Evolution of Texture and Grain Size during Equal Channel Angular Extrusion of Pure Copper and 6012 Aluminum

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Abstract
Among different SPD techniques, Equal channel Angular Pressing has attracted the most attentions, because of applying large strain to solid bulk materials. In this research, ECAP process up to 6 passes was carried out on a pure copper and a 6012 Al-Mg-Si alloy with B_C route in ECAP dies with Φ=120 and Ψ=20 and diameter of 20and 10 millimeter respectively. Moreover, X-ray diffraction (XRD) system was utilized to investigate changes of the crystallite size and texture variations using for calculating texture parameters and comparing the X-ray diffraction patterns of annealed samples and Ecaped samples. Dominant texture of (111) in annealed aluminum altered to a mixture plane. Also, dominant texture of annealed copper of (111) and (200) shifted to another plane. The opposite of copper samples, changes in diameter and length of aluminum specimens had no effect on diffraction pattern of them. The microstructures were evaluated by OM. The results showed that grain size of annealed aluminum after six passes of ECAP decreased by 75%.

Keywords
Aluminum Alloy 6012, Pure Copper, SPD, Equal Channel Angular Pressing, Texture

1. Introduction

Equal-channel angular pressing (ECAP) is a processing procedure whereby an intense plastic strain is imposed upon a polycrystalline sample by pressing the sample through a special die [1]. This procedure is capable of producing large fully-dense samplescontaining an ultrafine grain size in the sub micrometer or nanometer range [2-4]. ECAP is shown schematically in Fig. 1: a billet is pressed through two equal cross-section channels, intercepting at an angle □. The channel intersection curvature is defined by the angle Ψ. Since ECAP causes no change in the billet cross-section, the process can be repeated indefinitely, thus, deforming the material up to very high values of strain [5, 6].

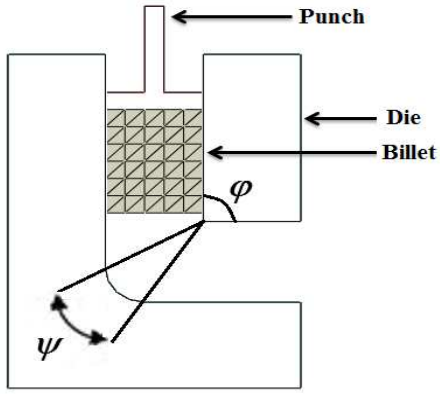

Figure1. Schematic diagram of ECAP die [7]

There are four routes for performing ECAP, Fig. 2 illustrates the four ECAP routes: route *A*, the billet is not rotated; route B_C, the billet is rotated 90° clockwise; route B_A, the billet is rotated 90° clockwise and counterclockwise alternatively; route *C*, the billet is rotated 180°.There is experimental evidence demonstrating that these ultrafine structures may exhibit, by comparison with large-grained polycrystals, major differences in some fundamental properties including the elastic moduli [8]and the Debye [2] and Curie [9] temperatures. Thus, ECAP appears to have the potential for changing the material properties in a significant and controlled way. The application of this procedure is currently under investigation for many different materials ranging from Al [12-13], Cu [14], Mg [15, 16]and Ni [6] alloys to eutectic and eutectoid alloys [17] and intermetallics [18].

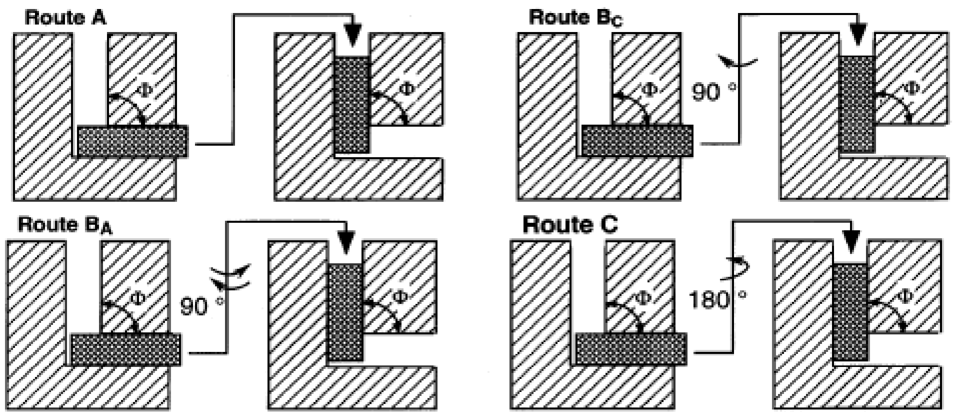

Figure2. Rotation schemes of four ECAP routes [19].

Despite this considerable interest, all work to date, with only one exception, has been devoted to an examination of the microstructures and the properties after subjecting selected materials to the ECA pressing procedure. The exception was a report in which experiments were conducted especially to investigate the microstructural evolution occurring in pure aluminum during pressing [20]. These latter experiments led to three important conclusions. First, the grain size of pure aluminum may be reduced from (1.0 mm) to the micrometer level (0.04 mm) after a single pressing through the die. Second, the microstructure in the early stages of pressing consists of parallel bands of sub grains and these sub grains subsequently evolve with further pressings into an array of grains separated by high angle grain boundaries. Third, a rotation of the sample by 180^0 between each pressing is advantageous because it leads to a more rapid evolution of the sub grain boundaries into high angle grain boundaries. This earlier work reported the first observations on the nature of the microstructural evolution taking place during ECA pressing [20] but the study was incomplete because it failed to provide microstructural information on the characteristics of the pressing including, for example, the orientations of the sub grain bands and texture changes with respect to the shearing direction and increasing passes. A study of the nature of the evolution of the predominant crystallographic orientations resulting from SPD is of primary importance in ECAP processing, especially since the crystallographic texture has a significant effect on many structure-sensitive properties so that a careful texture analysis makes it possible to estimate the mechanisms responsible for the progress of plastic straining and phase transformations. It is well known that the principle of plastic straining, which is a direct consequence of simple shear, is the basic process underlying the ECAP processing technique.

In this connection, a comparison was made between the textures developed during the ECAP processing of copper using route *A* and the established textures resulting from large strain simple shear [21]. It was demonstrated that there is a good similarity between these two cases both in terms of the main ideal components and their intensity variations. A peculiarity of the ECAP textures was also noted: the ideal orientations appear in significantly rotated positions, where these rotations are 10^0 after the first pass but decrease to 5^0 in the subsequent passes. An important difference between simple shear and ECAP textures is that the tilts of the components are in the opposite sense.

Investigations of the texture at several locations across the billet in Cu after the first ECAP pass [22] revealed significant spatial variations in texture, in good agreement with the results from OIM observations. The experimental study was extended for routes *A* and *C* up to a total of 16 passes. For route *A*, the textures in the upper and middle regions of the billet were similar, they remained much stronger than in the lower region and they strengthened with the number of passes. In route *C*, the textures in the upper and lower positions became similar after the second pass but they were quite different from the textures in the center of the billet. Furthermore, the heterogeneity in the texture was smaller for odd-numbered passes than for even-numbered passes.

In recent years, within the framework of modeling of the ECAP deformation, active work has been initiated on the simulation of texture evolution during processing [22, 23]. These simulations lead to the conclusion that the use of a combined finite element polycrystalline modeling approach is most effective in the prediction of texture, and this is true not only for the first pass in ECAP but also for multi-pass deformation [23].The aim of this study is to identify impact of geometry and property (nature of material) of materials in determining dominant texture, orientation and grain refinement occurs during ECAP processing. For this study, Two ECAP dies with different diameters were designed and manufactured from the tool steel X153CrMoV12.

2. Material and methods

In recent investigation aluminum alloy 6012 and pure copper are used. Samples were prepared in two dimensions of 20 and 10 mm diameter (according to diameter of die) and lengths of 50 and 70 mm for each specimen.The channel angle 'Φ' and outer corner angle 'Ψ' of the both dies were 120° and 20°, respectively.Samples were annealed for having a homogeneous structure.The copper samples were annealed in a vacuum condition at 600 °c for 2h and then cooled in the furnace. The

Al samples were annealed at 430 °c for 1h and then cooled down in air to room temperature.MOS_2 was used as lubrication and also routeB_c for rotating samples. All of the mentioned experimental factors used in the research are alsostated in the previous paper[24]. This paper is continued of previous work.

3. Results and discussion

3.1. Investigation of changes of orientation of crystallite planes before and after ECAP process

Figure3 and 4 show X-ray diffraction patterns of Aluminum samples before and after and after ECAP processing. Figure (3-a) shows result of diffraction pattern of annealed aluminum (refrence) with determining of its phase.Maximum intensity of plane in reference pattern is (111). Figure (3-b) and (3-c) indicate diffraction pattern of sixth pass Aluminum samples with same length of 70mm and two different diameter 10 and 20mm respectively. As is shown, the differene in size of diameter of Ecaped aluminum had no effect on their diffraction patterns. There is a remarkable difference when they are compared with reference pattern. So that, aftersix passes ECAPof aluminum samples with same length and diverse diameter, intensity of (111) has decreased dramaticlly and approximately can be ignored in comparison with intensity of otherpeaks. In contrast, intensity of plane which was mixture of Al (220) and Pb(222) and did not exist in reference pattern has maximum count after six passes ECAP. So, according above points it can be said which (111) is dominant texture for annealed sample. Also, after sixh pass dominant texture shifted to combined plane of (Al (220) Pb (222)). The Imoprtant point is that a great change in texture of specimens after six passes ECAP produced. Results displays ECAP has a major impact on orientation of crystalline planes.

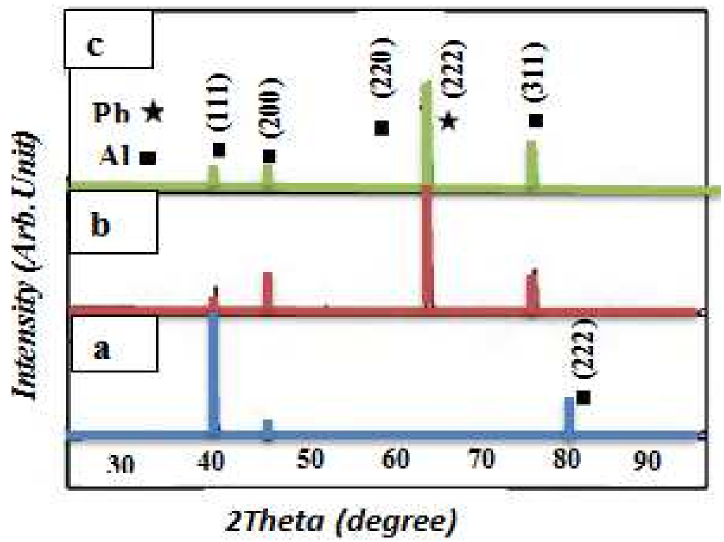

Figure3. Diffraction pattern of a) annealed aluminum b) Ecaped aluminum of with 10mm diameter and 70mm length after six pass c) Ecaped aluminum of with 20mm diameter and 70mm length after six pass.

Figure 4 expresses impact of length of sample on diffraction patterns of Aluminum specimens with similar diameter 20mm and two lengths 50 and 70mm after six passes ECAP. As can be seen, two samples have same diffraction pattern that claims length has no effect on the diffraction pattern of Aluminum samples. However, this point can not be unnoticed which diffraction pattern of mentioned Ecaped samples have changed in comparison with annealed sample.

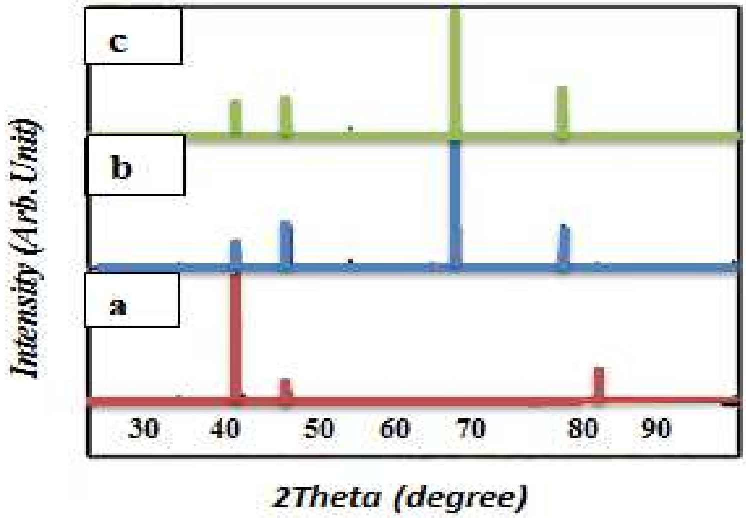

Figure4. Diffraction pattern of a) annealed aluminum b) Ecaped aluminum of with 20mm diameter and 50mm length after six pass c) Ecaped aluminum of with 20mm diameter and 70mm length after six pass

Diffraction pattern of copper samples are presented in Figure 5 and 6. Figure (5-a) presents all phases of annealed copper. It is obvious that maximum intensity of plane is (111) and after that (200) is in the second rank.

Figure (5-b) and (5-c) indicatediffraction pattern of Ecaped copper samples with same length and 10 and 20mm diameter, respectively. Firstly, it is clear which ECAP caused some changes in diffraction pattern of refrence sample. Secondly, in contrast to aluminum samples, the difference in amount of diameters led to a change in diffraction pattern of copper samples.

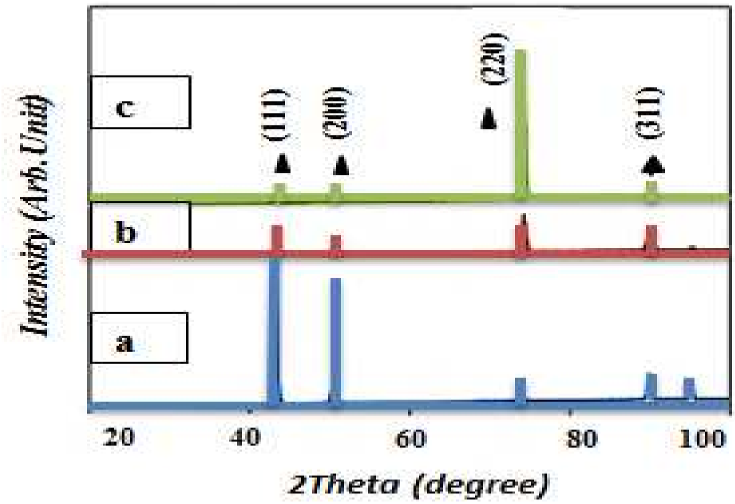

Figure5. Diffraction pattern of a) annealed copper b) Ecaped copper of with 10mm diameter and 70mm length after six pass c) Ecaped copper of with 20mm diameter and 70mm length after six pass.

After six passes ECAP on copper samples with same length and diameters of 10 and 20 mm, intensity of (111) and (200) reduced. Moreover, these peaks could be disregarded in comparison with other peaks. On the other hand, the intesity of (220) increased and altered to a dominant texture. As can be seen in Figure (5-b), copper sample with 10mm diameter has a littile increase in (220) plane. In that case we can not state surely that which plane is dominant texture, because intisity of all planes are almost close to eachother.Overall, there is no dominant texture in copper sample with 10mm diameter and 70mm length. Infact, it has a random texture.Although, copper sample with 20mm diameter and 70mm length has (220) as dominant texture. According to above results, it can be said that as diameter increase, ECAP process produce a dominant texture in copper sample.

As can be shown in Figure 6, With a decrease in length of copper samples with 20mm diameter from 70mm to 50 mm, intensity of (220) altered. So that, the shorter sample has a smaller intensity in (220)plane. It is clear that (220) is a dominant texture in both specimens. The important point is that there is a possibility wich this plane would reduce more when length decrease. Actually, mentioned plane might evolve to a random texture.

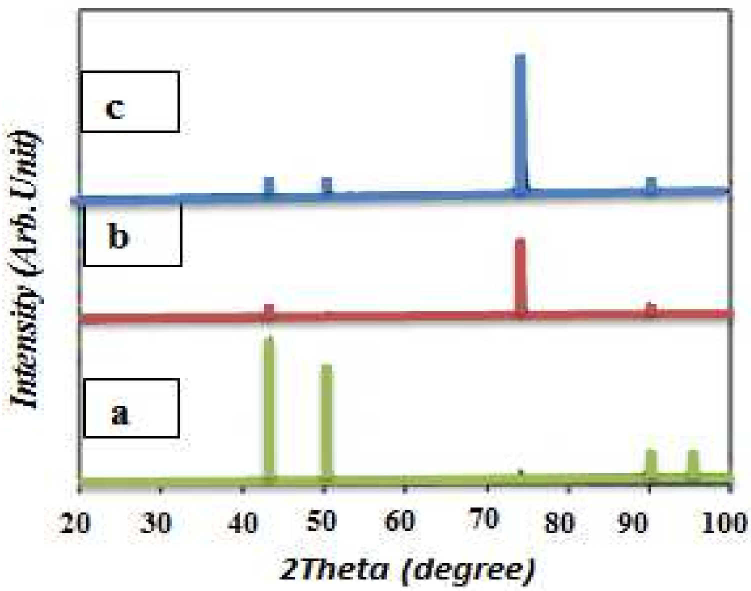

Figure6**.** Diffraction pattern of a) annealed copper b) Ecaped copper of with 20mm diameter and 50mm length after six pass c) Ecaped copper of with 20mm diameter and 70mm length after six pass

3.2. Study of microstructure of samples

Investigation and measuring of grainsize has a special importance in materials science. Grain size has a huge effect on grain property and also on physical property and phase. Diffraction patterns of samples utilize for recognizing of phases.Also, to ascertain amount of grain sizes by MAUD[1] software. In several recent researches [25-31], this sofware is used for identifying microscopic parameters of various materials such as lattice parameter and percentage of phase, crystallite size,lattice average microstrain, and residual microstrain.This shows importance, good validity and capability of mentioned software.MAUD has more accuracy than other softwares like Sigma Plot which usesformula Hall. With regarding to x-ray diffraction pattern of samples presented in previous section and with using MAUD software that has capility of analyzing x-ray diffraction pattern, subgrain size or average values of crystallite size and average values of microstrain of Ecaped and anealed samples were calculated. Results of mentioned calculations are presented in Table 1 and 2 for aluminum and copper specimens, respectively.

As can be shown in Table 1, after six passes ECAP of aluminum samples the value of crystallite sizes reduced from 964(nm) to 200(nm). In fact, this significant change shows 80% decline in crystallite size of aluminum samples.Table 2 offers same information for copper samples and indicates a reduction of 95% in subgrain size of them.

[1] Materials Analysis Using Diffraction

Table1 Calculated values of crystallite size and microstrain of aluminum samples

Average crystallite size (Å)	Average microstrain (×104)	sample
9641	21/7	Annealed aluminum
2983	35/9	Ecaped aluminum with ϕ =10mm and L=50mm
1910	49/7	Ecaped aluminum with ϕ =10mm and L=70mm
1670	96/7	Ecaped aluminum with ϕ =20mm and L=50mm
1927	64/6	Ecaped aluminum with ϕ=20mm and L=70mm

Table2 Calculated values of crystallite size and microstrain of copper samples

Average crystallite size (Å)	Average microstrain (×104)	sample
7891	8/3	Annealed copper
1189	15/01	Ecaped copper with ϕ =10mm and L=50mm
1071	13/11	Ecaped copper with ϕ =10mm and L=70mm
746	7/92	Ecaped copper with ϕ =20mm and L=50mm
746	9/02	Ecaped copper with ϕ=20mm and L=70mm

Results express which ECAP palys a significant role in reducing grain size of samples. Finally, in contrast with aluminum samples, a change in diameter of samples led to a shift in value of subgrain size and strain. Moreover, these results are in good conformity with XRD pattern. As seen, a change in diameter and length of aluminum samples had no effect on pattern of them. However, a difference in diameter of copper samples caused a change in XRD pattern and even eliminated the dominant texture. This subject prove importance of diamter in determining of amount of texture, grain size, and strain of copper specimen.

Electeropolish was performed to make a mirror surface and to minimize tiny scratches and residual stresses resulted from mechanical polish and grading. Ingrdient of etchant liquid were 6 (ml) chloric acid, 80 (ml) ethanl, 14(ml) distilled water and under voltage of 60(v)for 5 seconds. Eventually, microstructure of Ecaped 6012 aluminum evaluated with data of Optical Microscope. Images taken by analysis of Optical Microscope from annealed aluminum and after sixth pass are shown in Fig 9 and 10.Magnification of mentioned images are x1000 and are taken from cross-section of samples and in midlle of billets. As can be seen in Fig (7-a), OM picture is taken from microstructure of Aluminum6012 and in center of cross section.As can be shown, billethave a fairly homogeneous andequiaxed microstructure. Grain size is approximately 19.48 (μm) and with standard deviation of 0.48. Fig (7-b) states that after six passes ECAP of annealed aluminum through route B_c, grains became completely smaller and stretchedin comparison with reference samples. As a matter of fact, severe plastic deformation caused these changes. In this case, grain size is approximately 8.82(μm) and with standard deviation of 0.41.

Fig (7-a) shows microscopic pictures taken from microstructure of annealed aluminum6012 in area of far from center of cross section. In that case, grain size is around 29.12(μm) and with standard deviation of 0.13.

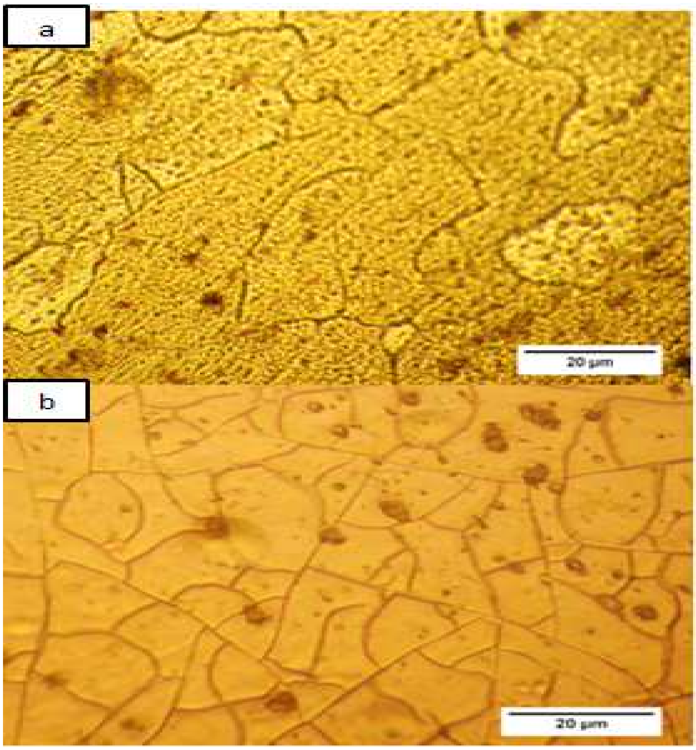

Figure7. Optical Microscope pictures taken from microstructure of Aluminum6012 in center of cross section of sample a) after anneal b) after six passes from die with B_c route

In Figure (8-b), grain size became 7.8(μm) and with standard deviation of 0.49. It is because of huge strain imposed to billet during 6 passes ECAP. As a result, ECAP affected samples in a way which after six passes grain size of them reduced around 75%.

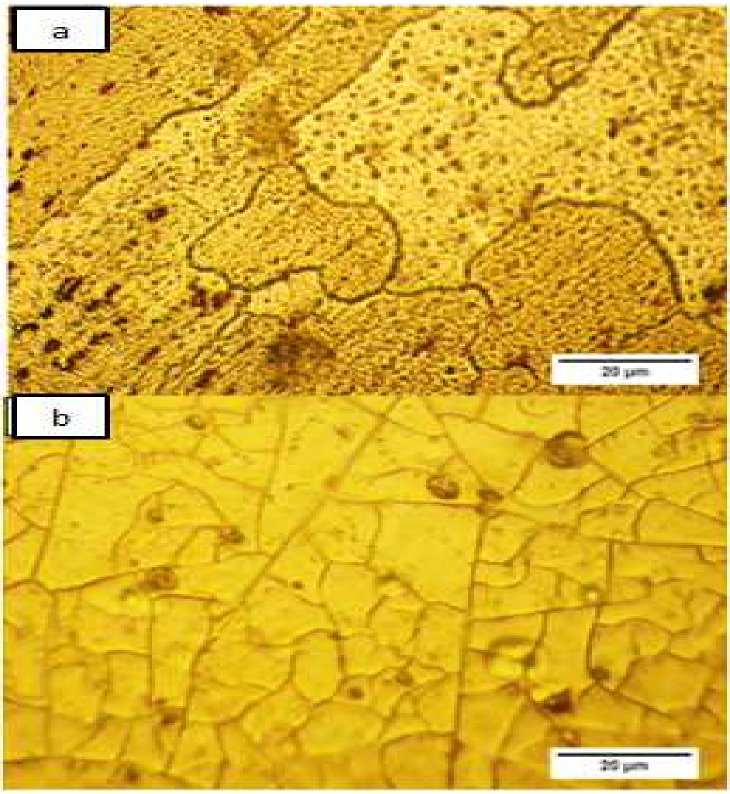

Figure8. Optical Microscope pictures taken from microstructure of Aluminum6012 in area of far from center of cross section of sample (in contact with upper edge of die) a) after anneal b) after six passes from die with B_c route.

4. Conclusion

Effect of geometry and property of materials on texture evolution investigated. After six passes ECAP on 6012Al and pure copper and comparing texture and structure of billets before and after ECAP it could be drawn that:

1. Dominant texture (111) in annealed aluminum after 6 passes ECAP changed to combined texture of ((220) Al and (222)Pb). Also, dominant texture of (110) and (220) in annealed copper altered to (220) for sample with 20mm diameter after 6 passes. While, dominant texture for Ecaped copper samples with 10mm diameter disappeared.
2. According to evaluating microstructure and microscopic images, after sixth pass grain size of billets reduced about 75 percent.
3. Mentioned differences in copper and aluminum samples can be related to variances in properties of them like diverse amount of Stacking Fault Energy of them and because of more convectional coefficient exists in copper than aluminum.

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