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Influence of the Dual Rolls Equal Channel Extrusion (DRECE) Process on the Microstructure and Mechanical Properties of Pure Copper

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Abstract: The paper presents the research results on the influence of the parameters of the Dual Rolls Equal Channel Extrusion (DRECE) process on the microstructure and mechanical properties of pure copper. The research was conducted for two variants of the DRECE channel geometry - with extrusion angles of 103° and 98°. Microstructure observations were performed using optical and electron microscopy with backscattered electron diffraction (EBSD). Mechanical properties were determined based on Vickers hardness measurements and tensile tests. The results showed that intense changes in mechanical properties occurred during the first DRECE pass, while subsequent passes led to further moderate strengthening. The values of yield strength (YS) and ultimate tensile strength (UTS) for both DRECE angles were characterized by low anisotropy. The results of the mechanical property tests indicate that, for pure copper, a lower extrusion angle provides higher ductility at comparable YS and UTS values. EBSD analysis showed noticeable microstructure segmentation and an increase in the proportion of fine grains. IPF maps showed a varied crystallographic orientation after DRECE, with no tendency to form a distinct texture direction.

Keywords: DRECE; copper; SPD; EBSD

1. Introduction

Conventional plastic processing methods, such as rolling, drawing, or forging, through plastic deformation mechanisms (increased dislocation density, grain refinement – Hall-Petch relationship), allow for a significant improvement in the strength properties of copper and other metals and alloys [1,2]. Studies have shown that properly controlled plastic processing allows for a compromise between the strength and conductivity of copper, which is crucial for engineering applications [3].

In addition to the aforementioned conventional plastic working methods, Severe Plastic Deformation (SPD) methods constitute a separate group. They provide new capabilities for creating microstructures and, consequently, mechanical properties that cannot be achieved by conventional methods [4]. An example is KOBO extrusion technology, where high strength properties can be obtained in copper alloys [5]. Another example is Repetitive Corrugation and Straightening (RCS), which, although it uses a different strengthening mechanism, also allows high strength to be obtained in copper alloys [6,7].

SPD methods allow for the introduction of intensive plastic deformations, in some cases without significant changes in geometric dimensions [8]. Processes such as Equal Channel Angular Pressing (ECAP) [9], Twist Channel Angular Pressing (TCAP) [10], C-ECAP [11,12], and Accumulative Roll Bonding (ARB) [13] create the possibility of obtaining ultra-fine-grained structures in the material, characterized by high dislocation density [14]. In the case of pure copper, it has been shown that SPD methods can significantly increase hardness and tensile strength while maintaining acceptable ductility [15].

Despite the many advantages of SPD techniques, their industrial application is limited due to their discontinuous character. The answer to these limitations is the use of continuous SPD methods, in particular Dual Rolls Equal Channel Extrusion (DRECE). The DRECE process involves extruding a metal strip through a roller system into a channel with a specific angle (Figure 1), which causes it to bend, thus generating deformation through intense shearing across the entire cross-section of the material [16, 17].

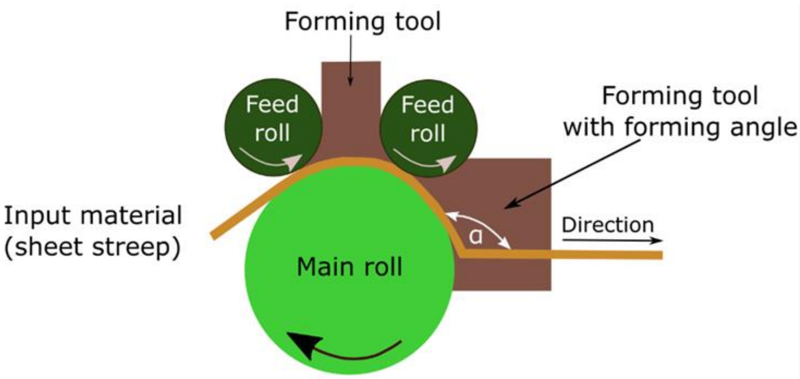

Figure 1. Schematic diagram of the DRECE process.

DRECE technology is currently used mainly for metallic materials with high potential for strengthening through intensive plastic deformation. The literature describes the beneficial effects of its application for low-carbon steels (DC01, DC03), in which an increase in hardness and improvement in mechanical properties were observed [18]. Also for aluminum and its alloys, the DRECE process enabled significant microstructural changes, resulting in increased strength properties [19]. Simulation studies of the DRECE process for low-carbon steel strips were also conducted, on the basis of which the uniformity of the deformation process was assessed [20]. The application of this method was also presented in the context of further industrial processes [21]. Previous research on DRECE technology has shown that after successive passes, the intensity of work hardening increased, leading to stronger grain refinement and a marked increase in strength properties [17,22,23].

Despite the growing number of studies on the DRECE process, most of them focus on the aforementioned steels and aluminum alloys, while research on pure metals remains limited. In particular, there is a lack of studies considering the simultaneous influence of the channel angle and the number of passes on the evolution of the microstructure and mechanical properties. The novelty of this work was to investigate the combined influence of DRECE channel geometry (angles of 103° and 98°) and the number of passes on the microstructure and mechanical properties of pure copper, along with the direct correlation of structural changes with mechanical properties.

2. Materials and Methods

The test material consisted of samples in the form of pure copper strips with a thickness of 15 mm, cold-rolled to a thickness of 2.0 mm and 1.5 mm with a total plastic deformation of approx. 90 %. The chemical composition of the samples was examined using an optical emission spectrometer (Table 1).

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Table 1. Chemical composition of pure copper samples (wt %).

Cu	Zn	Ag	Fe	Sn	Pb	Bi	As	P	S	others
99,9897	<0,0015	0,00057	0,00038	<0,0002	<0,0003	<0,0005	<0,0002	<0,0002	0,00085	rest

The material, strengthened after rolling, was then subjected to recrystallization annealing in an induction vacuum furnace at 350 °C for 1 hour in a protective atmosphere of 95 % N / 5 % H, and then actively cooled in a furnace with forced atmosphere circulation. As a result, samples in the initial state (IS) with a recrystallized microstructure and a hardness of approx. 50 HV01 were obtained (Figure 2).

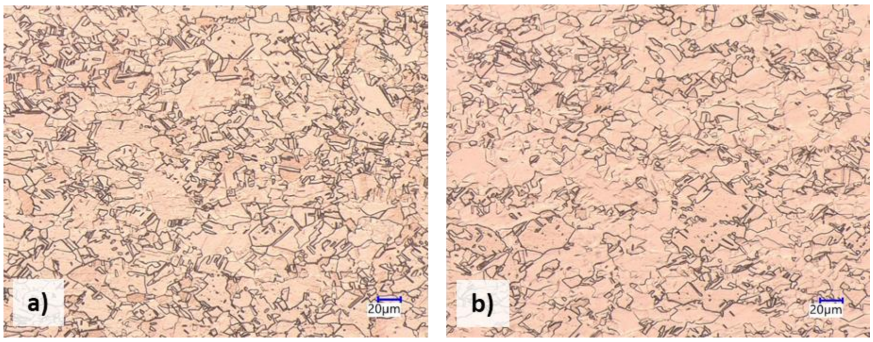

Figure 2. Microstructure of samples in the initial state for DRECE tests. Light microscopy: a) transverse cross-section; b) longitudinal cross-section.

The DRECE tests were performed in a device with a forming zone angle of 98° and 103°. The samples were subjected to 1, 2, and 3 passes, and after each pass they were rotated by 180° along the longer edge. The thickness of the strips was 1.5 mm and 2 mm for the 103° and 98° angles, respectively. The width and length of the strips in both variants were identical and measured 57 mm and 1000 mm, respectively. The process was performed at room temperature with an extrusion speed of 1 cm/s, which was kept constant for both analyzed channel angles. The strips were not preheated prior to processing. No lubrication was used, and the process was carried out under dry friction conditions between the strip and the die.

Changes in mechanical properties depending on the number of passes were determined in a tensile test and Vickers HV0.1 microhardness measurement. A static tensile test was performed on a Zwick/Roell Z100 device. In order to assess the anisotropy of strength properties, samples were taken from three directions – longitudinal, transverse, and approx. 45° to the DRECE extrusion direction (3 samples for each variant – Figure 3a) and then proportional samples were prepared, as shown in Figure 3b. HV0.1 microhardness measurements were performed using a Future-Tech FM-700 device.

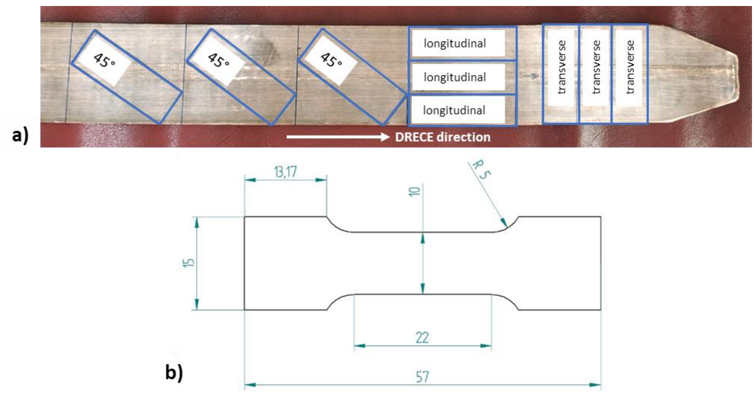

Figure 3. Stages of sample preparation for static tensile testing. a) locations from which samples were taken; b) geometric dimensions of the prepared samples.

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Microstructure observations were performed using a Keyence VHX-7000 optical microscope and a Carl Zeiss Supra 35 scanning electron microscope equipped with an electron backscatter diffraction (EBSD) detector. Due to the type of deformation in the DRECE method, the test samples were taken from the longitudinal direction to the extrusion direction. The images for optical microscopy were taken on samples etched with K2Cr2O2+NaCl+H2SO4+H2O reagent, while the EBSD maps were taken on samples electropolished in Struers D2 electrolyte.

3. Results

3.1. Mechanical properties

Figure 4 shows the distribution of HV0.1 microhardness of copper samples measured along the thickness for each pass on a device based on the DRECE method (0–3) passes. The distance between successive measurements was approximately 150 µm (Figure 4a). The range of values is marked with a long line.

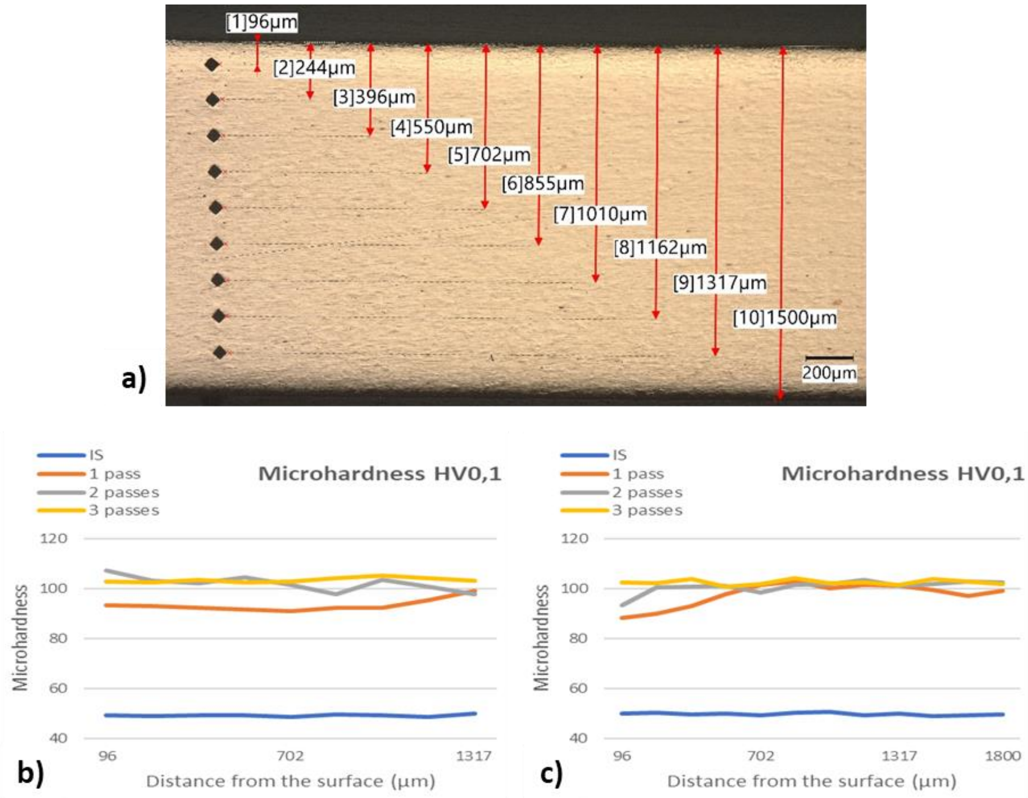

Figure 4. Microhardness measurement on the thickness of copper strips after 0 to 3 DRECE passes: a) measurement points; b) results for a DRECE angle 98°; c) results for a DRECE angle 103°.

Based on the microhardness distribution shown in Figure 4, it can be seen that the hardness of the strip in the initial state (IS) in both variants was similar (approx. 50 HV) and uniform across the thickness of the strip. The largest increase in microhardness, approx. 90 %, was observed after first DRECE pass - to approx. 94 HV for a strip with a thickness of 1.5 mm and a DRECE angle of 98° (Figure 4a) and 97 HV for a strip with a thickness of 2.0 mm and a DRECE angle of 103° (Figure 4b). After the second pass, a further, but significantly smaller increase to approx. 101 HV was observed for both variants. The third pass had no significant effect on the increase in microhardness, which was approx. 103 HV for both die angles. However, it can be noted that after the third pass, greater uniformity of hardness was obtained across the thickness of the strip. Based on the measure-

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ments performed, it can be concluded that for the tested copper, the size of the DRECE angle does not significantly affect the microhardness obtained on the longitudinal cross-section after subsequent passes. The peak value was obtained after the second pass, while the third pass mainly increased its uniformity across the thickness of the material. Table 2 shows the results of the static tensile test for the longitudinal, transverse, and 45° angles to the direction of DRECE extrusion, and Figure 5 shows a graphical interpretation of the anisotropy of the obtained properties - tensile strength, yield strength, and elongation - for the base sample and after the 1st and 3rd DRECE pass.

Table 2. Static tensile test. Effect of the DRECE extrusion angle and number of passes on the yield strength (YS), ultimate tensile strength (UTS), and elongation (% El). Values represent average of three tests.

	Longitudinal			Transverse			Angle 45°		
No. of passes	$R_{p0,2}$	R_m	A	$R_{p0,2}$	R_m	A	$R_{p0,2}$	R_m	A
	MPa	MPa	%	MPa	MPa	%	MPa	MPa	%
Initial State	82	234	72	85	220	69	84	234	70
DRECE, angle 103°									
1 pass	265	266	21	269	269	15	273	273	15
2 passes	282	283	17	285	288	13	287	289	12
3 passes	305	309	17	308	311	13	315	319	11
DRECE, angle 98°									
1 pass	260	264	23	271	272	35	252	256	23
2 passes	258	262	20	272	273	26	264	266	21
3 passes	278	279	19	293	296	21	301	304	19

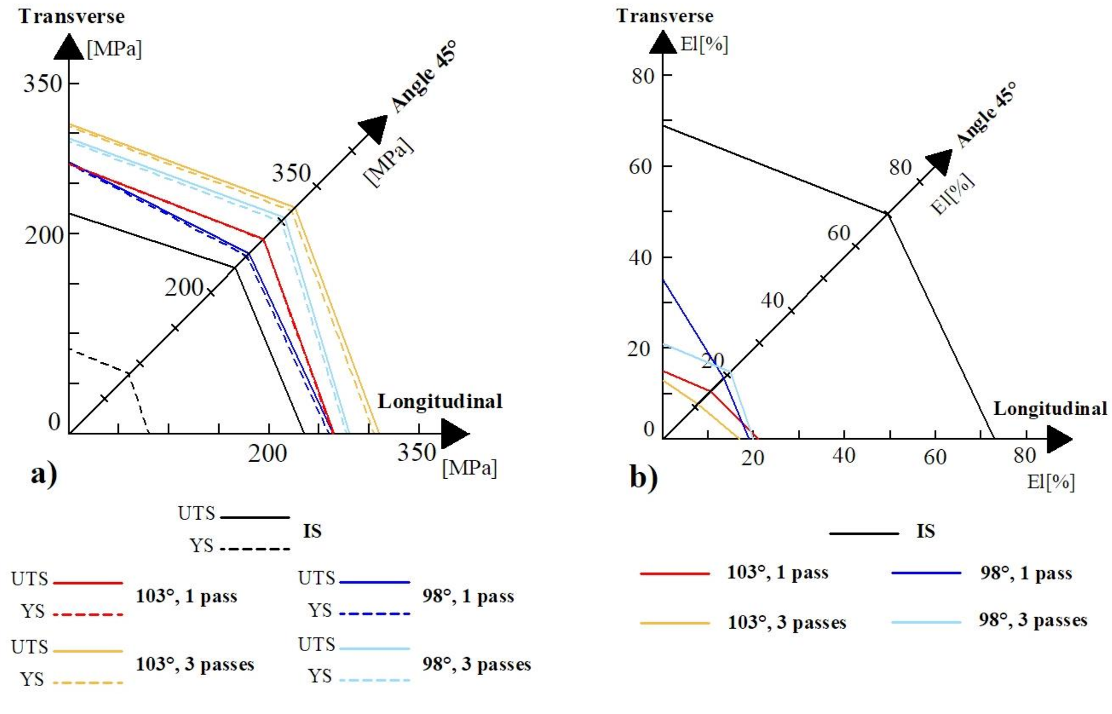

Figure 5. Graphical interpretation of the anisotropy of mechanical properties after 0, 1st, and 3rd DRECE passes: a) yield strength (YS), ultimate tensile strength (UTS) ; b) elongation (% El)

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Tensile tests performed in three orientations relative to the DRECE extrusion direction allowed for the assessment of changes in mechanical properties and their anisotropy. The first DRECE pass had the greatest impact on the strengthening of the material—the yield strength (YS) increased from approx. 82 MPa to approx. 260–270 MPa, and the ultimate tensile strength (UTS) reached values close to YS, which may explain the observed decrease in elongation (% El) from approx. 70 % in the initial state to (15–20) %. Subsequent passes (2nd and 3rd) resulted in a further, but significantly smaller increase in YS and UTS. After the 3rd pass, for both extrusion angles, YS values of (300–315) MPa were obtained, while UTS remained at a similar level. The values of yield strength and ultimate tensile strength were characterized by low anisotropy in the tested DRECE extrusion directions – longitudinal, transverse, and at an angle of 45° (Figure 5a). Noticeable differences in anisotropy were observed for elongation A – Figure 5b. For the DRECE extrusion angle of 103°, a small tendency for greater elongation A in the longitudinal direction was observed. More pronounced differences in elongation were observed for the smaller extrusion angle of 98°, especially after the 1st pass, where Figure 5b showed a significantly higher elongation in the transverse direction (% El ~35%), while for the other directions, % El was at 23 %. After the 3rd pass, these differences were no longer present and the anisotropy of elongation was negligible.

3.2. Microstructure – optical microscopy

Based on the slight differences in mechanical properties between the 2nd and 3rd DRECE passes for both extrusion angles, an initial state (IS) sample and samples after the 1st and 3rd passes were selected for microstructure observation. Figure 6 shows images of the microstructures obtained using an optical microscope for the selected samples.

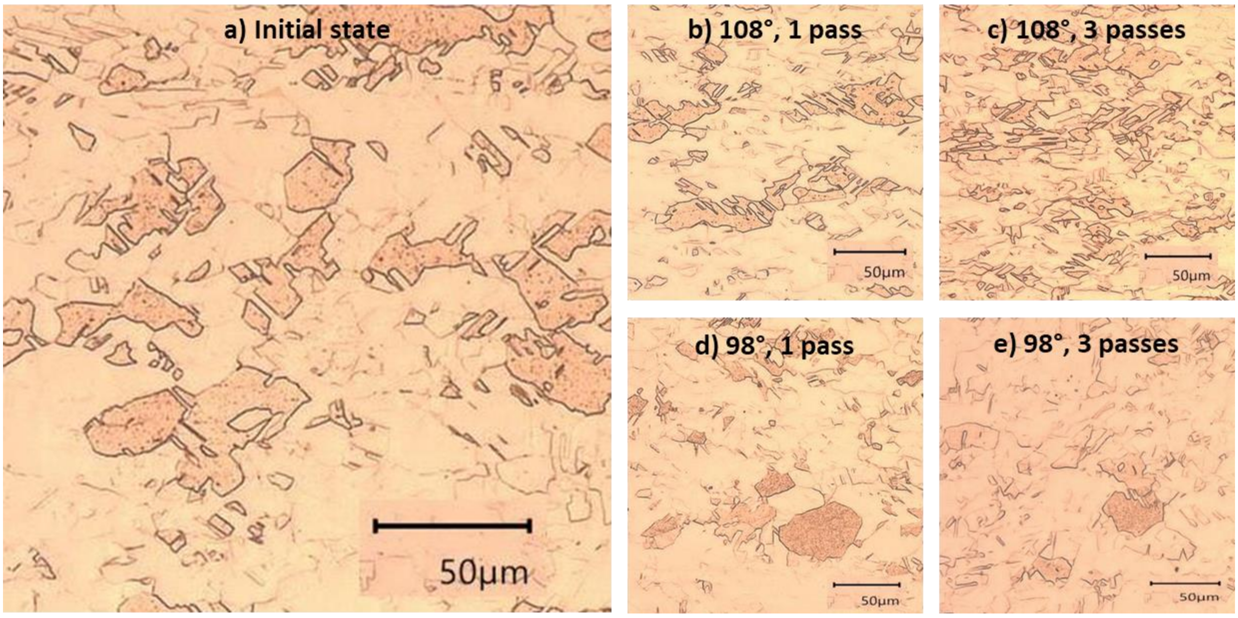

Figure 6. Images of the microstructure of samples in the initial state (IS) and after successive DRECE passes. Optical microscopy.

In the sample in initial state (Figure 6a), a microstructure with varied grain sizes was observed, typical for annealed copper after plastic processing. After subsequent DRECE passes, regardless of the extrusion angle and number of passes, no significant effect on grain fragmentation is observed. However, it can be seen that at a larger extrusion angle (103°), the grains are elongated in the direction of extrusion (Figures 6b-c). This effect is less noticeable for the 98° angle. For a more in-depth analysis of the microstructure, scanning microscopy using the EBSD technique was used.

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3.2. Microstructure – EBSD analysis

Figure 7 shows IPF maps of grain orientation for pure copper strip in the initial state (IS) and after the 1st and 3rd pass for DRECE extrusion angles of 103° and 98°. Combined with the grain size histograms (Figure 8), it was possible to evaluate the microstructural changes occurring during deformation in the DRECE method.

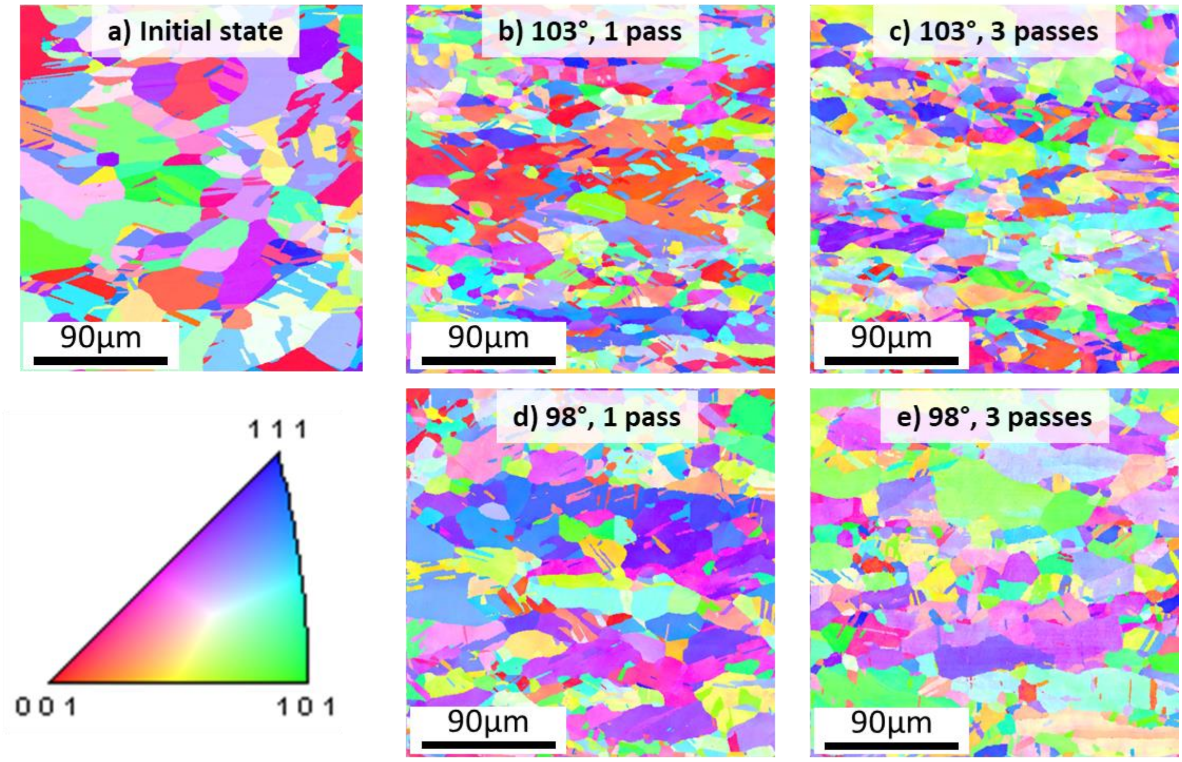

Figure 7. IPF (EBSD) maps showing microstructure evolution after DRECE extrusion.

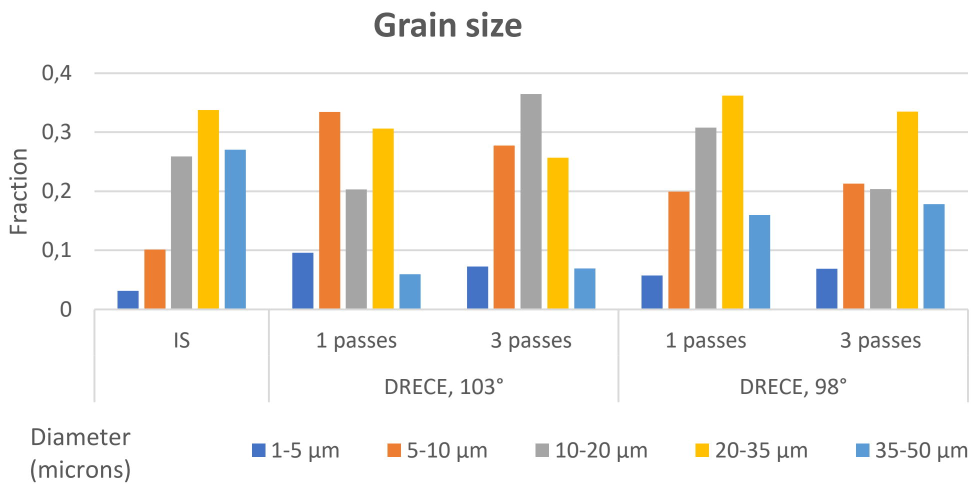

Figure 8. Characteristics of the microstructure after the DRECE process. Histograms of grain size distribution.

In the initial state (IS) sample, grains with a size of (10–50) µm were predominant (Figure 8). After the first DRECE pass, a noticeable shift in the grain size distribution towards smaller sizes was observed, which was

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particularly evident for the (5–10) μm range, whose share increased significantly for both deformation angles. At the same time, the proportion of the largest grains (35–50) μm was reduced, indicating the onset of grain refinement. The third pass no longer had a significant effect on the fraction of the smallest grains, which was at a similar level to the second pass. This suggests that the material reached a state in which further deformation no longer had a significant effect on the evolution of the microstructure. Observation of the IPF maps (Figure 7) shows a diverse crystallographic orientation of the grains, with no tendency to form a clearly oriented texture. A more pronounced effect of the DRECE method on grain orientation can be seen for the 103° extrusion angle after the first pass, where, compared to the other variants, grains with a <001> orientation were clearly visible (Figure 7b).

4. Discussion

The conducted research allowed for the assessment of the influence of the DRECE process – using two channel angle variants (103° and 98°) and after the 1st, 2nd, and 3rd pass – on the microstructure and mechanical properties of pure copper. The results obtained from tensile tests, Vickers microhardness HV0.1, optical microscopy, and scanning electron microscopy equipped with an EBSD detector were used to determine the changes in the material caused by intense shear deformation.

4.1. Mechanical properties

For both extrusion angles (103° and 98°), the highest increase in mechanical properties was observed after the first DRECE pass. Static tensile test showed that the yield strength (YS) increased from approx. 82 MPa (initial state) to approx. 265 MPa, while the ultimate tensile strength (UTS) increased from 234 MPa to approx. 266 MPa. Along with the increase in YS and UTS values, a decrease in elongation (El) from 70 % to (15–20) % was observed. The microhardness profile measured on the thickness of the samples also showed that the first DRECE pass had the greatest impact, after which an increase from 50 HV0.1 (initial state) to 94 HV0.1 was obtained for both extrusion angles. The second and third DRECE passes caused significantly smaller changes in tensile properties and microhardness. After the third pass, the YS values stabilized in the range of (305–315) MPa, UTS reached a level similar to YS, and microhardness slightly exceeded 100 HV0.1. Tensile tests on samples taken from three directions – longitudinal, transverse, and 45° to the DRECE extrusion direction – allowed the evaluation of the anisotropy of tensile properties. It was observed that the ultimate tensile strength and yield strength were characterized by low anisotropy for both DRECE angles. Larger differences were observed in elongation after the first pass, where there was a tendency for significantly greater values in the transverse direction for the 98° forming zone angle, as well as slightly greater values in the longitudinal direction for the 103° forming angle. After the 3rd pass, for both DRECE angles, the differences in the degree of elongation anisotropy disappeared. The tests carried out showed that for pure annealed copper, a lower die angle allows for obtaining material with higher plasticity (higher elongation) at comparable YS, UTS, and microhardness values. The obtained strengthening effect can be related to results reported for the Equal Channel Angular Pressing (ECAP) method, which is one of the most widely studied severe plastic deformation techniques. Similarly to ECAP, the DRECE process also resulted in a significant increase in mechanical properties. In the present work, this effect was achieved while maintaining relatively low anisotropy and allowing continuous processing of the material in the form of strips. This indicates that DRECE may represent an effective alternative to classical SPD methods, particularly in terms of process continuity and potential industrial applications.

4.2. Microstructure analysis

Microstructural observations conducted using optical microscopy did not reveal any significant changes in grain morphology on the longitudinal section when compared with the annealed material. The only noticeable effect of the DRECE process was a slight elongation of grains in the DRECE extrusion direction, which was observed mainly for the forming zone angle of 103°. In the case of the smaller angle of 98°, this effect was

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considerably weaker. This suggested that a larger channel angle resulted in more intense strain hardening and, consequently, more pronounced changes in grain shape. Apart from these differences, the remaining microstructural features observed by optical microscopy were similar for all variants, which justified the application of EBSD analysis for a more detailed evaluation of microstructural changes.

EBSD analysis showed that after the first DRECE pass, an increase in the fraction of the smallest grains (<10 μm) was observed, accompanied by a decrease in the fraction of larger grains (>15 μm) and an increase in the proportion of low- and medium-angle grain boundaries. These changes indicated progressive grain fragmentation and explained the observed improvement in mechanical properties. Analysis of the IPF maps (Fig. 8) for both DRECE angles revealed a diversified crystallographic orientation of grains, with no tendency to form a pronounced texture in a specific direction. A more pronounced effect of the DRECE process on grain orientation was observed for the 103° zone angle after one pass, where, in comparison with the other variants, grains oriented in the <001> direction accounted for a significant fraction.

The observed differences in microstructure and mechanical properties between the 98° and 103° channel angles can be related to variations in shear strain distribution and strain path across the material. Additionally, in SPD processes, the occurrence of redundant strain has been reported, which may locally influence grain elongation and crystallographic orientation. In the literature on severe plastic deformation processes [24,25], modifications of the strain path and the presence of redundant strain have been shown to affect grain refinement and texture evolution during repeated passes. Therefore, similar mechanisms may contribute to the observed differences in microstructure and mechanical properties between the 98° and 103° DRECE channel angles.

5. Conclusions

Mechanical property testing and microstructural observations carried out using optical microscopy and EBSD analysis made it possible to assess the potential role of the shear-dominated character of deformation in the DRECE process. It was concluded that:

1. The most significant changes in mechanical properties occurred during the first pass through the DRECE device, as confirmed by an increase in yield strength (YS) and ultimate tensile strength (UTS), accompanied by a decrease in elongation (El) and a pronounced increase in hardness. The observed strengthening was consistent with the Hall–Petch relationship.
2. The second and third passes resulted in further strengthening; however, the magnitude of this effect was smaller. After the third pass, the mechanical properties tended to stabilize.
3. Both extrusion angles (103° and 98°) resulted in comparable strengthening effects. Nevertheless, the results indicated that the 98° variant allowed similar values of YS and UTS to be achieved while maintaining a significantly higher elongation. This suggested that, for pure copper, a smaller DRECE angle enabled the attainment of material with comparable strength and a higher ductility.
4. The values of yield strength and ultimate tensile strength for both DRECE angles were characterized by low anisotropy.
5. EBSD analysis revealed pronounced microstructural fragmentation, particularly an increase in the fraction of grains smaller than 10 μm. IPF maps showed a varied crystallographic orientation after DRECE, with no tendency to form a distinct texture direction.

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