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Synthesis of copper –graphite composite using friction stir processing and evaluating parameters effecting hardness and wear

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Abstract

This report is strictly concerned with the fabrication of surface composites. Surface composites are a group of present day designed materials where the surface of the material is transformed by mixing reinforcement particles which also does not affect the change in basic structure and chemical composition. Friction stir processing has been utilized to create metal matrix composites by consolidating graphite particles in a copper material matrix using square pin tungsten carbide tool. Mechanical properties (i.e. tensile and hardness/micro hardness, wear resistance etc.) of the matrix metal matrix composites reinforced with graphite particles are calculated. The average micro hardness value within the stir zone decreased from 180 Hv in the base material to a minimum of 142Hv in a graphite reinforced composites. Wear resistance is enhanced with decrease in the friction coefficient from 0.490 of standard specimen to minimum of 0.168.

Keywords: Friction Stir Processing, parameters optimization, Mechanical Properties Improvement

1. Introduction

At first, FSP was utilized for microstructural refinement of aluminum [3] and magnesium [4] combinations. FSP advancement has facilitated led to the fruitful handling of composites of copper, titanium and steel. FSP has likewise exhibited its effectiveness in homogenizing powder metallurgy handled aluminum combinations, microstructural modification of metal matrix composites. There are numerous conventional methods for fabricating surface composites such as powder metallurgy, laser melt treatment, plasma spraying, stir casting etc. but these techniques lead to the deterioration of composite properties due to interfacial reaction between reinforcement and the metal matrix. FSP has definite advantages compared to other processes as shown in Fig.1. To start with, FSP is an immediate solid state preparing method that achieves microstructural change, densification, and homogeneity at the same time. By rewriting the tool design, FSP parameters, and dynamic cooling/heating the microstructure and mechanical properties of the handled zone can be decisively overseen. Having a broad capacity for the manufacture, handling, and amalgamation of materials FSP is a versatile strategy. The depth of the processed zone can be optionally controlled by altering the length of the tool pin. FSP is a green effective method without hurtful gas, radiation, and commotion since the heat contribution amid FSP originates from grinding and plastic twisting.

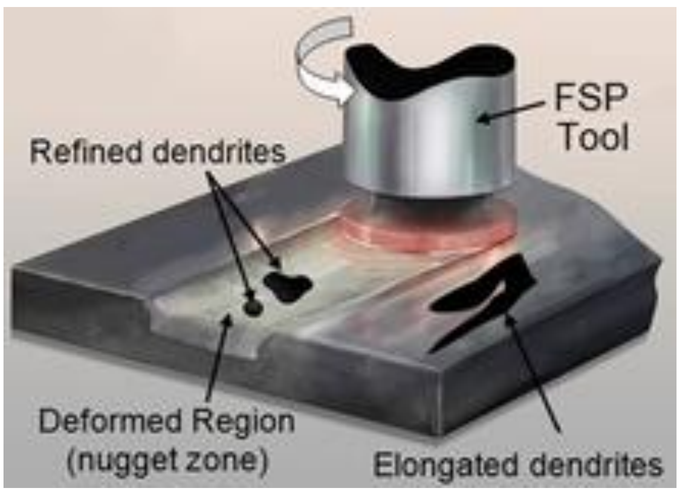

Fig-1: Friction Stir Processing [1]

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Copper and its alloys are broadly utilized as material for a few parts in electrical, thermal, substance, atomic and transportation ventures. They show great mix of electrical conductivity, thermal conductivity and workability. Enhanced wear resistance what's more, arcing resistance of copper are required in applications, for example, electrical contacts, nozzles, bearings and carbon brushes for motors and generators. Copper matrix composites (CMCs) have been gaining much attention owing to their good mechanical, thermal and tribological properties. In pure form it has poor quality, wear also, fatigue resistance and consequently is inadmissible for applications demanding high fatigue and wear resistance like contact terminals of electrical switches.

It has been recommended by a few analysts that FSP can largely portrayed as an in situ extrusion process and the mixing of material happened just at the surface layer of the prepared zone adjoining the rotating tool shoulder [1].

2. Literature Review

VJ Arulmoni et al. [2] studied the properties for FSPed copper whose outcomes got likewise showed that the choice of FSP parameters fundamentally impact the range of processed area for surface composite by the dispersion of material particles. Higher tool rotational speed and lower traverse speed deliver a magnificent dispersion of material particles and higher area of surface composite due to higher frictional heat, increased stirring and material. FSP technique has been effectively used to deliver surface composite layers on aluminum, magnesium, copper, steel and titanium [5]. R. Sathiskumar et al. [6] used FSP method to get ready copper surface composites reinforced with diverse ceramic particles, for example, SiC, TiC, B4C, WC and Al2O3. Mostly, different sorts of ceramic particles (carbides, oxides, borides and nitrides) were reinforced with copper to deliver CMCs. H. Sarmadi et al. [11] concentrated on friction stir processing (FSP) method, used to produce copper–graphite surface composites. Showed that the tool with triangular and square pin give rise to a better dispersion of graphite particles as compared to simple cylindrical pin tool. Mohsen Barmouz et al. [7] examined multi-pass grinding blend preparing (MFSP) which was utilized for development of microstructural and mechanical properties of in situ Cu/SiC composites. Mohammad Kazem et al**.** [8] 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. Sabbaghian et al. [9] watched pores around SiC and TiC particles individually in CMCs utilizing FSP. G.M. Karthik et al. [15] told that Friction stir alloying can be utilized for locally enhancing the strength/toughness of a structural member at the points of stress concentration. Rai et al. [10] brought up that the tungsten, molybdenum, and iridium are reasonable alternatives of tool materials as they possess high hardness, better hot strength and low reactivity with oxygen. These tool properties can be upgraded advance by the expansion of alloying components or coating the tool material with a hard and wear resistant material. Thus we opted for the tungsten carbide tool in our work. The combination of two parameters decides the rate of mixing of particles. An increment in rotational speed expands the capacity to scatter the particles productively into the copper matrix. On the contrary, an increase in traverse speed reduces the available stirring and results in poor distribution and formation of clusters [6]. Thus, the homogenous dispersion of graphite particles is an aftereffect of legitimate mix of process parameters picked in the work.

Welding parameter such as tool rotation, transverse speed and axial force have a significant effect on the amount of heat generated and strength of FSW joints. Microstructure evaluation of FSW joints clearly shows the formation of new fine grains and refinement of reinforcement particles in the weld zone with different amount of heat input by controlling the welding parameter [16, 17]

3. Experimental procedure

A pure copper (99%) base plate of 6 mm thickness, 200 mm length and 75mm breadth what's more, fine graphite powder with a normal molecule size of ~ 50 microns and maximum limits of impurities substance soluable in ethanol is 0.2% were utilized as the reinforcement materials. Initially, two rectangular groove of 1 mm wide and 2.5 mm depth was machined in the copper base plate via milling machine, After this, groove was properly cleaned with acetone which was along these lines firmly pressed with the graphite powder . A pressing (capping pass) was then performed over the graphite-filled groove using a pin-less tool (made of tungsten carbide , 20 mm shoulder diameter).The procedure parameters utilized for the pressing (capping pass) were: 1000 rpm tool rotation speed, 15 mm/min traverse speed, 0° tool tilt. The pressing (capping pass) was intended to close the section so that the graphite powder does not take off amid FSP. Following this, FSP was done over and along the pressed grove utilizing a tungsten carbide tool consisting of square pin of side 6mm and length of 3mm. five number of such plates were friction stir processed by varying the process parameters. For every pass, the specimen was allowed to be cooled to the room temperature and all the experiments were carried out at room temperature. The specimen was clamped on the hydraulic fixture with mild steel backing plate.

The FSP was carried out semi automatically on an indigenously built FSW machine (M/s RV Machine Tools, Coimbatore, India). The FSP was carried out randomly as per the parametric values as shown in Table 1. Different types of particles were successfully reinforced on the copper surface using various methods but several defects such as porosity, in homogenous distribution, formation of clusters and interfacial reaction were found prominent. Typical defects in the FSP process such as voids, tunnels and pin holes are not observed in the stir zone. The area of the stir zone is continuous without irregularities. It indicates that the composite was successfully synthesized. The machined groove disappeared totally after FSP. The copper experienced severe plastic deformation due to the frictional heat combined with the mechanical working of the tool.

The dimensions of the specimens for the micro hardness test are 10mm diameter cylinder which is carved through the Wire EDM process. The micro hardness of the bulk FSP pure Cu sample was measured along the middle-thickness of the processed zone with a 10 gram load for 10 seconds. Tensile specimens were machined from the processed zone in direction, parallel (longitudinal) to traverse direction. Large dog-bone-shaped tensile specimens with a gauge length of 33 mm, a gauge width of 6 mm and a gauge thickness of 6 mm were machined parallel to the FSP direction from the processed zone in the bulk sample. Specimens of size 10 mm× 10 mm were extracted from the friction stir processed plates via Wire EDM to evaluate microstructure. Thereafter mounting of specimens is done via resins and hardener for optical images. For polishing process specimens are rubbed over various different nos. emery paper. Then onwards it is rubbed with alumina powder with water over polishing machine whose wheel is rotating around 300-400 rpm. Afterwards specimens were etched with an etchant containing 20 g chromic acid, 2 g sodium sulfate, 1.7 ml HCl (35%) in 100ml distilled water and then dried through hot air via blower. The microstructural evolution during the FSP were characterized using an Olympus Optical Microscope (OM), model GX 41 equipped with image analysis software, a camera having 10x, 20x, 50x and 100x lens and a computer, field emission scanning electron microscope (CARL ZEISS-SIGMAHV). Wear samples of 10 mm diameter, which were cut from the stirred zone as discussed above, were small in size and difficult to hold in the wear testing machine. Therefore dummy pins were mounted on the wear samples. Mounting was done by drilling a hole of 4 mm diameter and 3 mm deep in the sample with the help of drilling machine; after that sticking of dummy pin to the processed wear samples was done with the help of Araldite and ultimately samples were kept drying for about 24 hours. Finally, filing was done on the flat surface of wear samples to avoid unevenness during wear test. In this way, total 10 wear samples were prepared for wear testing operation finally, filing was done on the flat surface of wear samples to avoid unevenness during wear test. The wear behavior of the surface composites was evaluated by a pin on disc tribometer. Wear test was conducted at load of 50 N, sliding speed of 2.5m/s and sliding distance of 3000m.

Table 1: Experimental Parametric Values

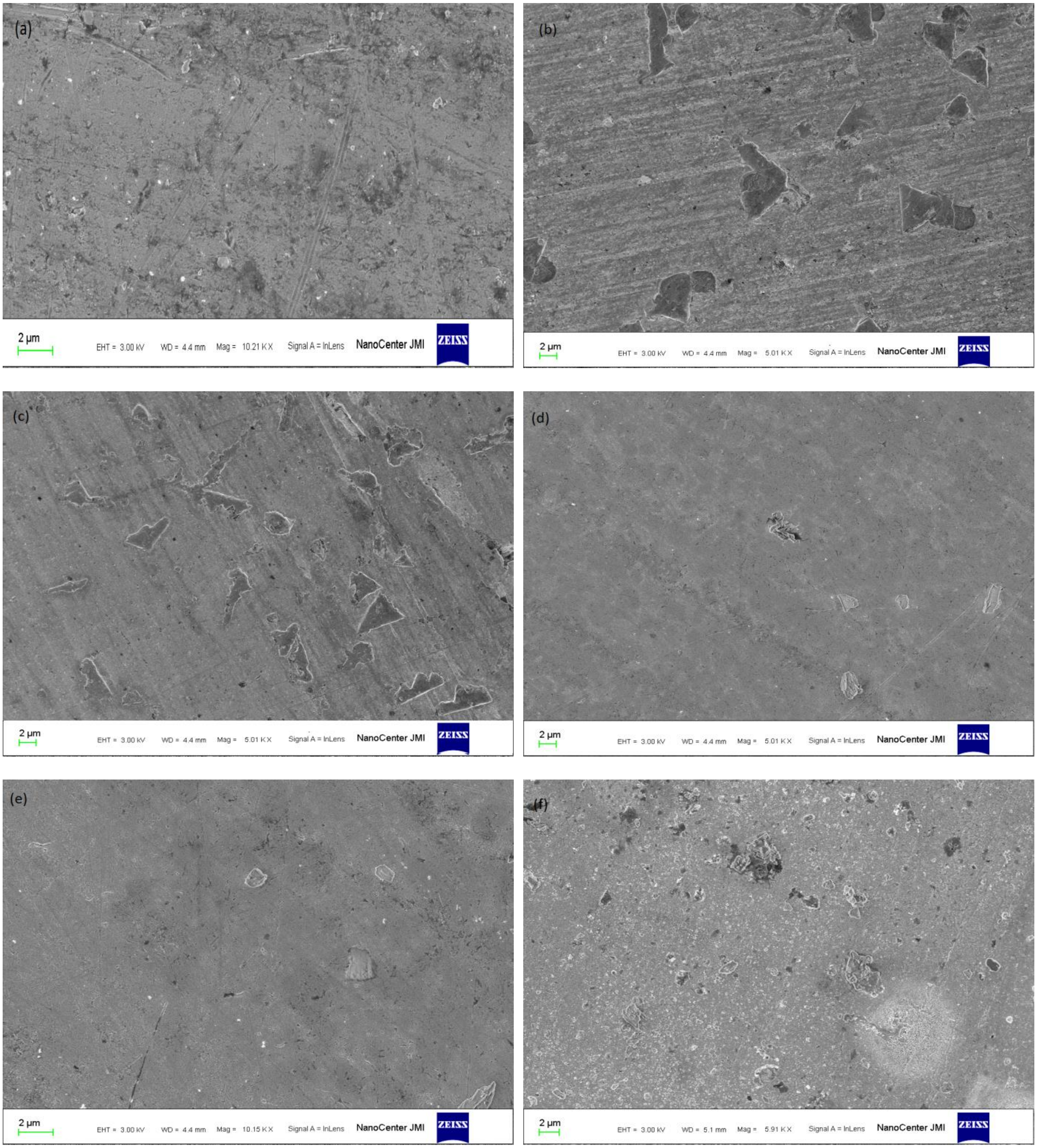
Specimen Nos.	Tool tilt	Rotational Speed	Traverse Speed
6	1.5	900	15
8	1.5	950	20
7	1.5	1000	25
1	2.0	900	20
2	2.0	950	25
9	2.0	1000	15
3	2.5	900	25
4	2.5	950	15
5	2.5	1000	20

4. Result and Discussions

One of the primary difficulties is to accomplish a uniform dispersion with a solitary pass, because of the material flow and intermixing modes which are forced by the FSP tool, The crown appearance implies that the trueness of the processed zone underneath it. Any deformation on the crown more often than not runs with a looking at imperfection in the processed zone [13]. The crown appearances of processed zone arranged copper with graphite particles are presented. The machined groove disappeared totally after FSP. The copper experienced severe plastic deformation due to the frictional heat combined with the mechanical working of the tool. An examination of the micrographs in Fig.3 reveals that the interface between graphite particle and the copper matrix is regular and uninterrupted. If the plasticized copper does not spread over the surface of reinforcement particle, it will result in the formation of pores. Presence of pores leads to weakening of interfacial strength [6]. The deformities in the crown amid trail runs can be reasoned through poor material flow amongst progressing and withdrawing side and lacking plasticization of copper. No pores and any other deformities were found after FSP. It indicates that the composite was successfully synthesized. The micrograph of the composite's surface (Fig.1) does not show any presence of onion rings. There is not much of clear indication for the presence of graphite particles and is similar to the standard specimen structure which proves the even mixing of graphite in the metal matrix. All the specimens named in the order with the resolution of 20x images. Redefined grains can be seen easily in images which were inherited via plasticization of copper.

4.1 Mechanical Properties

The influence of Graphite particles on the micro hardness of the surface Copper graphite CMCs is illustrated in Figure 3. The micro hardness was observed to be 180Hv at 0% volume graphite and 152Hv at 7.05% volume graphite. The fig.3 reveals that the incorporation of graphite particles is leads to decrease in the hardness of the surface composite. Hardness is observed to decrease with an increase in graphite volume fraction for constant traverse speed. It can be inferred from the plot that graphite particles lead to reduction in hardness of the composite. The subsequent factors can be ascribed to cause decrease in strength of the composite; first hardness of graphite particles, it is well known that the hardness of graphite particles is very low comparing to copper matrix. Second is the type of particle distribution, which found to be evenly dispersed in most of the specimens, only one specimen no.6 (shown in figure3) with an anomaly whose hardness (172Hv) is higher with high carbon content (4.87%) which may be due to the low rotational speed (900 rpm) and low tool tilt angle (1.50) used for its processing which leads to poor distribution consequently high hardness and third is the grain size. Smaller the grain size, higher will be the hardness of the composite. The refined grains contribute to improve the hardness but in this case graphite softness of particles is predominant in nature.


(a)
2 µm
EHT = 3.00 kV WD = 4.4 mm Mag = 10.21 K X Signal A = InLens NanoCenter JMI
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(b)
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(c)
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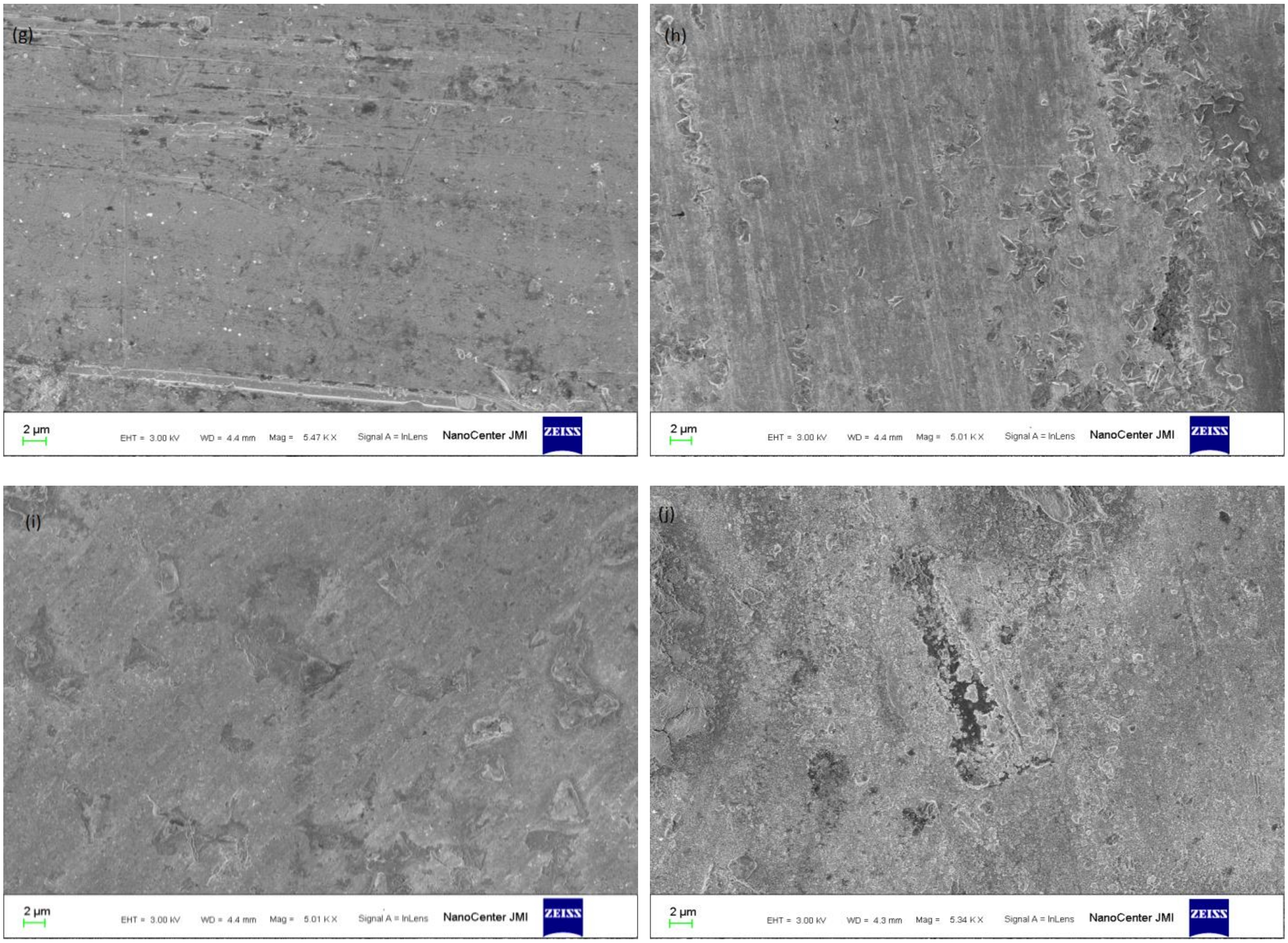

Fig.2: SEM images for various specimens

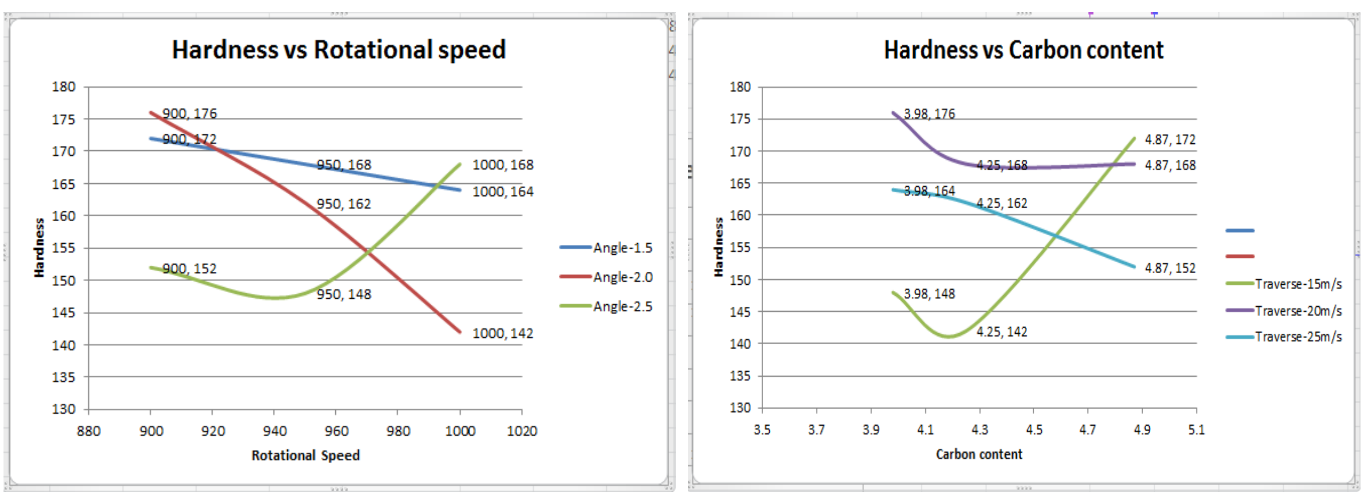

Fig.3: Hardness vs Rotational speed and Hardness vs carbon content

It is evident from Fig.3 that the micro hardness of the surface composite decreases when the rotational speed increases from 900 to 1000 rpm. The micro hardness was found to be 176Hv, 172Hv and 152Hv at 900 rpm and 142Hv, 164Hv and 168Hv at 1000 rpm for tool tilt angle 1.50, 2.00and 2.50 respectively. The presence of agglomerations causes a higher variation of hardness across the surface composite. This leads to higher hardness at 900 rpm. The decrease in hardness when tool rotational speed was increased can be attributed to the breaking up of agglomerations as discussed above. The distribution of particles becomes uniform at higher tool rotational speed. The possibility of indenter resting directly on the parent metal which may not be consist of graphite particles when hardness measurement was carried out on specimen 5, might be the valid reason for higher hardness. Another possible explanation would be the tool tilt angle 2.50 consequently plunge is increase for constant load (1000kg) condition, leads to dominant forging effect resulting increase in the hardness.

4.2 Wear

A homogenous distribution of reinforcement particles with good interfacial bonding is essential to sustain the wear resistance of the copper surface during service. It is evident from table 1 that the wear rate of the surface composite increases when the hardness decreases for constant rotational speeds 900, 950 and 1000rpm respectively. It is well known that the wear rate is inversely proportional to microhardness in metal matrix composites. The decrease in microhardness of the surface composite reduces the resistance to metal removal during sliding wear. The volume loss of material due to sliding wear is given by the following expression [12]

$$\text{Volume loss} = \frac{\text{Wear coefficient} \times \text{Applied load} \times \text{Sliding distance}}{\text{Hardness of material}}$$

The equation also tells us the relation between volume loss or wear rate and hardness of the specimen which are inversely proportional to each other. Although hardness of the composite decreases from the parent metal but among the decrease higher hardness specimen showed better wear resistance.

Table1: Showing hardness and wear rate values at different rotational speeds

Specimen No.	Rotational Speed	Hardness	Wear
1	900	176	7.30
6	900	172	9.40
3	900	152	10.33
8	950	168	8.53
2	950	162	9.36
4	950	148	10.10
5	1000	168	7.26
7	1000	164	5.26
9	1000	142	6.86
10	-	180	14.46

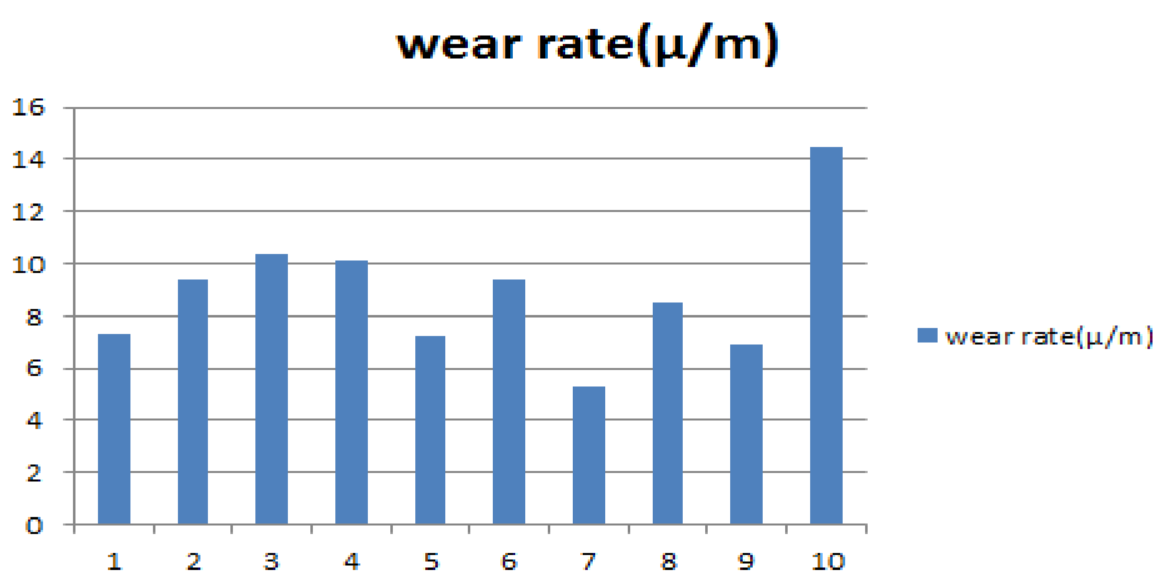

Fig.3: Wear rate in microns per meter sliding distance for each specimen

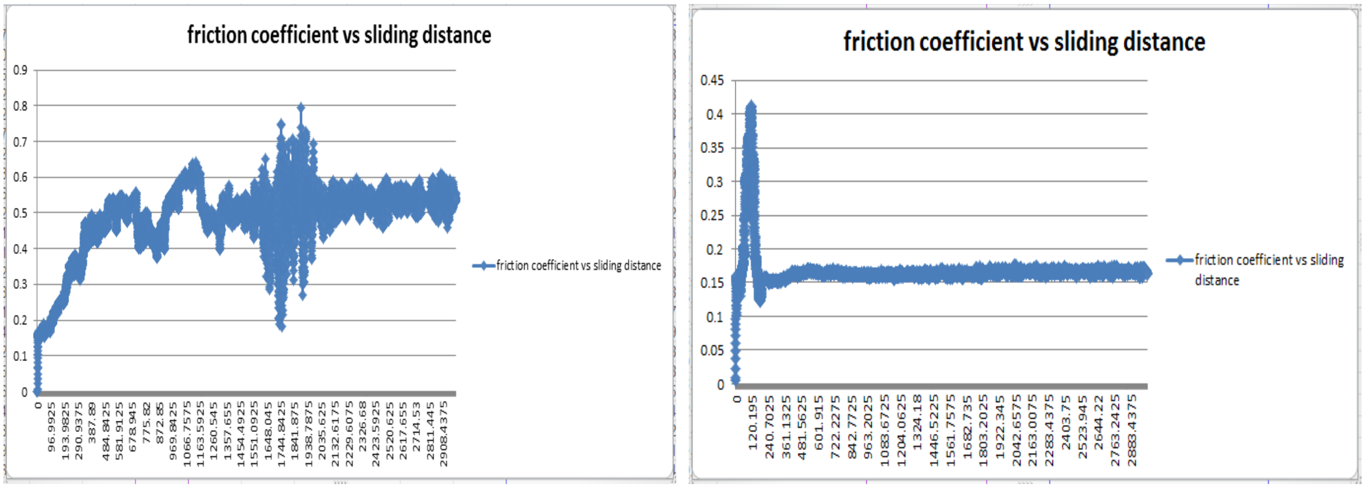

Fig.4: Friction coefficient vs sliding distance for Standard specimen and specimen no. 7

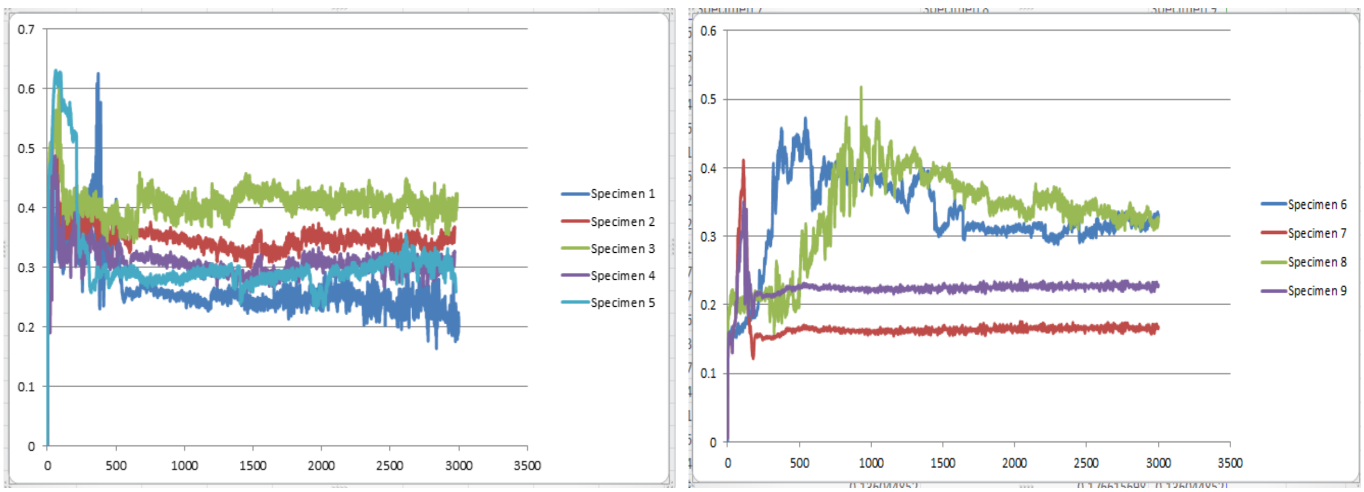

Fig.5: Friction coefficient vs sliding distance for various specimens

Wear rate for the parent metal copper is 14.46 microns/m which was reduced to the value 5.26 microns/m showed by specimen no. 7 with a coefficient of friction 0.168. The significant reduction can be deduced via lubricating property of graphite particles. This excellent wear behavior could be due to the low tilt angle for tool and high traverse speed which does not allow the particles to settle inside matrix at greater depths. Therefore all the particles quickly squeezed out over the surface while performing wear test. Shear strength of graphite is too low leading to sliding of the planes over each other and decrease in friction coefficient consequently wear rate. According to H. Sarmadi et al. [11] if matrix possesses low strength and high plastic deformation capability, squeezing out of graphite particles to surface will be eased. In this study the matrix is copper and has a good plastic deformation capability. Looking at plot for wear of parent metal i.e. copper shows earratic behavior in mid-range of sliding distance which can be due to the imperfection in structure. High wear rate is the well-known property for copper. Hence FSP also eliminates such imperfection which was initially present. Each plot has a big spike at the initial stages of sliding distance which becomes almost constant after the sliding distance of 500m for all specimens except specimens 6 and 8. These two has similarity in parametric values chosen for FSP i.e. tool tilt angle of 1.50 and low rotational speeds (900& 950 rpm) which may be also cause not proper distribution of graphite particles. It took significant sliding distance (1500m) to get the steady plot for these specimens. This spike is due to the initial metal (copper)-metal (wear plate) contact after some time because of the wear of the parent material, after all the graphite comes over the surface and induce a layer known as tribolayer which leads to steadiness in the plot and therefore reduced coefficient of friction and enhanced wear resistance as compared to standard raw-copper specimen. Kovacik et al. [12] contemplated the impact of graphite content on friction coefficient on copper–graphite composites and inferred that there are some key parameters affecting on friction coefficient such as particles size and spatial distribution of graphite particles.

In the plots for friction coefficient vs sliding distance the specimens 6 and 8 showed bit of the higher erratic plot which is due to parameters taken for them, therefore it is not viable to take 1.50 tool tilt angle along with lower rotational speeds (900-950 rpm). Due to these parameters along with solitary pass, proper distribution and size reduction of particles are not possible [11]. Therefore, particles weren't able to squeeze out of the matrix quickly.

5. Conclusions and Recommendation

The following conclusions were made during experimental investigation

1. The average micro hardness value within the stir zone decreased from 180 Hv in the base material to a minimum of 142Hv in a graphite reinforced composites. Hardness were found to decrease with increase in graphite content at constant traverse speed and higher hardness specimens showed better wear resistance at constant rotational speeds.
2. Wear resistance for the surface composite were greatly enhanced compared to pure copper. The values were reduced to 5.26 microns/m from 14.46 microns/m (pure copper).
3. Friction coefficient also reduced to 0.168 from 0.49 for pure copper specimens.
4. Lower tool tilt angle(1.50) and low rotational speeds (900 & 950 rpm) along with solitary pass doesn't give better plots for friction coefficient vs sliding distance so must be avoided. Therefore, tool tilt angles (2.00 and 2.50) gives consistent wear results with range of parameters taken in the work.

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