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

Mohammad Javad BAGHERİ[1,*], Payam SARAİAN[2]

[1]*Msc Student at Department of Mechanical Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran*

[2]*Assistant Professor at Department of Mechanical Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran*

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Abstract. Severe plastic deformation is a general concept for the explanation of a group of metal-working methods that apply many strains without changing the overall dimensions of the work piece. In other words, due to the special geometry of tools, the deformation of tools is restrained which applying this process will prevent the flow of materials, thus, creating enormous hydrostatic pressure. This pressure along with high shear strain results in high-density defects in the crystal lattice, leading to the formation of fine grains. The method of pressurizing under angular channels with identical cross sections (extrusion in angled cross section channels) is a suitable process for producing a material with fine structure, high strength, and desired physical properties. The aim of this research is to show that by decreasing the grain size of copper, its strength is increased. It was understood that the ECAP process appropriately decreased the grain size of the widely used metal, and in the eighth repeated test, reached the grain size number 11 14 falling into the standards of ASTM, which is equal to 2 – 8 micrometers.

Keywords: ECAP, Pure copper, Grain size

1. INTRODUCTION

The physical and mechanical properties of poly crystalline materials are influenced by many factors, which amongst them, grain size is considered of high importance and the utmost influential factor in most materials. In general, the process of decreasing the grain size is a relatively effective economical way to improve the mechanical properties of metals. In recent years, vast amount of research has been devoted to the process, structure, and mechanical behavior of materials towards submicron grains. Today, thermo-mechanical treatments are used to reduce the grain size of industrial and commercial alloys. These are thermal cycles combined with mechanical deformation. The minimum grain size created using such processes are approximately around a few microns and due to the thermal-mechanical nature of these processes, and the occurrence of the phenomenon of recovery and recrystallization, producing a sample with a grain size of less than one micron is very difficult. A lot of effort has been put towards trying to invent different ways of processing which will allow the production of UFG[1] material with a grain size less than one micron or even in nano-dimensions. The production of poly-crystal UFG material must be in such a way that in addition of only a fraction of a micron; coaxial fine grains in the structure are distributed homogeneously.

[1]- Ultra-Fine Grain

* Corresponding author. *Email address: bagheri@smc.iaun.ac.ir*

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These materials have special properties such as high strength at room temperature, super-plastic properties at low temperatures, high strain rate and excellent corrosion resistance. In further steps, the unique properties of nano-crystalline material (grain sizes less than 100 nm), in comparison with coarse materials are because nano-crystalline materials have many grain boundaries. In nano-crystalline materials a large fraction of atoms (up to 49%) are grain-boundary atoms. Therefore the structure of the interface (boundaries) plays an important role in the physical and mechanical properties of nano-crystalline materials [1].

Severe Plastic Deformation is a general concept for defining a group of metal-working methods that apply a lot of strain with no change in the overall dimensions of the work-piece. In other words, due to the special geometry of tools, the deformation of tools is restrained, preventing the flow of materials, which results in creating a lot of hydrostatic pressure. This pressure accompanied by a lot of high shear strain, causes crystal defects with high density, which leads to the creation of fine grains. When the tension applied to the sample has passed its elastic limit, the pattern of the alignment of crystals next to one another changes, and because the dimensions of the sample in SPD methods does not change, the pressure results in creating networks of fine grain in the volume of the substance. From methods of SPD we can name the following processes.

1. Extrusion/Equal-Channel Angular Pressing (ECAP/E).
2. High Pressure Torsion (HRP).
3. Twist Extrusion.
4. Accumulative roll bonding.

In the above methods, in the recent two decades, the process of ECAP with its special characteristics has attracted many researchers.

Some of the unique features of this method are listed as follow:

1. The substance produced using the ECAP method can be relatively large and can be used for industrial purposes. For example, manufactured parts produced by the templates used in this research, have a maximum length of 6cm and a fixed diameter of 1cm, considering the parts being pure copper, they can be used as a tool of spark, spot welding electrode, and etc.

2. Regardless of design and mold manufacturing stages, this method is relatively simple. The equipment needed can be found in most laboratories and can be easily accessed.

3. The ECAP method can be used on different types of materials with different crystalline structure and also on most alloys including heat treatable alloys and non heat treatable alloys, intermetallic compounds and even matrix composites.

4. In the case of appropriately controlling design parameters and friction UFG materials with relatively homogeneous structure can be produced [1].

Segal and his colleagues invented Equal-Channel Angular Pressing, which is also known as Equal-Channel Angular Extrusion, in the early 1980's, that is a suitable method for creating extreme plastic tensions, works based on creating simple cuts on the thinner layer of the clashed page of two channels with equal dimensions. The main purpose of this invention was to introduce a deformation method through which that could make possible the creation of large amounts of plastic strain (shear typed) in the substance. Then in the early 1990's Semiatin and his colleagues proved that this method had the capability of preparing UFG materials. This method gained a lot of attention to itself for the preparation of microstructures using severe plastic deformation technique. In this process by applying pressure to the piece via hydraulic press and passing it through a mold containing two channels with equal cross sections with an angle which had been defined beforehand (in this research a mold with an angle of 120 ° was used) come together, and severe plastic deformation occurs. This deformation occurs in the page encounter of the two channels. One of the benefits of this method is the possibility of the preparation of a lot of deformation of the simple shear form by using single or multiple step process without the need to modify mold cross section [2].

Generally, molds that are used in this method are obligated to have two channels with the same cross section (Figure 1).

The clash between these to channels result in forming an angle that creates the main angle of the mold. 90 and 120 degrees molds are the most used form of ECAP. By press pressure, work piece is entered through the vertical channel and at the time of entering the horizontal channel (in 90° molds), grains of the work piece are broken into finer microstructures and are converted into fine grains [3].

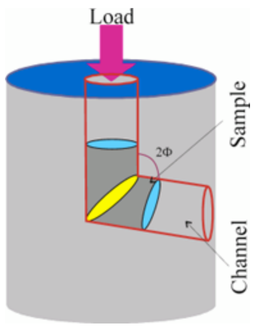

Figure 1. The process of ECAP [4].

2. RESEARCH METHOD

2.1 Materials

In this study according to the diameter of the ECAP mold available, copper coiled steel with special application from Kerman Bahonar Co. with a diameter of 10mm (99.9% purity), as the most suitable option was prepared. To understand the most effective factors (elements) on the hardness, grain size distribution and conduction; quantometric test was taken from the sample.

For performing this test, a device named Spark Emission Spectrometer, a production of Spectrolab Co. of the United States of America, owned by the Metallurgy Laboratory of Sharif University, was used. To study the vulnerability of repeated tests, and the device in use, sparking was repeated again (in different areas) which results of each one have been mentioned in one line of Table 1.

Table 1. Quantometric results show the high purity of copper produced.

Si	Ni	Fe	Mn	P
%	%	%	%	%
< 0.00040	< 0.0020	0.0041	< 0.00020	< 0.00010
< 0.00040	< 0.0020	0.0044	< 0.00020	< 0.00010
Cd	Sb	As	Cr	Mg
%	%	%	%	%
< 0.00010	< 0.00011	< 0.00010	< 0.00010	< 0.00010
< 0.00010	< 0.00010	< 0.00010	< 0.00010	< 0.00010
Be	S	Al	Co	Bi
%	%	%	%	%
< 0.00010	0.0012	< 0.00070	< 0.00060	< 0.00010
< 0.00010	0.0012	< 0.00070	< 0.00060	< 0.00010
Cu	Ti	B	Zr	
%	%	%	%	
99.975	< 0.00010	< 0.00010	0.00036	
99.976	< 0.00010	< 0.00010	0.00039	

2.2 Thermal Process

In order to make sure of the strength perspective of copper pieces with the available ECAP mold, tension test was done.

But the results of the tension test was not appropriate; due to the cold working and the accumulation of stress on copper pieces during the preparation of coiled steel throughout the production process, the sample was prepared for tension test, and the result was the occurrence of brittle fracture.

If these samples were placed in the ECAP mold, severe damage would have been caused to the ECAP mold. For this particular purpose, for destroying the cold working and stress stored in it, the copper pieces should be annealed. Due to the importance of the subject of research, to reduce the non-measurable factors influencing the results, after studies were done, the decision was made to prepare the heat treatment under vacuum in furnace. According to the references and the standard table, to achieve the softest structure, the highest annealing temperature for pure copper is (650 °C), and the annealing time for pure copper (90 mins) was considered [5]. After evaluation was done, heat treatment using furnace under vacuum made by The United states of America Materials Research Furnaces Co. owned by the Scientific Research Town of Isfahan, was used.

The vacuum pump of the furnace had the capability to generate vacuum quality of 10^2 mbar. To refine the entire atmosphere of the furnace and to free it of all gas elements remained from previous work cycles, Argon gas grade 5 (99.999 purity) was charged into the blast furnace and then vacuumed. For better results, the test was repeated.

2.3 The ECAP process and its theory

Considering the application of pure copper (spark tools or spot welding electrodes) the following is a review of available processes for hardening and increasing the strength of this metal.

Perhaps the first method to reduce the metal abrasion that comes to mind, is hardening them via heat treatment, resulting in the formation of martensite and is hardened; but because of the heat treatment (quenching) residual stresses is caused in the piece, if this piece was used as a tool in the EDM process, due to the existence of these tensions it is quickly worn away, subsequently this method is not suitable for strengthening spark tools [6]. The other reason that heat treatment is not suitable, is because this method needs a second element, therefore during quenching it will deposit as a solid solution, which will result in the hardness increasing. Any irregularity in the atomic frequency network of copper would result in the electrons spreading apart and causing electrical conductivity to decrease. Element solutions in the copper network with different atomic size causes areas with localized elastic strain, which causes the reduction of conductivity.

For increasing copper hardness, three methods have been studied:

1. Adding phosphorus to copper leads to the increase of its hardness; but phosphorus leads to the reduction of electrical conductivity, having this feature is not suitable for such a piece capable of the mentioned applications. Also it should be mentioned that such an alloy does not have the heat treatment capability.
2. Adding aluminum to copper allows it to be heat-treated. In the best case, copper alloy containing 11.8% aluminum can be hardened up to 240 Brinell by quenching and then immediately re-heating up to 30mins, but this process severely reduces its electrical conductivity [5&7].
3. Cold work is another method for increasing the hardness of metals. During cold work process in each step depending on the type of treatment the grains are stretched or made finer which this increases the hardness of the metal. Using the ECAP process for making the structure of copper tools smaller, and increasing the strength of this widely used metal is the first suggestion of this study. By using this method in each step of the process the metal grains can be reduced in size over and over again. The process that leads to fine grain, results in the increase of hardness and also appears that during metal wear, which ECAP process had been applied to it, smaller particles from the tool and the work piece separate, compared to the metal with coarse grain size. This issue can be effective and reduces the wear of spark tools and spot welding electrodes [6].

In this study pure copper is fine-grained via using the equal-channel angular pressing process. These types of structures can be made using severe plastic deformation methods. Such methods are designed in a way that the dimension of the sample does not change and remains fixed while the process is taking place. Extrusion in equal angular channels (ECAP) is a process used to increase the strength of metals. In general molds used in this method, are required to have two equal channels. The clash of these two channels together, creates an angle that is the molds main angle. 90° and 120° molds are the most used ECAP molds. Via press pressure, piece work enters the vertical channel and when entering the horizontal channel (in 90° molds) grains of the work piece are broken into smaller structures and are fine-grained [3].

Equal-channel angular pressing (extrusion in angular equal channels), is a decent process to produce a material with fine structure, high strength, and suitable physical properties. This fine-grained structure is produced by severe plastic deformation. The ECAP process acts in such a way that throughout the process, the dimension of the sample remains the same and does not change.

Basically, the design of the ECAP mold is done in such a way that it has two channels with equal cross sections via the colliding angle of Φ and an external curvature angle of Ψ are attached to one another. While the substance passes through the mold and is compressed towards the inside, at the cross section of the two channels, severe shear deformation of the substance occurs. By the samples cross section staying fixed, in each pass and by the strains being reserved in the substance, grain boundaries are changed and result in the size reduction of grain networks and change the structure of the sample. Strain equal to (ε_N) after N stages (repeated tests on the sample) of the ECAP process, equation 1 is calculated [3, 8, 9].

$$\varepsilon_N = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi\csc\left(\frac{\phi}{2}+\frac{\psi}{2}\right)\right] \quad (1)$$

In this research an ECAP mold owned by the Department of Mechanical Engineering of Isfahan University of Technology was used. The mold had an angle of 120° and a diameter of 10mm. The mold is made of the steel of X 153 Cr Mo V12 cold working tools with a standard of DIN 1.2379. Figure 2 shows the schematics of the mold that was used.

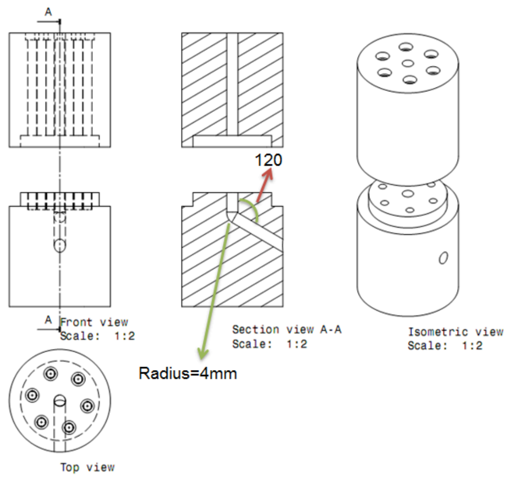

Figure 2. Map of ECAP mold that was used

Figure 3. Copper work piece Exiting the mold.

By employing 100-ton hydraulic press owned by the Islamic Azad University of Najaf Abad, copper pieces under pressure were guided into the ECAP mold. Due to the molds channel length (160mm), if 3 copper work pieces were placed consecutively, large amount of force would be applied to the mold. To reduce the applied force of the press being applied to the matrix walls and the molds mandrel, lead pieces were used to bring out the copper pieces (because of the higher plasticity of lead compare to copper the exerted force was reduced). Furthermore, to reduce the adhesion force between the copper pieces and the matrix walls 20% molybdenum disulfide compound in lubricant was used. The way that the piece is fitted into the mold is called route of processing. In this study route B_c was used, that according to it, each time the pieces exited the mold, they were rotated in line with the axis 90° and for being processed in the next several repeated tests were put back into the mold [10]. By repeating the process, pieces with repeated test amounts of 2, 4, 6, 8, and 10 were obtained.

2.4. Metallography

Pieces complying with the following standards: Preparation of metallography samples[2], micro etch of metals[3], optical microscope[4] images were prepared for the metallography test. The etching agent was made from combining hydrochloric acid, ferric chloride and alcohol.

The images obtained from the optical microscope are shown below (Figure 4 - 9).

[2] ASTM E 3-11:2013
[3] ASTM E 407-07$^{\varepsilon 1}$:2013
[4] ASTM E 883-11:2013

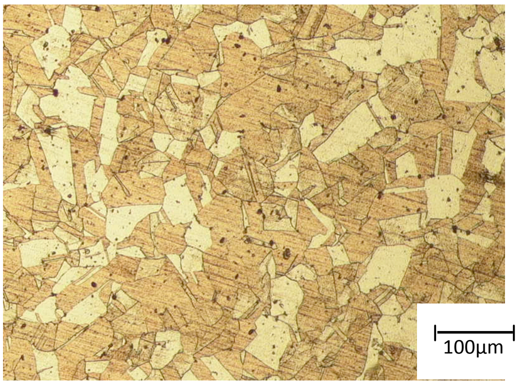

Figure 4. Piece not gone through ECAP process

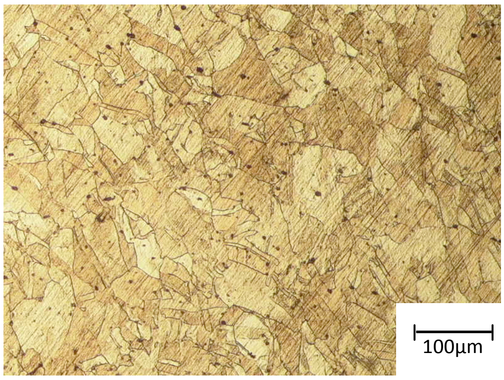

Figure 5. Piece processed through 2 stages of ECAP

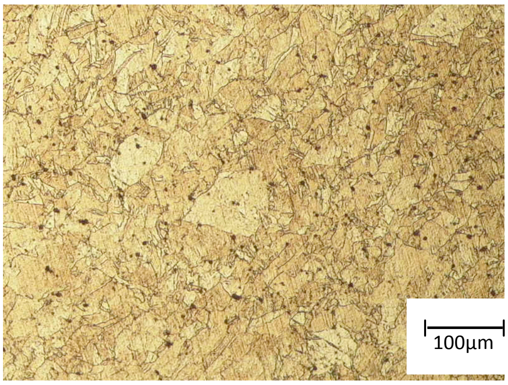

Figure 6. Piece processed through 4 stages of ECAP

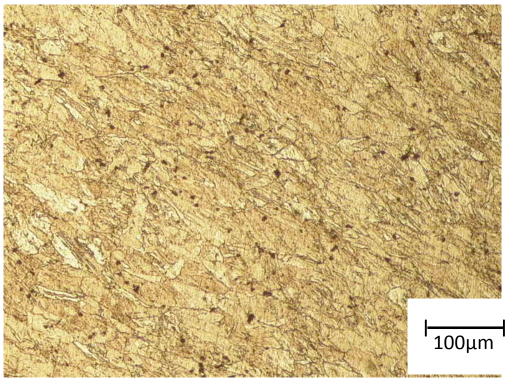

Figure 7. Piece processed through 6 stages of ECAP

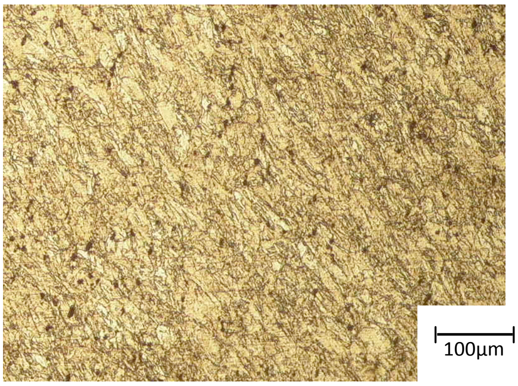

Figure 8. Piece processed through 8 stages of ECAP

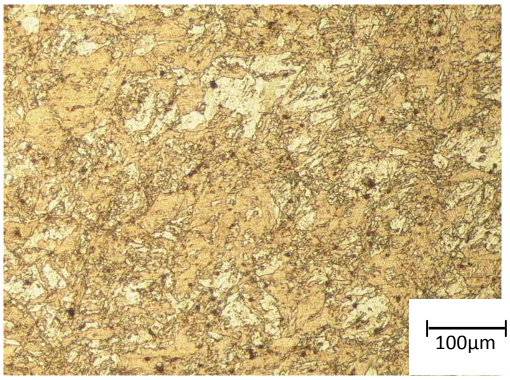

Figure 9. Piece processed through 10 stages of ECAP

3. DİSCUSSİON AND CONCLUSİON

In Figure. 4 – 9, the only existing phase is Alpha. As it is observed by the increase of each piece being tested several times, the grain size is reduced to finer grains. This increases the metal strength.

In repeated tests of 6, 8 and 10 (each piece tested repeatedly) due to the increase of mechanical work on the copper pieces dislocation–twin boundary strengthening mechanism are formed. Due to differing grain boundaries in different planes, the etching agent will not be able to identify some of the grain boundaries.

On the other hand, the ECAP process has been able to coordinately reduce grain size and also reduce the difference between the smallest and the biggest grain size. For example, the ECAP piece that has not been processed has an average grain diameter size in the range of 31.8 – 63.5 μm, which this amount is 2 – 8 μ for a piece that has been through 8 stages of the ECAP process.

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