu\text{m}$. Figure 5c presents the grain boundary distribution map, where black, red, and green lines represent high-angle grain boundaries (HAGBs), low-angle grain boundaries (LAGBs), and $\Sigma 3$ coincidence site lattice (CSL) grain boundaries, respectively. A large number of annealing twins are observed within the recrystallized grains. These twins exhibit different morphologies, including throughout twins, partially throughout twins, and intragranular twins [18]. The grain boundary misorientation distribution shown in Figure 5d reveals that the fraction of HAGBs reaches 81.15%, which is significantly higher than that in the cold-drawn samples with deformation degrees of 84% and 99.75%. This increase is primarily attributed to the occurrence of recovery and recrystallization during annealing, during which deformation-induced substructures bounded by LAGBs progressively transform into recrystallized grains separated by HAGBs [19]. Moreover, a pronounced misorientation peak is observed near 60° , with a corresponding fraction of 30.74%, which is substantially higher than that at other misorientation angles. Since a misorientation angle of approximately 60° is characteristic of $\Sigma 3$ CSL grain boundaries in face-centered cubic metals, this result indicates the formation of a high density of annealing twin boundaries in the recrystallized oxygen-free copper [20]. This observation is consistent with the grain boundary distribution shown in Figure 5c. The formation of abundant $\Sigma 3$ CSL grain boundaries is closely

associated with the relatively low stacking fault energy of copper, which facilitates annealing twinning during grain boundary migration and recrystallization. Compared with conventional HAGBs, $\Sigma 3$ CSL boundaries possess lower interfacial energy and higher atomic matching at the interface, enabling them to form readily and remain thermodynamically stable during annealing [21,22].

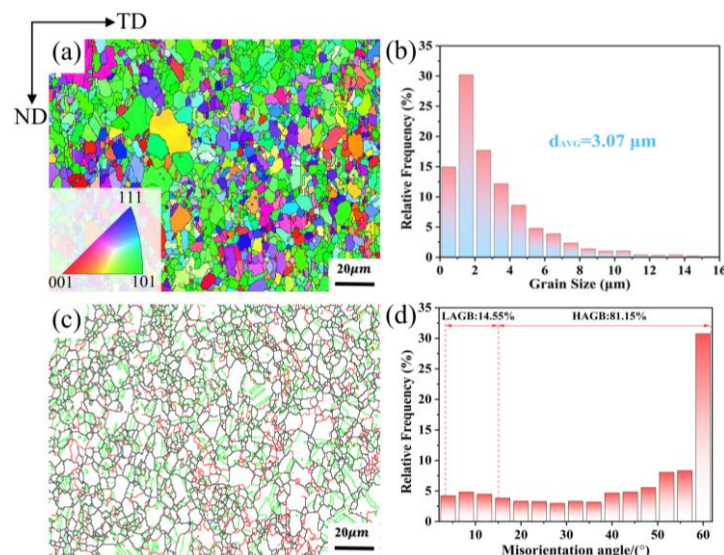


Figure 5. EBSD maps of oxygen-free copper with 99.75% deformation after annealing in the radial direction: (a) grain orientation map; (b) grain size distribution map; (c) grain boundary distribution map; (d) grain boundary misorientation map.

Figure 6 presents the recrystallization characteristics of annealed oxygen-free copper after severe cold drawing (WA 1.0). In the recrystallization maps, red, yellow, and blue regions represent deformed grains, subgrains, and recrystallized grains, respectively. As shown in Figure 6a, most of the severely deformed microstructure undergoes substantial recovery and recrystallization during annealing, resulting in the formation of a large fraction of recrystallized grains together with a small amount of subgrains. Only a limited number of regions remain in the deformed state. The extensive recrystallization behavior indicates that the severely cold-drawn oxygen-free copper stores a high density of deformation energy, which provides a strong driving force for recrystallization during subsequent annealing treatment. The recrystallization fraction statistics shown in Figure 6b reveal that recrystallized grains account for 82.7% of the microstructure, whereas subgrains and deformed grains account for 13.7% and 3.6%, respectively. The high recrystallized fraction further confirms that recovery and recrystallization dominate the microstructural evolution during annealing, leading to the elimination of deformation-induced dislocations and the formation of a more stable recrystallized microstructure.

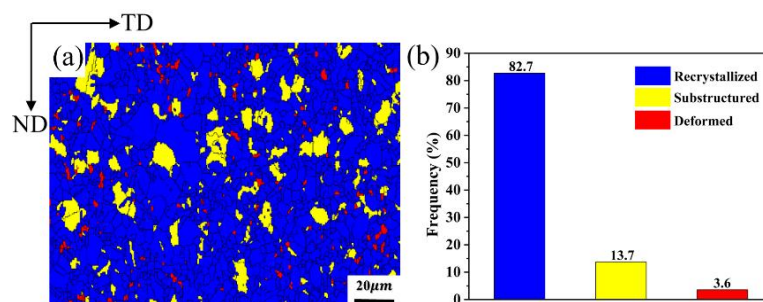


Figure 6. Recrystallization maps of oxygen-free copper with 99.75% deformation after annealing in the radial direction: (a) recrystallization distribution map; (b) recrystallization fraction map.

3.3. Mechanical Properties and Electrical Conductivity During the Drawing Process

Figure 7 presents the evolution of mechanical properties and electrical conductivity curves of oxygen-free copper with different drawing deformations. The as-cast oxygen-free copper exhibits a hardness of 42.78 HV, with an electrical conductivity of 100.6% IACS and a tensile strength of 154 MPa. After cold drawing to a deformation of 84%, the hardness and tensile strength increase sharply to 115.05 HV and 403 MPa, whereas the electrical conductivity slightly decreases to 98.2% IACS. With further deformation to 99.75%, the tensile strength continuously increases to 486 MPa, accompanied by a further reduction in electrical conductivity to 96.7% IACS. The continuous increase in hardness is primarily attributed to severe strain hardening induced by grain refinement and rapid dislocation accumulation during drawing deformation. In contrast, the decrease in electrical conductivity mainly results from enhanced electron scattering caused by the high density of dislocations, subgrain boundaries, and lattice distortion. After subsequent annealing treatment, recovery and recrystallization substantially reduce the dislocation density and eliminate deformation substructures. Consequently, the hardness and tensile strength decrease significantly to 70.03 HV and 187 MPa, while the electrical conductivity recovers to as high as 101.5% IACS.

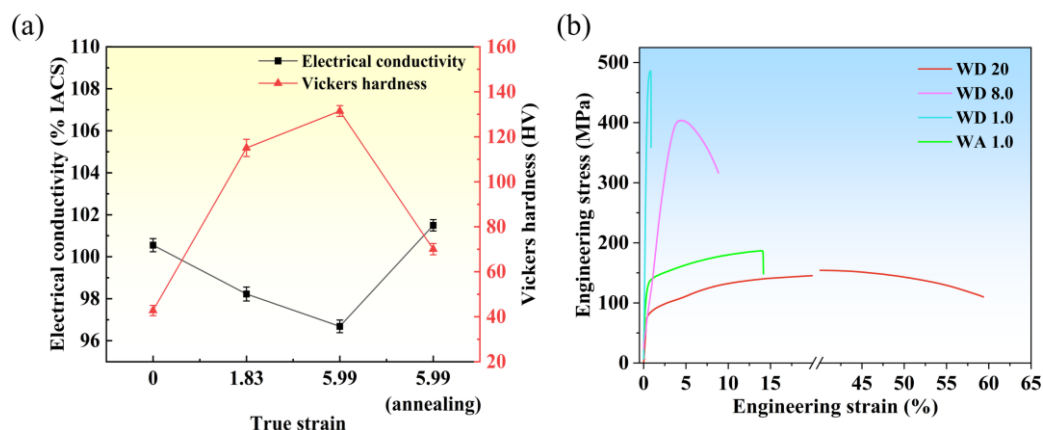


Figure 7. Mechanical properties and electrical conductivity curves of oxygen-free copper with different drawing deformations: (a) Electrical conductivity and hardness; (b) Tensile strength.

4. Discussion

4.1. Strengthening Mechanisms

The strengthening mechanisms of metals mainly include solid solution strengthening, dislocation strengthening, grain boundary strengthening, precipitation strengthening and texture strengthening [23–25]. Since oxygen-free copper contains no alloying elements and has extremely low oxygen content, the strengthening mechanisms of oxygen-free copper are primarily grain boundary strengthening, dislocation strengthening, and texture strengthening. The yield strength of oxygen-free copper can be expressed as follows:

$$\sigma_{YS} = \sigma_{GB} + \sigma_{DIS} + \sigma_{Tex} \quad (3)$$

where σ_{YS} is the yield strength; σ_{GB} is the yield strength caused by fine grain reinforcement; σ_{DIS} is the yield strength caused by the dislocation density generated; σ_{Tex} is the yield strength caused by the preferred orientation of grains.

4.1.1. Grain Boundary Strengthening

According to the Hall-Petch formula [26], the grain strengthening increment can be expressed as follows:

$$\sigma_{GB} = \sigma_0 + K_y/\sqrt{d} \quad (4)$$

where σ_0 is the total resistance to dislocation motion in a metal single crystal, also known as the lattice friction. For copper, σ_0 is 20 MPa [27]; K_y is the Hall-Petch constant, with a value of 110 MPa· $\mu\text{m}^{1/2}$; d is the average grain size of the metal. According to the Hall-Petch formula, within a certain grain size range, finer grains contribute more to the yield strength. The grain sizes of oxygen-free copper samples with different drawing deformations and their corresponding yield strengths are shown in Table 2.

Table 2. Yield strength of oxygen-free copper with different drawing deformations.

Diameter/mm	d/ μm	σ_{GB}/MPa	$\rho_{\text{GND}}/\times 10^{14} \text{ m}^{-2}$	$\sigma_{\text{DIS}}/\text{MPa}$	$f_{\langle 111 \rangle}/\%$	$f_{\langle 100 \rangle}/\%$	$\sigma_{\text{Tex}}/\text{MPa}$	$\sigma_{\text{Exp}}/\text{MPa}$
WD 20	339.18	25.97	0.27	56.08	/	/	/	85
WD 8.0	13.08	50.42	10.12	343.36	53.3	46.7	0.73	392
WD 1.0	2.12	95.55	12.30	378.54	63.1	36.9	5.13	478
WA 1.0	3.07	82.78	0.35	63.86	/	/	/	138

Note: σ_{Exp} refers to the yield strength obtained under experimental conditions.

4.1.2. Dislocation Strengthening

During the drawing process of oxygen-free copper, many dislocations are generated within the grains. According to Taylor's formula, the yield strength increment caused by dislocations can be expressed as follows [28]:

$$\sigma_{\text{DIS}} = M\alpha Gb\sqrt{\rho_{\text{GND}}} \quad (5)$$

where M is the Taylor factor, which is approximately 3.06; α is a constant and is usually taken as 0.3 for copper [29]; G is the shear modulus and is taken as 45.6 GPa; b is the Burgers vector, and the value is usually 0.256 nm for face-centered cubic metals; ρ_{GND} is the geometrically necessary dislocation density.

The average geometrically necessary dislocation density ρ_{GND} can be expressed as follows:

$$\rho_{\text{GND}} = 2KAM_{\text{ave}}/\mu b \quad (6)$$

$$KAM_{\text{ave}} = \exp [(1/N)\ln KAM_{R,i}] \quad (7)$$

where μ is the step size selected for the EBSD experiment, taken as 0.7 μm ; N represents the number of test points within the radial section of oxygen-free copper; KAM_{ave} is the average KAM value for the selected region of oxygen-free copper radial grains; $KAM_{R,i}$ is the local KAM value at point i inside the radial section of oxygen-free copper [30]. Figure 8 shows the KAM maps of oxygen-free copper radial grains under various deformation degrees. The color gradient in the legend indicates different misorientation angles, ranging from 0° to 5°. A larger misorientation angle indicates a higher degree of strain, namely a higher geometrically necessary dislocation density. The misorientation angles of different grains were obtained through Channel 5 v5.10 software. The corresponding geometrically necessary dislocation density was calculated. Accordingly, the yield strength caused by dislocation strengthening was determined. The results are shown in Table 2.

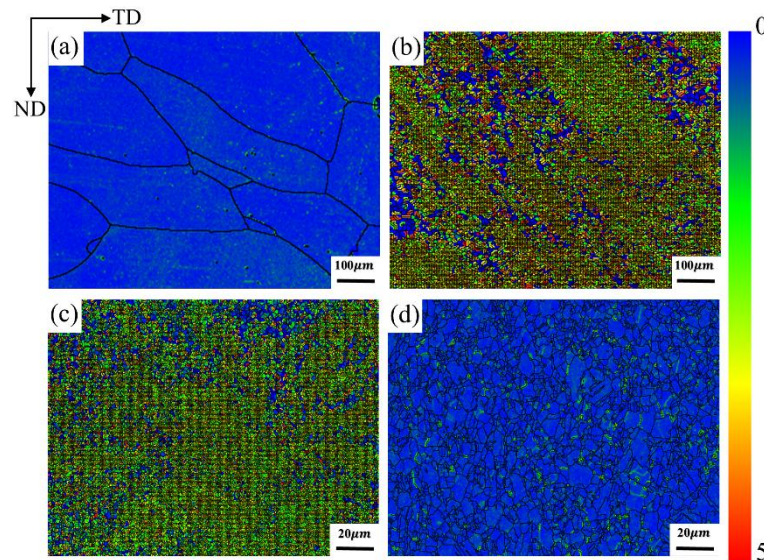


Figure 8. Local average misorientation of oxygen-free copper grains in the radial direction with different drawing deformations: (a) WD 20, $\varepsilon = 0\%$; (b) WD 8.0, $\varepsilon = 84\%$; (c) WD 1.0, $\varepsilon = 99.75\%$; (d) WA 1.0, $\varepsilon = 99.75\%$.

4.1.3. Texture Strengthening

During the drawing process of oxygen-free copper, grains gradually transform from random orientations to $\langle 111 \rangle$ and $\langle 100 \rangle$ orientations, as shown in Figure 2, which forms a microstructure with preferred orientation known as texture. Texture is an important strengthening mechanism in deformed metals, and its calculation formula is given as follows [31]:

$$\sigma'_{YS} = \sigma_{Tex} + \sigma_{GB} \quad (8)$$

$$\sigma'_{YS} = f_{\langle 111 \rangle} \sigma_{\langle 111 \rangle} + f_{\langle 100 \rangle} \sigma_{\langle 100 \rangle} \quad (9)$$

where σ'_{YS} represents the sum of texture strengthening and grain boundary strengthening; $f_{\langle 111 \rangle}$ and $f_{\langle 100 \rangle}$ are the proportion of $\langle 111 \rangle$ oriented grains and $\langle 100 \rangle$ oriented grains in cold-drawing oxygen-free copper wires, respectively, as shown in Table 2; $\sigma_{\langle 111 \rangle}$ and $\sigma_{\langle 100 \rangle}$ are the yield strength increment contributed by $\langle 111 \rangle$ oriented grains and $\langle 100 \rangle$ oriented grains, respectively.

Schmid's law [32] describes the relationship between the critical resolved shear stress and the yield strength, which can be expressed as follows:

$$\tau_s = \sigma_s \cdot \Omega \quad (10)$$

The relationship between the critical resolved shear stress and yield strength for $\langle 111 \rangle$ and $\langle 100 \rangle$ oriented grains is thereby obtained [33], which is expressed as follows:

$$\tau_{\langle 111 \rangle} = \sigma_{\langle 111 \rangle} \cdot \Omega_{\langle 111 \rangle} \quad (11)$$

$$\tau_{\langle 100 \rangle} = \sigma_{\langle 100 \rangle} \cdot \Omega_{\langle 100 \rangle} \quad (12)$$

where $\tau_{\langle 111 \rangle}$ and $\tau_{\langle 100 \rangle}$ are the critical resolved shear stresses for the $\langle 111 \rangle$ and $\langle 100 \rangle$ oriented grains, respectively, which are identical during the cold drawing of oxygen-free copper; Ω is the Schmid factor, with $\Omega_{\langle 111 \rangle}$ and $\Omega_{\langle 100 \rangle}$ being 0.272 and 0.408, respectively.

The critical resolved shear stress of polycrystalline pure copper can be expressed by the following formula:

$$\sigma_{GB} = \tau_{GB} \cdot M \quad (13)$$

According to Formulas (9)–(13), σ_{Tex} can be expressed as follows:

$$\sigma_{\text{Tex}} = \sigma_{\text{GB}} \left[\frac{1}{M} \cdot \left(\frac{f_{\langle 111 \rangle}}{\Omega_{\langle 111 \rangle}} + \frac{f_{\langle 100 \rangle}}{\Omega_{\langle 100 \rangle}} \right) - 1 \right] \quad (14)$$

Based on Equation (14), the contribution of texture to the yield strength of oxygen-free copper under different deformation degrees can be calculated, and the results are presented in Table 2.

As summarized in Table 2, the grain size of oxygen-free copper decreases progressively with increasing drawing deformation, accompanied by a continuous increase in dislocation density and proportion of strong $\langle 111 \rangle$ texture. Consequently, grain boundary strengthening, dislocation strengthening, and texture strengthening are significantly enhanced during the cold drawing process. Among these strengthening mechanisms, the contribution from dislocation strengthening is dominant due to the rapid accumulation and entanglement of deformation-induced dislocations under severe plastic deformation. After annealing treatment, recovery and recrystallization are activated in the severely deformed oxygen-free copper. During this process, recrystallized grains nucleate and grow, while a large proportion of deformation-induced dislocations are annihilated or rearranged into low-energy configurations. As a result, the dislocation density decreases markedly, leading to a significant reduction in both grain boundary strengthening and dislocation strengthening effects.

5. Conclusions

To clarify the deformation-induced microstructure evolution and property response of oxygen-free copper during severe cold drawing, oxygen-free copper wires with different drawing strains were systematically investigated. This work establishes the correlation between deformation microstructure evolution and property variation in oxygen-free copper during severe cold drawing, providing theoretical guidance for the fabrication of high-strength and high-conductivity copper conductors. The main technical conclusions are listed below.

- (1) With increasing drawing deformation from 0% to 99.75%, the grains are progressively elongated, fragmented, and refined, forming a typical fibrous deformation microstructure. The grain size decreases from 339.18 μm to 2.12 μm . After annealing, fine recrystallized equiaxed grains with abundant annealing twins are formed, with an average grain size of 3.07 μm .
- (2) Severe plastic deformation promotes rapid dislocation accumulation and entanglement, leading to the formation of dense dislocation cells, substructures, and nanoscale twins. With increasing deformation, dislocation cells gradually evolve into subgrains, while localized recovery and partial recrystallization occur in regions with high stored deformation energy.
- (3) The texture evolves significantly during cold drawing. The as-cast oxygen-free copper initially exhibits random grain orientations, whereas increasing deformation promotes the gradual formation of preferred $\langle 111 \rangle$ and $\langle 100 \rangle$ textures. After annealing, the recrystallization $\langle 101 \rangle$ texture becomes dominant.
- (4) Increasing drawing deformation continuously enhances the strength of oxygen-free copper due to grain refinement and dislocation strengthening, while the electrical conductivity decreases slightly because of increasing electron scattering from deformation defects. At 99.75% deformation, the tensile strength reaches 486 MPa with an electrical conductivity of 96.7% IACS. After annealing, the conductivity recovers to 101.5% IACS, accompanied by a tensile strength decrease to 187 MPa.

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References

1. Lu, L.; Shen, Y.F.; Chen, X.H.; Qian, L.H.; Lu, K. Ultrahigh Strength and High Electrical Conductivity in Copper. *Science* **2014**, *304*, 422–426.
2. Yang, H.S.; Chen, Y.L.; Feng, H.; Yang, P.; Zhang, J.; Shu, B.P. Optimized corrosion resistance in pure copper via surface mechanical attrition treatment and subsequent annealing. *Mater. Today Commun.* **2023**, *35*, 106069.
3. Lee, B.; Li, W. Performance of different layers of graphene as protective coating for copper wire. *Mater. Lett.* **2020**, *273*, 127875.
4. Sakai, Y.; Schneider-Muntau, H.-J. Ultra-high strength, high conductivity Cu-Ag alloy wires. *Acta Mater.* **1997**, *45*, 1017–1023.
5. Chen, C.H.; Lee, P.I.; Chuang, T.H. Microstructure evolution and failure mechanism of electromigration in Ag-alloy bonding wire. *J. Alloy Compd.* **2022**, *913*, 165266.
6. Rojas, H.A.G.; Egea, A.J.S.; Hameed, S.; Bolmaro, R. An Ultra-Fast Annealing Treatment by Electropulsing during Pure Copper Wire Drawing. *Metals* **2019**, *9*, 1253.
7. Han, Z.Y.; Li, C.; Chen, Z.S.; Ning, H.Y.; Li, F. Plastic recovery and strength retention of cryogenic rolled pure Cu via high density electropulsing. *Mater. Sci. Eng. A* **2026**, *956*, 149902.
8. Dong, J.C.; Liu, H.T.; Li, W.Q.; Hua, Y.X.; Zhang, S.Y.; Song, K.X. Effect of microalloying with different contents of Al and Sc on the microstructure and properties of pure copper. *J. Mater. Res. Technol.* **2025**, *39*, 1480–1493.
9. Fan, X.Y.; Hou, J.P.; Lu, Y.; Wang, S.; Liu, Z.Q.; Duan, Q.Q.; Wang, Q.; Zhang, Z.J.; Zhang, Z.F. Strengthening mechanisms and strength prediction of directionally solidified Cu wire processed by cold-drawing. *Mater. Today Commun.* **2025**, *42*, 111421.
10. Liu, P.S.; Tong, L.Y.; Wang, J.L.; Shi, L.; Tang, H. Challenges and developments of copper wire bonding technology. *Microelectron. Reliab.* **2012**, *52*, 1092–1098.
11. Zhang, Z.H.; Liu, H.T.; Hua, Y.X.; Zhang, B.F.; Hou, Z.G.; Li, W.Q.; Song, K.X. Study on the effect of phosphorus on anti-softening performance of oxygen-free copper. *J. Mater. Res. Technol.* **2024**, *33*, 2612–2623.
12. Dobatkin, S.V.; Szpunar, J.A.; Zhilyaev, A.P.; Cho, J.-Y.; Kuznetsov, A.A. Effect of the route and strain of equal-channel angular pressing on structure and properties of oxygen-free copper. *Mater. Sci. Eng. A* **2006**, *462*, 132–138.
13. Sun, P.F.; Zhang, P.L.; Hou, J.P.; Wang, Q.; Zhang, Z.F. Quantitative mechanisms behind the synchronous increase of strength and electrical conductivity of cold-drawing oxygen-free Cu wires. *J. Alloy Compd.* **2021**, *863*, 158759.
14. Waryoba, D.R.; Kalu, P.N.; Crooks, R. Grain-boundary structure of oxygen-free high-conductivity (OFHC) copper subjected to severe plastic deformation and annealing. *Mater. Sci. Eng. A* **2008**, *494*, 47–51.
15. Huang, K.; Logé, R.E. A review of dynamic recrystallization phenomena in metallic materials. *Mater. Des.* **2016**, *111*, 548–574.
16. Liu, L.; Baker, I. The dependence of recrystallization temperature and stored energy on rolling strain in polycrystalline copper. *Scr. Metall. Mater.* **1993**, *28*, 197–200.
17. Shigematsu, T.; Morita, K.; Fujii, Y.; Shigi, T.; Nakamura, M.; Yamaguchi, M. Investigation of annealing effects of ultra pure copper. *Cryogenics* **1992**, *32*, 913–915.

18. Mahajan, S.; Pande, C.S.; Imam, M.A.; Rath, B.B. Formation of annealing twins in f.c.c. crystals. *Acta Mater.* **1997**, *45*, 2633–2638.
19. Cho, J.-H.; Oh, K.H.; Rollett, A.D.; Cho, J.-S.; Park, Y.-J.; Moon, J.-T. Investigation of Recrystallization and Grain Growth of Copper and Gold Bonding Wires. *Metall. Mater. Trans. A* **2006**, *37*, 3085–3097.
20. Liu, J.S.; Zhou, Y.; Wang, S.W.; Chen, S.F.; Song, H.W.; Zhang, S.H. Exploring the evolution of texture and properties of ultrafine copper wire during high strain drawing process. *J. Cent. South Univ.* **2025**, *32*, 1973–1994.
21. Randle, V.; Owen, G. Mechanisms of grain boundary engineering. *Acta Mater.* **2006**, *54*, 1777–1783.
22. Brandon, D. The structure of high-angle grain boundaries. *Acta Metall.* **1966**, *14*, 1479–1484.
23. Han, S.Z.; Choi, E.-A.; Lim, S.H.; Kim, S.; Lee, J. Alloy design strategies to increase strength and its trade-offs together. *Prog. Mater. Sci.* **2020**, *117*, 100720.
24. Li, X.; Zhou, Y.J.; Liu, Y.H.; Li, S.L.; Song, K.X.; Feng, C.L.; Wu, B.A.; Zhang, P.F.; Wu, H.J.; Gu, J.H.; et al. Texture evolution and its influence mechanism on properties of single crystal copper and polycrystalline copper during cumulative deformation. *J. Mater. Res. Technol.* **2023**, *24*, 6808–6819.
25. He, J.Y.; Wang, H.; Huang, H.L.; Xu, X.D.; Chen, M.W.; Wu, Y.; Liu, X.J.; Nieh, T.G.; An, K.; Lu, Z.P. A precipitation-hardened high-entropy alloy with outstanding tensile properties. *Acta Mater.* **2016**, *102*, 187–196.
26. Niels, H. Hall–Petch relation and boundary strengthening. *Scr. Mater.* **2004**, *51*, 801–806.
27. Dasharath, S.M.; Koch, C.C.; Mula, S. Effect of stacking fault energy on mechanical properties and strengthening mechanisms of brasses processed by cryorolling. *Mater. Charact.* **2015**, *110*, 14–24.
28. Wang, Z.W.; Lu, W.J.; Zhao, H.; Liebscher, C.H.; He, J.Y.; Ponge, D.; Raabe, D.; Li, Z.M. Ultrastrong lightweight compositionally complex steels via dual-nanoprecipitation. *Sci. Adv.* **2020**, *6*, 9543.
29. Starink, M.J.; Qiao, X.G.; Zhang, J.W.; Gao, N. Predicting grain refinement by cold severe plastic deformation in alloys using volume averaged dislocation generation. *Acta Mater.* **2009**, *57*, 5796–5811.
30. Gubicza, J.; Chinh, N.Q.; Lábár, J.L.; Dobatkin, S.; Hegedűs, Z.; Langdon, T.G. Correlation between microstructure and mechanical properties of severely deformed metals. *J. Alloy Compd.* **2008**, *483*, 271–274.
31. Skrotzki, W.; Scheerbaum, N.; Oertel, C.G.; Arruffat-Massion, R.; Suwas, S.; Tóth, L.S. Microstructure and texture gradient in copper deformed by equal channel angular pressing. *Acta Mater.* **2007**, *55*, 2013–2024.
32. Chen, J.; Yan, W.; Liu, C.X.; Ding, R.G.; Fan, X.H. Dependence of texture evolution on initial orientation in drawn single crystal copper. *Mater. Charact.* **2011**, *62*, 237–242.
33. Hou, J.P.; Li, R.; Wang, Q.; Yu, H.Y.; Zhang, Z.J.; Chen, Q.Y.; Ma, H.; Wu, X.M.; Li, X.W.; Zhang, Z.F. Three principles for preparing Al wire with high strength and high electrical conductivity. *J. Mater. Sci. Technol.* **2019**, *35*, 742–751.

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