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ORIGINAL ARTICLE

Texture Characterization of The Copper Produced by ECAP Process Using Neutron Diffraction Technique

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ABSTRACT – Texture and hardness characterization have been carried out on market copper samples that have gone through the equal channel angular pressing (ECAP) process. Neutron diffraction technique had been used for obtaining an average crystalline texture in a particular volume non destructively to the sample. The ECAP process is carried out once (1 pass) with some parts that have been plastically deformed and some parts that have not been deformed. Crystalline texture and hardness were observed in the deformed and non-deformed parts. Initial characterization was carried out by X-ray diffraction (XRD) followed by measurement of crystal texture using the neutron diffraction technique, while hardness was tested using the Vickers method. Rod-shaped sample with a diameter of 10 mm. Texture observations were performed at the center of the sample with a neutron beam limiting slit of 5x5 mm^2. There was an increase in hardness in the deformed position compared to the undeformed one. The texture that occurs is in the form of fibers with different directions and indexes, sequentially as follows: position 1, [111] of 4.96 m.r.d., position 2, -[111] of 1.86 m.r.d. and position 3 [010] of 2.44 m.r.d. , position 4 orientation is distributed on [011], [013], [115], [235] fibers with a texture index range of 1.07–1.33 m.r.d.

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INTRODUCTION

Copper and its alloys have high thermal and electrical conductivity. Its high corrosion resistance and ease of work make it attractive in manufacturing applications such as the automobile, rail, electrical and electronic industries.

The severe plastic deformation (SPD) technique was introduced decades ago to create sub-micron grain sizes which known as ultrafine-grained (UFG) [1]–[3]. Several types of SPD that are known and well known include high-pressure torsion (HPT), twist extrusion (TE), and Accumulative roll bonding (ARB). The ECAP technique is an effective tool for producing ultra fine grained materials and is relatively easier to apply to efficiently render UFGs than the others. The ECAP technique can be applied to various workpiece shapes, but is usually cylindrical or billet.

The ECAP technique is carried out by pressing the workpiece throught a tunnel which making certain angle between input line to the output line. These two channels are of the same shape and size as the channel into which the workpiece enters. This is where the origin of the naming of this equal channel comes from. These two channels intersect and form the angle of curvature. To get the desired grain size, pressing is conducted repeatedly until the desired grain size is obtained.

Texture characterization of the ECAP results mainly were carried out using the electron backscattering diffraction method, EBSD. Where to obtain a texture distribution pattern with EBSD method, the sample must be slices considering this method only provides a pattern of texture on the surface of the material. Therefore, this method can be classified as a destructive method [4]–[6].

Neutron diffraction can be used to characterize the crystalline texture of materials non-destructively. This can be done because neutrons have great penetrating power in almost all materials. Measurements by neutron diffraction are possible at volumes measuring several tens of mm^3 or even cm^3. This is in stark contrast to area measurements performed with EBSD which are generally only a few mm^2 or even less. Thus the results of the evaluation of the crystal texture obtained by neutron diffraction will be much more representative of the actual conditions of the observed material.

Indonesia has a neutron diffractometer that can be used to characterize the crystalline texture of materials, namely DN1 (Neutron diffractometer for residual stress measurement) and DN2 (Four-circle Neutron diffractometer). The DN1 is connected to the neutron beam tube S6 in the GA Siwabessy multipurpose nuclear reactor (RSG-GAS). In addition, the DN1 is also equipped with additional equipment that allows the measurement of residual stresses using neutron diffraction. DN1 has a incident slit of 5x5 mm^2, while DN2 has a incident slit of 20x20 mm^2 with wavelengths of 1.75 Å and 0.96 Å, respectively [7].

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EXPERIMENTAL METHOD

Materials and Instruments

The samples used in this work are commercially available in the market with a diameter and length of 10 mm and 70 mm, respectively. The schematic of the dies and the resulting samples are shown in Figure 1. The universal press machine made by Shimadzu model AG-X/R has been used in this works.

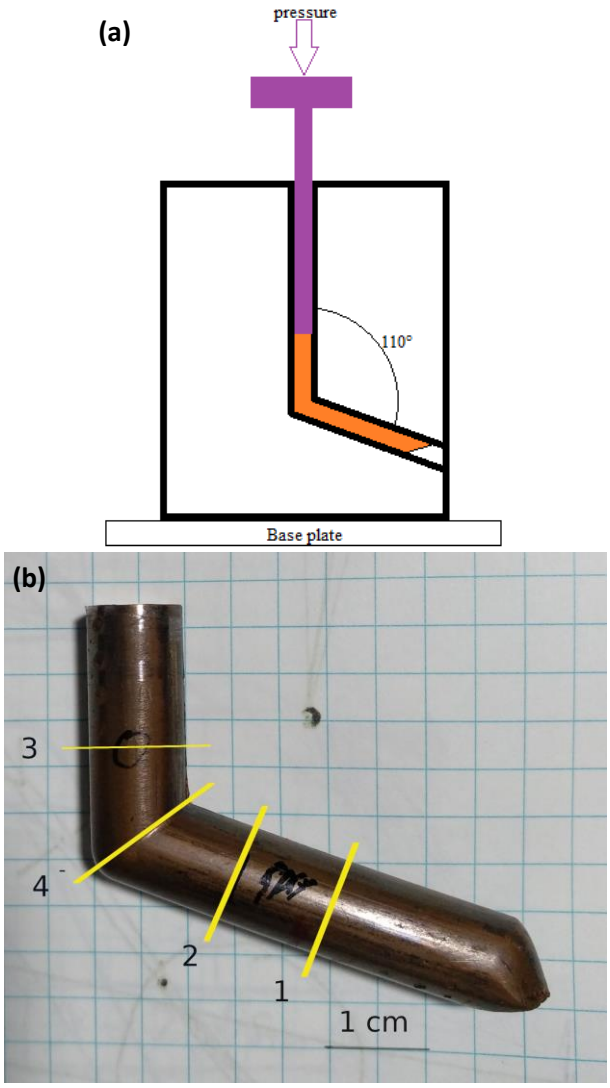


Figure 1. (a) Schematic of the dies and (b) the resulting sample and measurement positions 1–4

Crystal texture measurements were carried out using a neutron diffractometer, DN1, made by Kuroda Co. Japan. Texture measurements were carried out at 4 positions, namely before the bend (3), at the turn (4) and 2 points after the turn (1&2). The slit size used for measurement is 5x5 mm^2. The measurement position is shown in Figure 1(b).

X-ray diffraction (XRD) measurements were carried out using a Shimadzu XD-610 XRD by using Cu-Kα as a target with a wavelength of 1.54 Å. To limit the beam hitting the sample, a slit with a diameter of 1 mm was used. In addition, to limit the irradiated area in the sample, masking using Pb sheets with a thickness of 0.5 mm was used. The PG monochromator is installed between the sample and the detector. A parallel collimator with 1° divergence is installed between the X-ray source and the sample [8].

Crystalline texture measurements were performed at DN1. The displacement of the measurement positions 1–4 is done manually by removing the sample and reattaching it so that the measurement position is precisely in the neutron beam. The installation of the sample on the sample table and the measurement axes are shown in Figure 2 [9].

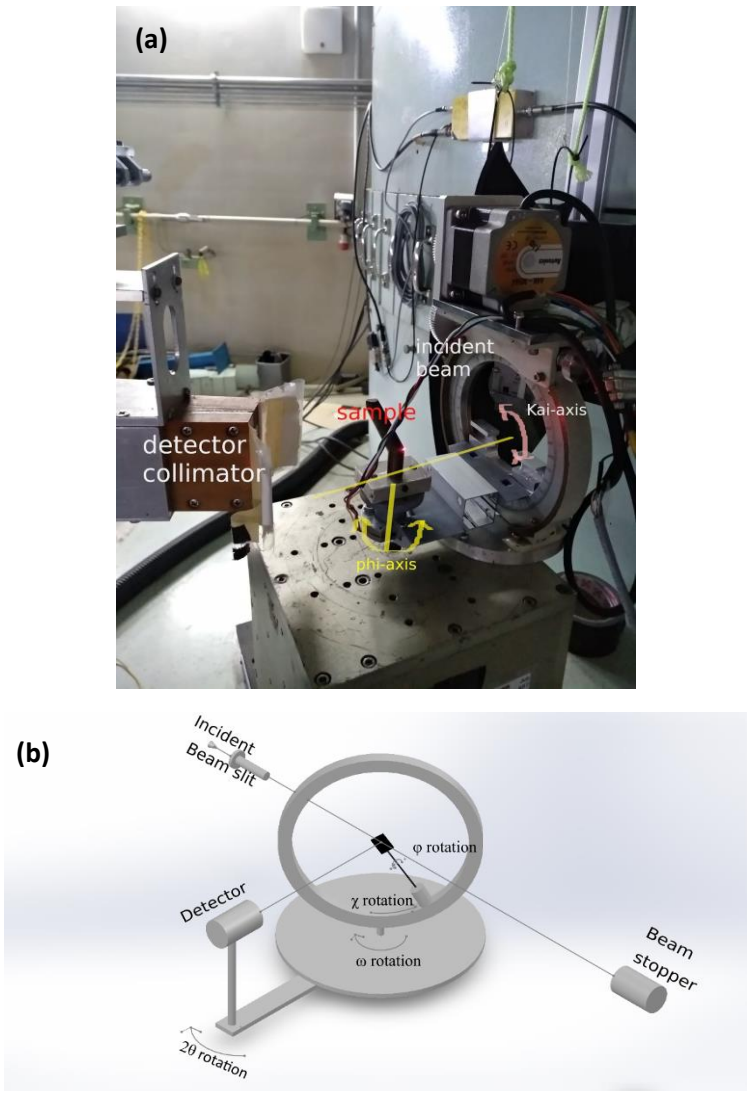


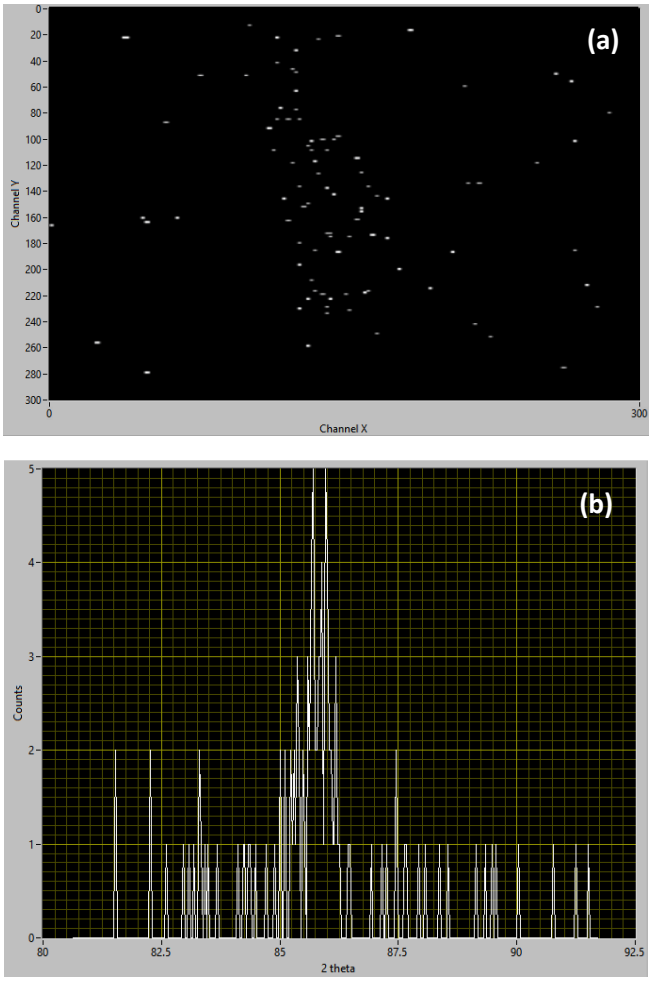
Figure 2. (a) The position of the sample installation on the DN1 diffractometer table and (b) the main axes of the sample texture measurement

Method and Procedure

Measurements were made on the crystal plane (111), (200) and (220) with the positions of the diffraction angles (2θ) of 49.3°, 57.5°, and 86°, respectively. Scan measurements of φ-χ with φ = 0° - 90° steps of 10° and χ = 0° - 180° steps of 10° for each crystal plane. Measurements were carried out using the fix-count mode with a count of $4.8e^5$ neutrons or in the range of 2 minutes for each particular φ-χ position.

The distribution of neutrons detected by the DN1 detector at each particular φ-χ position is shown in Figure 3(a). Then the data is converted into a 2θ vs intensity curve as shown in Figure 3(b). Then the curve of 2θ vs intensity is integrated to get the total intensity (integrated intensity) for a particular φ-χ position.

Crystal texture calculations were carried out using Labosoft's LaboTex software version 3.0. Hardness measurements were carried out using a hardness tester Microhardness tester model HV 1000. The indenter used was in the form of a diamond prism with an angle of 136° with a weight of 0.1 kg. Measurements were carried out 5 times for each position.



N total	Mean	Std. dev	Sum	Min	Median	Max
70	1.04	1.35	73	0	0.5	5

Figure 3. (a) The raw data, distribution of neutrons detected by the detector with 300x300 counter channels and (b) the results of converting raw data into a 2θ vs intensity curve and its integrated intensity

RESULT AND DISCUSSION

The results of XRD measurements at various positions as in Figure 1 are shown in Figure 4. Figure 4 shows that the copper sample used is crystalline with the observed crystal planes being (111), (200), (220) and (311). Before and after deformation, the crystals remained intact in their shape and size, which were indicated by the same pattern and the position of the peaks did not change.

From the intensity data on the results of the XRD pattern, it can be seen that certain fields (hkl) have different intensities for different positions. This indicates a non-uniform texture at the measurement positions. However, due to the limited penetrating power of X-rays only a few microns below the sample surface, these data only indicate the presence of surface texture in the sample. To find out the texture in the material, a neutron beam is needed to characterize it without damaging it.

The presence of other phases in the copper sample used can also be clearly observed in Figure 4, as shown in the unindexed peaks. However, the existence of these phases will not be discussed in this report.

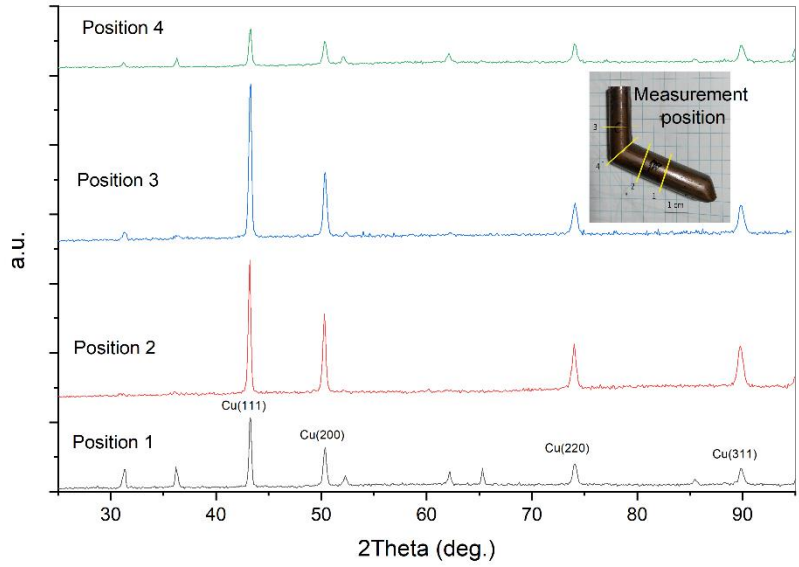


Figure 4. X-ray diffraction pattern of ECAP copper samples at various posi-tions. X-rays use a Cu target with a wavelength of 1.54 Å

The results of microhardness measurements are shown in Figure 5.

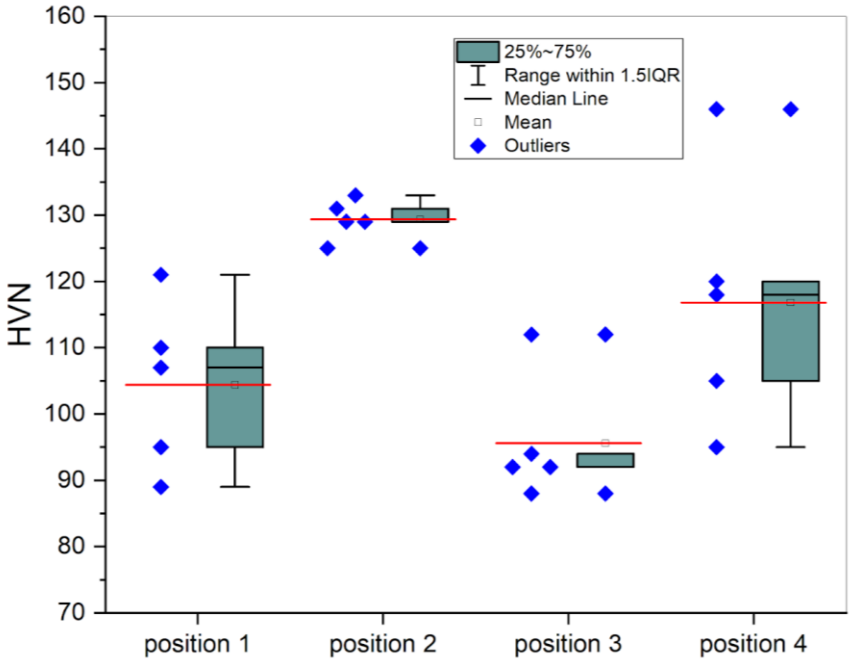
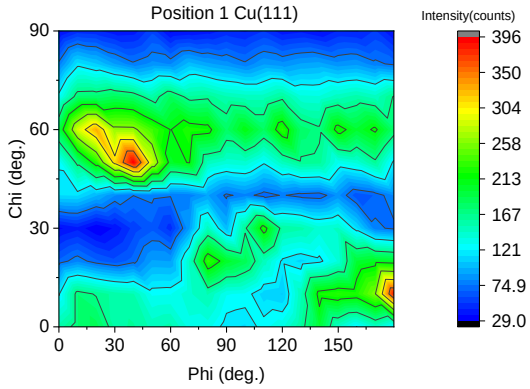
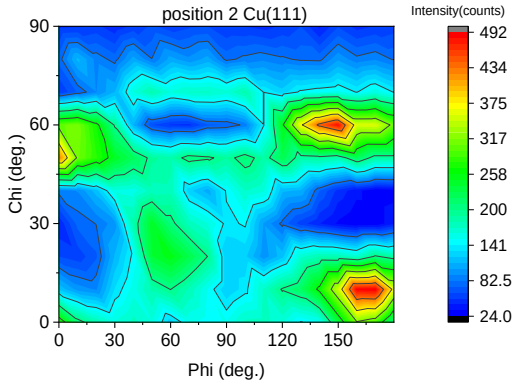
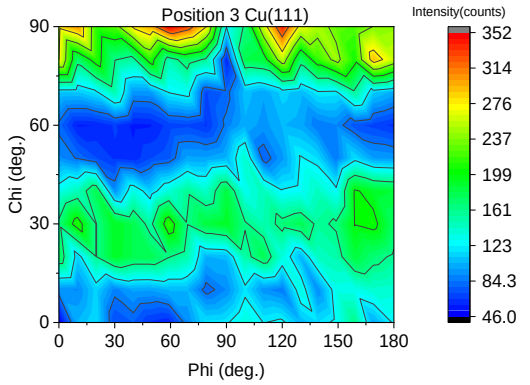
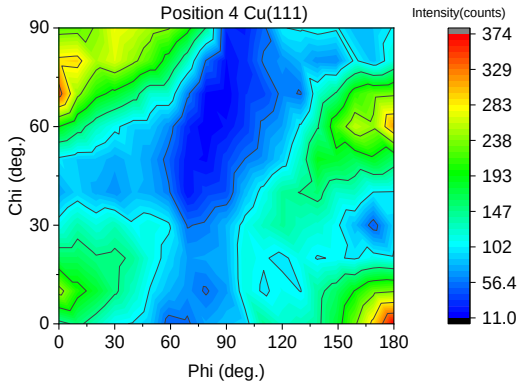


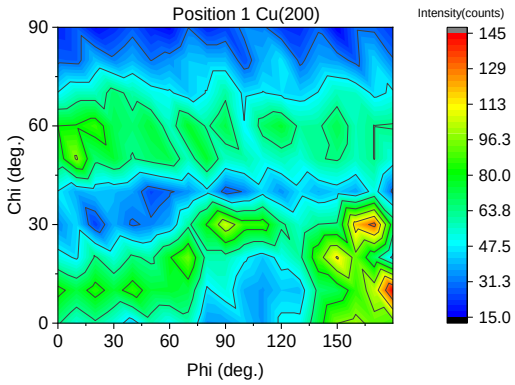
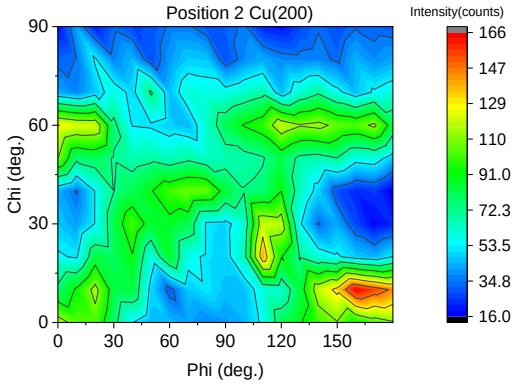
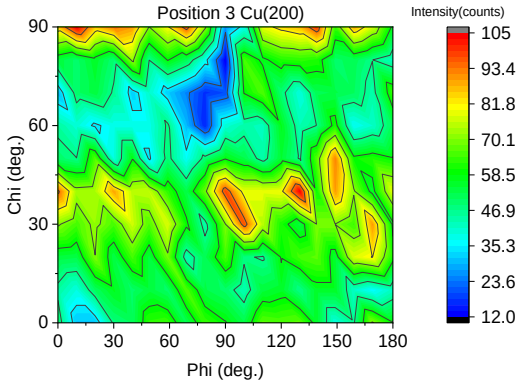
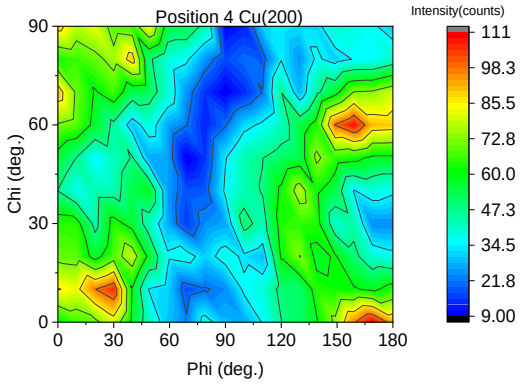
Figure 5. Vickers hardness measurement results at various measurement positions (each measurement point was carried out 5 times)

The hardness test results at position 3 showed the lowest value, 95 HVN. Hardness increases to 115 HVN at position 3 where the sample begins to enter the granulation region. The data at position 3 has a large variance due to the transition before and after the deformation. The highest hardness of 130 HVN was measured at position 2 and slightly decreased to 105 HVN at position 1. The hardness that occurred at position 2 was still within the range of the hardness at position 4, which indicates that the hardness after the sample underwent deformation persists.

The results of texture measurements at each measurement point are shown in Table 1.

Table 1. Raw data of -χ measurement results at measurement positions 1-4 for the (111), (200), (220) planes

Plane (hkl)	Position	Pole figure
(111)	1	
	2	
	3	
	4	

(200)	1	 Position 1 Cu(200) Intensity(counts) 145 129 113 96.3 80.0 63.8 47.5 31.3 15.0 Chi (deg.) 0 30 60 90 0 30 60 90 120 150 Phi (deg.)
	2	 Position 2 Cu(200) Intensity(counts) 166 147 129 110 91.0 72.3 53.5 34.8 16.0 Chi (deg.) 0 30 60 90 0 30 60 90 120 150 Phi (deg.)
	3	 Position 3 Cu(200) Intensity(counts) 105 93.4 81.8 70.1 58.5 46.9 35.3 23.6 12.0 Chi (deg.) 0 30 60 90 0 30 60 90 120 150 180 Phi (deg.)
	4	 Position 4 Cu(200) Intensity(counts) 111 98.3 85.5 72.8 60.0 47.3 34.5 21.8 9.00 Chi (deg.) 0 30 60 90 0 30 60 90 120 150 180 Phi (deg.)

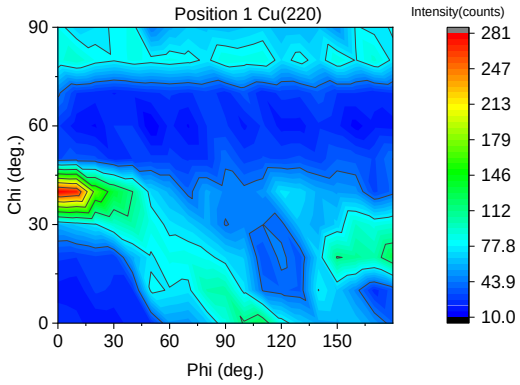
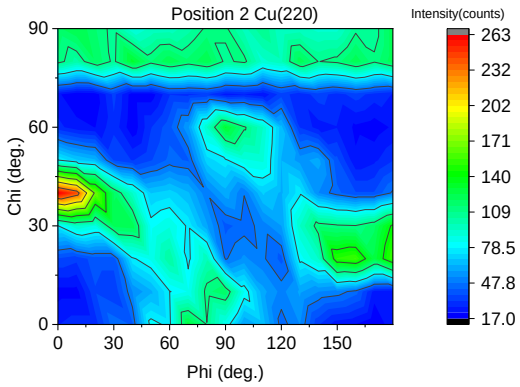
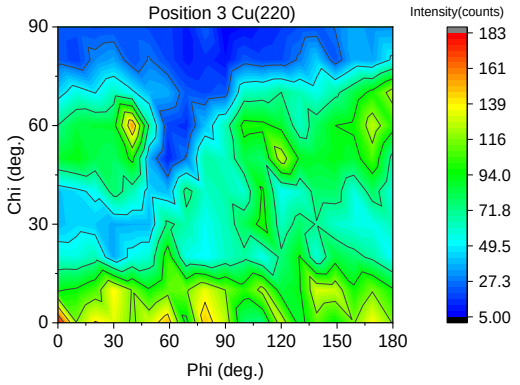
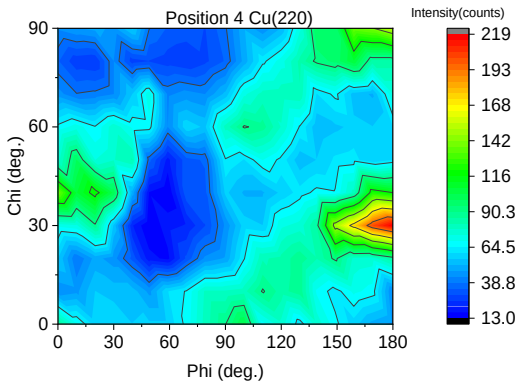
(220)	1	
	2	
	3	
	4	

Table 1 shows experimental data of (χ-ϕ) scan. It uses to investigate characteristic poles in the pole figures. From (χ-ϕ) scan, at positions 1,2 and 3 some poles appear at a particular position along the transverse direction. However, at position 4 (bending position), poles appear in the radial direction. Poles at a particular (χ-ϕ) position show a crystallite orientation (texture). The (χ-ϕ) data as experimental data convert into a calculated pole figure (CPF). Based on the Labotex data format, the CPF is calculated using the orientation distribution function (ODF) to obtain a recalculated pole figure (RPF). Furthermore, the RPF data were analyzed to determine texture characteristics such as type, direction, and strength.

Figure 6 is the Recalculated Pole Figure (RPF) (111), (200), and (220) which shows <hkl>fiber crystal orientation. Analyzing the pole figures generally shows similar fiber orientations at positions 1 and 2. Figures 6(a) and 6(b) show the crystal orientation of the [111] fiber at positions 1 and 2, respectively. Figure 6(c) shows the orientation of [110] at position 3. Figure 6(d) shows [013] fiber at position 4 [10], [11].

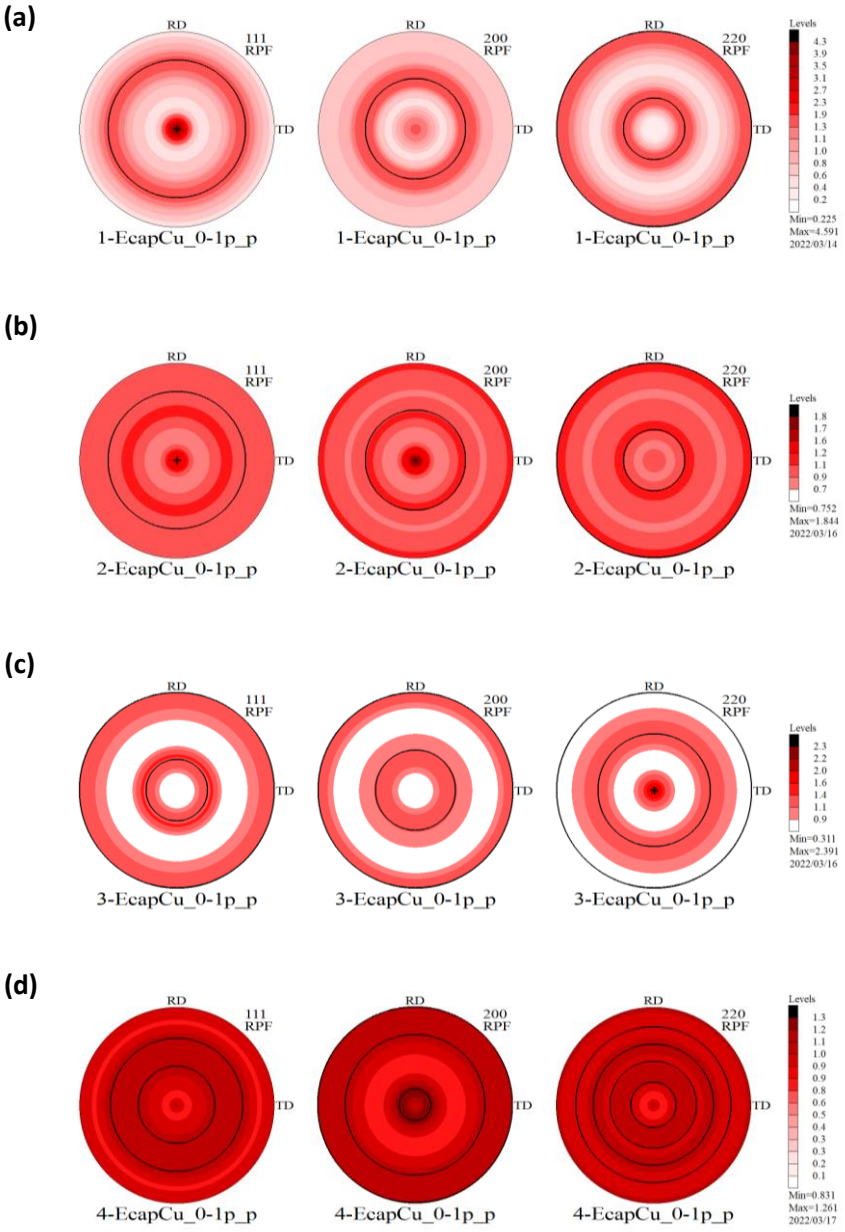


Figure 6. Recalculated Pole Figure (RPF) (111), (200), and (220) at position (a) 1, (b) 2, (c) 3, (d) 4 for ECAP copper

Figure 7 shows the orientation distribution function (ODF) at four positions. At position 1 (horizontal channel position), the projection φ1 in Euler space (φ1,Φ,φ2) shows that the crystal orientation (texture) is [111] fiber with a texture index is 4.96 m.r.d. Due to the pressing process, position 1 has the largest stress, grain at position 1 becomes denser than position 2, so that deformation occurs, where most crystallite orientation to <111> fiber. The compression process at position 1 causes a change in the direction of deformation at position 2 [12]–[13]. So that there is a change in crystal orientation from [111] fiber at position 1 distributes to [111] fiber, [018] fiber, and [001] fiber at position 2 with texture indexes are 1.80, 1.86, and 1.78 m.r.d, respectively. This is similar to the explanation of A. I. Alateyah *et al.* where the strong texture was completely transformed after the first ECAP pass towards the simple shear texture with an intensity of about 4 times random [14].

Furthermore, at position 4, (bending position). A compression process forms an angle of 110°. It causes the grain size to be larger than of it at position 2 and the other positions, At the bending position, the crystal orientation appears at [011] fiber, [013] fiber. [115] fiber and [352] fiber with a texture index of 1.07, 1.30, 1.31, and 1.33 m.r.d, respectively [15]–[17].

Position 3 is the last compression process.Due to the compression process, the grain size at position 3 is larger than that at position 1. It causes the texture towards the [101] fiber with a texture index of 2.44 m.r.d.[18].

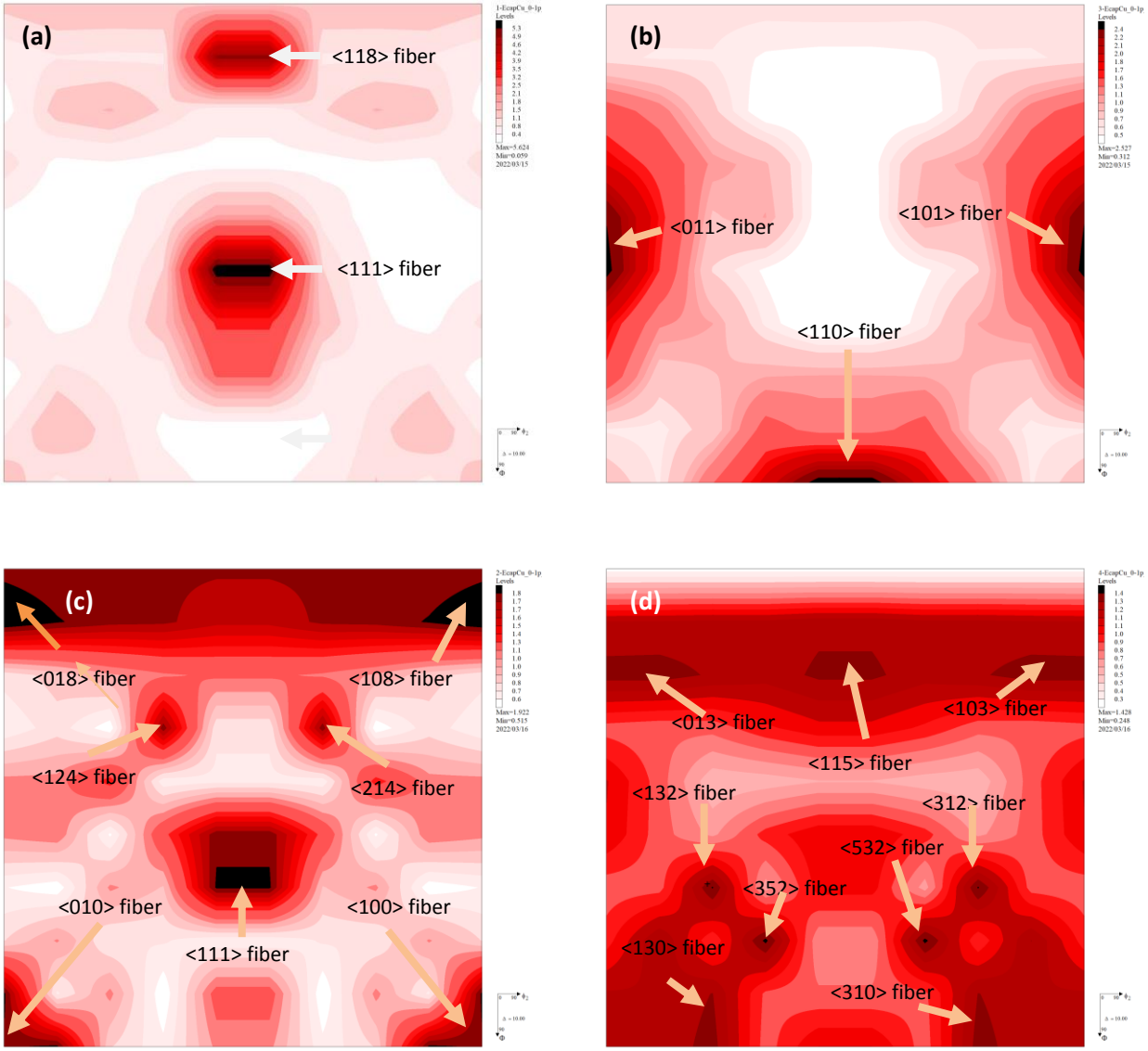


Figure 7. Orientation Distribution Function (ODF) of Cu ECAP at positions a. 1, b.2, c. 3 d. 4 with φ1 projection in Euler space (φ1,Φ, φ2)

CONCLUSION

The hardness of the part that has not been deformed (position 3) is lower than that of the part that has been deformed (position 1,2) and that is being deformed (position 4).

There has been deformation in different directions due to the compression process. It occurs because of the stress and strain on the material. In position 1 is the initial compression position where the material experiences the highest stress, the strongest texture fiber formed in the direction of [111] fiber with a texture index equal to 4.96 m.r.d. At position 2, texture distributed to [111] fiber, [010] fiber, [018] fiber, and [124] fiber with almost the same texture index, it is between 1.78-1.86 m.r.d. At position 3, there is a change in the crystal orientation direction to [010] fiber with a texture index of 2.44 m.r.d.

At position 4 (bending position), the crystal orientation distributes to [011] fiber, [013] fiber. [115] fiber and [235] fiber. The texture indexes ranged from 1.07-1.33 m.r.d. The low texture indexes at position 4 indicate that grains size at position 4 is more prominent than grain size at position 2 or the other positions.

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