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Effect of Single and Multiple-Pass Friction Stir Processing on Microstructure, Hardness and Tensile Properties of a 99.99% Cu with Carbon Nano Tubes

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Abstract
Friction stir processing (FSP), a relatively new solid-state microstructural modification technique can also be viewed as a severe plastic deformation technique because of the high processing strain involved. It is also unique from the viewpoint of its applicability to a localized region. Here the states of development of FSP for processing of Copper with carbon nanotubes are addressed. This paper investigates the parameters affecting the friction stir processed copper with carbon nano tubes and enhancement of the microstructure, hardness and tensile properties of the composite material. The behaviour of Copper with carbon nano tubes has been studied with single pass, double passes and triple passes. The SEM results are also presented for each case.

1. Introduction

Friction stir processing (FSP), a relatively new solid-state microstructural modification technique can also viewed as a severe plastic deformation technique because of the high processing strain involved. It is also unique from the viewpoint of its applicability to a localized region. FSP was developed for microstructural modification of metallic materials. In FSP, a rotating tool is plunged into a material and high plastic deformation is produced. FSP is used to enhance ductility, induces super plasticity and improve corrosion resistance properties. Dynamic recrystallization of the deformed zone forms an ultrafine-grained structure. FSP has been successfully applied to various cast aluminium and magnesium and copper alloys to eliminate casting defects and there by improve their mechanical properties [24, 25]. Two modes of metal transfer during friction stir processing have been discussed [15]. The first mode of metal transfer is generated between the tool shoulder and the plate and takes place as layer-by-layer deposition of metal one over the other. The second mode of metal transfer is generated by the extrusion of metal around the tool pin, when it reaches a state of sufficient plasticity. Metal transfer, generated between the tool shoulder and the plate, plays an important role in influencing the mechanical properties during friction stir process [29]. Modes of metal transfer are clearly visible in the microstructure characteristics, but they are not too distinct in macrostructure of most processed samples. Friction stir processing can be applied as a single-pass for processing a small area. For large engineering components in which the contact areas are relatively large, single pass FSP may not be adequate. Multi-pass FSP with a certain level of overlap between the successive passes is required for large contact areas. For both single and multi-pass processes, it is important to assess the microstructural evolution and its influence on the mechanical properties [2, 7, 17, and 23]

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Copper is a mostly used industrial and functional metal for various thermal, electrical and electronic applications, i.e. electronic packaging, electrical contacts and resistance welding electrodes. This is because of good thermal and electrical conductivity, high plasticity and excellent resistance to corrosion and oxidation. Nevertheless, low mechanical strength and poor wear resistance limit its applications [3, 10, 16, 26, and 27]. Carbon nano tubes are unique tubular structures of nanometer diameter and large length/diameter ratio. The nanotubes may consist of one up to tens and hundreds of concentric shells of carbons with adjacent shells separation of _0.34 nm. The carbon network of the shells is closely related to the honeycomb arrangement of the carbon atoms in the graphite sheets. The amazing mechanical and electronic properties of the nanotubes stem in their quasi-one dimensional (1D) structure and the graphite-like arrangement of the carbon atoms in the shells. Thus, the nanotubes have high Young's modulus and tensile strength, which makes them preferable for composite materials with improved mechanical properties. The nanotubes can be metallic or semiconducting depending on their structural parameters. This opens the ways for application of the nanotubes as central elements in electronic devices including field-effect transistors (FET), single-electron transistors and rectifying diodes. Possibilities for using of the nanotubes as high-capacity hydrogen storage media were also considered for experiment purpose [26].

Here the states of development of FSP for processing of Copper with carbon nano tubes are addressed. This paper investigates the parameters affecting the friction stir processed copper with carbon nanotubes and enhancement of the microstructure, hardness and tensile properties of the composite material. The behaviour of Copper with carbon nano tubes has been studied with single pass, double passes and triple passes The SEM results are also presented for each case.

2. Literature Review

Bahram A. Khiyavi, et al [1] produced copper reinforced metal matrix composite (MMC) using micron sized chromium particles via friction stir processing (FSP) in order to study effects of adding Cr particles to copper based matrix by FSP. Microstructures, micro hardness and wear properties were studied in order to evaluate the microstructures and mechanical properties of fabricated composites. H.R. Akramifard et al [4] in their investigation, pure Cu sheets were reinforced with 25μm SiC particles to fabricate a composite surface layer by friction stir processing (FSP). In order to improve distribution of reinforcing SiC particles, a net of holes were designed by drill on the surface of pure Cu sheet. H. Sarmadi, et al [5] focused on friction stir processing (FSP) used to produce copper–graphite surface composites. Five tools with different pin profile were employed in order to achieve a comprehensive dispersion. Results showed that the tool with rectangular pin give rise to a better dispersion of graphite particles. Furthermore, four copper–graphite composites containing different graphite content were prepare dusing rectangular tool through repeating the process passes. Friction and wear performance of the composites were also studied using a pin-on-disctribometer. It was indicated that the friction coefficients of composites were lower than pure annealed copper and decreased with increase in graphite content.

I. Galvao, A. Loureiro and D. M. Rodrigues [6] In their work, 1 and 3 mm-thick copper-DHP plates were processed with the aim of simulating surface (SFSP) and bulk (VFSP) processing. The influence of the processing conditions on the microstructure and mechanical properties of the processed materials was analyzed. It was found that the tool geometry, which has a close relation with the plastic deformation and dynamic recrystallization kinetics inside the stirred volume, the processing parameters and the heat exchange conditions, which determine the extent of dynamic recrystallization and annealing phenomenon, are determinant in FSP. Kudzanayi Chiteka [8] studied making a choice in selection of friction stir welding / processing (FSW/P) tool material which has become an important task in determining the quality of the weld produced. The tool material selection depends on the operational characteristics such as temperature, wear resistance and fracture toughness that determine the type of materials to be joined. Soft materials can be easily welded using tool steels while harder materials need harder tool materials such as carbide based materials and polycrystalline cubic boron nitride (PCBN).K. Surekha, A. Els-Botes [9] the objective of their study was to obtain a high strength, high conductivity copper by friction stir processing. Three millimeter thick pure copper plate was friction stir processed to a depth of 2.8 mm at low-heat input conditions by varying the travel speed from 50 to 250 mm/min at a constant rotation speed(300 rpm) to obtain fine grains.

Mohsen Barmouz, et al [11]studied multi-pass friction stir processing (MFSP) which was used for improvement of micro structural and mechanical properties of in situ Cu/SiC composites. Field emission scanning electron microscopy and optical microscopy images indicate that multi-pass FSP notably enhances the separation and dispersion of SiC particles and also reduces the grain size in the composite matrix, SiC particles size and porosity contents. Mohsen Barmouz, et al [12] their study was to produce copper reinforced metal matrix composite (MMC)layers using micron sized SiC particles via friction stir processing (FSP) in order to enhance surface mechanical properties. Microstructural evaluation using optical microscopy (OM) and scanning electron microscopy (SEM) indicated that an increase in traverse speed and a decrease in rotational speed cause a reduction in the grain size of stir zone (SZ) for the specimens friction stir processed without SiC particles. With the aim of determining the optimum processing parameters, the effect of traverse speed as the main processing variable on microstructure and micro hardness of MMC layers was investigated. P. Xue, et al[13]worked with large-area bulk ultrafine grained (UFG) pure Cu which was successfully prepared by multiple-pass overlapping friction stir processing (FSP) under additional rapid cooling. It was observed that overlapping FSP does not exert a significant effect on the microstructure and mechanical properties of the FSP UFG Cu. Similar average grain size was achieved in the transitional zone (TZ) of the multiple-pass FSP sample compared to that in the nugget zone of the single-pass FSP sample, and the TZ exhibited a strong {111} (112) type A fiber shear texture. Q. Zhang, et al [14] investigated situ Al3Ti/Al–5.5Cu composites fabricated by powder metallurgy and subsequent forging which were subjected to multiple pass friction stir processing (FSP) with and without active cooling. Valentin N. Popov [26] studied Carbon nanotubes: properties and application. V Jegannathan et al. [25] investigated the parameters affecting the friction stir processed copper and enhancement of the mechanical properties of the composite material. The results showed that the grain size of fabricated composite reduce, also it is indicated that in comparison to base metal (copper) micro hardness of friction stir processed composites in stir zone (SZ) increase significantly. The results obtained also indicated that the selected FSP parameters significantly influence the area of surface composite by the distribution of material particles. Higher tool rotational speed and lower processing speed produce an excellent distribution of material particles and higher area of surface composite due to higher frictional heat, increased stirring and material transportation. Ranganath M S et al [21] conducted the experimental studies for assessing the tribological performance of aluminium at the sliding contacts with mild steel plate, using a pin-on-disk tribometer as per ASTM-G 99. The study has been done in order to explore the friction and wear behaviors at the interface of tribo-pair. The tribological properties as coefficient of friction and specific wear rate of aluminium 6061are investigated. The Tribological tests are carried out at 500, 1000, 1500 rpm for 1000 meters in dry condition based on Response Surface Methodology. Track diameter, rotating speed and normal load are considered as the design parameters. Using central composite design, the problem is converted into single response optimization problem and the optimum combination of design parameters are found as 50mm track diameter, 500 rpm of rotating speed and 0.5 kg of normal load. The ANOVA result shows that the rotational speed is the most significant factor, followed by load and Track diameter for co-efficient of friction. Whereas the Track diameter is the most significant factor, followed by rotating speed and normal load for specific wear rate. Finally, microscopic images are investigated to identify the wear mechanism.

R. Sathiskumar, et al [18] applied Friction stir processing (FSP) to fabricate boron carbide (B4C) particulate reinforced copper surface composites. The effect of FSP parameters such as tool rotational speed, processing speed and groove width on microstructure and micro hardness was investigated. A groove was contrived on the 6mm thick copperplates and packed with B4C particles. FSP was carried out using five various tool rotational speeds, processing speeds and groove widths. R. Sathis kumar, et al [19] applied the friction stir processing technique to fabricate boron carbide particulate reinforced copper surface composites and investigate the effect of B4C particles and its volume fraction on microstructure and sliding wear behave or of the same. A groove was prepared on 6 mm thick copper plates and packed with B4C particles. The dimensions of the groove was varied to result in five different volume fractions of B4C particles (0, 6, 12, 18 and 24 vol. %). R. Sathis kumar, et al [20] In this work FSP technique was applied to prepare copper surface composites reinforced with variety of ceramic particles such as SiC, TiC, B4C, WC and Al2O3. Empirical relationships were developed to predict the effect of FSP parameters on the properties of copper surface composites such as the area of the surface composite, micro hardness and wear rate. The FSP parameters which influence the properties of surface composite are shown in Fig. 1. Salar Salahi and Vahid Rezazadeh [22] studied Fracture mechanism in metals using Friction Stir Processing (FSP) which was a challenging investigation and made by means of a rotating tool inserted in a work piece providing heat transfer and plastic deformation. In their paper, improving ductility during FSP was determined as a purpose and the microstructure and fracture mechanism of samples were investigated during Friction Stir Processing (FSP) of pure copper.

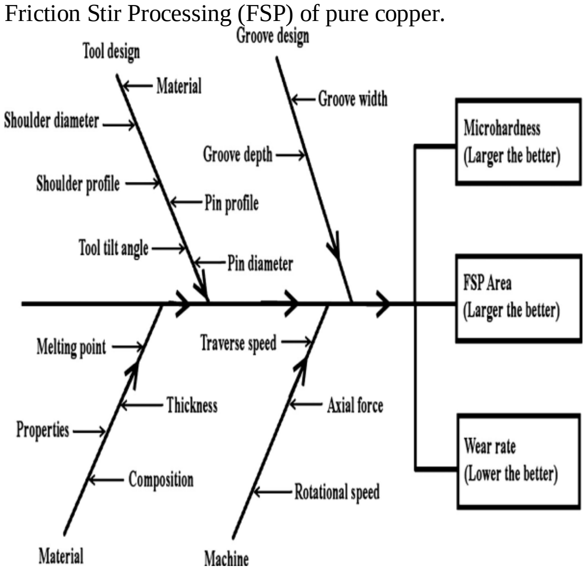


Fig: 1. FSP parameters influencing the properties of surface composite. [20]

3. Experimental Setup

The machine used for Friction stir processing was a special FSW machine is shown below.

Fig: 2. Friction stir welding / processing machine (Central workshop, Delhi Technological University)

Fig: 3. Tools used during Friction stir Processing

The specimen size of the copper plate that is used for processing 200 mm x 74 mm x 5 mm. One groove of 1mm width and 2 mm deep was made on the 99.99% pure Copper Plates using horizontal milling machine with a 1mm (width) saw cutter was cut in the middle of the specimen plate for processing. The tool Material used is H13 steel with shoulder diameter 15mm, threaded pin diameter 8 mm, pin length 2.5 mm with tool rotational speed 960 rpm, tool angle 2^0 and table traverse speed 25 m / min. Initially without carbon nanotubes one specimen plate was processed with single pass. Then second specimen plate was processed after filling carbon nanotubes in the groove cut (single pass). Then third specimen plate was processed after filling carbon nanotubes in the groove cut (double pass). Finally the fourth specimen plate was processed after filling carbon nanotubes in the groove cut (triple pass). The processed pieces were taken for the following tests: Tensile Strength Test, Brinell hardness test, Microstructure test.

4. Tensile Strength Test

After the FSP the specimen for tensile testing were cut from the job. The specimens are as shown in figure. The specification of specimen is as following:

- Length of specimen- 101.6 mm

- Gauge Length –25.4 mm
- Gauge width- 6.00mm
- Gauge thickness- 5.00mm

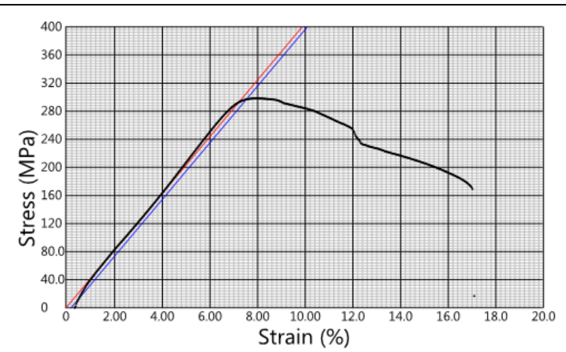
The specimens were chosen marked with marker on their ends. Care was taken to ensure that the specimens did not have any notching or cracks from manufacturing or any surface defects that would adversely affect the tensile tests. Before loading the specimens in the Instron machine, the computer system connected to the machine was given inputs such as gauge length and width of the specimen. The computer system was then prepared to record data and output necessary load-deflection graphs. The specimens were loaded into the Instron machine, and a tensile test was performed. The data was recorded electronically in text files and the load-deflection curve was shown on the computer screen as a visual representation. The average of different values of 3 specimens each from 2 jobs and parallel material was taken as the final values. The stress strain graphs and load elongations graph are shown for the ultimate strength of the specimen. The ultimate tensile strength of the processed material comes out to be lesser than the parent material.

Table: 1a. Specimens Details and Output Results during Tensile Tests

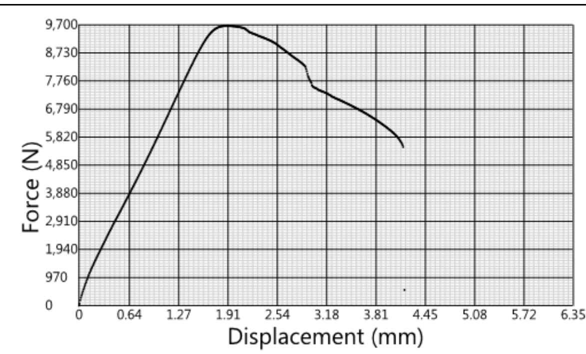
Out Put (Generic metals tensile from position	**Without processing**	**Single Pass Without CNT**
Width (mm)	6.00	6.00
Thickness (mm)	5.40	5.30
Gauge Length (Initial) (mm)	25.0	25.0
Gauge Length (Final) (mm)	28.7	31.8
Area (mm^2)	32.4	30.8
Ultimate Force (N)	9670	5650
Ultimate Stress (MPa)	298	183
Offset @ 0.2% (N)	9620	1240
Offset @ 0.2% (MPa)	297	40.2
TE (Auto) (%)	17.1	26.3

Table: 1b. Specimens Details and Output Results during Tensile Tests

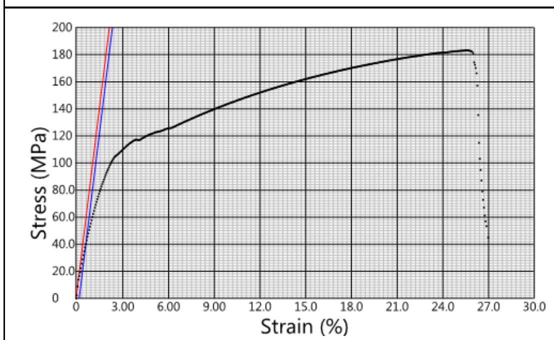
Out Put (Generic metals tensile from position	**Single Pass With CNT**	**Two Pass With CNT**	**Three Pass With CNT**
Width (mm)	6.00	6.00	6.00
Thickness (mm)	5.14	5.05	5.00
Gauge Length (Initial) (mm)	25.0	25.0	25.0
Gauge Length (Final) (mm)	26.8	27.4	30.4
Area (mm^2)	30.8	30.3	30.0
Ultimate Force (N)	2800	4860	6380
Ultimate Stress (MPa)	90.7	160	213
Offset @ 0.2% (N)	2550	2120	4540
Offset @ 0.2% (MPa)	82.7	70.0	151
TE (Auto) (%)	7.28	9.72	22.2



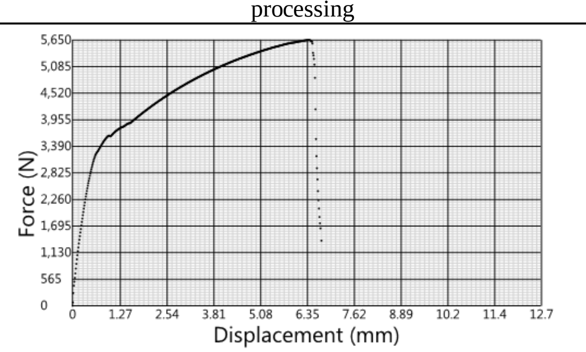
(a) Stress-strain curve for specimen without processing



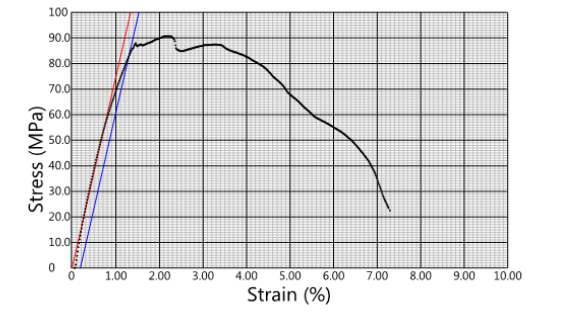
(b) Force-displacement curve for specimen without processing



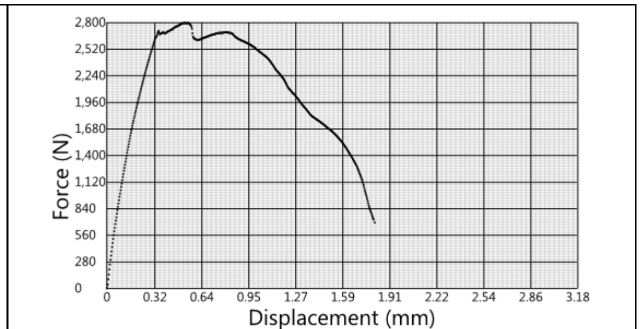
(c) Stress-strain curve for specimen without cnt single pass processing



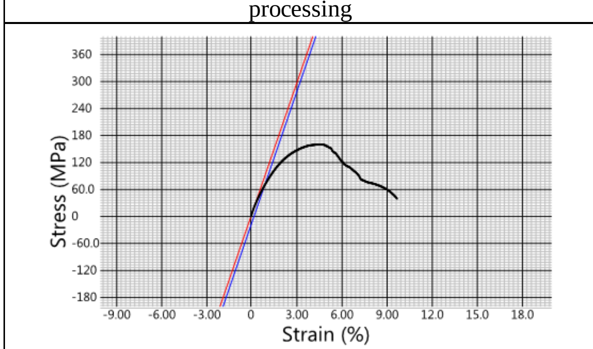
(d) Force-displacement curve for specimen without cnt single pass processing



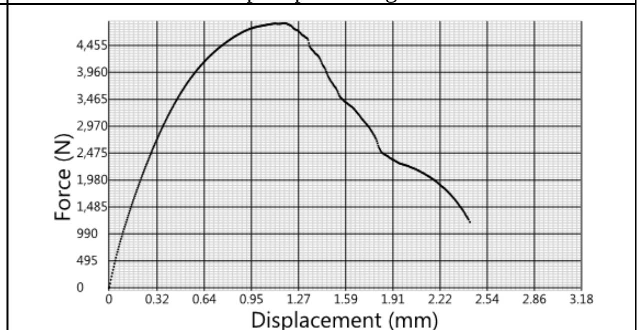
(e) Stress-strain curve for specimen with cnt single pass processing



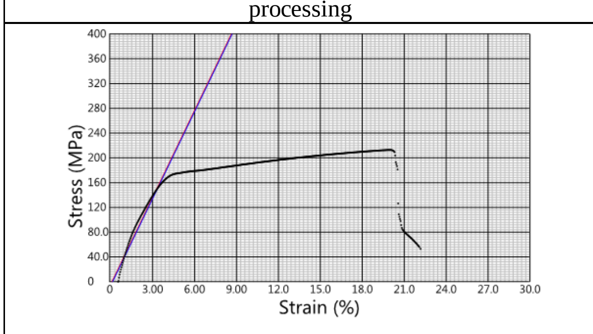
(f) Force-displacement curve for specimen with cnt single pass processing



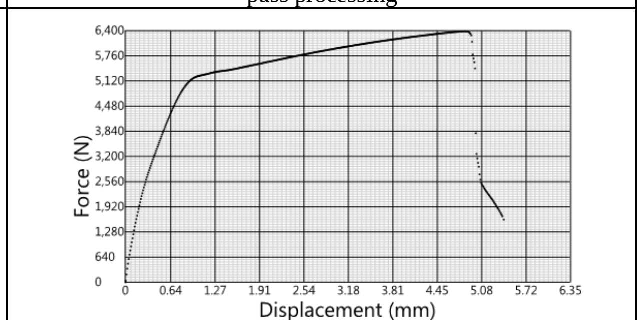
(g) Stress-strain curve for specimen with cnt double pass processing



(h) Force-displacement curve for specimen with cnt double pass processing



(g) Stress-strain curve for specimen with cnt triple pass processing



(h) Force-displacement curve for specimen with cnt triple pass processing

Fig: 4. output results during tensile tests after Friction stir processing

5. Brinell Hardness Test

The Brinell scale characterizes the indentation hardness of materials through the scale of penetration of an indenter, loaded on a material test-piece. It is one of several definitions of hardness in materials science. The brinell hardness was conducted on all specimens .As we can see the brinell hardness of the processed specimen increases as the number of passes are increased due to the more compact microstructure. The brinell hardness number comes out to be highest for third pass in comparing with single and double passes.

Table: 2. Specimens Details and Output Results during Tensile Tests

Expts	Description	Depth of indendation	Brinell hardness BHN
1	Without carbon nanotube and without processing	2.8 mm	11.14
2	single pass processing Without carbon nanotube	3.09	8.93
3	Single pass With carbon nanotube	3.47	6.823
4	Double passes With carbon nanotube	3.40	7.162
5	Triple passes With carbon nanotube	3.00	9.55

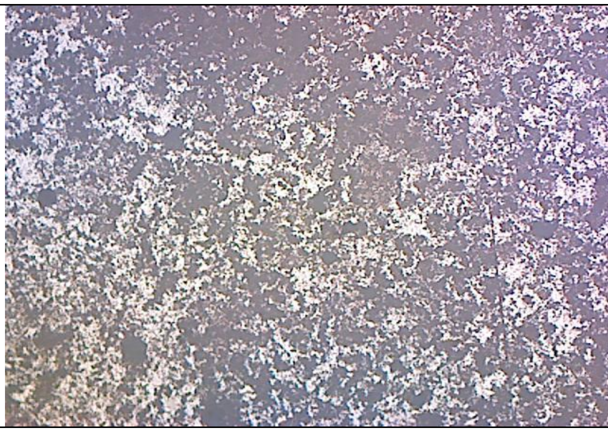
6. Microstructure Ananlyis

The optical microscope is the principal tool used to characterize the internal grain structure of steel. Traditionally, the structure revealed by the microscope is called the microstructure. The mechanical properties of given steel are strongly influenced by its microstructure. An optical microscope uses reflected light to generate an image.

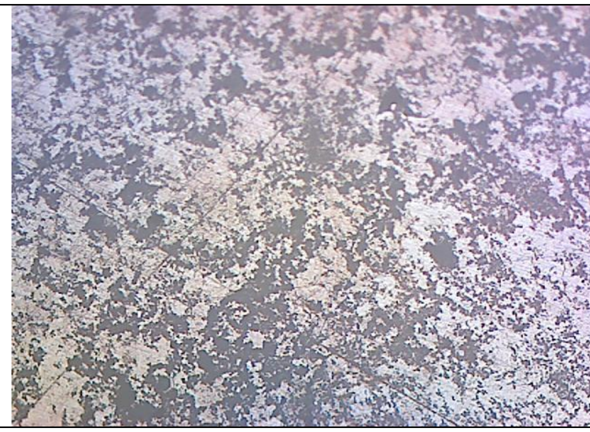
A beam of light is directed down onto the surface and the image is generated either on film or the eye by light reflected along the same direction.This is performed on specimens either cut to size or mounted in a resin mold. The samples are polished to a fine finish, normally one micron diamond paste, and usually etched in an appropriate chemical solution prior to examination on a metallurgical microscope. For microstructure test we had five specimens. There are two examination methods in metallography:

(i) Macroscopy
(ii) Microscopy

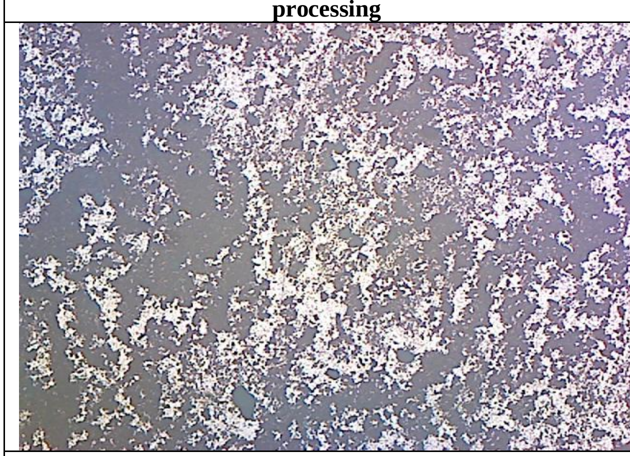
In macroscopy the examination of the structural characteristics or chemical characteristics of a metal or an alloy is done by the unaided eye or with the aid of a low-power microscope or binocular, usually under l0x. In microscopy similar examination is done with the prepared metal specimens, employing magnifications with the optical microscope of from 100 x to as high as 2000x. Figure 5 represents the microscopic results of all 5 specimens after processing



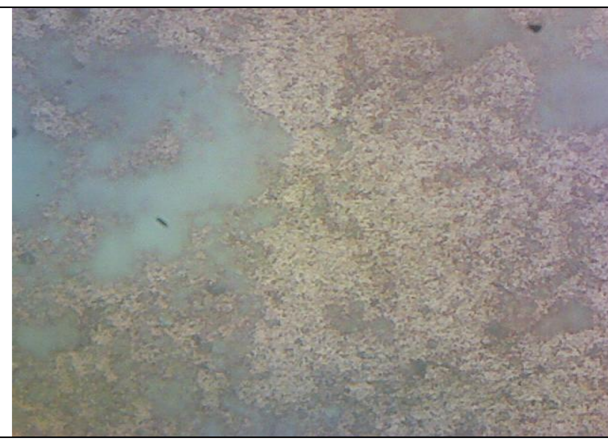
(a) Specimen 1--10x Base metal microstructure without processing



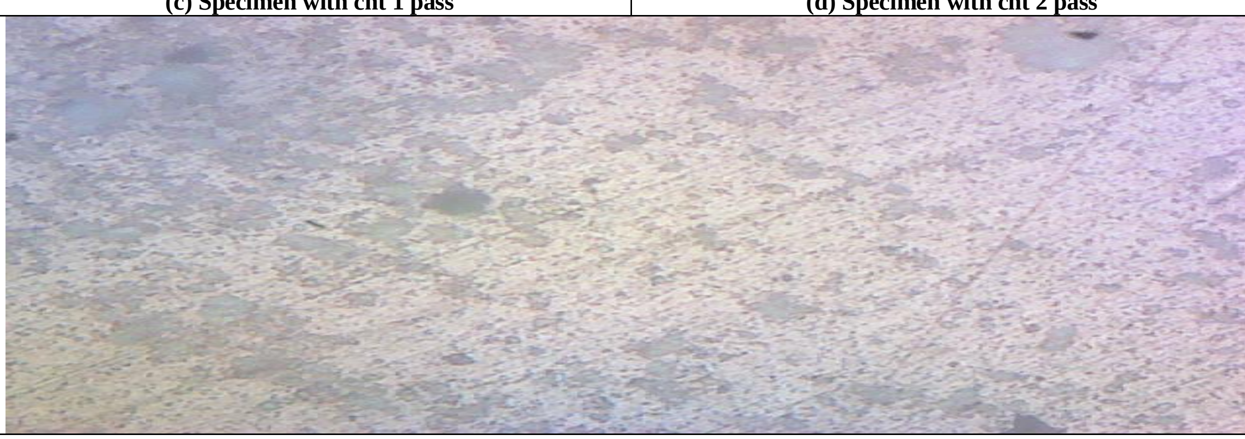
(b) Specimen 1--20x Specimen without cnt with 1 pass



(c) Specimen with cnt 1 pass



(d) Specimen with cnt 2 pass



(e) Specimen with cnt 3 pass

Fig: 5. a-e. Microscopic Results of all Specimens After Friction Stir Processing

7. Conclusions

The effect of friction stir processing (FSP) parameters on copper material with carbon nano tubes as composite material has been studied. Tensile strength, microstructure and micro hardness tests were conducted on the specimens prepared at the central workshop of Delhi Technological University, India. The following conclusions have been made from present investigation.

- Fabrication of copper–carbon nano tube composites by friction stir processing (FSP) is possible.
- The microstructure for different specimens showed that after single, double and triple passes the microstructure gets more compact and observed no defects after processing.
- The brinell hardness number comes out to be highest for third pass in comparing with single and double passes.

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