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# Investigation of Mechanical and Microstructure of Fine Graincopper via Friction Stir Processing Method

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## Abstract

**Objectives:** In this study, Friction Stir Processing method is used to decrease grain size and increase the materials strength. **Methods/Analysis:** Produced with this process are the production of parts with fine-grained structure without porosity, being environmentally friendly, availability and ease of having mechanical properties. Application of coarse grain copper in medical science is an important problem, because release of dangerous materials during the time, while this problem could be removed by decrease of grain size and increase its applications. Process varying conditions are included as size of tools shoulder, speed and number of passes, each one possesses its specific effects. **Findings:** By increasing of improvement speed and number of passes besides decrease of rotational speed, grain size decreased while strength and consolidation increased. **Novelty/Improvement:** Investigating the effects of friction stir processing on the mechanical and microstructure changes of copper.

**Keywords:** Dynamic Re-Crystallization, Fracture Strength, Hardness, Pure Copper, Severe Plastic Deformation

## 1. Introduction

Fine grain materials are paid so attentions because of having desirable mechanical properties and some methods are introduced for production of these parts. Fine grain materials are produced in a two general methods including bottom-up and top-down approaches. In the bottom-up approach, arrangement is done atom-by-atom and layer-by-layer to form a nanostructure. The problem of this method is about creation of porous structures which cannot be applied in the industry. In the top-down approach, the coarse grain material is converted to a nanostructure, by severe plastic deformation[1]. The basis of severe plastic deformation is about increase of dislocation density via a severe uniform deformation of material and formation of dense walls of dislocations and conversion of dislocation walls to the high angle borders. The materials produced by the severe plastic deformation because of having porosity-free structures, high mechanical properties like higher strength and toughness and also having proper dimensions for physical and mechanical testing are so considerable.

Because of high ratio of strength to weight, these parts have proper conditions to be used in the different industries like Automobile. There are many methods applied for severe plastic deformation including: Equal Channel Angular Extrusion (ECAE) (also known as Equal Channel Angular Pressing (ECAP)), Accumulated Roll Bonding (ARB), High Pressure Torsion (HPT), Repetitive Corrugation and Straightening (RCS), Constrained Groove Rolling (CGR), Constrained Groove Pressing (CGP) and Friction Stir Processing restricted in pieces.

Among the available techniques, use of High Pressure Torsion (HPT) and Equal Channel Angular Extrusion (ECAE) are infeasible for production of fine grain parts in the form of sheets. Although the Repetitive Corrugation and Straightening (RCS) is designed for production of fine grain material form a sheet. In this way creation of successive connections among the sheets is required, so that if one complete connection would not be created among the sheets while rolling, the remained interface can cause decrease of mechanical properties. In the Constrained Groove Rolling process, desired mechanical properties

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are not obtained, probably because of the bending created while deformation.

Friction stir processing is a solid state technology for modification of microstructure and mechanical properties of materials[3]. During this process, temperature of stirring area does not reach the materials melting point, but the severe Dynamic Recrystallization (DRX) is happened in the zone and the materials changes to a semi-solid and paste form. This process is done using the Friction Stir Processing (FSW).

So that the tools with specific geometry including pin and shoulder are entered into the surface of the desired metal by high rotational speed and submerged in a depth somehow lower than pin length. The shoulder tools is connected to the metal surface and the created temperature caused by the friction of tools shoulder and metal surface and mixing of materials by pin, causes the formation of a paste in the treated zone and on the other hand by simultaneous forging and extrusion occurred at the stirring zone while improvement of the tools, the materials undergoes a severe Dynamic Recrystallization and a homogenous, fine grain, defect free and uni-axial structure is formed in the material.

Regarding the ongoing growth of the modern technologies in the world, the simplicity, accessibility, low cost and being environmentally friendly of these technologies are from the main factors in selection of methods of work[2]. Friction Stir Process has unique characteristics in this case which put it in the first level for implantation. Friction Stir is a completely green process (environmentally friendly) that because of requiring low cost and accessible equipment (milling system and tools made by the researcher) is paid considerable attention by the researchers in the past decade.

This method can create considerable reformations in the materials structure in the low dimensions with simple processes. On the other hand copper is one of the frequently used industrial materials in the world that was always considered because of its high electrical and thermal conductivity. But its main problem was about low strength and stiffness which lead to fatigue phenomenon in the structure during the time[4,5]. Because of that, investigation of effects of friction stir processing on the mechanical and microstructure changes of copper can open the doors for the future investigations.

The purpose of this research was to decrease the size of copper grain to control its mechanical properties and increase of copper hardness for increasing its resistance to wear, corrosion and fatigue.

## 2. Materials, Tools and Apparatus

### 2.1 Materials

In this study, the pure commercial copper (99.9%) was used as the base material. The 6 mm thick rolled copper sheets with dimensions of about 100 × 6 were prepared. As shown in Figure 1 in the four sides of the mentioned parts, 8 mm diameter holes were embedded. The 20 mm thick steel sheet (St-37) with dimensions of 250 × 250 with flat and grounded surface was used as the samples holder, so that by means of four M8 screws, they were attached to the four sides of the steel plate. Table 1 shows some of the general physical and mechanical properties of the pure copper.

### 2.2 Tools of Friction Stir Process

In this study, tools with shoulder diameter of 20 mm and a cylindrical pin with diameter and height of about 5 and

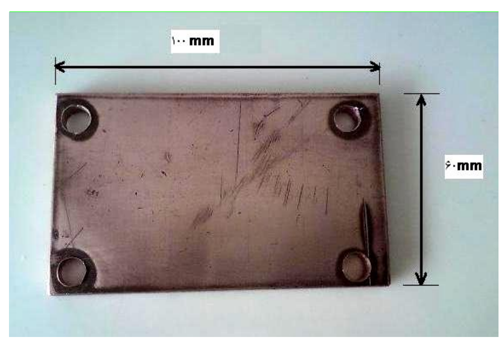


**Figure 1.** Image of a copper sample with holes for being fixed on fixture.

**Table 1.** Physical and mechanical properties of pure copper

| Material | Density (g/cm3) | Melting point (C°) | Coefficient of linear expansion (1/K 6-10) | Coefficient of thermal conductivity (KJ/m · h · K) | Thermal conductivity Ω.m))/ 108 | Vickers hardness (HV) | Tensile strength (MPa) | Pressure strength (MPa) |
|---|---|---|---|---|---|---|---|---|
| copper | 8.96 | 1083 | 16.6 | 1384 | 58 | 80-90 | 216 | - |

3 mm were provided. The type of these tools were from hot work steel (H13) that using heat treatment, they were hardened to 54 Rockwell.

Figure 2 shows an image of Friction Stir Process tools used in this process. The available grooves in the environment are embedded for increasing the tools body surface with the air that causes more heat transfer from the body and also by preventing the increase of upstream temperature of the tool, prevents the damage of the system[2].

Also, a tool with the same characteristics but with different shoulder diameter (from 20 to 14 mm), was also prepared to investigate the effect of internal temperature on the samples.

### 2.3 System for Friction Stir Processing

In this study, the vertical milling system was converted to a Friction Stir Processing system by addition of fixture on the apparatus desk. Fixture is designed so that becomes able to keep the parts fixed on the forces caused by the processing. The milling machine used in this study is a Universal FP4M model, made in Tabriz. Figure 3 shows this apparatus together with its tools and embedded parts.


**Figure 2.** Friction Stir Processing tool with the shoulder diameter of 20 mm, used in this study.

**Figure 3.** A view of the apparatus, tools, fixture and embedded part,

In order to implementation of Friction Stir Process, fixture was put on the system desk and closed firmly to prevent movement of the fixture caused the vibration caused by the tools movement into the parts. In the next step, tools fixed on the system are conducted to the spices with low speed and then the tools penetrated the spices slowly for 0.4 mm.

Figure 4 shows the steps of tools movement into the working spices and also the finalized sample of the work. After a short stop of 20s, the ongoing movement in the longitude path is started and finally, the tool is pulled out of the spices (Figure 5).

## 3. Investigated Parameters of the Process

In order to investigate the parameters affecting the microstructure and mechanical characteristics of the samples and selection of the proper parameter for manufacturing of perfect samples, different parameters are used in implementation of process, including:

### 3.1 Progress Speed

The speed of progress in the study ranged from 40 to 315. The changes were applied to achieve the desired conditions in the construction sample.

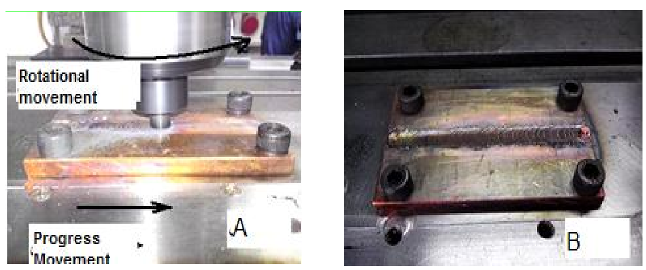



**Figure 4.** Image of copper sample, **(a)** While ongoing operation of tools on the spices, **(b)** After completion of work.

**Figure 5.** Images of the created samples via Friction Stir Process.

### 3.2 Rotational Speed

The rotational speed of the tool in this research varied in the range between 710 to 1120. The rotational speed of the tool is provided by rotation of spindle of milling system. Table 2 shows the different progress and rotational speeds rotational speeds used in this study.

### 3.3 Number of Passes

To investigate the effect of passes on the materials mechanical and microstructure properties, samples were prepared using one and two passes. In this way, the process is repeated by the number of passes, in the direction of past pass and with the same rotation direction.

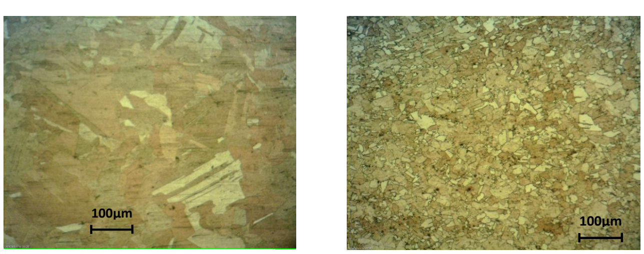
## 4. Results and Discussions

### 4.1 Microstructure Analysis

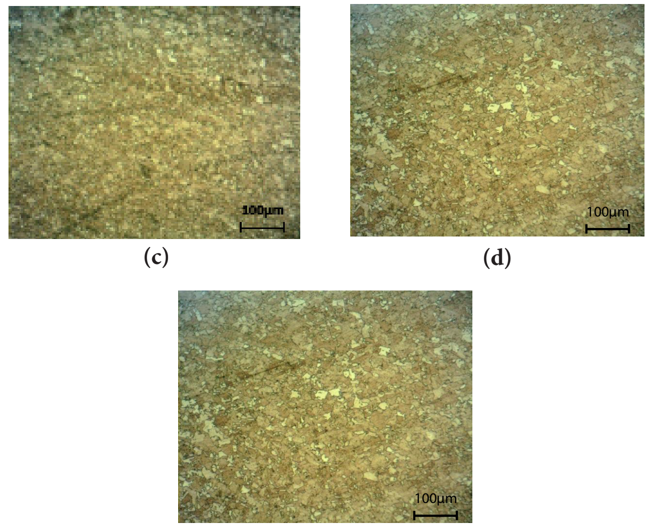
Figure 6 shows the stir zone of the process using a light microscopy in terms of process (rotational speed and tools progress). Figure 6 (a) shows the microstructure of pure copper, so that the picture is clear and amorphous region include coarse grain respectively. While Figure 6 (b) involves a fineness and homogeneous microstructure, respectively. Grain size reduction in Friction Stir Processing samples due to continuous Dynamic Recrystallization of plastic deformation and severe mechanical stresses simultaneously with increasing temperature due to frictional contact of the shoulder tools and copper sheet and continuous flow of material is located in the stir area.

**Table 2.** Variation domain of rotational speeds in this study

| Final speed | | Initial speed | |
|---|---|---|---|
| 1120 | ← | 710 | Rotational speed (rpm) |
| 315 | ← | 40 | Progress speed (mm/min) |




(a) (b)




(c) (d)

(e)

**Figure 6.** Micrograph of the stirring area in the process, **(a)** The base material, **(b)** And **(c)** Rotational speed of about 710 rpm and progress speed of 40 and 160 mm/min, respectively, **(d)**, **(e)** Rotational speed of 1120 rpm and progress speeds of 40 and 160 mm/min, respectively.

These reasons led to the creation of new nucleation sites and by increase of the number of grains in the stir zone, density of grain boundaries has also increased and grain growth has been stymied and delayed, thus grain size is considerably reduces. Another important parameter affected the grain size is the heat input in this area during the process and increased the size of grains during the annealing process[7]. But as results have shown, the dominant factor during the process, which reduces the size of particles is the effect of Dynamic Recrystallization. Figure 6 (b) and Figure 6 (c) included materials fine structure at constant rotational speed of 710 rpm and progress speeds of 40 and 160 mm per minute. Obviously in both constant rotational speeds, grain size in the stir zone has considerably decreased by increase of progress speed of the tools. Two main factors affect the grain size in the stir zone:

- Dynamic Recrystallization which leads to nucleation and increase of dislocation density and as a result prevents the sliding of grain boundaries and reduces the grain size.
- Annealing effect of the input temperature which leads to formation of coarse grains.

At higher progress speeds, effect of the first factor is intensified, because by increase of the progress speed, material undergoes a severe mechanical compressive strength and experiences a severe Dynamic Recrystallization which leads to increase of nucleation sites and raises the number of grains and as a result reduces the grain size. Also, increase of tools progress speed leads to decreasing the effects of the second factor and input temperature is reduced because the tools have passed the region with a higher speed and duration of stir process is considerably decreased.

Decrease of annealing effect is caused by reduction of input temperature while grain size is not raised. Regarding mentioned points, it could be said that by increase of the tools progress speed, both factor affecting the grain size are considerably reduced which leads to considerable reduction of grain size[9,10].

Figure 7 shows the SEM images of the stirring area of the prepared sample with the rotational and progress speed of about 710 and 315 rpm. Obviously, grain size in this region is considerably decreased and from about 45 micrometer for the base metal was decreased to about 5 micrometer for this sample, which was happened because of the high progress speed of the sample and intensity of the first agent on the grain size.

Figure 8 shows that microstructure of the stir area via light microscopy in different conditions of rotational and tools progress speed. The processing zone in this case shown in Figure 8 (a-d) is composed of a microstructure with fine and uniform grains. Reduction of grain size in

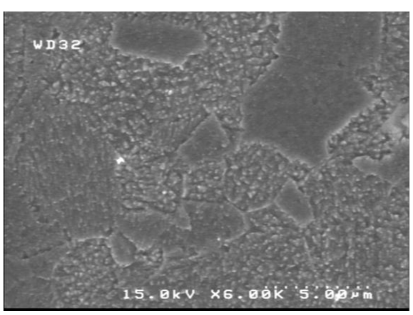
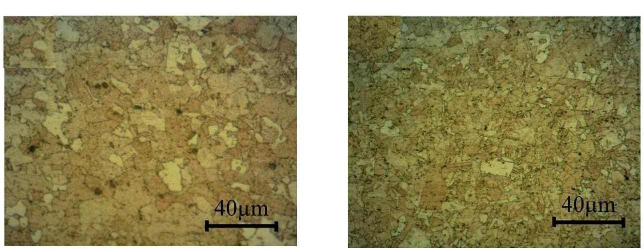
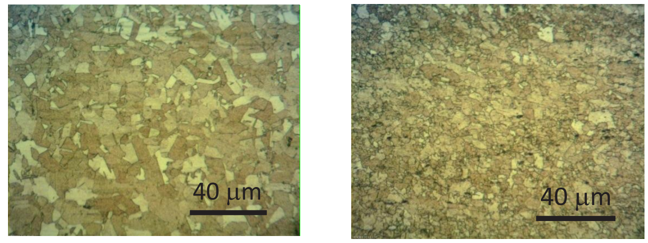



**Figure 7.** SEM images of the stir region of the process in the rotational speed of 710 rpm and progress speed of 315 mm/min.




**(a)** **(b)**




**(c)** **(d)**

**Figure 8.** Micrograph images of the stir zone of the process produced by **(a)** 14 mm tools and **(b)** Rotational speed of 710 rpm and progress speed of 40 and 160 mm/min, **(c and f)** Rotational speed of 1120 rpm and progress speed of 40 and 160 mm/min.

the samples undergo the friction stir process are located in stir zone because of continuous Dynamic Recrystallization caused by the plastic deformation and severe mechanical stresses simultaneous with increase of temperature caused by friction contact of shoulder tools with the surface of copper sheet and continuous flow of materials. Figures 8 a and b are contained of materials microstructure at the constant rotational speed of 710 rpm and different progress speeds of 40 and 160 mm/min and Figure 8 c and d have shown the materials microstructure at the constant rotational speed of 1120 rpm and progress speeds of 40 and 160 rpm. Clearly, in both rotational speeds, grain size has considerably decreased by increase of progress speed in stir area.

Figure 9 shows a SEM image of the stir area of the produced sample with the rotational and progress speeds of about 710 rpm and 315 mm/min. Obviously, grain size is considerably reduced in this area from 45 micrometer for the base metal to about 3 micrometer in this sample, because of the high progress speed and intensity of the first factor effect on the grain size. Clearly, size of shoulder tools has a considerable effect on the grain size in the stir area and can reduce it.

## 4.2 Effect of Pass Number on the Microstructure of the Samples in Friction Stir Process

Figures 10 (a and b) shows the image of light microscopy of the friction stirred samples with 2 passes using 20 and 14 mm tools in rotational and progress speeds of 900 rpm and 160 mm/min, respectively. Obviously, increasing the number of passes leads to considerable reduction of grain size and also the formed structure at the higher passes is a complete uniform and uni-axial structure[8].

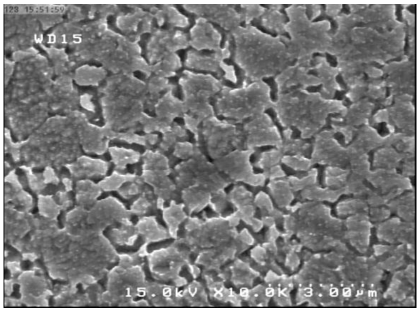



**Figure 9.** SEM image of the created stir zone by the tools with shoulder diameter of 14 mm at rotational speed of 710 rpm and progress speed of 315 mm/min.

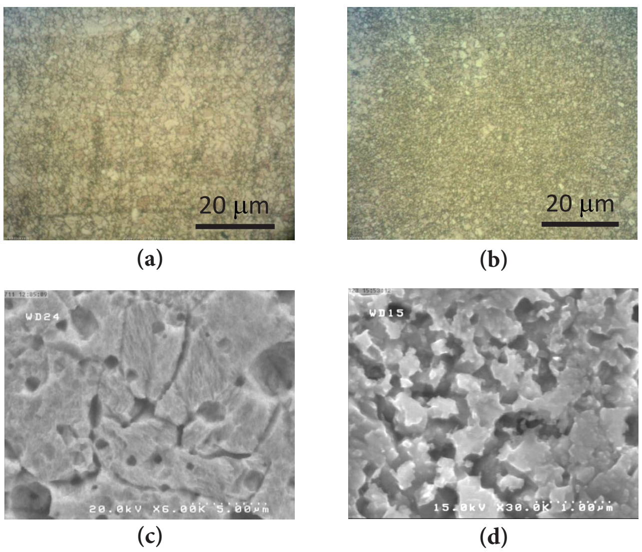



**Figure 10.** **(a and b)** Metallographic images of the samples produced by 2 passes and tools of 20 and 14 mm, respectively, **(c and d)** SEM images of sampled produced by 2 passes with 201 and 14 mm, respectively.

Table 3 shows the changes of grain size in different passes during the Fiction Stir Process. Obviously, a severe decrease of grain size in 2 passes of both shoulders with shoulder diameters of 14 and 20 mm are shown. This can show that by increase of pass numbers, grain size have reduced and the main reduction of grain size are happened in the initial passes. This reduction for the first pass is about 85%, while in the second pass it's about 40%.

For investigation of samples hardness, the Vickers microstructure evaluator was used, so that in the distance of 1 mm from processes sample surface, about 10-14 diffusion points were embedded to study the materials hardness behavior in the width of the regions of HAZ, TMAZ and SZ.

**Table 3.** Variation of grain size with number of passes in the samples produced via Friction Stir Process

| Diameter of shoulder tools (mm) | Number of pass | Size (micrometer) |
|---|---|---|
| 20 | 1 | 9.3 |
| | 2 | 4.3 |
| 14 | 1 | 7.5 |
| | 2 | 1.8 |

Figure 11 shows the variation of Friction Stir Processed materials at the constant rotational speed of 900 rpm and different progress speeds. Obviously, materials produced in these conditions have interesting hardness behavior, so that microstructure of the samples by 40 and 80 mm/min progress speed, in relation to the basic hardness of the copper, 55 Rockwell, has decreased in the stir zone. This phenomenon happened while grain size in these samples showed reduction in relation to the basic metal. Generally, main factors in behavior of produced samples can be attributed to:

### *4.2.1 Grain size*

So that by decrease of grain size, because of the Dynamic Recrystallization effect in the grain boundaries movement region and dislocations becomes more difficult because of increase of density and as a result, materials hardness is increased.

- *Annealing Effect*

Input heat to the spices causes softening and reduces materials hardness.

In the sample produced with the progress speed of 160 mm/min, samples hardness was increased in relation to the base metal and it could be said that the dominant factor in control of materials hardness behavior, is the first factor named grain size reduction. In other words, in this sample because of high progress speed, input temperature was severely decreased leading to weakness of the effect of second factor in control of materials hardness.

Figure 12 shows the trend of variation of samples hardness with variation of tools rotational speeds. As mentioned before, at the progress speed of 40 mm/min, hardness of the samples were lower than the base copper metal. Obviously, by reduction of rotational speed from 900 to 710 rpm, materials hardness was increased and

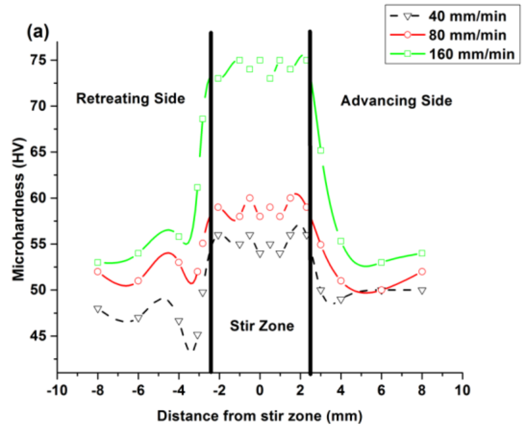



**Figure 11.** Variation of micro-hardness at different progress speeds for the samples processes via Friction Stir Methods by 20 mm diameter tools.

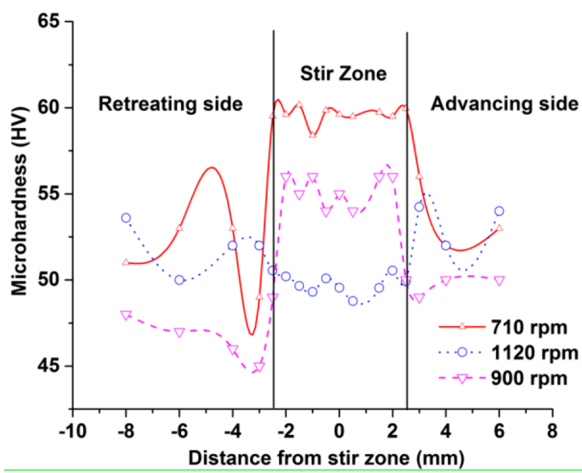



**Figure 12.** Graph of samples hardness showing the effects of rotational speed on the hardness of processed samples with progress speed of 40 mm/min.

also by increase of rotational speed to 1120 rpm, hardness is decreased. It can be interpreted so that as mentioned above, different factors affect the materials hardness including grain size, temperature and density of dislocations[6]. In this section, it should be said that by decrease of grain size and input temperature, hardness is increased but for the samples with higher rotational speeds despite more stirring and higher Dynamic Recrystallization at upper regions, but the input temperature is considerably increased and makes the last factor useless.

- Micro-hardness behavior of samples processed by friction stirring via tools with 14 mm of shoulder diameter.

Figure 13 shows the hardness variation trend of the samples produced by tools with 14 mm of shoulder diameter at the constant rotational speed of 900 rpm and different progress speeds. Clearly, despite the samples produced by 20 mm tools diameter, no reduction of hardness was observed in these samples even at the low progress speeds where production temperature is high.

These results have shown that the input diameter of the processed zone is important for control of hardness. On the other hand, as shown in microstructure part, samples produced by 14 mm tools have a smaller grain size distribution in relation to the samples produced by 20 mm tools, which is a positive factor for increase of hardness.

In addition, investigation of these figures have shown that in such diameter, by increase of progress speed from 40 to 160, samples hardness showed a considerable increase, which was more sensible than the increase happened in 20 mm sample. In reality, effect of increase of progress speed is more sensible at the lower shoulder diameters, which is caused by low input temperatures to the processes zone, by using the tools with smaller shoulder diameter.

Figure 14 shows the effect of rotational speed on the hardness of the samples produced by tools with 14 mm of shoulder diameter. Obviously, for these samples also the effect of rotational speed is similar to that of samples produced by 20 mm tools that show a considerable increase by reduction of rotational speed and by increase of rotational speed, no change was happened in the samples hardness.

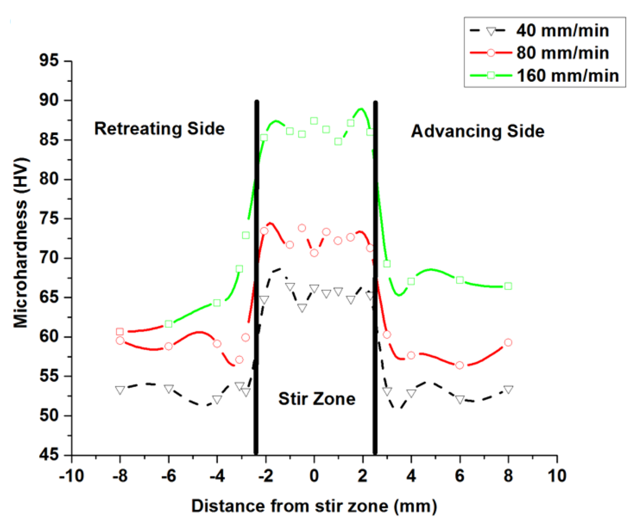



**Figure 13.** Graph of samples hardness showing the effect of progress speed on the hardness of processed samples with rotational speed of 900 rpm.b

- Effects of pass numbers on the hardness behavior of samples processed by the friction stir method.

Figure 15 shows the hardness variation of the samples produced by Friction Stir Process via 20 mm tools by increase of pass numbers to 2 in the rotational and progress speeds of 900 rpm and 160 mm/min, respectively.

This figure shows that by increase of pass numbers, samples hardness show a considerable increase, because by increase of pass numbers, as mentioned before in the microstructure topic, grain size shows a severe decrease

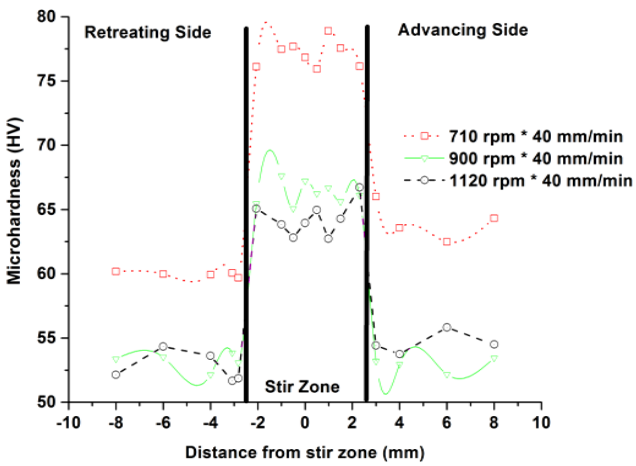



**Figure 14.** Samples hardness produced by 14 mm tools and the effect of rotational speed on the hardness of the samples processes by 40 mm/min of progress speed.

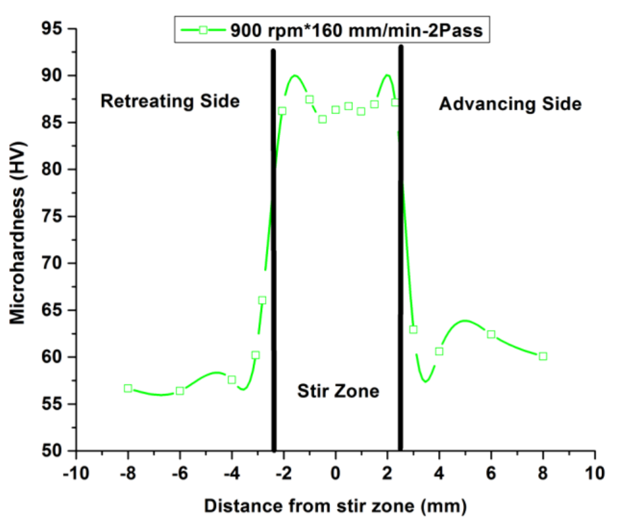



**Figure 15.** Hardness of the sample produced by 20 mm tools under 2 passes of Friction Stir Process with the rotational and progress speeds of 900 rpm and 160 mm/min, respectively.

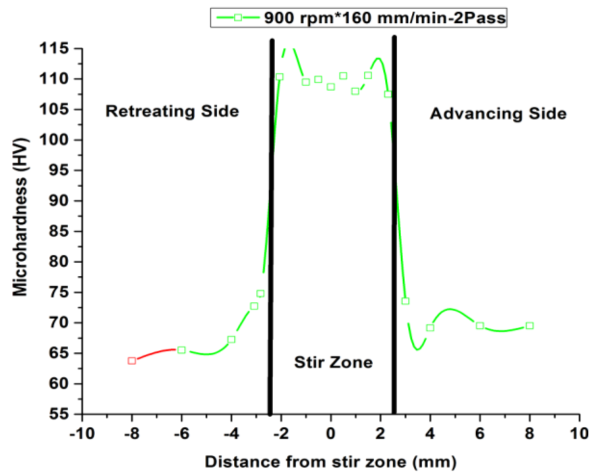



**Figure 16.** Hardness of the sample produced by 14 mm tools at two passes under Friction Stir Process with rotational and progress speeds of 900 rpm and 160 mm/min.

which leads to increase of grain boundaries resistance and dislocations against the sliding and transporting, which is a mechanism leads to increase of samples micro-hardness.

Figure 16 shows the hardness variation of samples produced via Friction Stir Process with 14 mm tools by increase of pass numbers to 2 at rotational and progress speeds of 900 rpm and 160 mm/min. Obviously, increase of hardness is more sensible in the sample produced by 14 mm tools because of increase of pass. The reason is about the very small grain size in these samples which leads to increase of grain boundaries density and materials hardness.

## 5. Conclusion

In this paper, the friction stir process was done on the pure copper to modify the microstructure and mechanical properties. By change of the process conditions, optimum conditions were obtained for modification of surface structure and gaining the desirable mechanical properties in the pure copper. The parameters used for investigation of microstructure and mechanical properties of produced samples were included of: progress speed, rotational speed and number of passes.

Obtained results can be concluded in the cases mentioned below:

- The Friction Stir Process can considerably reduce the grain size in the produced samples.

- Grain size in the Friction Stir Processes samples is independent of diameter of tools shoulder with increase of progress speed and decrease of rotational speed.
- The rotational speed during the Friction Stir Process is more effective than the rotational speed for control of grain sizes.
- Decrease of the tools shoulder diameter leads to severe decrease of the input temperature of the stir zone and severely decreases the grain size.
- Increase of the number of passes of Friction Stir Process leads to decrease of grain size and increase of samples hardness. Also, the obtained structure at the higher passes has a uniform morphology and uniaxial grains.
- Samples hardness is increased by the decrease of grain size, except of the cases where the input temperature to the zones was high and annealing is the dominant factor.

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