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Investigation on the effect of Equal-Channel Angular Pressing process on the pure copper grain size by EBSD

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Abstract. ECAP is a suitable technique for producing ultrafine-grained high-strength materials with desirable mechanical properties. In this study, high-purity copper bars were first processed through the ECAP method. The microstructure of the copper was changed due to the induced hydrostatic pressure and shear strain. Combined with a very high shear strain, this pressure generates high-density crystallographic defects that, in turn, result in the creation of fine grains. Initially, conventional techniques including optical microscopy metallography and FESEM were used to measure the grain size changes; however, they failed to determine the grain boundaries. Finally, changes in the grain size induced by ECAP were measured using EBSD. According to the EBSD results, grain size decreased from 60.8 μm, for a conventional sample, to 5.4 μm after eight passes of ECAP.

Keywords: pure copper, ECAP, grain size, FESEM, EBSD

1. INTRODUCTION

Severe plastic deformation is a general term describing a group of metal working methods resulting in severe strains without introducing any significant change in the overall dimensions of the workpiece. According to another definition, tool deformation is controlled due to its specific geometry. This prevents material flow and, in turn, generates a high hydrostatic pressure. Combined with sever shear strains, this pressure generates high-density crystallography defects. The defects result in the creation of fine grains. When a stress induced on a specimen exceeds its elastic limit, it changes the pattern by which crystals are placed next to each other. Since severe plastic deformations do not change the dimension of a specimen, this pressure creates fine grain networks throughout the material [1].

Metallography refers to the process of preparing specimens for microscopy and studying microstructures aimed at determining the physical and mechanical properties of a given pure alloy. Metallography is in fact the initial stage of observing objects' surface. It prepares surfaces for aided eye observations. It took many years until scientists and researchers could find a solution for magnification of objects. They succeeded to capture a power that even Nano scales became a study arena for the scientists of material sciences. Magnification, however, was not the sole observation problem. The investigations of most scientists revealed that observing the structure of surface texture demands a series of preparatory operations. When the surface of a sample is prepared, it is possible to identify the structure and, consequently, the physical and mechanical properties of materials. The preparation process consists of the following steps: acquiring a piece of metal, cutting a small piece out of it, placing the piece inside mount substances (handling of the pieces in difficult due to their small size), grinding its surface with

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different fine grinders, polishing the surface until all scratches induced by the final grinding process are eliminated, exposing the final piece to a corrosive environment (generally an acid), and observing the surface under a microscope. The above steps are classified into four main steps:

1- Fine grinding

2- Coarse polishing

3- Final polishing

4- Etching

1.1 Fine grinding

The main objective of the first three steps is to reduce the thickness of the deformed layer beneath the sample surface. Cutting, grinding, and grinding operations introduce significant changes to the structure of the surface-adjacent layer. The actual structure of a metal appears when the deformed layer is completely removed from the surface. Since every step deforms the surface, grinder in each any step should be finer than that of the previous one. Every grinder removes the deformed layer produced in the previous step and, at the same time, produces a distorted shallow layer. In the fine grinding step, the surface of samples is grinded using silicon carbide powders mounted on special papers.

1.2 Coarse Polishing

This is a very crucial process. Currently, diamond powder with a grain size of about 3 to 6 microns is used for coarse polishing purposes. Diamond powder is reserved and handled within an oil-soluble paste. In this step, a small amount of the paste is rubbed on a rotating wheel covered with a plastic fabric. Special oil serves as the lubricant during polishing process. The sample is pressed against the wheel at a high pressure. The sample is not kept in a particular position of the wheel. Instead, it is moved in opposite direction around the wheel. This results in a more uniform polishing. Diamond particles are very sharp cutters and effectively remove the deep deformed layer produced in the initial grinding process. A 6-microne diamond powder can remove the deformed layer produced by silicon carbide at the final stages of the initial grinding step.

1.3 Final polishing

This process removes the very fine scratches and distortions produced in coarse polishing step. Alumina powders with a grain size of 0.05, 0.3, and 1 micron are generally used for final polishing. This powder is poured in a rotating wheel covered with a fabric where distilled water serves as the lubricant. Unlike lint-free plastic fabrics used in coarse polishing step, this step uses fabrics with lint. If this step and the previous one are performed accurately, a visible scratch-free surface will be formed with almost no detectable distorted metal.

1.4 Etching

After final polishing, the grain structure of metallography samples is not generally identified through microscopy. The thickness of grain boundaries of a metal is as high as the thickness of several atoms at its best while the detection power of a microscope is too low than required level for identifying grain boundaries. Grain boundaries are detectable only in metals in which

different crystals with different colors contact with each other. To make grain boundaries visible, the prepared metallography samples are etched. This is done by immersing the surface of the polished sample inside an etching solution (a weak acid or base). Nital is the most conventional etchant (etching solution) for steels. It contains 2% acid nitric solved in alcohol. In some cases, it is possible to conduct the etching process by gently rubbing a piece of cotton smeared with the etchant on the sample surface. In any form of etching, parts of the surface is solved and removed. If a suitable etchant is used, the surface will not be solved homogenously. Sometimes, the etching agent attacks grain boundary quicker than grains surface. Etchant solves different grains depending on their orientation. After etching, grain boundaries appear as shallow stairs on the surface. The vertical walls of the stairs serve as almost smooth crystal surface and prevent the reflection of light into the objective lenses of microscopes. Therefore, the location of crystal boundary can be identified under the microscope.

1.5 Electrical Polishing and Etching

Mechanical polishing is not suitable for some metals such as stainless steel, titanium and zirconium where the elimination of distorted layers is a very difficult task. Therefore, at the final step, electric polishing is performed on the metals. During electric polishing, the sample serves as the anode while a non-soluble substance serves as the cathode while both are placed in a suitable electrolyte bath. If a suitable current density is used, the surface of sample can be solved to result in a proper polished surface. In this method, the electrolyte bath and current density should be controlled separately in a manner that a smooth surface with no up and down is obtained. However, it is possible to produce a surface with suitable up and downs for etching process through changing chemical composition of the bath and the current density [2]. In this study, the grains of pure copper bars were refined after 4 and 8 passes of ECAP. Then, changes in grain size induced by ECAP process were determined using different methods including optical microscopy metallography, FESEM microscopy, and EBSD (electron back scattered diffraction).

2. MATERIALS AND METHODS

2.1 ECAP Process

A die with an angle of 120^{o} and channel diameter of 10 mm was used for grain refinement of the pure copper samples. The ECAP die was made of cold-worked tool steel (X153 CrMoV12) as per DIN 1.2379. Fig.1 shows the die during ECAP process. Using a 100-ton hydraulic press, the samples were directed into the ECAP die. To further refine the grains, ECAP was performed in several successive passes [3]. This study used route Bc where each time the pieces were removed from the die, they were rotated by 90^{o} and relocated inside the die to apply the next pass [4, 5]. This process was repeated for several times, and copper parts were processed by 4 and 8 passes of ECAP. In this study, the ECAP process was performed at room temperature [6].

Figure 1. Copper workpiece exiting the die.

2.2 Metallography and Optical Microscope

Observing related standards, conventional samples and samples processed by 4 and 8 passes of ECAP were prepared for metallography tests. The standards include preparing metallography samples [7], metals micro-etching [8], optical microscopy images [9] and measuring the means size of grains [10]. A mixture of hydrochloric acid (30 ml), Iron chloride III (10 grams) and alcohol (120 ml) was used as the etchant [2]. During the etching process, the metal surface was continuously analyzed using optical microscopy, and the samples were immersed into the etching solution, if necessary. Finally, the best surface was obtained after immersing the sample in the etchant for 35 seconds. The microstructure of ECAPed copper samples was studied using the NEOPHOT 32 optical microscope manufactured by CARL ZEISS JENA.

2.3 FESEM

Preparing stages were as the same of section 2.2. The etching duration and solution were changed several times as the grain boundaries were not detectable.

2.4 EBSD

EBSD is a new technique for determining the grain size of samples. This technique has remarkable capabilities in the qualitative and quantitative analyses of microstructures. The development of EBSD has recently attracted the attentions of research and industrial centers so that it has been turned into the main technique for microstructure analysis in most centers. This technique can be used for calculating the crystalline direction of microstructure, identification of phases and the extent of their distribution.

EBSD is also known as an OIM (Orientation Imaging Microscopy). Currently, there are two commercial products in the market made by two competitors: EDAX-TSL and Oxford-HKL. Products with the EBSD test ability are directly offered only to a few countries due to their special applications and reverse engineering abilities. Fig. 2 shows the EBSD principles and the way of placing samples in SEM housing. It illustrates the fundamentals of this technique in a simple manner. A beam of electrons hit a sample rotated by 70^{o} around its lateral axis. The backscattered electrons generate a pattern on a phosphor plate. The pattern contains lines similar to Kikuchi bands in TEM. The geometrical configuration and the position of lines play a role in determining the structure and the orientation of crystals of the zone contributed to scattering.

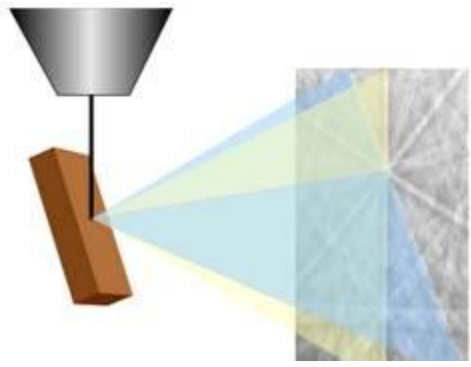


Figure 2. A schematic view of EBSD imaging [11].

A well-prepared sample is necessary to obtain a high quality EBSD pattern. The surface of the area swept by electrons should be flat and free of any contamination and oxide layer. Optimal roughness depends on the grain size of the microstructure. Finer microstructure requires higher roughness. For a nanometer microstructure, for example, the required roughness should be in the nanometer scale. Electrochemical, mechanical and ion-based techniques are used to achieve such a quality [11].

2.4.1 Preparation of the ECAPed Samples

Since optical microscopy metallography failed to determine the grain size of the cold-worked samples, this study used the EBSD technique only to define the microstructure of the samples processed by 4 and 8 passes of ECAP. First of all, the samples were cut preciously into two halves using a cutter in order to find the center of the ECAPed samples. During this process, the generated noise indicated the difference of hardness and strength of the ECAPed samples. Then, the surface of samples was polished by sandpaper. Since copper is a soft metal, these steps were practiced very slowly and patiently. Then, hand polisher and alumina suspension were used to polish the samples. The samples were not mounted since a reliable electric connection was necessary in later steps. Between the two steps, the samples were continuously washed by ethanol and dried by high pressure wind. The quality of the prepared samples was then checked by optical microscopy. Fig. 3 and Fig.4 illustrate the surface images of copper samples after polishing (before electro polishing step). To conduct the EBSD tests, the scratches shown in figures 3 and 4 had to be removed using electro polishing.

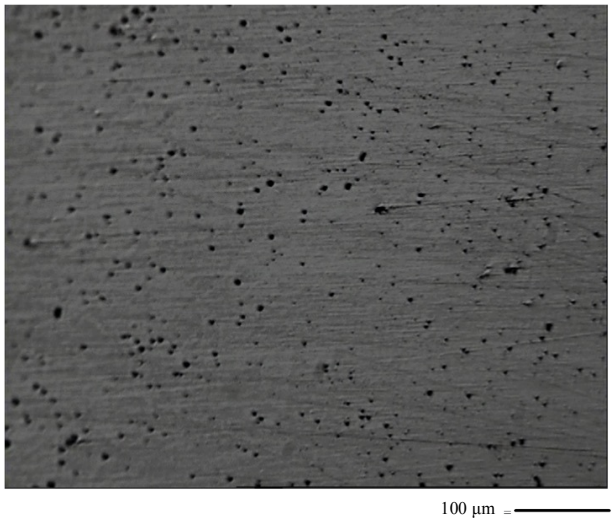


Figure 3. Optical microscopy images of samples processed by 4 passes of ECAP before electro polishing.

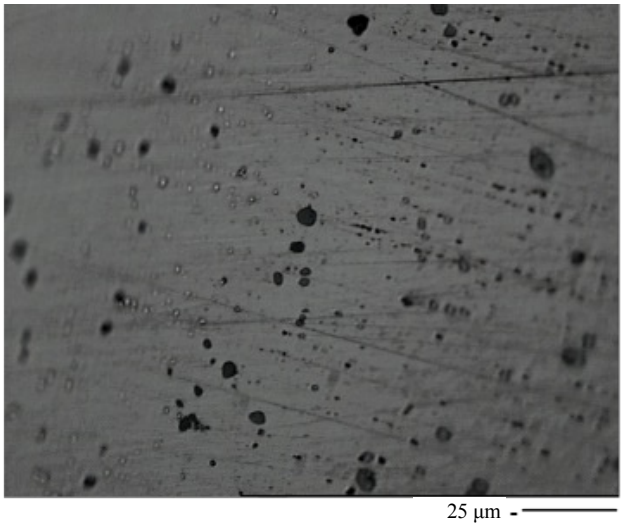


Figure 4. Optical microscopy images of samples processed by 8 passes of ECAP before electro polishing.

An electrolyte with a given percentage of phosphoric acid, ethanol, and ammonium persulfate was used for polishing. The electro polishing process was conducted after establishing a suitable voltage. Fig. 5 shows the optical microscopy image of the samples processed by 8 passes of EACP after electro polishing. This figure shows that all unpleasant scratches and lines have been eliminated after polishing. It should be noted that the cavities that are seen in the microstructure had no negative impact on EBSD test results because the EBSD test investigates cavity-free areas of surface (Fig. 6).

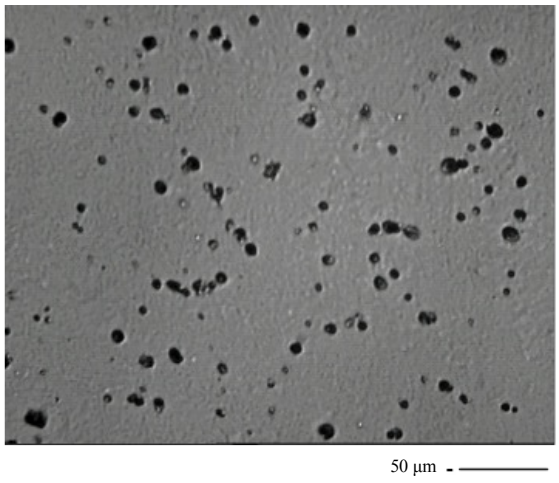


Figure 5. Optical microscopy images of samples processed by 8 passes of ECAP after electro polishing.

Figure 6. A sample processed by 8 passes of ECAP is located in EBSD test chamber.

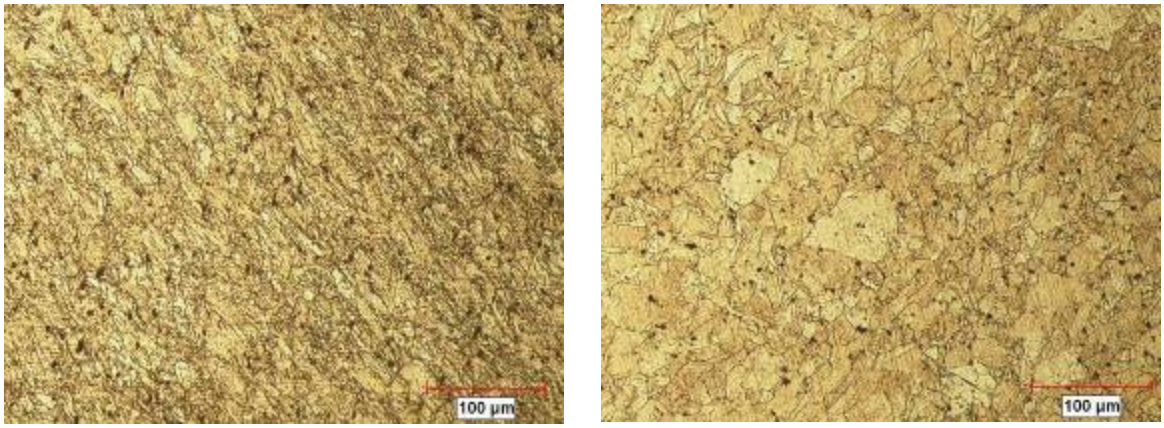
3. RESULTS AND DISCUSSIONS

a. Optical Microscopy Metallography Results

Fig. 7 shows a schematic view of a conventional sample. The mean grain size can be calculated in this sample since the grain boundaries are obvious. According to measurements, the mean diameter of the grains ranged from 31.8 μm to 89.8 μm as per ASTM 4-7 with an almost identical frequency distribution. The mean grain size of the unECAPed sample is 60.8 micrometer.

Figure 7. The unECAPed sample with a grain size of 60.8 micrometer.

Fig. 8 shows optical microscopy images of the samples after 4 and 8 ECAP passes. The boundary of real grains cannot be determined by etching due to cold working process.



b **a**

Figure 8. Optical microscopy metallography: a) sample after 4 ECAP passes b) sample after 8 ECAP passes.

b. FESEM Metallography Test Results

Cold working and the consequent etching process removed grain boundaries to a large extent. Nevertheless, comparing the images of samples processed by 4 and 8 passes of ECAP revealed that the reduction of grain size was higher in samples processed by 8 passes of ECAP as compared to samples processed by 4 passes of ECAP.

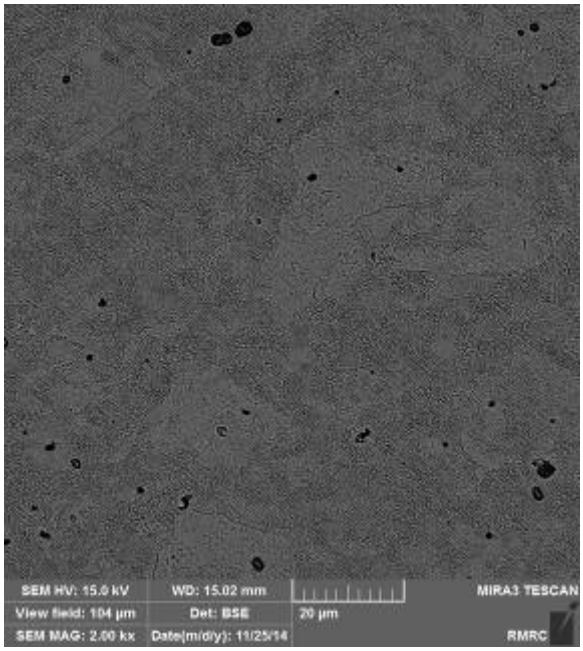


Figure 9. Samples processed by 4 passes of ECAP

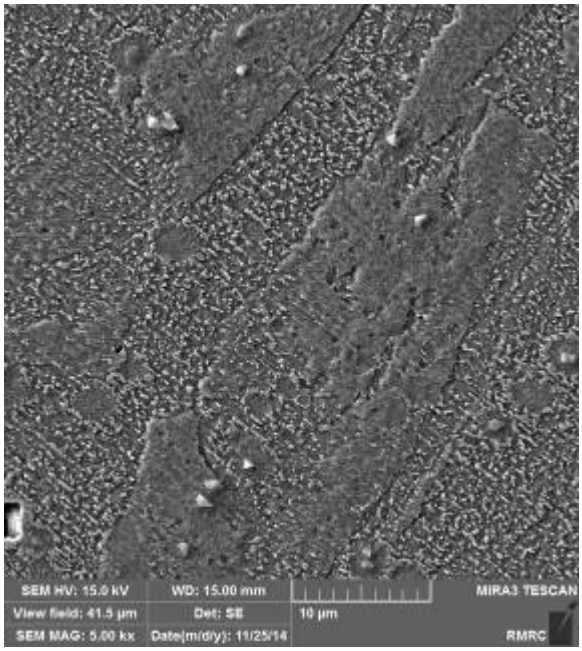


Figure 10. Samples processed by 4 passes of ECAP

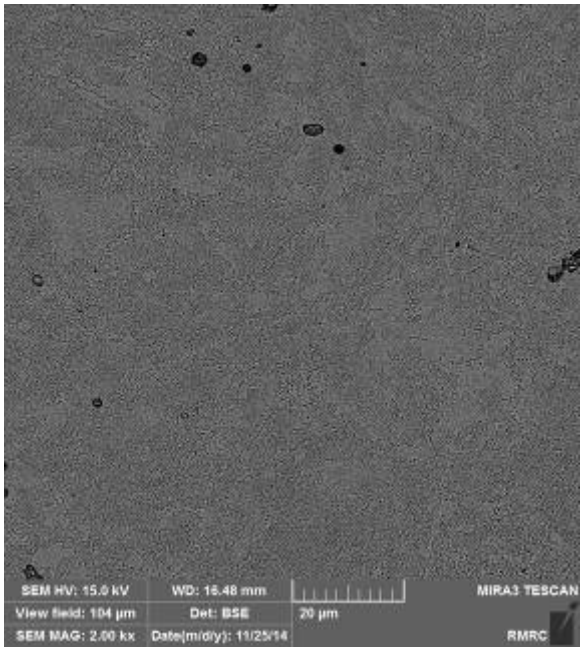


Figure 11. Samples processed by 8 passes of ECAP

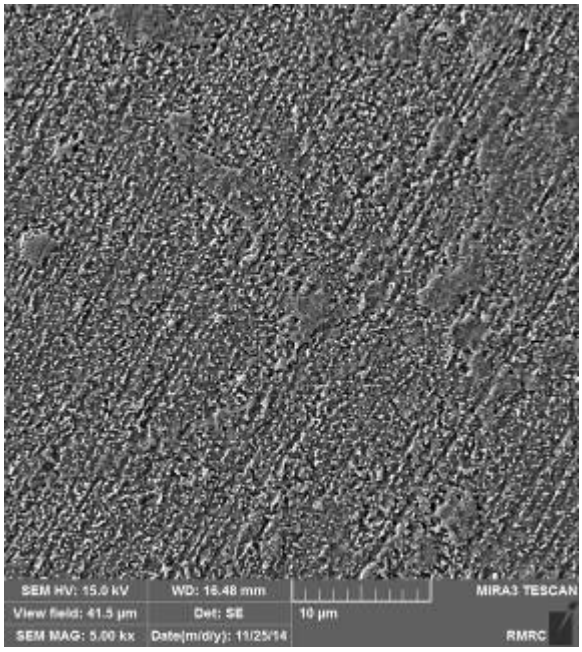


Figure 12. Samples processed by 8 passes of ECAP

c. EBSD Test Results

Using a particular mechanical preparation technique this method avoids the change in or elimination of grain boundaries and presents grain size as the output values of the test device.

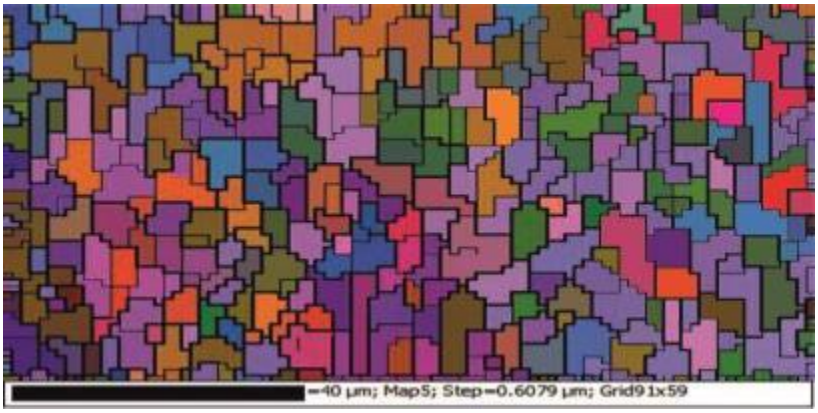


Figure 13. EBSD test in the sample processed by 4 passes of ECAP passes; mean grain size=9.84 micrometer.

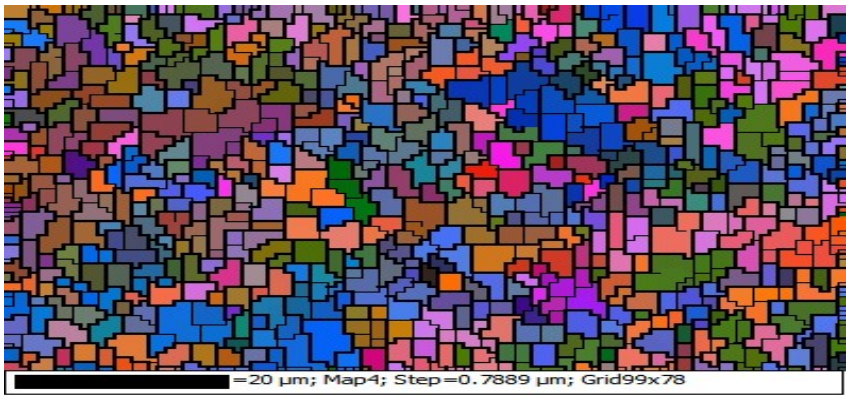


Figure 14. EBSD test in the sample processed by 8 passes of ECAP passes; mean grain size=5.48 micrometer.

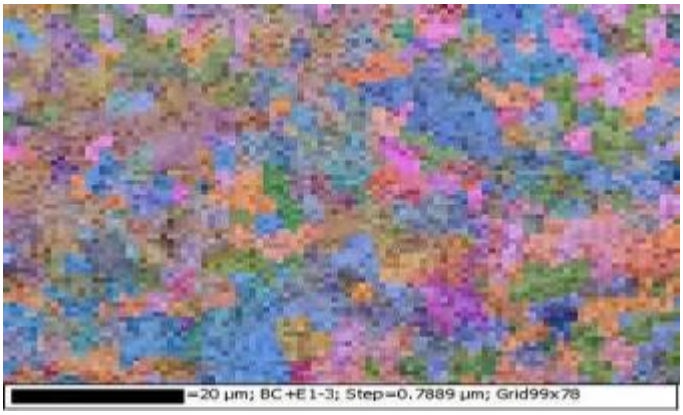


Figure 15. The main image of sample processed by 8 passes of ECAP obtained from the EBSD test.

Both hardware (EBSD detector attached to electron microscope chamber) and software (Aztec-W) tools are required to conduct the EBSD test. This image is the main source of Aztec-W. This software had been installed on the hardware system by the manufacturer of the EBSD test. Following processing the images, Aztec-W presents the results as the images shown in Fig. 14 in order to enhance the quality of output data. It should be mentioned that this study used the EBSD test in order to find the grain size of samples. This is why a great area of samples was scanned. If we wished to have a high quality image (that was not justifiable considering the objectives of this study) scanning steps should be decreased to 0.1 µm. This would raise the test duration 8 times as much as current time for every sample i.e. from 4 hours to 32 hours.

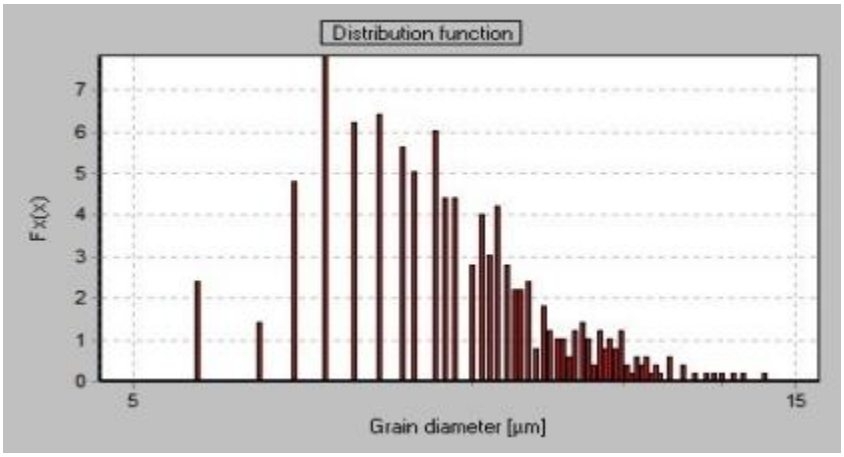


Figure 16. Frequency distribution of grain size of samples processed by 4 passes of ECAP derived from the EBSD test.

The vertical (y) axis represents the frequency distribution of the grain size. The statistical analyses of the data indicated that the mean grain size of samples after 4 ECAP passes is 9.84 µm (output image of Aztec-W).

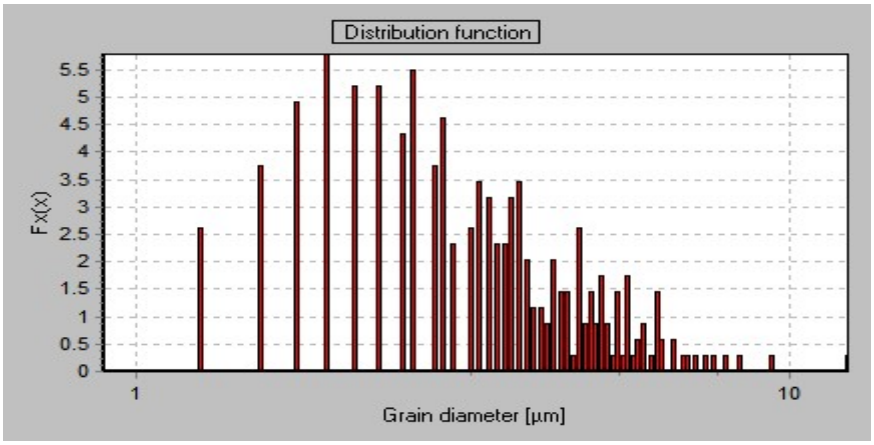


Figure 17. Frequency distribution of grain size of samples processed by 8 passes of ECAP derived from the EBSD test.

The vertical (y) axis represents the frequency distribution of the grain size. The statistical analyses of the data indicated that the mean grain size of samples after 8 ECAP passes is 5.48 µm (output image of Aztec-W).

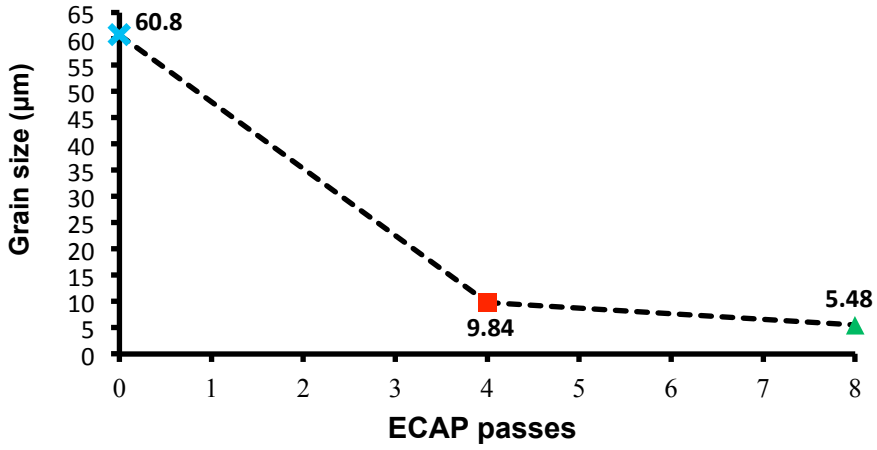


Figure 18. Variations of mean grain size during the ECAP process

4. CONCLUSION

To investigate the impact of the ECAP process on the microstructure of pure copper, pure copper grains were refined through the extrusion process in channels with the equal cross sections. The surface of the samples was then studied using different techniques. It was concluded that:

- Chemical surface preparation-based metallography techniques (etching) do not produce proper images for measuring the grain size of pure cold-worked copper.

- Due to its nature and since it does not require chemical preparation (only mechanical preparation is necessary)[1], the EBSD technique can provide acceptable results for the granulation of ECAPed copper samples.

- Considering the orientation of each grain, this technique can differentiate grains. It assigns a given color to every grain depending on its angle. These play a key role in determining the granulation of cold-worked samples.
- The ECAP process can decrease the grain size of pure copper samples from 60.8 µm (grain size of a conventional copper sample) to 5.48 µm within 8 minutes indicating a 90.99% decrease in the grain size after 8 passes of ECAP. The decrease was as much as 83.82% and 44.31% in the first four passes and the second four passes, respectively. According to results, a decreased grain size decreased the reduction rate in the mean grain size.

REFERENCES

[1] Iwahashi, Y. Horita, Z. Nemoto, M. and Langdon, T., The process of grain refinement in equal-channel angular pressing, Acta Materialia, Volume 46, Issue 9, Pages 3317-3331, 1998.

[2] ASM handbook, Volume 9., Metallography and microstructures, ASM International, The materials information company, 2004.

[1] Electropolish is a mechanical technique whereas etching is a chemical technique

[3] Han, Y. Li, J. Huang, G. Lv, Y. Shao, X. Lu, W and Zhang, D., Effect of ECAP numbers on microstructure and properties of titanium matrix composite, Materials & Design, Vol 75, pp. 113-119, 2015.

[4] Hoseini, M. Meratian, M. Toroghinejad, M.R. and Szpunar, J.A., Texture contribution in grain refinement effectiveness of different routes during ECAP, Materials Science and Engineering, Vol 497, Issues 1–2, pp. 87-92, 2008.

[5] Bian, L. Liang, W. Xie, G. Zhang, W. and Xue, J., Enhanced ductility in an Al–Mg2Si in situ composite processed by ECAP using a modified BC route, Materials Science and Engineering, Vol 528, Issue 9, pp. 3463-3467, 2011.

[6] Vijayashakthivel, A.T. Srikantha Dath, T.N. and Krishnamurthy, R., Response of copper to Equal Channel Angular Pressing with different processing temperature, Procedia Engineering, Vol 97, pp. 56-63, 2014.

[7] ASTM E3 - 11, (2013), Standard Guide for Preparation of Metallographic Specimens, ASTM International, West Conshohocken, PA, 2013.

[8] ASTM E 407-07ε1, (2013), Standard Practice for Microetching Metals and Alloys, ASTM International, West Conshohocken, PA, 2013.

[9] ASTM E ASTM E 883-11, (2013), Standard Guide for Reflected–Light Photomicrography, ASTM International, West Conshohocken, PA, 2013.

[10] ASTM E 112-12, (2013), Standard Test Methods for Determining Average Grain Size, ASTM International, West Conshohocken, PA, 2013.

[11] Oxford Instruments, (2013), Introduction to EBSD, http://www.ebsd.com/. EBSD.com is an educational site by Oxford Instruments.