ISSN (Print) : 0974-6846
Indian Journal of Science and Technology, *Vol 9(7), DOI: 10.17485/ijst/2016/v9i7/87778, February 2016* *ISSN (Online) : 0974-5645*

Study the Microstructures of Nano-Composite Copper/zirconium Dioxide under the Process of FSW (Friction Stir Welding)

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Abstract

Objective/Analysis: For the first time, the FSW / PP were used in the aluminum alloys that the recent developments in equipment for operation at higher temperatures, the processhas also been used for copper base alloys, iron and nickel. **Methods:** In this study, a copper plate is used as the base metal, after that by adding ZrO2 ceramic powder to it and using a non-consumable tool for mixing powder in forms one, two and four-pass, Nano-composite Cu/ZrO2 has been created. Thenthe mechanical properties of the created Nano-composites were examined and compared with the base metal. Also in another part of this study, a samplewas prepared with the same conditions but without using ZrO2 powder.And in order to compare with other samples, its properties were studied. **Findings:** The results of abrasion tests, micro-hardness and micro-structure reviews which were done by using optical and electron microscopes showed that incomparison with base metal, a very desirableabrasion and hardness properties are obtained and fine aggregate copper which already has been considered is fully achieved.It should be noted that in the case of a four-pass process along with using powder, the processed composite layer has more uniformity and homogeneity and higher mechanical properties than the base metal and powder-free samples. Based on the results, the maximum amount of hardness is about 160 Vickers which was obtained in four-pass mode and by using the powder, in compared with the base metal hardness which was about 80 Vickers, a twofold increase is shown. **Application/Improvements:** The lowest coefficient of friction is associated withthefour-pass process sample by usingpowder, which has the amount of47/0; it hasdemonstrated a significant improvement about abrasion propertiesin comparing with the friction coefficient of the base metal which was about 81/0.

Keywords: Abrasion Test, Copper, Friction Stir Processing, Microstructure, Nano-Composites, Zirconium Dioxide

1. Introduction

Friction stir processing is a method of article commotion which has recently been developed based on the principles of friction stir welding, with the difference that goal of this process is not to connect two pieces of metals with each other but to change and modify the microstructure and improvement of mechanical properties of materials as a result. This solid state process can cause the creation of homogenized granulation and also reduce the porosity of hydrogen that may occur during the production of the alloys. A non-consumable tool is used in the process which by a circular movement and appropriate onrush gets into the seam that is filled with amplifier powder and resulted in mixing without melting the material[1]. Figure 1 shows the overall picture of this process.

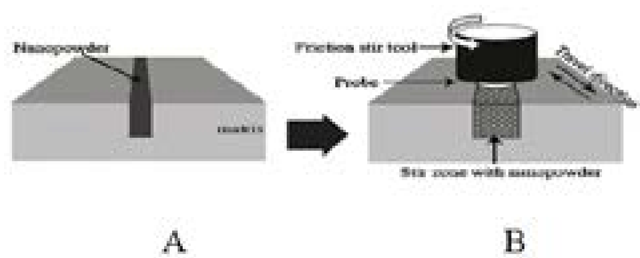

Figure 1. The overall picture offriction stir processing
A) The seam filled with amplifier nanoparticles.
B) Applying the friction stir processing process.

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The instrument used in friction stir processing consists of two parts ofthe pin and shoulder. Pin has the task of creating frictional heat and plastic strain in effect, ofinstrument rotation, and then extrusion and the volatilityof the material from the front of the pin to the back in effect of combination of rotating and onrush move of the instrument.So in this part of the tool, we can determine a suitable material for pin to prevent it from abrasion and increase its lifetime, and also by determining the appropriate parameters for the pin, the friction and extrusion of granulated materials can be controlled in effect of heat[2]. The most important role of the shoulder is to focus the frictional heat by preventing the escape of a granulated material away from the turbulent region.During this process,the powder which is spilled along the pin movement line is mixed with granulated material; and in case of optimization of the parameters, it will be mixed into the material quite homogeneous and the composite will be created[3]. The temperature caused by the friction in the process at most gets up to 80% of the melting temperature of the base metal (0.8 TM). This is because the higher temperature the higher softness and pasty material and thus prevent further friction. So by lowering the friction, material temperature will be reduced and the melting of the material will be prevented. Given the importance of the tool effect, tool material should be considered in a manner that can tolerate high temperatures[4]. As with many new technologies,new terminology is used for description of detailed observations;in the process of FSW/P also new terminology is necessary to appropriate and adequate description of the created microstructures after this process. In general, the processed microstructures are divided into four districts as follows:

- Stir Zone: Stir Zone is a full crystallized zone, the material in this zone goes extremely under plastic deformation and the microstructure of granulation can be seen in this zone.
- Thermo-Mechanically affected zone: The distortion of granulation in the zone cause to relent and formation of coaxial granulation in the interfacial of turbulence and thermo-mechanical zone. Also recrystallization doesn't happen in this zone due to the effects of strain[5]. In comparing with stir zone, less heat is applied to the material, but ithas plastic deformation in thiszone.
- Heat-affected zone: This zone is closest to the center of FSW/P and is influenced by the heat that causes to modify the mechanical properties of the microstructure, although plastic deformation does not occur in this zone[6, 7].
- Base Metal zone: This zone is located far from FSW/P zone where there is virtually no deformation. Although this zone may be affected by the generated heat,but has no effect onchanging the structural or mechanical properties of that zone[8, 9, 10].

Figure 2 shows the different zones of microstructure after the FSP.

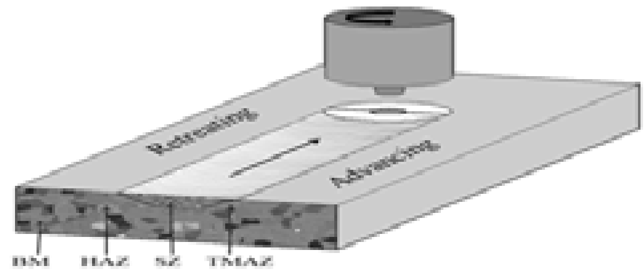


Figure 2. Different microstructure zones in friction stir processed piece.

2. Research Objectives and Hypotheses

The main hypothesis of the study is as follows:

- After the FSP process, the added particles to the context form the surface composite.
- The surface composite which has been formed increases the surface hardness.

The study also follows these objectives:

- Increase the copper usage in the industry while their strength is lower than some other metals.
- Increasethe replacement duration of parts resulting in increased maintenance duration.

3. Materials and Methods

In this study, the FSP process has been discussed that includes the base metals of copper and zirconium dioxide Nano-powder. Then the sample preparation, required equipment including tools, machinery and related variables to each of them are described.In the following, how the process is done on the samples is explained, and the studies and tests done on the processed samples that include metallographyby using optical and scanning electron microscopes and also micro-hardness and abrasion tests are given at the end.

3.1 Base Metal

In this study, friction stir processing is done on sheets of copper with a thickness of 5 mm. The chemical composition of copper used in the process is determined by using emission spectrometry test (Quant metric) and the results are given in Chapter IV.

To carry out the process,at first two copper sheets with dimensions 400 × 70 mm was prepared through a cutting action by milling machines. In an example which is intended to add theamplifier particles, a groove is created with a width of 1 mm and a depth of 2 mm by using a milling saw blade to a thickness of 1 mm in the middle and in the longitudinal direction of the sheet (Figure 3).

After creating the groove on the sample, in order to remove possible corners caused by the fold in the edges of the groove, which causes to holdingand wasting the powder sanding operation has been done. The necessary plans to prepare the plates are given in figures (3) and (4).

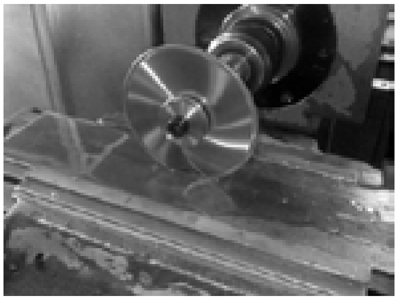


Figure 3. Creating a groove to a depth of 2 mm on the samples.

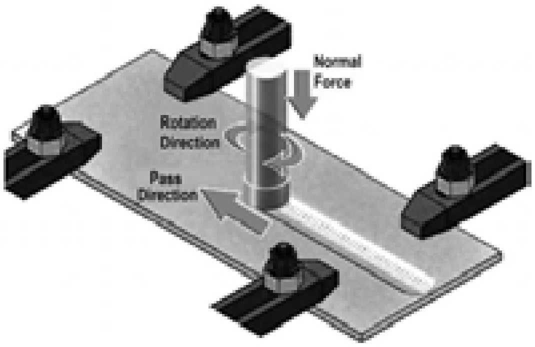


Figure 4. A pattern of the clamping work piece in friction stir processing.

3.2 Machines

In this study, a vertical milling machine is used to do the FSP process. The milling machine is the type of TOS FA5U and its properties are presented in Table 1.

Table 1. Technical characteristics of the milling machines used in friction stir processing

Technical Specifications	Unit of measurement	Value
Length course along the X axis	Mm	1000
Length course along the Y axis	Mm	300
Vertical course along the Z-axis	Mm	400
Rotary axis speed range	RPM	18-2000
Onrush speed range	Mm in minutes	14-320
Engine power	KW	7.5
The overall weight of the machine	Kg	2840
Table length	Mm	1600
Table width	Mm	315

3.3 Method of Processing Performance

The study examines and compares the effect of FSP process without and with amplifier particles on copper samples. At first, the piece of copper without groove was fastened well on the machine worktable by fitclamping. It should be noted that the accurate fasten of the components is very important for a successful friction stir process, because incoming forces are very high in this process and therefore fastening the parts should be in the way that can resist against these forces and prevent their movement during the process. The pattern of clamping the work piece in the FSP process is shown in Figure 4.

The piece surface was cleaned well with alcohol, in order to remove grease, dust and other contaminants. Then the second type tool was fastened in the machine tool holderand 3degreedeviation was added to the machine rotary axis (the head). In this mode, the tool shoulder will penetrate in the sheet about 1.0 to 15.0 mm. Due to the limitations of the research, the rotational speed of 700 RPM and theonrush speed of 14 mm per minute were selected. Also the numbers of tool passes were considered as 1, 2 and 4 pass. In some previous studies, a separate piece is considered for each of tool pass numbers, this requires frequent opening, closing and adjustment of pieces, but in this research, the number of tool passes on a piece is done integrated;so that the 1pass, 2pass and 4pass are respectively considered over throughout the work piece, over two-thirds of the work piece and over a one third of the work piece. An example of the friction stir processing steps without using powder on the copper sheet is shown in Figure 5.

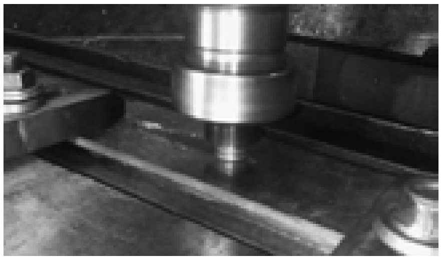


Figure 5. Picture of friction stir processing without using powders on a copper sheet.

After the process on the piecewhich has no groove, the sample was opened the table and the grooved piece that was prepared by adding the amplifier particles was fastened to the table. In order to obtain the correct posture and undeviating move of the pin in the groove, the piece was accurately set in the line of tool onrush movesby using the measurement hour. After setting, fastening and cleaning the workpiece, the piece's groove was filled well with Zirconiaamplifier particles and the first tool was placed in the machine toolholder in order to coverthe groove. By applying a proper vertical load to the spinning tool to penetrate into the layer of the workpiecesurface and then starting the tool onrush, groove got closed and amplifier particles were trapped below the workpiece surface (Figure 6).

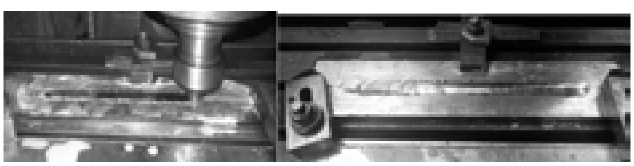


Figure 6. The operation of closing the groove containing the powder.

Then, the second type tool is placed in the machine tool holder. The tool gets closed in the beginning of the groove that contains powder by rotational speed of 700 RPM and get loaded graduallyuntil the pin tool penetrate deep into the workpiece. In this case, a few minutes are given to the rotating and penetrating tool till the material gets in a paste form. Then,in order to perform the friction stir processing, the tool will getonrush speed of 14 mm per minute. A picture of the steps of this process is shown in Figure 7.

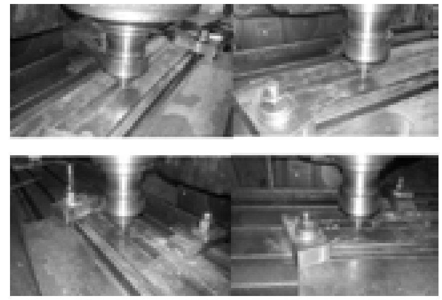


Figure 7. Various shots of friction stir processing.

At the end of this step, a composite layer of zirconium oxide nanoparticles is created on copper. At the Following steps, evaluation and comparison of microstructure and mechanical properties of the processed parts without and with amplifier particles by doing the related tests. The characteristics of variables used in the process are given in Table 2.

Table 2. The characteristics of variables used in the process

Process variables	Values description
Rotational speed of the tool	700 RPM
Onrush speed of the tool	14 mm / min
Deviation angle of the tool	3 °
The number of tool passes	1-2-4 Pass
The geometry of the tool	Cylindrical
Shoulder diameter, length and diameter of the pin	18-5-3 mm
Penetration depth of the tool	0.2 mm
Kind of nanoparticles	$ZrO2$

3.4 Tests Conducted on the Samples

In order to carry out the tests and studies, processed samples were cut up transversely and perpendicular to the tool onrush direction and divided into smaller pieces.

3.4.1 Microstructure Reviews

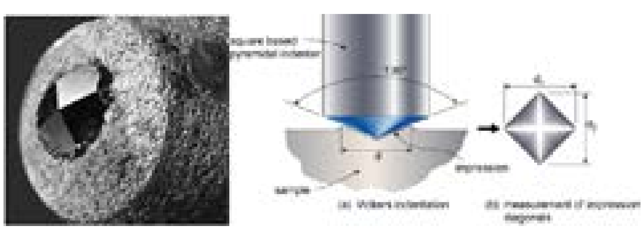
Before examining the microstructure, the samples went under the mounting action (locating in the circular shape of the plastic material). In order to achieve a perfect smoothness of desired levels, the cross-section of all samples, including base samples, processed with and without powder was sanded by using the silicon carbide sanding papers from No.600 to 2000 and then were polished by felt plates.Weight combine the ingredients of the etching solution consists of 30 ml of HCl, 10 g $FeCl_3$ and 120 ml H_2O. Next, the samples were shooting by using optical microscopy (OM) in order to study the microstructure and distribution of particles in the copper context. Also, in order to investigate the microstructure and measure the granulation in a more accurate and more magnification way, the field emission scanning electron microscope (FESEM) was used. A picture of the equipment,sample used in metallographyis shown in Figure 8.

A B

Figure 8. The equipment used in the microstructure reviews: A) Optical microscope with imaging camera B) Field emission scanning electron microscope.

3.4.2 Micro-Hardness Test

Hardness is one of the characteristics of the material which is usually defined as the material resistance against the indentor object. Many methods such as Brinell, Rockwell, Vickers, Nop, Scleroscopeand Moohez were developed to measure the hardness of the materials. One of the most common methods is Vickers method. In Vickers method a pyramid with a square base is used as a mandrel. Because of the shape of the mandrel in this method, it is also called as diamond pyramid hardness test (Figure 9).



A B

Figure 9. A picture of indentor and its effect on the Vickers test. A) A grandiosity picture of indentor B) A scheme of indentor effect in the sample.

Vickers hardness number (HV) is equal to the amount of load, divided by the sinkingarea, and is available by equation (1). Sinking area is calculated by macroscopic quantities of the drip effects.

Equation (1)

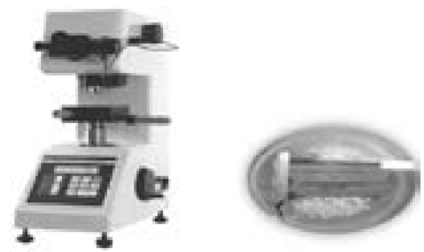
P = Load in kilograms

d = Mean diameters in millimeters

θ = The angle between opposite faces of diamond (136 °)

The Vickers hardness test is one of the most common micro-hardness tests. The method principles of Vickers micro-hardness of the test are same asa Vickers standard test with the difference that 1-1000 gf force range is used instead of the several pounds. It should be noted that prior to this test, the sample's surface must be polished in a roughness range of Ra = 0.4 to 0.8 μm, so that surface preparation would not change the structure.

The prepared samples in metallography step that have an appropriate surface flatnessare used in this test. An image of the device and a sample of the parts used in the micro-hardness test are shown in Figure 10.



A B

Figure 10. Micro-hardness test equipment A) Parts used in micro-hardness test B) Vickers micro-hardness tester.

The hardness evaluation of different areas of FSP process in the samples has been doneat the piecescross-section, perpendicular to the process path and in distance of 5.0 mm from below the surface. The amount and duration of incoming force were respectively selected by the device as 1kg and 15seconds. Also impacts intervals from each other were considered as 25/0. Some images of this test are shown in Figures 11 and 12.

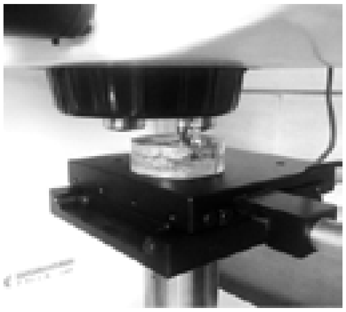
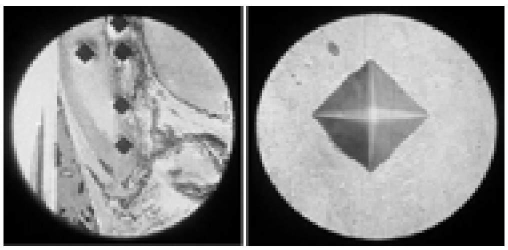


Figure 11. Micro hardness testing of Vickers methods.



A **B**

Figure 12. Observed images of Vickers device microscope A) The indenter effect with 200 times magnification B) The indenter effect with a 100 times magnification.

3.4.3 Abrasion Test

In this study, in order to measure the abrasion resistance and determine the friction coefficient of samples, pin on disk method is applied among the existing methods. In this method,a pin prepared from the sample is placed on a rotating plate by force exertion after measuring its initial weight, and its weight is measured again after a certain distance. Thus, the abrasion amountis obtained due to weight loss. A scheme of abrasion test device by pin on disk method is shown in Figures 13 and 14.

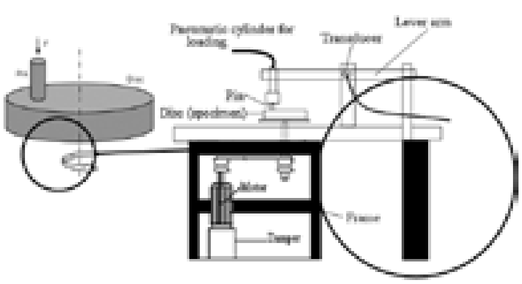


Figure 13. A scheme of pin on disk test machine.

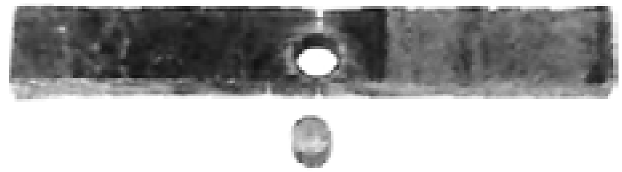


Figure 14. Pin sample prepared for abrasion test.

In this study, a cylindrical pin with a diameter of 5 mm and a thickness of a workpiece was separated from the processed area by using wire electrical discharge machines (Figure 13). In the next step, the surfacesprepared from pins got under the sanding and polishing operations. Each of the pins was cleaned using acetone solution and went under ultrasonic cleaning machine, then was carefully weightwith an accuracy of 0.0001. In order to compare the results with thebase metal, a pin with the same conditions and size was prepared from the base metal.

Required terms and variablesfor abrasion test are taken into account according to ASTM G99-05 standard. The condition includes a force of 10N, speed of 0.5ms^{-1}, sliding distance of 1000m and path radius of 15mm. It should be noted that the prepared pins had been tested on a sheet of steel 52100 with a hardness of 800 Vickers.

4. Conclusion

4.1 The Results of Copper Quant Metric

The chemical composition of copper has been determined using emission spectrometry (Quant metric), according to DIN EN 15079-07 standard, at the temperature of 20°C and 30% humidity. The results of this test are shown in Table 3.

4.2 The Results of Dimensional Review and Determination of the Chemical Composition of Zirconium Dioxide Particles

The particle size is determined by scanning electron microscope and the chemical composition by X-ray diffraction method.The results of these studies are shown in Figure 15.

Table 4. The tool chemical compositions used in friction stir processing

The chemical composition of hot work steel 1.2344									
Element Name	Vanadium (V)	Carbon (C)	Silicon (Si)	Manganese.(Mn)	Phosphorus (P)	Sulfur (S)	Chromium (Cr)	Molybdenum (Mo)	Iron (Fe)
Wt	0.85-1.15	0.35-0.42	0.8-1.2	0.25-0.5	Max 0.03	Max 0.02	4.8-5.5	1.2-1.5	Base

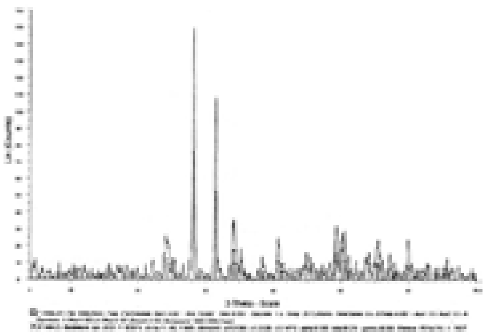


Figure 15. X-ray diffraction pattern of the zirconium dioxide powder.

According to the conducted surveys found that the average particle size is about 100 nm, and also according to the pattern of X-ray diffraction, the particles are single phase and have ZrO2 chemical composition.

4.2.3 Chemical Composition of the Tool

The chemical composition of tools used in this study is shown in Table 4. The chemical composition of silicon and chromium results in an appropriate resistance to oxidation at high temperatures. Also,according to carbon, vanadium and molybdenum elements in these tools, they have a high abrasion resistance and are suitable for working in hot conditions.

4.3 Microstructure Reviews

4.3.1 The Results of the Optical Microscope

Thepictures of the microstructure of the base metal (BM) and processed territories in samples with and without amplifier particles which are observed by using an optical microscope with magnification of 200 times are shown in Figures 16 to 22. It should be noted that in this study, the granulation changes and scattering patterns of amplifier particle are considered during each time of tool pass; therefore determination of granulation size and existence of amplifier particles will be studied in the next section.

The microstructure of pure copper used in this study is shown in Figure 16. As can be seen pure copper has a rough and nonuniformmicrostructures, and its microstructures mean size is 22microns. It is absolutely clear in Figures 17, 18 and 19 that due to plastic severe deformation caused by friction stir process and recrystallization mechanism in each tool pass, the granulation size is smaller and more uniformed in the processed areas, so that the granulation size in the fourth tool pass is very smaller and more uniform than the initial granulation.Also,it is observed in Figures 20, 21 and 22 whichare related to the samples containingamplifier particles that the accumulation of particles is reduced and particles are scattered in the base metal with more uniformity by every pass of the tool. ZrO2 particles cause to prevent thegrowth of dynamic recrystallized granulation under the effect ofsevere deformation. Also, these particles create more places to increase the germination in the structure and causeto break the largergranulation into smaller ones. Hence, by increasing the number of particles passing, followed by a greater fragmentation of particles in the context and preventing thegranulation growth, the granulations size will be much smaller.

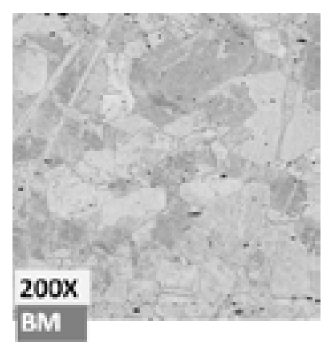


Figure 16. Base metal microstructure at a magnification of 200 times.

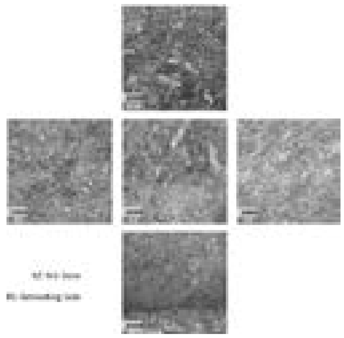


Figure 17. Micro-structure of the processed areas in the sample without amplifier particles and at the first tool passes with 200 times magnification.

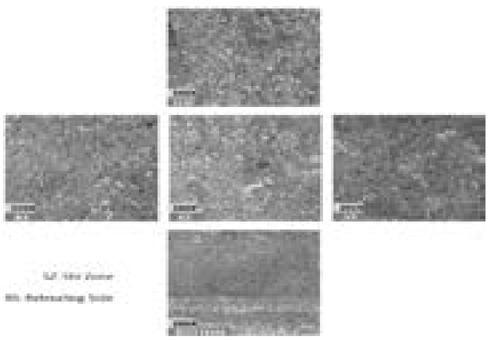


Figure 18. Micro-structure of the processed areas in the sample without amplifier particles and at the second tool passes with 200 times magnification.

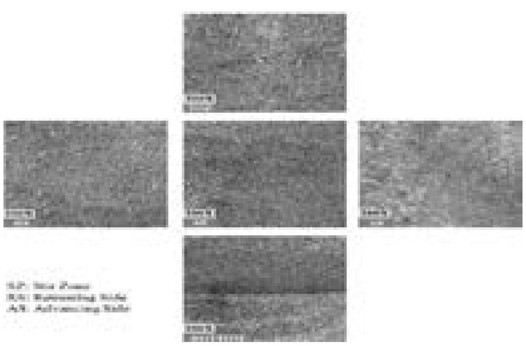


Figure 19. Micro-structure of the processed areas in the sample without amplifier particles and at the fourth tool passes with 200 times magnification.

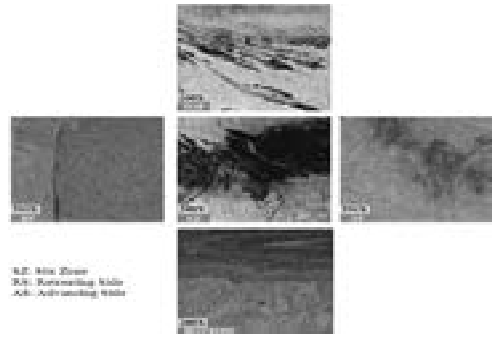


Figure 20. Micro-structure of the processed areas in the sample with amplifier particles and at the first tool passes with 200 times magnification.

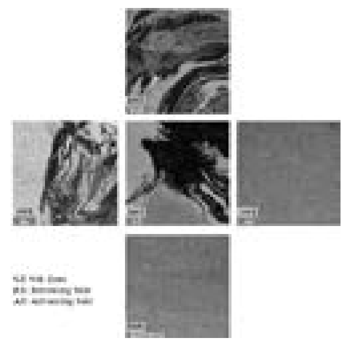


Figure 21. Micro-structure of the processed areas in the sample with amplifier particles and at the second tool passes with 200 times magnification.

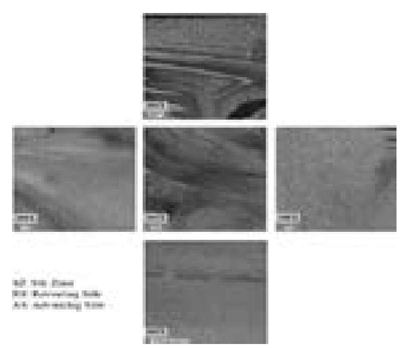


Figure 22. Micro-structure of the processed areas in the sample with amplifier particles and at the fourth tool passes with 200 times magnification.

4.3.2 The Results of Field Emission Scanning Electron Microscope

The observed images of the base metal microstructures, stir zone (SZ) and the area affected by thermo-mechanical (TMAZ) in processed samples without amplifier particles and samples containingan amplifier by electron microscope are shown in Figures 23 and 24.

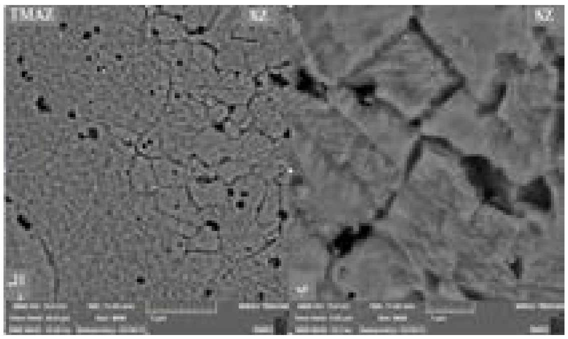


Figure 23. The microstructure of processed area in the sample without amplifier particles in the first tool pass. A) Stir areas and affected by thermo-mechanical B) Stir area.

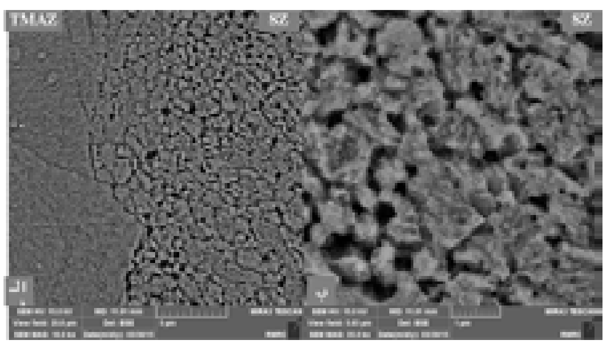


Figure 24. The microstructure of processed area in the sample without amplifier particles in the second tool pass. A) Stir areas and affected by thermo-mechanical B) Stir area.

According to the images prepared by FESEM and a study made by ImageJ software, Tables 5 and 6 are available. The values of granulations size and amplifier particle size after each tool pass are given in these tables. The microstructure of processed area in the sample for the various tool pass are illustrated in figures 25-28 in two cases including without and with amplifier particles

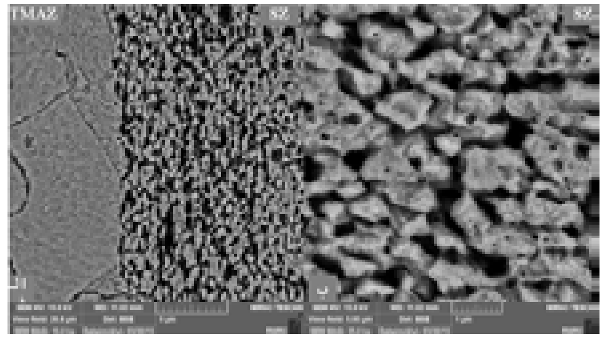


Figure 25. The microstructure of processed area in the sample without amplifier particles in the fourth tool pass. A) Stir areas and affected by thermo-mechanical B) Stir area.

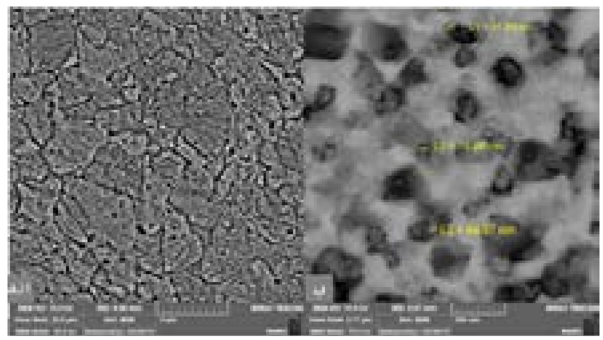


Figure 26. The microstructure of processed area in the sample containing amplifier particles in the first tool pass. A) Stir area with a magnification of 10kx B) Stir area with a magnification of 75kx.

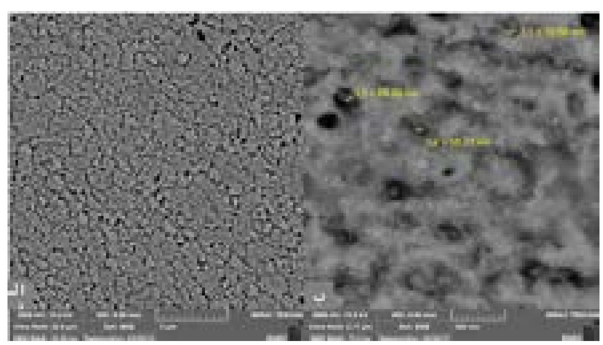


Figure 27. The microstructure of processed area in the sample containing amplifier particles in the second tool pass. A) Stir area with a magnification of 10kx B) Stir area with a magnification of 75kx.

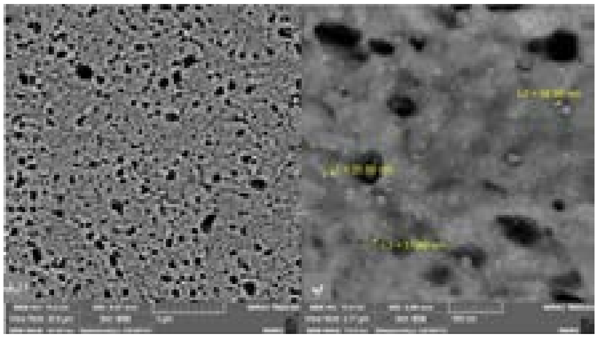


Figure 28. The microstructure of the processed area in the sample containing amplifier particles in the fourth tool pass. A) Stir area with a magnification of 10kx B) Stir area with a magnification of 75kx.

Table 5. The average grain size of base metal and stir areas in the processed samples

The average grain size by micrometers (μm)						
Base metal	Without amplifier particles			With amplifier particles		
	1th tool pass	2th tool pass	4th tool pass	1th tool pass	2th tool pass	4th tool pass
22	2.20	1.23	0.80	0.75	0.61	0.43

Table 6. The average size of Zirconia particles

The average size of amplifier particles by micrometers (nm)			
Before the process	1th tool pass	2th tool pass	4th tool pass
100	77.37	62.37	48.22

Given the above analysis, it is evident that with every passing of the tool, aggregation get smaller, powder accumulation get reduced,and material become more homogeneous;and as noted previously, it can be due to the severe plastic deformation, breaking down of ZrO2 particles at each tool pass and alsoseparating the powder particles from each other and preventing the growth of recrystallized granulations.

4.4 Hardness Reviews

As explained before, all samples after proper preparation go under the Vickers micro-hardness test from the cross-section. The hardness of a base metal containing a large and rough grains microstructure is about 80 Vickers. According to theHall patch equation, hardness amount has aninverse relationship with grain size. Therefore, a grain size reduction in the under process area than the primary substance, can increase the hardness. An average hardness of each sample is given in Table 7.

5. Conclusion

- Performing the friction stir processing by the parameters ofrotational speed of 700rpm and an onrush speed of 14mm per minute, on its own on copper cause to reduce the context grain size, and a more fine- aggregate structure obtained by increasing the toolpass from 1 to 4.
- Use of ZrO2 amplifier particles to produce Cu/ZrO2 Nano composite by friction stir processing results in more reductionof grain size in comparison with the processed samples without containing ZrO2 particles.
- By microstructure fine- aggregating during the friction stir process increases the hardness of the processed samples than the base metal. The hardness amount in composite samples compared to samples without amplifier particles has a significant increase; so that the greatest hardness is observed in composite samples of four passby 166Vickers hardness, which is about twice the hardness of the base metal.
- Increase the steps of friction stir processing by passing the tool up to four-pass results in reduction of the amplifier particleconcentration and by more uniform dispersion of the particles cause to form a homogeneous structure.
- The friction stir processing can increase the abrasion resistance by reducing the coefficient of friction; so that by four stages of processing, the composite samples will have the highest abrasion resistance among the processed samples.

6. Proposals

Some proposals are presented for future studies in the field of friction stir process:

- Increase the number of passes without adding amplifier material to determine the production potential of the amorphous structure.
- Study the electrical conductivity changes of copper by increasing the number of passes and adding amplifier particles.
- Study the super-plasticity behavior of copper after applying the friction-stir process.
- Study the microstructure and abrasion properties of copper after performing the process of friction - stir in immersion state.

- Study the effect amplifierparticles type on the conductivity of the composite material produced by friction stir process.
- Study the effect of friction stir process on the properties change of copper-based alloys.

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