VETOR

Hardness and Texture of Electrolytic Copper Processed by ECAP at Cold and Warm Temperatures☆

Dureza e Textura do Cobre Eletrolítico Processado por ECAP em Temperatura Ambiente e a Morno

Hiron Akira Yamada Magalhaes[1,†], Talita Gama de Souza[1], Rodrigo Felix de Araujo Cardoso[2], Bruno Rangel da Silva[2], Luiz Paulo Brandao[1]

[1]*Materials Engineering Section, Military Institute of Engineering, Rio de Janeiro, Brazil*
[2]*Instrumentation and Mechanical Technology Laboratory, Brazilian Center for Physics Research, Rio de Janeiro, Brazil*

[†]**Corresponding author:** hiron.akira@gmail.com

Abstract

Among several severe plastic deformation (SPD) methods, the Equal Channel Angular Pressing (ECAP) process is one of the most popular. This process's main characteristic is producing materials with ultra-fine or nanometric grains. Due to these microstructural changes, it is possible to improve mechanical properties such as strength and ductility. In this perspective, the aim of the present work was to evaluate the variations of the mechanical hardness property associated with microstructural and textural changes of pure copper as a function of its processing by SPD via ECAP. For this, the material was submitted to four passes through routes A (the sample is repetitively pressed without any rotation between each pass) and Bc (the sample is rotated in the same sense by 90° between each pass) at cold and warm temperatures. Through the obtained result, it was verified that the ambient temperature of the Bc route was the one that promoted greater homogeneity in the microstructure and weakening of the texture after the fourth pass. On the other hand, warm processing of copper by ECAP promoted a softening of the samples and a homogeneous distribution of hardness in both routes.

Keywords

Copper • ECAP • Crystallographic Texture • Dislocation Density • Microhardness

Resumo

Dentre os diversos métodos de deformação plástica severa (DPS), o processo de extrusão por canal equiangular (ECAP – Equal Channel Angular Pressing) é um dos mais populares. Este processo tem como principal característica produzir materiais com grãos ultrafinos ou até mesmo grãos nanométricos. Devido a estas mudanças microestruturais é possível gerar melhoria em algumas propriedades mecânicas como a resistência e ductilidade. Nesta perspectiva, pretendeu-se no presente trabalho avaliar as variações da propriedade mecânica dureza associada às alterações microestruturais e texturais do cobre puro, em função das variações de parâmetros

☆ This article is an extended version of the work presented at the Joint XXIV ENMC National Meeting on Computational Modelling and XII ECTM Meeting on Science and Technology of Materials, held in webinar mode, from October 13th to 15th, 2021.

do processo por DPS via ECAP. Para isto o material foi submetido a quatro passes através das rotas A (a amostra é pressionada repetidamente sem qualquer rotação entre cada passagem) e Bc (a amostra é rotacionada no mesmo sentido em 90° entre cada passagem) em temperatura ambiente e a morno. Por meio do resultado obtido verificou-se que na temperatura ambiente a rota Bc foi a que promoveu maior homogeneidade na microestrutura e enfraquecimento da textura após o quarto passe. O processamento do cobre por ECAP a morno promoveu um amolecimento das amostras e uma distribuição homogênea da dureza em ambas a rotas.

Palavras-chave

Cobre • ECAP • Textura Cristalográfica • Densidade de Discordâncias • Microdureza

1 Introduction

Several techniques are proposed to modify a material's mechanical properties, one of which is through the severe plastic deformation (SPD) methods, which have wide application in metallic materials. Through this process, a microstructure with ultra-fine and nanostructured grains is obtained. These microstructural modifications result in a material with different properties from those presented before being processed and with coarse grains, such as a higher hardness value and, consequently, a higher tensile strength limit and no significant loss of ductility [1].

Processing by SPD is defined as any method of metallic forming that imposes a high deformation. The equiangular channel extrusion process (ECAP) is one of the SPD processes whose main characteristic is that the shaped part does not change its shape. Another interesting feature of this process is the ability to produce exceptional grain refinement to sizes that would not be achievable by conventional thermomechanical treatments [2]. In this way, SPD methods are used to convert coarse-grained metals and alloys to ultra-fine-grained materials. Through this process, these materials have improved mechanical and physical properties, such as increased mechanical strength, which are of great interest for commercial purposes.

Among the various types of severe plastic deformation, the equiangular channel extrusion process (ECAP) is considered to be one of the most used, not only because it requires little compression load but also because of its simple geometry tool that can be easily assembled in a laboratory to carry out experimental tests [3].

By processing the material by ECAP, high magnitudes of plastic deformation are imposed on the material, which results in a refinement of the microstructure and, consequently, a change in mechanical properties. These modifications happen due to the level of severe induced plastic deformation and the number of passes, the route and the processing temperature [4].

This severe plastic deformation method was developed by Professor Segal in 1977 in Russia and later by Valiev et al. in 1991 [2] with a more scientific approach, analyzing the correlation of nanostructured materials with severe plastic deformation methods.

In this present research work, the material was chosen to study of microstructural changes and, consequently, mechanical behavior in ECAP processing was pure copper, as it has a medium stacking defect energy, good formability, and low cost. In this sense, through the SPD method via equiangular channel extrusion, we sought to study of the microstructural evolution of pure copper and hardness values as a function of the sequence of the number of passes in two different routes and at cold and warm temperatures.

2 Material and Method

The material for this research consisted of round bars with a diameter of 9.56 mm of electrolytic copper (99.998% Cu and 0.002% O) supplied by the company Paranapanema. Copper bars were supplied after continuous extrusion, followed by drawing by automatic machines. The circular section bars received were sectioned into 80 mm long test specimens to prepare the samples. The samples were classified according to the number of passes, chosen route (Route A or Bc) and cold and warm temperature condition, for example, processed via ECAP with 2 cold passes via route A and processed via ECAP with 4 passes via warm route Bc.

To carry out the cold ECAP process, this study used a die designed and developed for this purpose. It was made with H13 tool steel, its channels were nitrided to ensure greater wear resistance. This die has geometric parameters of the channel $\Phi = 120°$, $\psi = 23°$, $R = 4$ and $r = 2$ and a diameter of 10 mm, resulting in an equivalent true deformation in each pass of 0.67. The samples were processed by routes A (all passes of the specimen always with the same orientation) and Route Bc (rotated 90° counterclockwise with each pass) and 4 passes in each route.

To carry out the warm ECAP tests, the same die and a homemade furnace with a constructive principle using halogen lamps were used. Samples were processed at approximately 350 °C (homologous temperature of 0.48), which was chosen based on studies in the literature [5, 6]. During the ECAP process, a copper and molybdenum disulfide (MoS_2) based grease was used under both temperature conditions. Furthermore, the samples were machined and sanded to a 2500 mesh sandpaper after each pass.

The characterizations were carried out by analyzes obtained through the X-ray diffraction (XRD), microstructural and Vickers microhardness techniques. To perform the Vickers microhardness test, a LECO microhardness tester was used with a load of 10 kgf for a period of 15 s. Fifteen indentations were performed, which were divided into three microhardness profiles with five measurements near the edges and middle of the specimen.

A scanning electron microscope (SEM) Quanta FEG 250 was used for the microstructural analyses. The analyzes were performed after the samples were sanded, mechanically polished with diamond paste and finally chemically etched with a solution containing 5 parts of nitric acid PA (HNO_3), 5 parts of acetic acid PA (CH_3CO_2H) and 1 part of phosphoric acid PA (H_3PO_3) [7].

The XRD technique was used to obtain the dislocation density and crystallographic texture values. Dislocation density values were determined using the Convolutional Multiple Whole Profile (CMWP) software from the analysis of the diffractograms of each sample. The diffractograms were obtained using the PANalytical X'Pert Pro MRD diffractometer in the focus point configuration, voltage, and current of 40 kV and 40 mA, respectively. For the texture analysis, initially, the pole figures were obtained from measurements in the planes (111), (200) and (220). Then, the crystallographic texture analysis results of were observed using the texture severity factor (TSF) and the crystalline orientation distribution functions (ODF) obtained using the PopLa software. The TSF factor is a parameter proposed by Kallend & Davies [8] who determines a measure of the standard deviation of the crystalline orientation distribuction function (ODF) compared to an untextured sample that would have a null value.

3 Results and discussion

3.1 Cold ECAP – Route A and Bc

In order to study the microstructural homogeneity at the end of the process by ECAP, microhardness profiles were carried out along the length of the samples. The results obtained are presented in Fig. 1, where the graphs of the hardness distribution of the sequence of the number of passes for route A and route Bc processed by ECAP at ambient temperature are shown.

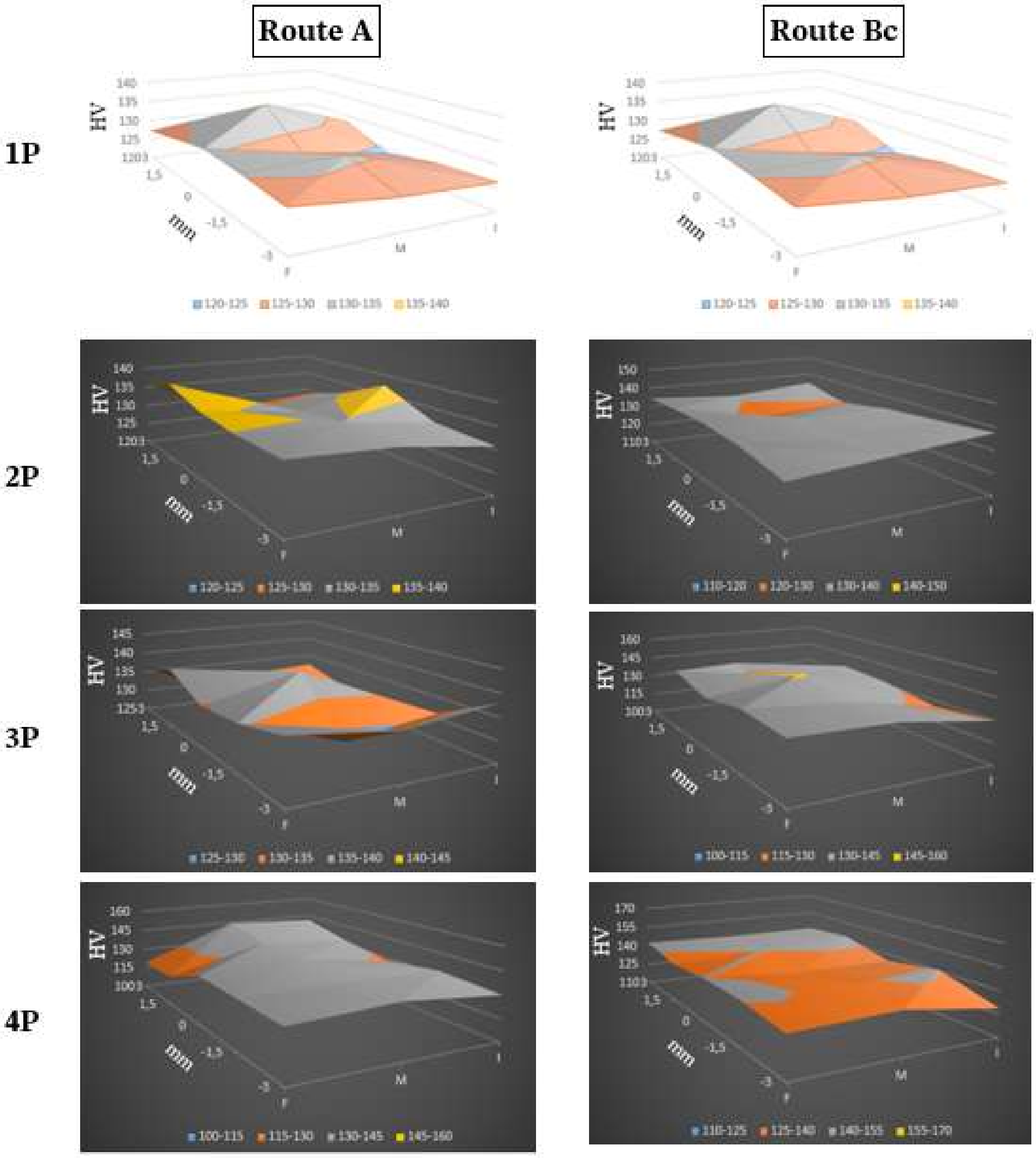


Figure 1: Surface hardness distribution of the four pass sample processed by ECAP at ambient temperature.

It was observed that through route A, a less homogeneous distribution of hardness was obtained when compared to the sample processed by route Bc. The results corroborate the studies by Valiev & Langdon, who demonstrated [9] that of all routes, Bc presents greater microstructural homogeneity, leading the material processed by this route to behave more isotropically during mechanical tests. The hardness and dislocation density obtained in samples processed by ECAP at ambient temperature are shown in Fig. 2.

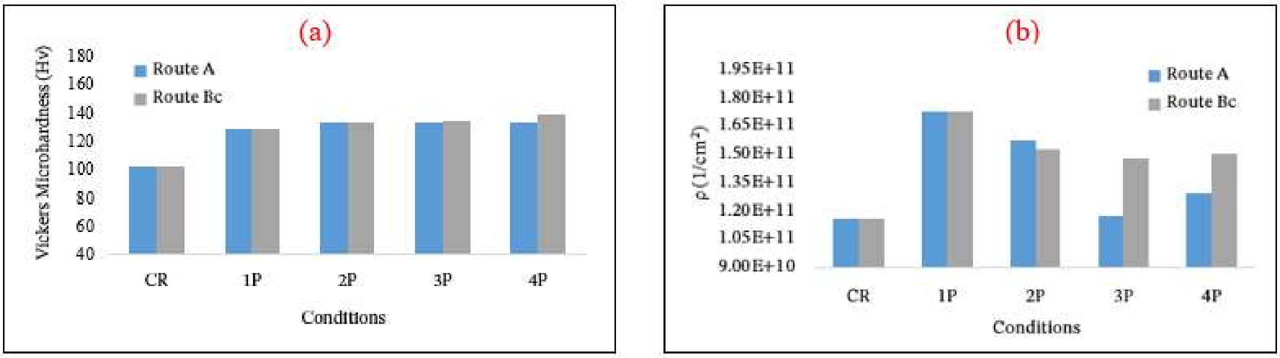


Figure 2: Copper samples processed by ECAP via route A and Bc at ambient temperature. (a) Vickers microhardness; (b) Dislocation Density.

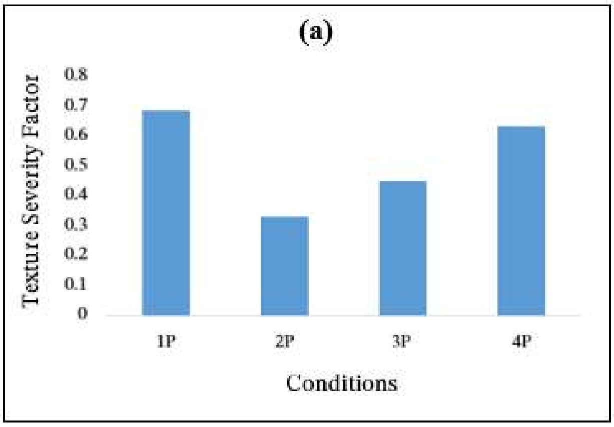
The results show that the most expressive increase in hardness (Fig. 2a) was only after the first pass in both routes. It is known that several characteristics could lead to an increase in the material's hardness after its first pass processing. As for the dislocation density (Fig. 2b), it is observed that there was a drop right after the first pass for both routes, showing a slight tendency to increase after the third pass. Because the material has a high purity level and as the increases in dislocation density were not so expressive, the increase in hardness is attributed to the grain refinement. Another aspect that was taken into account was the initial condition of the material, where it had not been annealed and although had some deformation resulting from its previous manufacturing process. Thus, after the first pass, the dynamic copper recovery process was possibly activated, which may have contributed to the drop in the dislocation density in the subsequent number of passes in both routes [10].

After the first pass, the material processed by route A had an almost constant hardness value up to the fourth pass. On the other hand, the hardness exhibited a slight increase at the end of the fourth pass through the Bc route, which can also be attributed to the grain refinement since the value of dislocation density remained practically constant.

As said, the condition of the starting material also influenced the evolution of the dislocation density. Since the material showed some deformation and had not been annealed before processing by ECAP, it led to a saturation of its number of dislocations right at the beginning of the first pass. Furthermore, different from the values found in the literature [11, 12] where the order of magnitude of the dislocation density increases as the sequence of passes by ECAP increases, in the present study, it remains constant in the order of magnitude of 10^{11} cm^{-2} throughout all four runs of the test at ambient temperature condition. This fact can be attributed to a probable dynamic recovery process suffered by the material. Even with an increase in its deformation due to the sequences of passes by ECAP, this was likely assisted by the competition between multiplication and dislocation annihilation due to dynamic recovery [13]. Similar behavior was observed by Alawadhi et al. [10]; they verified a reduction in the dislocation density due to the dynamic recovery suffered by copper during its processing by ECAP.

Alawadhi et al. [10], Sousa et al. [12], and Wen et al. [14] found for the processing of pure copper through the Bc route via ECAP at ambient temperature, dislocation density in the order of magnitude of 10^{10}-10^{12} cm^{-2}. The difference between the present research values and these can be attributed to the initial condition of the material and the recovery process it possibly underwent. Another factor that should be considered for this discrepancy can be correlated to the material used in this research presenting a high degree of purity compared to the others mentioned above, which had traces of other chemical elements, differently from the electrolytic copper used in this investigation. As the pure material is known, in this case, copper, it probably has higher stacking defect energy [15], which resulted in a more significant recovery process during its processing. Huang et al. [16], Liu et al. [17], and Zi [18] found similar behavior for pure materials, where they verified that the reduction of the stacking defect energy hinders the dynamic recovery, which, consequently, leads to dislocation density values higher.

In Fig. 3, the analysis of the texture evolution as a function of the sequence of passes using the texture severity factor (TSF) is presented.



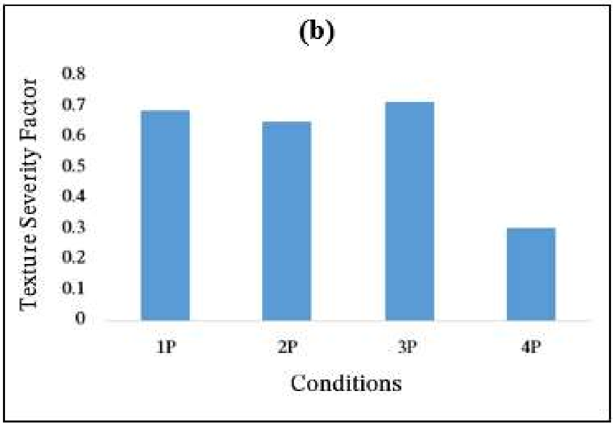


Figure 3: Evolution of the texture of Cu samples as a function of the number of passes through cold ECAP. (a) Route A; (b) Route Bc.

The texture severity factor provides an index to the degree of texture of the material, which means the more intense the degree of texture as a whole. For example, it can be observed that through route A, right after the second pass, the copper presented a reduction in its texture. However, it presented a constant increase trend until the fourth pass. On the other hand, through the Bc route, copper presented practically a constant texture index until the third pass and a significant reduction after the fourth pass, which is a common characteristic of this route that presents a weakening of the texture at the end of its processing [19].

Another texture analysis was performed using the X-ray diffraction technique (XRD) and later with the data treatment, the crystallographic orientation distribution functions (ODF's) were generated. Thus, they were performed for each condition of processing the samples by ECAP as a function of the number of passes and routes at both analyzed temperatures. The Bunge notation was used to interpret the ODF for the analysis of the texture results of the present research, based on the characteristic texture components for Face-centered Cubic (FCC) materials deformed by simple shear. According to the texture variations presented by the material as a function of its recovery, they were also used to assist in the analysis of the texture results of the present research, the recrystallization texture components for FCC materials, components typically found in copper after recrystallization, but also as in the present work, eventually observed in deformed materials. It is shown in Fig. 4, the ODF is only at $\phi = 0°$ and $\phi = 45°$ for the sequence of number of ECAP passes in the route A and Bc in the ambient temperature condition.

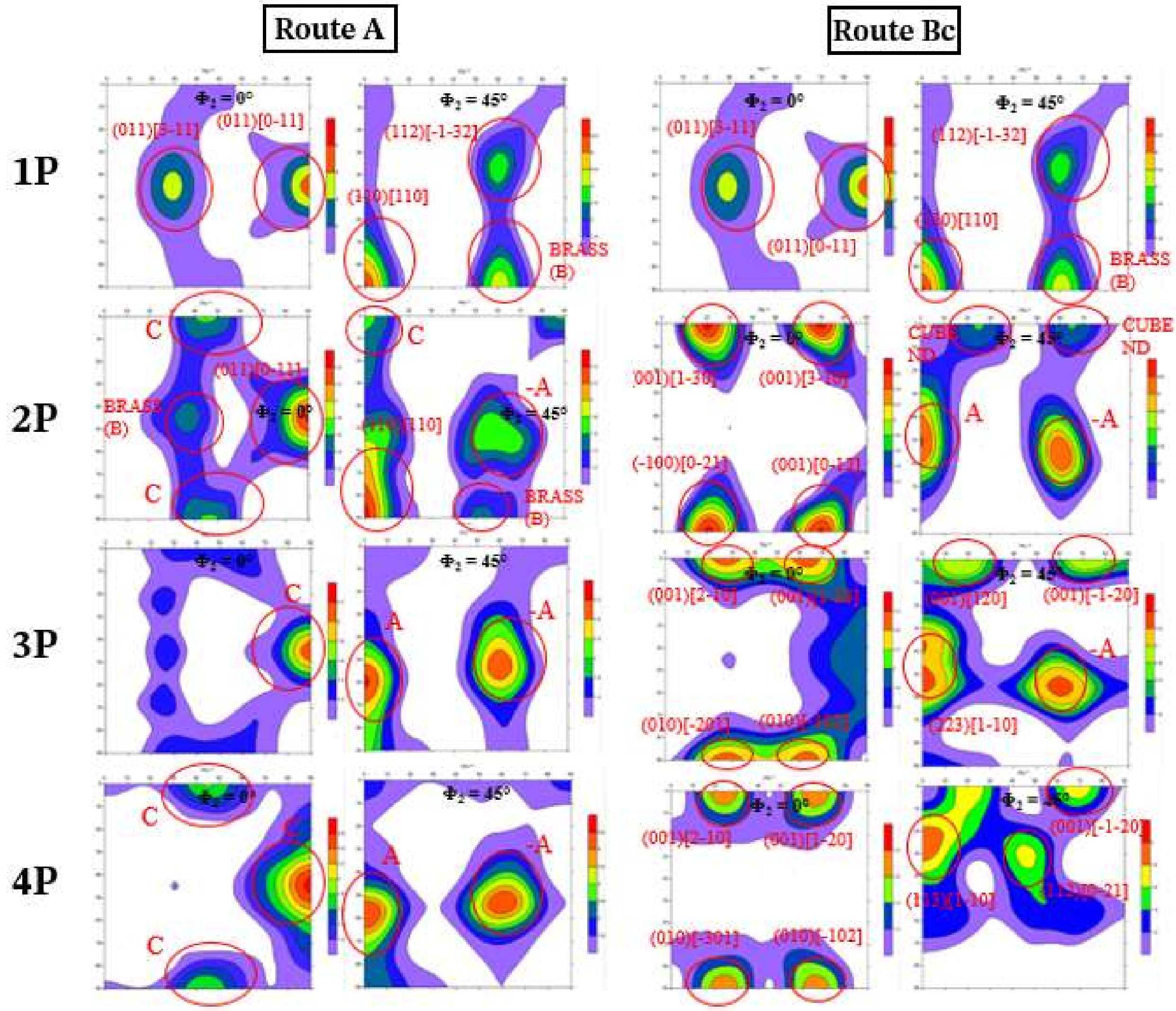


Figure 4: ODF of Cu samples cold processed by ECAP through Route A and Bc.

As can be seen, using the ODF of cold processed samples by ECAP, the texture presented by the evolution of passes through route A, it was found that right after the first pass, the Brass component (B) (110) [1$\bar{1}$2] appeared of recrystallization texture and after the second pass, the components C (001) [1$\bar{1}$0] and $A/\bar{A}$ (111) [110] / (111) [110] also appeared in the material, characterizing a shear texture. Through this route, there was a decrease in texture intensity right after the first pass, although it increased again consecutively in the last two passes. From the second pass through route A, there is practically the intensification of component C in $\phi_2 = 0°$ and $A/\bar{A}$ in $\phi_2 = 45°$ to the fourth pass. Although through the Bc route, along the sequence of passes right after the initial, in $\phi_2 = 45°$ there is the appearance of a recrystallization texture, the Brass component (B) (110) [1$\bar{1}$2] in $\phi_2 = 45°$. After the second pass it also appears in $\phi_2 = 45°$ to component Cube ND (001) [$\bar{1}\bar{3}$0], another recrystallization texture component and another two pure shear texture components $A/\bar{A}$ (111) [110] / (111) [110]. Component A intensifies after the third pass, although it disappears at the end of the last pass.

It can be said that the results obtained are following those found in the literature, as can be seen in the research carried out by Higuera and Cabrera [20]. They processing copper by ECAP via the Bc route at ambient temperature and demonstrated that after the first pass was carried out, the material preferentially exhibited deformed fiber B, due to a high proportion of component C with a moderate presence of components $B/\bar{B}$ and $A/\bar{A}$. After the fourth pass, these researchers further reported that copper developed additional components and guidance indicating the presence of a dynamic recrystallization process during the ECAP test.

3.2 Warm ECAP – Route A and Bc

Through Fig. 5, the results obtained from the hardness distribution wtih the passes for routes A and route Bc processed by ECAP at warm temperature are presented.

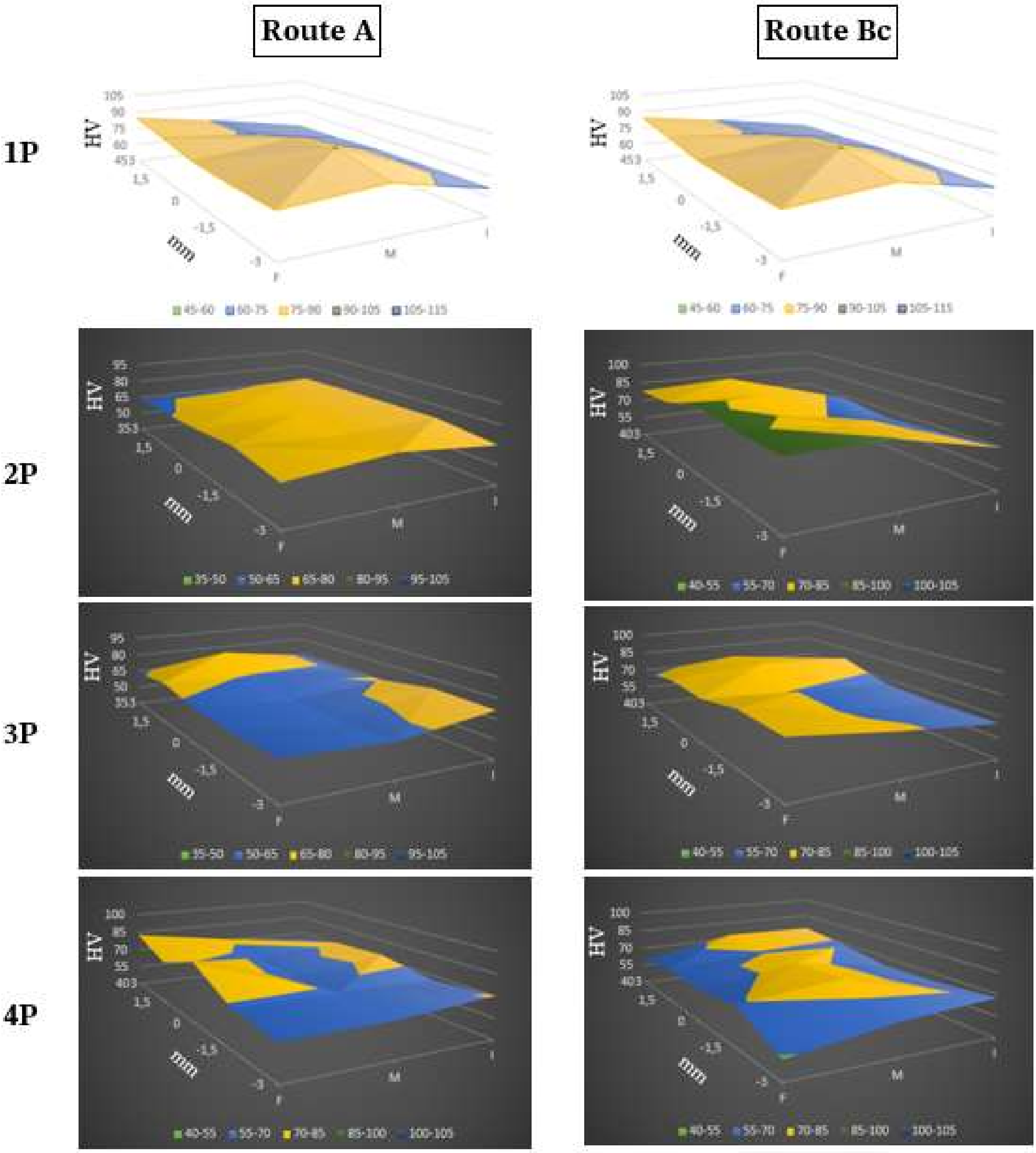


Figure 5: Surface hardness distribution of the fourth pass sample processed by ECAP at warm temperature.

Regarding the hardness distribution along the analyzed surface of the warm-processed samples, it could be observed that both routes presented homogeneous characteristics. Different when compared to the profile presented by the distribution of hardness in the cold condition. Other researchers [21] also found a microstructure with homogeneous hardness distribution for pure copper, which was processed by ECAP up to the fourth pass via the Bc route in a warm test at 200 °C. This uniform hardness distribution was attributed to the high deformations achieved through multiple-pass processing, which stabilized the sample's internal structure. The hardness and dislocation density results obtained in the samples procesed by warm ECAP are presented in Fig. 6.

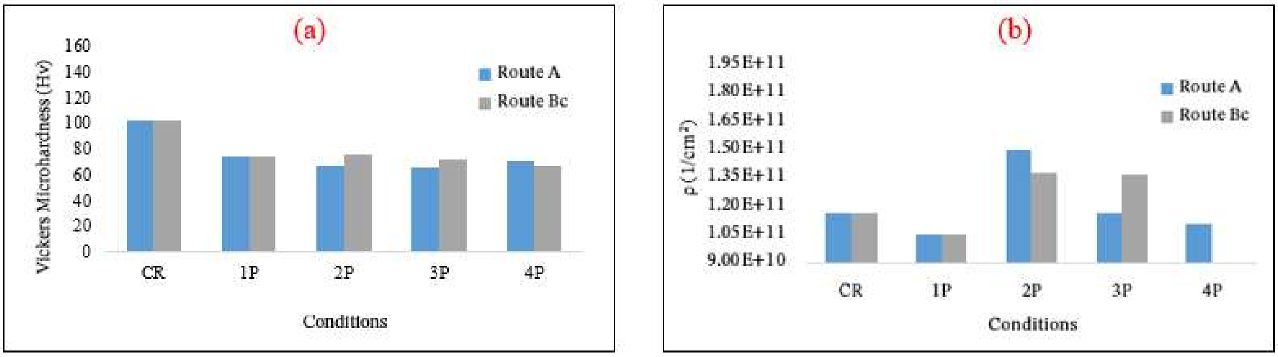


Figure 6: Copper samples processed by ECAP via route A and Bc at warm temperature. (a) Vickers Microhardness; (b) Dislocation Density.

From the observed data from the warm processing by ECAP, both routes practically presented a constant hardness value (Fig. 6a) in the subsequent passes after the first pass. As for the dislocation density (Fig. 6b), a certain variation was observed along with the passes for both routes. Although they presented an increase after the first pass, they presented a reduction after the third pass, being more expressive for the Bc route. Thus, it was observed that the material underwent a recovery process within the temperature range at which it was processed, resulting in a process whereby, in a way, an equilibrium occurred in relation to the multiplication and the annihilation of dislocations. It is observed with the first pass, the hardness value decreased and remained approximately constant for successive passes.

For both routes, the hardness values remained practically constant from the second pass onwards, showing only a small variation as a function of the sequence of the number of passes. Furthermore, dislocation densities did not show an increase compared with the variation of hardness values, which may also be correlated with the grain size reduction characteristic of ECAP processing.

The copper recovery temperature is approximately 270 °C and the recrystallization temperature is in the range of 450-500 °C. The purer and greater the degree of deformation of the material, the lower the minimum recrystallization temperature [22]. Thus, the warm tests in this research gave the material a recovery process, which may have been justified by its reduction in the hardness value presented by the passage of the material through ECAP under temperature.

Based on the results obtained, the sharp reduction in hardness of the samples in the present study was attributed to a recovery suffered by the material during the ECAP process, where this was carried out in a temperature range similar to that in the literature [12, 23]. It is also suggested that the material may have started a recrystallization process, as can be seen in Fig. 7, in which the microstructure can be seen with evidence of the formation of some equiaxed grains.

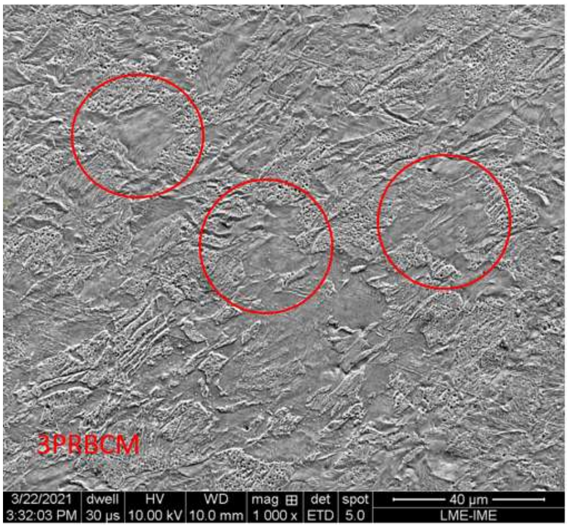


Figure 7: Sample processed with three passes through the route Bc to warm showing a possible start of recrystallization, as highlighted by the circles (magnification of 1000x).

With the ECAP process carried out at a high temperature, simultaneously with the increase in the dislocation density generated by the deformation, restoration mechanisms possibly acted in the material and, as a consequence of this fact, a sharp drop in its hardness values was expected. This fact may have been enhanced by the accumulation of internal energy in the material due to its deformation in the initial condition. As copper is a material with medium stacking defect energy, the softening presented may have been due to the dynamic recovery process [24].

In Fig. 8, it is shown how the texture developed as a function of the two processing routes through warm ECAP. In the same way, as for the cold ECAP, the texture evolution was analyzed as a function of the sequence of the number of passes using the texture severity factor for the warm condition. Thus, it was observed through route A that copper tends to reduce texture intensification along the sequence of passes. The same behavior was verified for the processing through the Bc route, where the material presented this texture reduction even more expressively. However, a slight increase in the texture intensity was observed after the fourth pass.

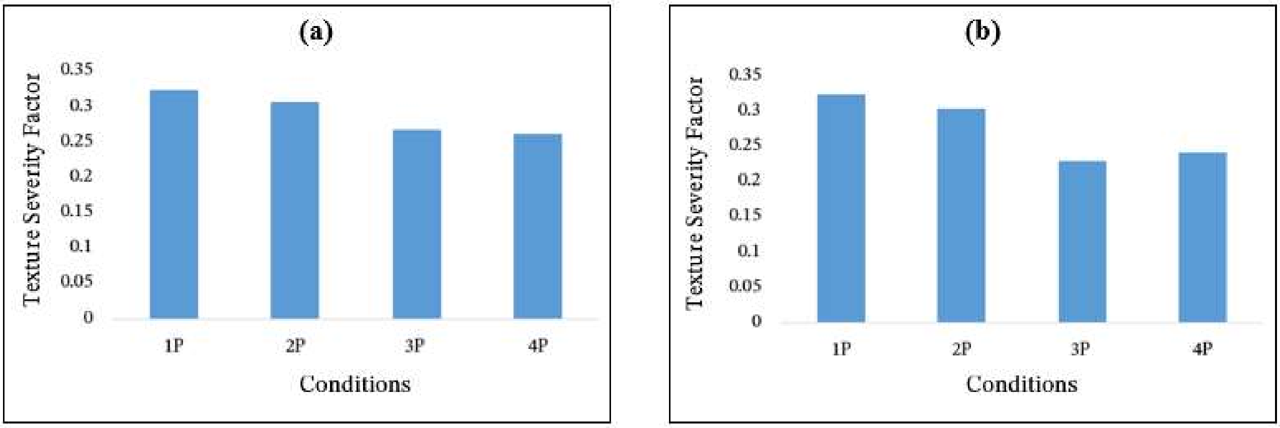


Figure 8: Evolution of the texture of Cu samples as a function of the number of passes through warm ECAP. (a) Route A; (b) Route Bc.

From the ODF of cold processed samples by ECAP, the texture components present during the evolution of passes processed by the warm condition were also evaluated. In Fig. 9, the ODF data for the sequence of ECAP passes in routes A and Bc in the warm condition are presented.

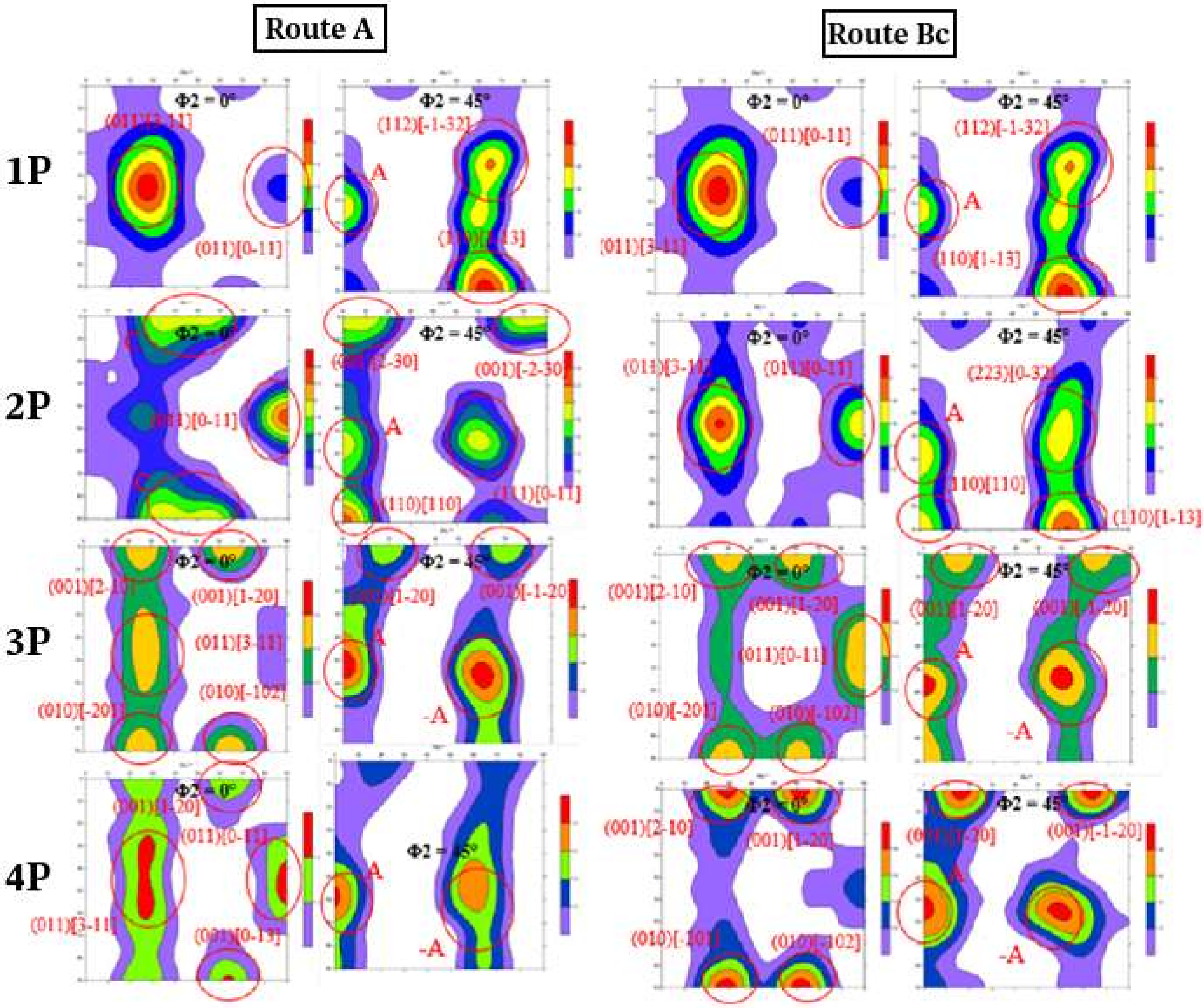


Figure 9: ODF's of Cu samples warmly processed by ECAP through Route A and Bc.

Through the texture analysis performed, it was observed that after the first pass, the texture components A/$\bar{A}$ (111) [110] / (111) [110] appeared, both components of pure shear texture. The texture generated through route A, showed a slight intensification after the second pass where the C (0$\bar{1}$0) [101] component appears at $\phi_2 = 0°$ and the A (111) [110] component at $\phi_2 = 45°$. In the subsequent passes, the attenuation occurs consecutively in the last two passes, keeping the A component and the texture components that appeared in the previous passes. On the other hand, through the Bc route, this route remains with a constant texture intensity after the second pass, oscillating in consecutive passes. The components A/$\bar{A}$ (111) [110] / (111) [110] also appear after the second pass, both pure shear texture.

The texture results agree with the literature [25], where the copper was submitted to ECAP processing up to four passes through the Bc route with an operating temperature of 350 °C. After the passages of the specimens, a weak texture was observed, which could be attributed to the presence of texture component C {001} <110>, together with components B/$\bar{B}$ (112) [110] / (112) [110] and A/$\bar{A}$ (111) [110] / (111) [110]. The texture components A/$\bar{A}$ were found after processing the copper samples in the present research.

4 Conclusions

From the objectives determined for this research and through the results obtained, it can be concluded that:

- Through cold ECAP, it was possible to observe that the increase in hardness and dislocation density were not significant. Furthermore, it can be verified that the Bc route promoted a more homogeneous microstructure and a weakening of the crystallographic texture after the fourth pass of ECAP;

- By means of warm ECAP, a softening phenomenon can be observed concomitant to the deformation process due to the dynamic recovery undergone by the material in the used temperature range;
- By processing electrolytic copper by ECAP at a homologous temperature of 0.48, it was possible to promote a homogeneous distribution of hardness along the microstructure of the material by both routes;
- With the increase in the number of passes through the processing of electrolytic copper by ECAP, in the temperature range of 0.48, it was possible to promote a decrease in the hardness value and in the dislocation density;
- Through both routes via cold ECAP, they presented shear texture components. The Bc route, along the sequence of the number of passes, also showed recrystallization texture components;
- Through both routes via warm ECAP, they presented pure shear texture components.

References

[1] G. Faraji, H. S. Kim and H. T. Kashi, "Mechanical properties of ultrafine-grained and nanostructured metals," in *Severe Plastic Deformation*, Elsevier, 2018, p. 223-257. Available at: https://doi.org/10.1016/b978-0-12-813518-1.00007-2

[2] T.G. Langdon, "Processing by severe plastic deformation: historical developments and current impact," *Materials Science Forum*, vol. 667-669, pp. 9-14, 2010. Available at: https://doi.org/10.4028/www.scientific.net/msf.667-669.9

[3] S. N. Alhajeri, N. Gao, T. G. Langdon, "Hardness homogeneity on longitudinal and transverse sections an aluminum alloy processed by ECAP", *Materials Science and Engineering A*, vol. 528, pp. 3833-3840, 2011. Available at: https://doi.org/10.1016/j.msea.2011.01.074

[4] A. T. Vijayashakthivel, T. N. Srikantha Dath and R. Krishnamurthy, "Response for copper to Equal Channel Angular Pressing with different processing temperature," *Procedia Engineering*, vol. 97, pp. 56-63, 2014. Available at: https://doi.org/10.1016/j.proeng.2014.12.224

[5] S. Ranjbar Bahadori, K. Dehghani and F. Bakhshandeh, "Microstructure, texture and mechanical properties of pure copper processed by ECAP and subsequent cold rolling," *Materials Science and Engineering: A*, vol. 583, pp. 36-42, 2013. Available at: https://doi.org/10.1016/j.msea.2013.06.061

[6] K. Neishi, Z. Horita and T. G. Langdon, "Achieving superplasticity in a Cu–40%Zn alloy through severe plastic deformation," *Scripta Materialia*, vol. 45, no. 8, pp. 965–970, 2001. Available at: https://doi.org/10.1016/s1359-6462(01)01119-8

[7] G. F. Vander Voort, *Metallography: principles and practice*. 2nd ed. New York: McGraw-Hill, 1984.

[8] J. S. Kallend and G. J. Davies, "The development of texture in copper and copper-zinc alloys," *Texture*, vol. 1, no. 1, pp. 51-69, 1972. Available at: https://doi.org/10.1155/tsm.1.51

[9] R. Z. Valiev and T. G. Langdon, "Principles of equal-channel angular pressing as a processing tool for grain refinement," *Progress in Materials Science*, vol. 51, no. 7, pp. 881-981, 2006. Available at: https://doi.org/10.1016/j.pmatsci.2006.02.003

[10] M. Y. Alawadhi, S. Sabbaghianrad, Y. Huang and T. G. Langdon, "Direct influence of recovery behavior on mechanical properties in oxygen-free copper processed using different SPD techniques: HPT and ECAP," *Journal of Materials Research and Technology*, vol. 6, no. 4, pp. 369-377, 2017. Available at: https://doi.org/10.1016/j.jmrt.2017.05.005

[11] M. Y. Alawadhi, S. Sabbaghianrad, Y. Huang, and T. G. Langdon, "Evaluating the paradox of strength and ductility in ECAP at ambient temperature ultrafine-grained oxygen-free copper processed by ECAP at ambient temperature," *Materials Science and Engineering: A*, vol. 802, article 140546, 202'. Available at: https://doi.org/10.1016/j.msea.2020.140546

[12] H. Wen, Y. Zhao, T. D. Topping, D. Ashford, R. B. Figueiredo, C. Xu, T. G. Langdon, E. J. Lavernia, "Influence of pressing temperature on microstructure evolution and mechanical behavior of ultrafine-grained Cu processed by Equal-Channel Angular Pressing," *Advanced Engineering Materials*, vol. 14, no. 3, pp. 185-194, 2012. Available at: https://doi.org/10.1002/adem.201100080

[13] M. P. P. Leis, S. S. Carvalho, T. G. Sousa, S. I. V. Santana and L. P. Branda, "Comparative study by TEM, EBSD and microhardness of the microstructure of copper wiredrawn at 77 K and 295 K," *Tecnologia em Metalurgia, Materiais e Mineração*, vol. 16, no. 2, pp. 279-283, 2019. Available at: https://doi.org/10.4322/2176-1523.20191731

[14] T. G. Sousa, V. L. Sordi and L. P. Brandão, "Dislocation density and texture in copper deformed by cold rolling and Ecap," *Materials Research*, vol. 21, no. 1, 2018. Available at: https://doi.org/10.1590/1980-5373-mr-2017-0515

[15] I. L. Dillamore, R. E. Smallman and W. T. Roberts, "A determination of the stacking-fault energy of some pure FCC metals," *The Philosophical Magazine: A Journal of Theoretical Experimental and Applied Physics*, vol. 9, no. 99, p. 517-526, 1964. Available at: https://doi.org/10.1080/14786436408222963

[16] Y. Huang, S. Sabbaghianrad, A. I. Almazrouee, K. J. Al-Fadhalah, S. N. Alhajeri and T. G. Langdon, "The significance of self-annealing at room temperature in high purity copper processed by high-pressure torsion," *Materials Science and Engineering: A*, vol. 656, pp. 55-66, 2016. Available at: https://doi.org/10.1016/j.msea.2016.01.027

[17] F. Liu, H. Yuan, J. Yin and J. T. Wang, "Influence of stacking fault energy and temperature on microstructures and mechanical properties of fcc pure metals processed by equal-channel angular pressing," *Materials Science and Engineering: A*, vol. 662, pp. 578-587, 2016. Available at: https://doi.org/10.1016/j.msea.2016.03.022

[18] A. Zi, "Pure copper processed by extrusion preceded equal channel angular pressing," *Materials Characterization*, vol. 61, no. 2, pp. 141-144, 2010. Available at: https://doi.org/10.1016/j.matchar.2009.11.03

[19] A. M. Kliauga, W. R. Sussai, M. Ferrante, V. L. Sordi, R. E. Bolmaro, "Texture and lankford factor of AA1050 alloy deformed by angular channel extrusion and heat treated", pp. 1214-1223, in *67th ABM international congress*, 2012.

[20] O. F. Higuera and J. M. Cabrera, "Texture analysis in ultrafine grained coppers processed by equal channel angular pressing," *Materials Research*, vol. 16, no. 3, pp. 619–624, 2013. Available at: https://doi.org/10.1590/s1516-14392013005000032

[21] A. I. Alateyah, M. M. Z. Ahmed, Y. Zedan, H. A. El-Hafez, M. O. Alawad, and W. H. El-Garaihy, "Experimental and numerical investigation of the ECAP processed copper: microstructural evolution, crystallographic texture and hardness homogeneity," *Metals*, vol. 11, no. 4, pp. 607, 2021. Available at: https://doi.org/10.3390/met11040607

[22] A. P. Gulháev, *Metals and their Alloys – Volume 1 – Structures, properties and industrial application (General and structural metallography).* Moscow: Mir, 1981.

[23] F. Liu, T. Fa, P. H. Chen and J. T. Wang, "Steady-state characteristics of FCC pure metals processed by severe plastic deformation: experiments and modelling," *Philosophical Magazine*, vol. 100, no. 1, pp. 62–83, 2020. Available at: https://doi.org/10.1080/14786435.2019.1671621

[24] A. Rohatgi, K. S. Vecchio and G. T. Gray, "The influence of stacking fault energy on the mechanical behavior of Cu and Cu-Al alloys: Deformation twinning, work hardening, and dynamic recovery," *Metallurgical and Materials Transactions A*, vol. 32, no. 1, pp. 135–145, 2001. Available at: https://doi.org/10.1007/s11661-001-0109-7

[25] G. Carro, A. Muñoz, B. Savoini, M. A. Monge and R. Pareja, "Processing, microstructure and mechanical characterization of dispersion strengthened Cu-1%Y," *Fusion Engineering and Design*, vol. 138, pp. 321–331, 2019. Available at: https://doi.org/10.1016/j.fusengdes.2018.11.058