



## Exploring the Impact of Deformation on Strain Energy of Ultrafine-Grained Copper through Equal Channel Angular Pressing: Insights from X-Ray and Neutron Diffraction

Muhammad Rifai<sup>1,\*</sup>, Mujamilah<sup>2</sup>, Andon Insani<sup>2</sup>, M. Refai Muslih<sup>2</sup>, Tri Hardi Priyanto<sup>2</sup>, Iwan Sumirat<sup>2</sup>, Bharoto<sup>2</sup>, Grace Tj. Sulungbudi<sup>2</sup>, Ahadi Damar Prasetya<sup>2</sup>, Ridwan<sup>2</sup>, Muhamad Saparudin<sup>3</sup>, Diene Noor H.<sup>4</sup>, Sairun<sup>2</sup>, Rifky Apriansyah<sup>3</sup>, Wildan Z. Lubis<sup>3</sup>

<sup>1</sup>Research Center for Accelerator Technology, National Research and Innovation Agency of Indonesia (BRIN), Indonesia

<sup>2</sup>Research Center for Nuclear Beam Analysis Technology, National Research and Innovation Agency of Indonesia (BRIN), Indonesia

<sup>3</sup>Directorate for Nuclear Facility Management, National Research and Innovation Agency of Indonesia (BRIN), Indonesia

<sup>4</sup>Research Center for Nuclear Reactor Technology, National Research and Innovation Agency of Indonesia (BRIN), Indonesia

\*E-mail: muha084@brin.go.id

**Abstract.** The mechanical behavior of materials at the nanoscale level has attracted significant attention in materials science research. This study investigated the impact of deformation on the strain energy of ultrafine-grained (UFG) copper produced by equal channel angular pressing (ECAP). X-ray and neutron diffraction techniques were employed to analyze the changes in strain energy of the copper samples subjected to varying levels of deformation. The results of the study indicate that the strain energy of the UFG copper increased with increasing levels of deformation due to the formation of dislocations with high density in the copper crystal structure. The study also found that the increase in strain energy was more significant in the initial stages of deformation and then reached a plateau as deformation progressed. This study provides valuable insight into the mechanical behavior of UFG copper and the role of deformation in altering its strain energy. The results can contribute to developing new materials with improved mechanical properties for various aerospace, automotive, and biomedical applications. The study highlights the importance of using advanced techniques such as X-ray and neutron diffraction to accurately investigate the nanoscale mechanical behavior of materials.

**Keywords:** *deformation; equal channel angular pressing; neutron diffraction; strain energy; X-ray diffraction.*

## 1 Introduction

Ultrafine-grained (UFG) copper is a material of particular interest in regard of the present study due to its potential for use in various industries such as aerospace, automotive, and biomedical. The mechanical properties of UFG copper can be significantly altered through deformation, making it an attractive material for various applications. Many researchers have demonstrated that the microstructure of copper is significantly refined after deformation processes, which leads to an improvement in the strength and ductility of the material [1-4]. The mechanical properties of copper can be altered through various processes, including deformation, heat treatment, and alloying [5-7]. Studying them provides insight into the effects of grain size on the mechanical properties and deformation behavior of copper. The deformation of materials can significantly alter their mechanical properties, including their strain energy, as demonstrated in [8-11].

The study of strain energy is crucial for understanding a material's mechanical behavior. ECAP is a popular deformation technique used to alter the mechanical properties of metals such as copper. ECAP involves repeatable pressing of a metal billet through a die with two channels that intersect at a specific angle. ECAP is a widely used deformation technique for producing UFG metals with unique mechanical properties. The metal billet undergoes severe plastic deformation through successive passes in the die, which generates a high density of dislocations within the crystal structure. The dislocations cause the grains of the material to break down into smaller grains, resulting in a UFG microstructure. The unique microstructure produced through ECAP typically results in enhanced strength and hardness while retaining a reasonable balance of ductility, depending on the extent of microstructural refinement. The improved mechanical properties make ECAP metals attractive for various industrial applications, including aerospace, automotive, and biomedical. Valiev and Langdon [13] discuss the principles of ECAP and its potential as a processing tool for grain refinement. They emphasize the importance of the reduction in strain energy to refine grains and improve the mechanical properties of materials. Li *et al.* [14] investigated the strain path effect on microstructure evolution and deformation behavior of copper during ECAP. They found that changing the strain path could alter the deformation behavior and microstructure evolution of copper, leading to different mechanical properties. Sivaprasad *et al.* [15] employed ECAP for studying the deformation behavior of a medium carbon steel. They observed an increase in hardness and tensile strength due to the significant reduction in grain size and changes in the

dislocation density and substructure during ECAP. Hosseini *et al.* [16] conducted a comparative study to investigate the effect of back pressure and rotation on microstructure and texture evolution during severe plastic deformation of pure copper by ECAP. They found that the back pressure could induce more strain homogeneity, while rotation could refine the grains more effectively. Huang *et al.* [17] explored the formation and evolution of shear bands of pure copper processed by high-pressure torsion. They observed a strong correlation between the shear band formation and the initial texture, deformation temperature, and strain rate during the processing of pure copper by high-pressure torsion.

X-ray and neutron diffraction techniques are commonly used to investigate the changes in the crystal structure of metals such as copper subjected to deformation through ECAP. Zhang *et al.* [18], through an in-situ X-ray diffraction (XRD) study, found that the Ti sample exhibited a continuous increase in strain with increasing torsion cycles and significant grain refinement was observed. Li *et al.* [19] observed that the Cu sample underwent a continuous increase in strain with increasing torsion cycles and significant changes in microstructure and texture were detected. Song *et al.* [20] conducted in-situ neutron diffraction and found that the alloy exhibited cyclic softening during the first few cycles, followed by a gradual increase in strain hardening. Hong *et al.* [21] performed in-situ neutron diffraction on a Ti-48Al-2Nb-2Cr alloy during compression and observed that the alloy exhibited a gradual increase in strain with increasing compressive stress, and significant changes in texture and phase transformation were detected. Cakmak *et al.* [22] investigated the deformation behavior of a metastable austenitic steel using in-situ neutron diffraction. The steel sample exhibited a continuous increase in strain with increasing deformation and a significant change in microstructure was observed due to the formation of deformation twins.

In this study, the impact of deformation on the strain energy of UFG copper through ECAP was explored in detail. X-ray and neutron diffraction techniques were applied to investigate the changes in the crystal structure and strain energy of the copper samples subjected to varying levels of deformation. This study aimed to provide valuable insight into the mechanical behavior of UFG copper and the role of deformation in altering its strain energy. The results of the study may contribute to the development of new materials with improved mechanical properties for various industrial applications.

## 2 Experimental Procedure

Copper samples were prepared by cutting 10-cm long rods of commercially available oxygen-free high conductivity (OFHC) copper into cylindrical billets

with a diameter of 10 mm using a high-precision saw. The billets were then annealed at 600 °C for 2 hours to remove any residual stress and ensure a homogenous initial microstructure. ECAP was used to deform the copper billets to produce UFG copper samples. Deformation was carried out using a die consisting of two channels intersecting at an angle of 110°. The billets were pressed through the die using a hydraulic press, and the pressing was repeated for a total of five passes through the die. Prior to ECAP processing, the copper billets exhibited a coarse-grained structure with a grain size of 25 to 30  $\mu\text{m}$ , as typically observed in annealed OFHC copper. This condition was used as the reference state before the evolution toward ultrafine grains during ECAP. The angle between the channels was maintained at 110° to ensure a consistent deformation throughout the sample.

X-ray and neutron diffraction experiments were carried out to investigate the changes in the crystal structure and strain energy of the copper samples subjected to varying levels of deformation. XRD experiments were done at room temperature using a Rigaku SmartLab X-ray diffractometer with a Cu K $\alpha$  radiation source. The diffraction patterns were recorded in a  $2\theta$  range of 20° to 80° with a step size of 0.02°. Neutron diffraction experiments were carried out at the Neutron Scattering Laboratory, GA Siwabessy Nuclear Reactor. The copper samples were loaded into a cylindrical sample holder and placed in the neutron beam. The diffraction patterns were recorded using a position-sensitive detector over a  $2\theta$  range of 20° to 120°. Rietveld refinement using FullProf software was done for analyzing the X-ray and neutron diffraction patterns. The crystal structure of the copper samples was determined from the diffraction patterns, and the lattice parameters were calculated. The dislocation density and strain energy were calculated using the Williamson-Hall method. In this work, the term UFG copper refers to the microstructure refined to crystallite sizes below 100 nm after multiple ECAP passes, as confirmed by the X-ray and neutron diffraction results.

### 3 Results and Discussions

Severe plastic deformation introduced by ECAP leads to the accumulation of a high density of crystal defects, predominantly dislocations, which store elastic energy within the lattice. This stored energy, commonly referred to as strain energy or defect energy, represents the driving force for subsequent recovery and recrystallization phenomena and therefore provides a fundamental metric to quantify the extent of deformation. In ultrafine-grained metals processed by ECAP, diffraction peak broadening observed in X-ray and neutron diffraction patterns arises from both crystallite refinement and lattice distortions associated with dislocation structures. Consequently, diffraction-based line profile analysis offers a reliable pathway to quantitatively evaluate the internal strain state and

the associated stored energy. To establish a quantitative link between diffraction observables and strain energy, we adopted a dislocation-based formulation in which the stored strain energy density is directly related to the dislocation density generated during deformation. The diffraction peak broadening was analyzed using the Williamson–Hall method to separate the contributions of crystallite size and microstrain. The extracted microstrain and crystallite size were subsequently used to estimate the dislocation density, which in turn allowed calculation of the stored strain energy. This framework provides a physics-based interpretation of the diffraction results and enables direct correlation between ECAP deformation level and the evolution of strain energy in ultrafine-grained copper.

The strain energy density stored due to dislocations is expressed as:

$$U = 1/2 G b^2 \rho$$

where  $U$  is the stored strain energy per unit volume,  $G$  is the shear modulus of copper,  $b$  is the Burgers vector, and  $\rho$  is the dislocation density. The dislocation density  $\rho$  is obtained from diffraction peak broadening through the Williamson–Hall approach:

$$\beta \cos\theta = (k \lambda / D) + 4 \varepsilon \sin\theta$$

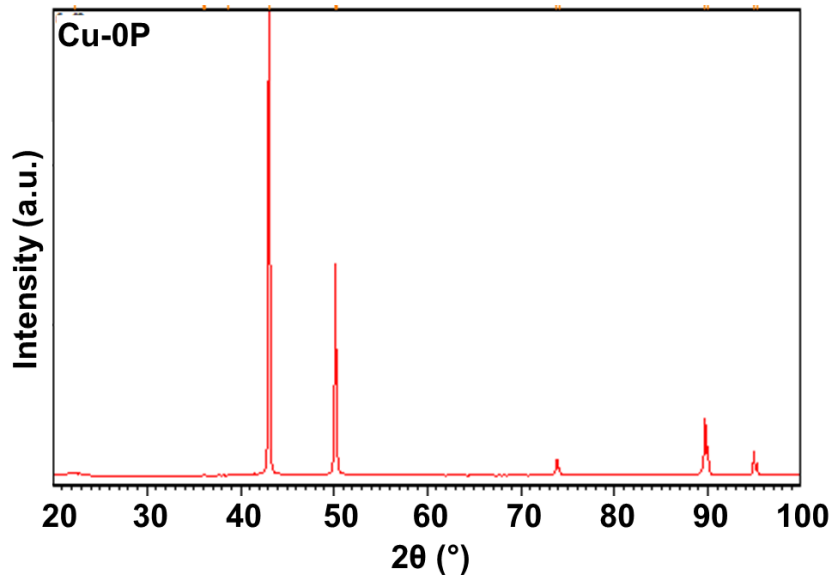
where  $\beta$  is the full width at half maximum (FWHM),  $D$  is the crystallite size,  $\varepsilon$  is the microstrain,  $\theta$  is the Bragg angle, and  $\lambda$  is the radiation wavelength. Following established models for severely deformed FCC metals, the dislocation density is related to microstrain and crystallite size by:

$$\rho \approx (2\sqrt{3} \varepsilon) / (b D)$$

By substituting  $\rho$  into the first equation, the stored strain energy can be quantitatively evaluated directly from X-ray and neutron diffraction data.

### 3.1 X-Ray Diffraction

XRD analyses were used in this work to study the microstructure and texture evolution of copper during the deformation process. The XRD patterns of the UFG copper samples showed the presence of the face-centered cubic (FCC) crystal structure of copper. The diffraction peaks corresponding to the (111), (200), (220), and (311) planes were observed in the XRD patterns, indicating the presence of a single crystal phase, as shown in Figure 1.



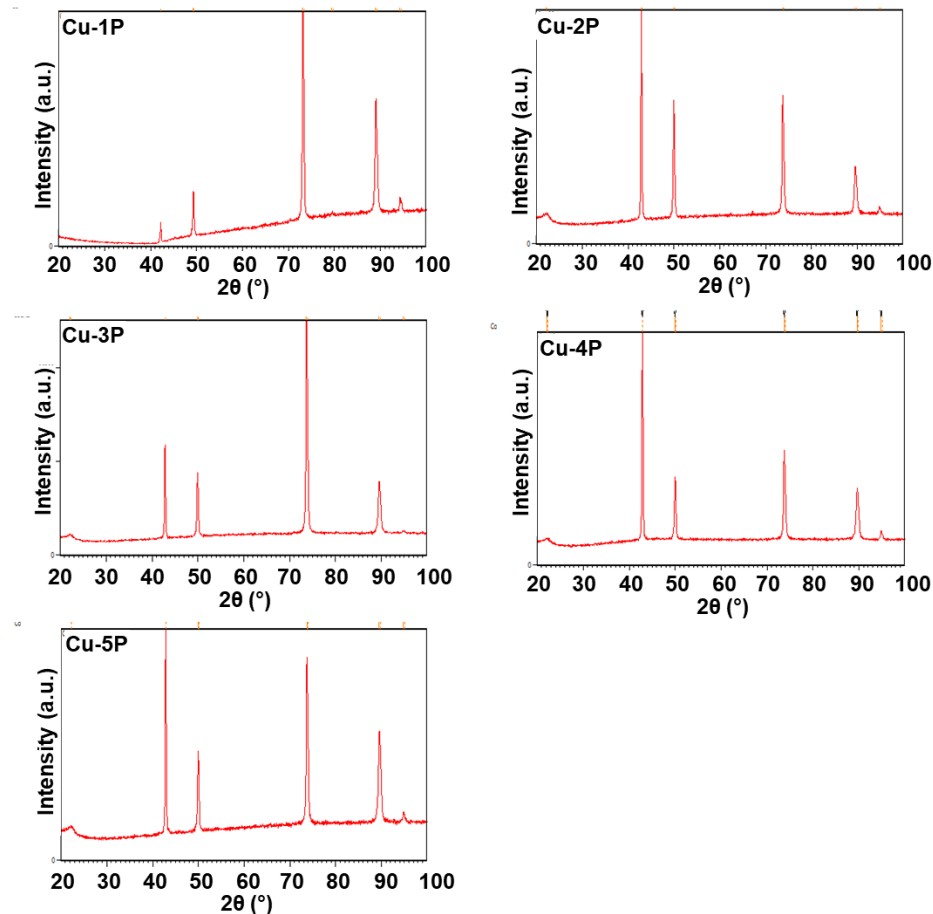
**Figure 1** Ultrafine-grained copper XRD patterns.

The XRD patterns in Figure 2 also show a reduction in the peak intensity and an increase in peak width with an increase in the number of ECAP passes, indicating a reduction in grain size. The reduction in grain size was confirmed by the Scherrer equation, which showed a decrease in grain size from 494 nm to 69 nm with an increase in the number of ECAP passes from 1 to 5. The XRD patterns were analyzed using the Rietveld refinement method to determine the crystal structure and strain energy of the samples. The refinement results showed that the lattice parameter of the FCC crystal structure of copper decreased with an increase in the number of ECAP passes, indicating grain size reduction.

The analysis also showed an increase in dislocation density of the samples with an increase in ECAP pass cycles, confirmed by the broadening of the XRD peaks, as shown in Figure 3. The XRD analysis of the UFG copper samples furthermore revealed the presence of a high density of stacking faults in the samples. The density of stacking faults increased with an increase in the number of ECAP passes.

The XRD analysis of the UFG copper samples also showed an increase in the strain energy of the samples with an increase in the number of ECAP passes. The increase in strain energy was attributed to the stored energy within the material resulting from the deformation through ECAP. The XRD analysis of the UFG copper samples produced by ECAP revealed the presence of the FCC

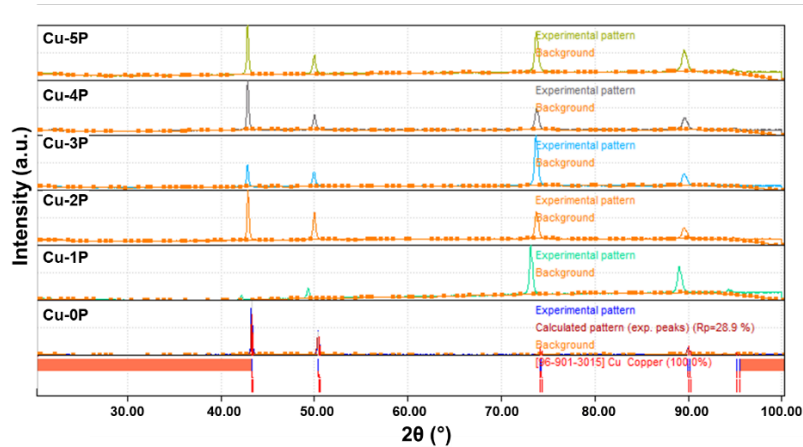
crystal structure of copper. The reduction in grain size and increase in dislocation density observed in the samples contributed to the increase in strain energy observed in the samples. The results of the study can contribute to the development of new materials with improved mechanical properties for various industrial applications. Future studies can build upon the results of this study by investigating the impact of deformation on other metals and alloys using similar techniques.



**Figure 2** Reduction in the peak intensity and the increase in peak width with an increase in the number of ECAP passes.

The progressive broadening of the X-ray diffraction peaks with increasing ECAP passes reflects the continuous rise of lattice microstrain ( $\epsilon$ ), which

originates from the accumulation of dislocations during severe shear deformation. According to the Williamson–Hall analysis, the extracted microstrain and crystallite size enable estimation of the dislocation density ( $\rho$ ), which provides a direct quantitative measure of defect storage. By applying the dislocation-based strain energy formulation introduced in Section 3.1, the stored strain energy density can be determined. Therefore, the observed increase in peak broadening and microstrain in Figures 2 and 3 is not merely indicative of grain refinement but quantitatively corresponds to an increasing stored strain energy within the copper lattice. This establishes a physics-based interpretation linking diffraction peak evolution to internal energy storage rather than a purely descriptive trend.

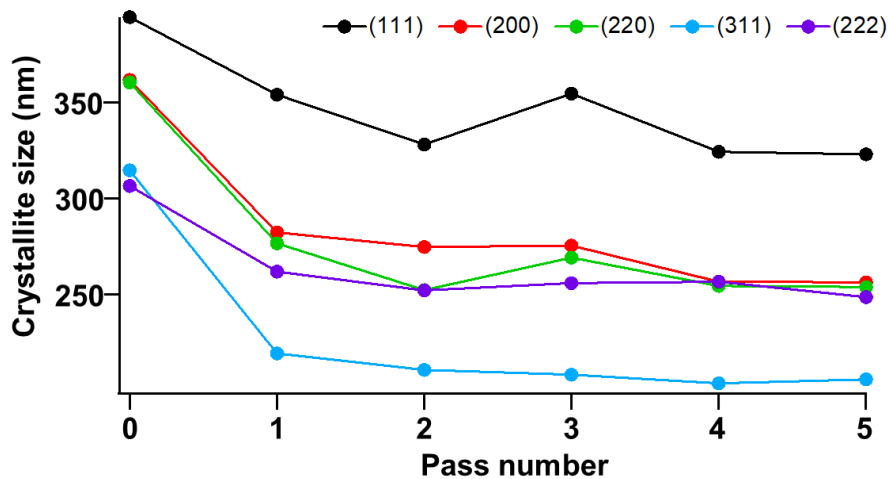


**Figure 3** Increase in the dislocation density of the samples with an increase in the number of ECAP passes.

### 3.2 Neutron Diffraction

The neutron diffraction patterns of the UFG copper from ECAP showed a significant broadening of the diffraction peaks, indicating a higher degree of lattice distortion and microstrain in the material. The broadening of the diffraction peaks was due to the small grain size and high dislocation density, which create a high degree of lattice strain in the material. The microstrain was estimated using the full-width at half-maximum (FWHM) of the diffraction peaks according to the Scherrer equation. The average crystallite size of the UFG copper was calculated using the Debye-Scherrer equation. The results showed that the crystallite size decreased with increasing deformation, indicating that the material underwent severe plastic deformation during ECAP, as shown in Figure 4. The decrease in crystallite size is due to grain refinement that occurs during the deformation process, which leads to the formation of

smaller grains with a higher density of dislocations. It should be emphasized that the crystallite size presented here corresponds to diffraction-coherent subgrain domains rather than full grain size. Under SPD, dislocation walls reorganize into cell/subgrain boundaries, leading to the observed refinement trend, while the increase in microstrain arises from high internal lattice distortions.

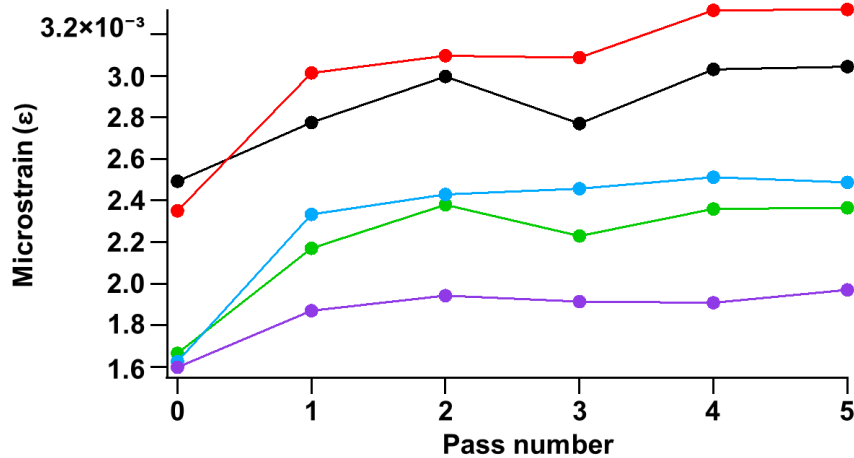


**Figure 4** Decrease in crystallite size as function of ECAP passes.

The lattice parameter of the UFG copper was also determined from the neutron diffraction data. The lattice parameter was found to decrease with increasing deformation, indicating the presence of compressive stresses in the material. This compressive stress is due to the accumulation of dislocations during deformation, which causes the crystal lattice to contract. The microstructural properties of the UFG copper were further analyzed using the Williamson-Hall method. The Williamson-Hall method is a widely used technique for analyzing the microstrain and crystallite size of materials from X-ray or neutron diffraction data. This method separates the contribution of the microstrain and crystallite size to the broadening of the diffraction peaks, allowing for a more accurate determination of these properties.

Compared with XRD, the microstrain values derived from neutron diffraction were consistently higher, indicating that the bulk region experiences more severe dislocation accumulation than the near-surface area. This highlights the unique capability of neutron diffraction to probe the true deformation state throughout the ECAP-processed material thickness. The results obtained from the Williamson-Hall analysis showed that the microstrain increased with

increasing deformation as shown in Figure 5, while the crystallite size decreased. The microstrain was found to be the dominant contributor to the broadening of the diffraction peaks, indicating a high degree of lattice distortion and structural disorder in the material. The neutron diffraction analysis of the UFG copper from ECAP revealed a significant increase in microstrain and a decrease in crystallite size due to the high density of dislocations and grain refinement induced by the deformation process. The technique allowed for a more accurate determination of these microstructural properties, which are important factors in determining the mechanical behavior of the material. The decreasing slope of strain-energy evolution after the 4th ECAP pass indicates that the material approaches a saturation state of stored energy. Although dislocation density continues to increase, the broadening rate of diffraction peaks becomes less pronounced at higher deformation levels, suggesting that a fraction of dislocations reorganize into more stable configurations. Such rearrangements are consistent with the formation of dislocation cell structures and subgrain boundaries produced during severe shear deformation. These recovery-like processes reduce the contribution of newly generated dislocations to stored elastic energy, leading to the observed plateau. This is in agreement with classical SPD behavior reported in ECAP-processed copper, where dynamic recovery partially balances defect accumulation at advanced passes.



**Figure 5** Increase in microstrain as a function of ECAP passes.

The microstrain values obtained from neutron diffraction exhibited systematically higher magnitudes than those derived from X-ray diffraction, indicating a larger dislocation density in the bulk interior of the ECAP-processed specimens. By substituting the neutron-derived microstrain into the

dislocation density relation and subsequently into the strain energy, the stored strain energy evaluated from neutron diffraction was found to be higher than that obtained from X-ray measurements. This difference highlights the capability of neutron diffraction to probe the true internal deformation state throughout the sample thickness, whereas X-ray diffraction predominantly reflects near-surface conditions. Consequently, the neutron diffraction results provide a more representative quantification of bulk strain energy accumulation during ECAP deformation.

### 3.3 Texture

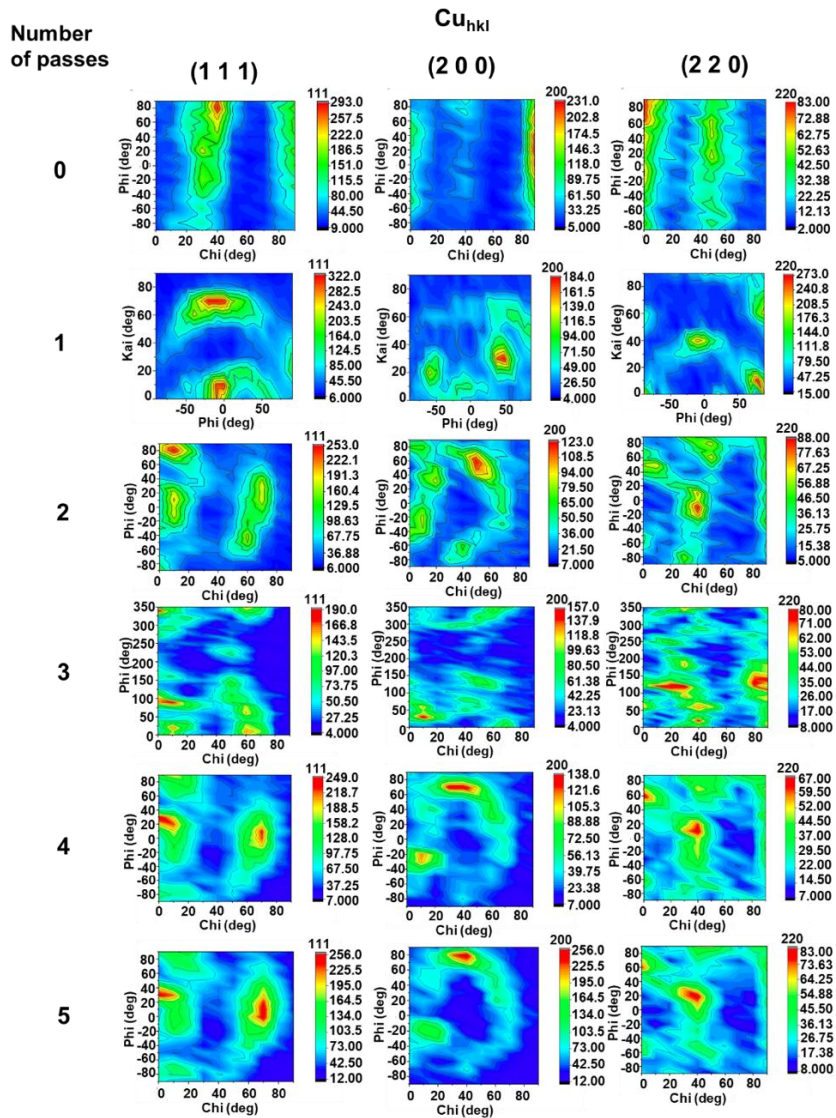
Texture analysis can be used to study the preferred orientation of the crystallites in a polycrystalline material. In this study, the texture of the UFG copper samples produced by ECAP was analyzed using neutron scattering techniques. The neutron diffraction patterns of the UFG copper samples revealed a weak texture in the samples, indicating that the samples were mostly isotropic. However, a slight texture was observed in the (200) plane of the FCC crystal structure of copper, with a maximum intensity along the transverse direction of the samples.

The texture of the samples was analyzed using the orientation distribution function (ODF) and the inverse pole figure (IPF) methods (Figure 6). The ODF analysis revealed a random texture in the samples with no preferred orientation of the crystallites. The IPF analysis showed a weak texture in the (200) plane of the FCC crystal structure of copper, with a maximum intensity along the transverse direction of the samples. The texture analysis of the UFG copper samples also revealed a change in texture with an increase in the number of ECAP passes. The texture of the samples was observed to become more isotropic with an increase in the number of ECAP passes.

The texture analysis also revealed a decrease in the intensity of the (200) plane with an increase in the number of ECAP passes. The decrease in the intensity of the (200) plane was attributed to the reduction in the grain size of the samples with an increase in the number of ECAP passes. In addition to the ODF and IPF analysis, the texture of the samples was also analyzed using pole figure analysis. The pole figure analysis revealed a slight texture in the (200) plane of the FCC crystal structure of copper, with a maximum intensity along the transverse direction of the samples.

The pole figure analysis also revealed a change in texture with an increase in the number of ECAP passes. The texture of the samples was observed to become more isotropic with an increase in the number of ECAP passes. The texture

analysis of the UFG copper samples produced by ECAP revealed a weak texture in the samples, indicating that the samples were mostly isotropic. However, a slight texture was observed in the (200) plane of the FCC crystal structure of copper, with a maximum intensity along the transverse direction of the samples. The texture of the samples was observed to become more isotropic with an increase in the number of ECAP passes.



**Figure 6** Texture analysis of the UFG copper samples produced by ECAP.

The results of the study can contribute to the development of new materials with improved mechanical properties for various industrial applications. Future studies can build upon the results of this study by investigating the impact of deformation on the texture of other metals and alloys using similar techniques. The preservation of a weak (200) shear-related texture component may facilitate the activation of multiple slip systems in bulk copper during ECAP, which is beneficial for maintaining a reasonable level of ductility despite substantial strengthening. Such texture-supported deformation mechanisms are typically undetectable from surface-dominant XRD but are captured by bulk-sensitive neutron scattering. Such microstructural evolution is highly advantageous for industrial copper applications that demand concurrent strength and high conductivity performance, such as high-current electrical conductors, compact heat-conducting components, and wear-resistant electromechanical parts. The weak (200) texture retained during ECAP can promote multiple slip activation, helping copper maintain the acceptable ductility required for forming and service reliability.

It is noteworthy that the rate of strain energy increase becomes progressively smaller after the fourth and fifth ECAP passes, indicating the onset of a saturation regime in stored energy. At advanced stages of severe plastic deformation, further dislocation generation is partially balanced by dislocation rearrangement into low-energy configurations such as dislocation cell structures and subgrain boundaries. This dynamic recovery-like process reduces the net contribution of newly generated dislocations to stored elastic energy, leading to the observed plateau in strain energy evolution. Such stored-energy saturation behavior is consistent with classical severe plastic deformation theory, where a steady-state balance between defect accumulation and recovery mechanisms is achieved at high imposed strains. The present neutron diffraction results therefore provide direct experimental evidence of strain energy saturation in ultrafine-grained copper processed by ECAP.

#### **4 Conclusion**

This study quantitatively established the relationship between ECAP deformation and the evolution of stored strain energy in copper using complementary X-ray and neutron diffraction methods. Increasing ECAP passes caused progressive crystallite refinement and a significant rise in dislocation density and microstrain, leading to continuously increasing strain energy. Importantly, strain energy quantitatively evaluated using the dislocation-based model  $U = \frac{1}{2}Gb^2\rho$  confirmed stored-energy saturation after 4–5 ECAP passes that recovery-like dislocation rearrangement mechanisms (cell/subgrain boundary formation) start to balance further defect storage. Neutron diffraction revealed stronger bulk lattice distortion than detected by XRD, confirming its

essential role in capturing the true deformation state throughout the material thickness. These results provide new insight into the stored-energy saturation phenomena in severely deformed copper and demonstrate that ND–XRD combination enables reliable quantification of defect accumulation mechanisms during SPD. The findings support ECAP as a promising pathway for developing UFG copper with improved strength while maintaining a reasonable ductility balance.

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