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EFFECT OF GRAPHENE AND GRAPHENE-FERROMOLYBDENUM ON MECHANICAL, ELECTRICAL, AND THERMAL PROPERTIES OF FSPED COPPER HYBRID COMPOSITES

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SUMMARY

Copper has good electrical and thermal conductivities and is used in numerous electrical and electronic applications as well as thermal management applications, but not for mechanical applications because of its low mechanical strength. In this research work, fabrication of graphene and graphene ferromolybdenum (Gr-FeMo) reinforced copper surface hybrid composites via Friction Stir Processing (FSP) technique is presented, which has high multifunctional properties. Optimum FSP conditions for surface reinforcement of commercially pure copper plates with graphene and graphene ferromolybdenum (1:1) hybrid mixture, respectively, at various volume fractions of 5, 15, and 25% have been performed. The surface hybrid composites thus fabricated have been characterized by SEM, energy-dispersive spectroscopy (EDS), Vickers microhardness, electrical and thermal conductivity. The microstructure analysis indicates that there is no defect formation in the composites due to severe plastic deformation caused by FSP technique. The highest microhardness (62.56 HV) was reached by the copper composite containing 5% of graphene, being 24% greater than the microhardness of untreated copper (50.60 HV), due to grain refinement, Orowan effect, and effective load transferring mechanism. The electrical conductivity of the hybrid composite 5% graphene-ferromolybdenum had the highest value of 1691.34 S/m while untreated copper had the value of 940.24 S/m. Thus, hybrid reinforcing had a positive impact on electron transport properties. Besides, the copper composite having 25% graphene had the highest thermal conductivity value equal to 1204.41 W m⁻¹ K⁻¹ compared to thermal conductivity of untreated copper (549.26 W m⁻¹ K⁻¹) due to good thermal conductivity properties of graphene. From the results received, it could be concluded that reinforcement with graphene and graphene-ferromolybdenum hybrid provides for a balanced reinforcement in terms of strength, electrical and thermal conductivity.

Key words: *friction stir processing (FSP), copper matrix composites, graphene, graphene-ferromolybdenum, surface hybrid composites, electrical conductivity, thermal conductivity.*

INTRODUCTION

Copper exhibits excellent electrical conductivity, thermal conductivity, corrosion resistance, and has good workability. All of these characteristics make copper an appropriate material for electrical, electronic, and thermal applications [1][2]. It is used in the electrical industry, heat exchangers, electronics, and power system applications. Copper, on the other hand, has relatively low ductility and low-strength copper material. This limits the application of copper in high conductivity and high mechanical performance applications [3][4]. In an effort to overcome these limitations, researches have been conducted towards the innovation of copper matrix composites with the addition of reinforcing materials [5]. As a solution to the above limitation, researches have been conducted towards the innovation of copper matrix composites with reinforcing materials [6]. Some of the reinforcing materials that have been added include ceramic particles, carbides, oxides, and metallic particles to increase hardness, strength, wear resistance, and durability of copper [7]. Copper matrix composites can be fabricated by conventional methods such as stir casting and powder metallurgy methods, but many of the composites fabricated suffer from some deficiencies such as agglomeration of the reinforcement, porosity, improper dispersion of particles, and poor particle/matrix bonding [8].

High-quality surface composites can effectively utilize FSP as a solid-state process. In contrast to traditional casting processes, FSP does not suffer from many of the problems that come with casting processes [9]. When friction stir processing occurs, there is severe plastic deformation that will result in dynamic recrystallization that helps to refine the grain size and evenly disperse the particles in the composite [10][11]. As such, the mechanical properties are enhanced without affecting the excellent electrical conductivity of the copper matrix. Many studies have shown that the introduction of reinforcement materials like silicon carbide, aluminum oxide, titanium carbide, tungsten, and nanoparticles has helped improve the performance of the copper composites produced through the friction stir processing technique [12]. These reinforce particles offer improved hardness, tensile strength, wear and corrosion resistance [13][14]. However, many of the ceramic reinforcing materials reduce electrical and thermal conductivity since they interfere with the movement of electrons and heat through the copper matrix [15]. In other words, it is difficult to design copper composites which have improved mechanical and functional properties [16]. Graphene can be regarded as one of the most prospective reinforcing agents as it has excellent mechanical, electrical and thermal properties. It is highly durable and has high electron mobility and thermal conductivity [17]. However, graphene can be considered an appropriate reinforcement material for copper composites as the material has such properties. Yet, graphene has high surface energy which results in agglomeration of graphene during manufacturing [18].

Key Contributions of the Research

- Used FSP technique to fabricate graphene and graphene-ferromolybdenum reinforced copper surfaces with different amounts of reinforcements (5%, 15%, and 25%) in order to create defect-free composites.
- Conducted studies to evaluate the effect of the kind and number of reinforcements on the microstructure, hardness, electrical conductivity, and thermal conductivity of produced composites using SEM and EDS techniques.
- Successfully proved that FSP-based composites have better multifunctionality with hardness as high as 24% higher than conventional composites.

The organization of the remainder of the paper is as follows: Literature review related to Friction Stir Processing and Cu Matrix Composites is provided in Section II. Section III covers the material used, experimental setup, parameters of friction stir processing and the characterization techniques applied in this work. Experimental results of the fabricated composites including microstructure, microhardness, electrical conductivity and thermal conductivity of the fabricated composites are reported in Section IV. The results observed and the effects of graphene and graphene – ferromolybdenum reinforcement on the properties of the fabricated composites are discussed in Section V. Finally, Section VI summarizes the major findings of the work and concludes with directions for future research.

LITERATURE REVIEW

Copper-graphene nanocomposites have been synthesized via FSAM by Khorram et al. [19] in order to improve the properties of copper. As a result of this study, defect-free microstructures with uniform distribution of graphene sheets were observed which caused 56% improvement in tensile strength and increase in electrical conductivity. The study stated that solid-state processing technique increases the bonding between graphene and copper without losing conductivity.

Copper/graphene surface composites have been fabricated via friction stir processing by Naik et al. [20], and the influence of processing conditions on microstructures and properties of fabricated surface composites have been investigated. It is observed that almost 40% increment in the hardness value has been achieved without tunnel defects, while the electrical conductivity is about 97% of IACS. From this research work, it can be seen that FSP is an effective technique to improve the mechanical properties of copper without electrical conductivity degradation.

Cui et al [21] The combined cold spray additive manufacturing and FSP process was performed on alumina dispersion-strengthened (DS) Copper. The results they have obtained were tremendous increases in tensile strength, ductility and electrical conductivity due to the development of ultrafine grains and even distribution of reinforcements. In addition, it is stressed that the FSP process is a critical manufacturing process for creating multifunctional and balanced properties of copper composites.

Guo et al. [22] produced copper-steel bimetallic sheets by Friction Stir Additive Manufacturing (FSAAM) and studied the morphology and mechanical behavior of the developed sheets. It is noted that the fabricated composites possessed good metallurgical bonding, fine microstructure and very high tensile and shear strength than the conventional processed samples. They have revealed that the solid-state processing process is an effective way to produce high-performance copper-based materials with good structural integrity.

Li et al. [23] by FSSP. In this process, nano-TiC particles were dispersed uniformly in the copper matrix, leading to substantial grain refinement and thermal stability. With TiC particles as effective barriers to grain size, the grain growth was effectively inhibited during heat treatment of the composites. The copper composite exhibited tensile strength and elongation values of 294.1 MPa and 41.5% respectively. From the above conclusions, the authors concluded that dynamic recrystallization and uniform dispersion of nano-TiC particles were responsible for the improved mechanical behavior. This suggests that FSSP is a suitable method for the production of copper composites.

Influence of Pre-Deformation and Heat Input on Cu-W Composites Fabricated by FSP was investigated by Oliaei et al. [24]. It was found that through proper optimization of the parameters of processing, the grain structure of the composites turned out to be fine and homogeneous, which positively impacted their hardness and tensile strength. In addition, the samples with optimized parameters had high electrical conductivity because of the reduced number of defects and improved binding between the particles of tungsten and the copper matrix. Thus, it can be stated that the control of heat input and pre-deformation plays a key role in obtaining good properties in Cu-W composites.

Liu et al. [25] have examined the progress of graphene-copper composite materials in terms of processing technology, interface engineering, reinforcement principles, and multifunctionality. According to the review, graphene has been recognized as the promising reinforcement material for copper because of superior mechanical, thermal, and electrical properties. Yet, the problem of the uniform dispersion and good bonding of graphene at the interface is critical and should be studied in detail using hybrid reinforcements and advanced solid-state processing technologies.

In this context, recent literature on Cu-based FSP composites is summarized in table 1. Recent studies on Cu-based friction stir processed (FSP) composites are presented in table 1. This study is focused on the improvement of mechanical, electrical and thermal properties of copper with different types of reinforcement material such as graphene, nano-TiC, alumina, and tungsten. Defect free composites with

refined microstructures have been developed through different solid state processing techniques including FSP, FSSP and FSAM techniques.

Table 1. Summary of recent research on friction stir processed copper-based composites

Authors	Methodology	Dataset / Simulation Environment	Research Gap
Khorram et al. [19]	Fabricated copper–graphene nanocomposites using Friction Stir Additive Manufacturing (FSAM) and evaluated microstructure, tensile strength, and electrical conductivity.	Experimental study using copper reinforced with graphene nanosheets under optimized FSAM conditions.	Focused only on graphene reinforcement; hybrid graphene–metal reinforcement and thermal conductivity analysis were not investigated.
Naik et al. [20]	Produced copper/graphene surface composites using Friction Stir Processing (FSP) and examined hardness, microstructure, and electrical conductivity.	Experimental investigation with different FSP parameters and graphene reinforcement.	Limited evaluation of multifunctional properties such as thermal conductivity and hybrid reinforcement systems.
Cui et al. [21]	Combined Cold Spray Additive Manufacturing (CSAM) with FSP to fabricate alumina dispersion-strengthened copper composites.	Experimental characterization including tensile, ductility, electrical conductivity, and microstructural analysis.	Used only alumina reinforcement; graphene-based hybrid composites were not considered.
Guo et al. [22]	Fabricated copper–steel bimetallic sheets through FSAM and evaluated interfacial bonding and mechanical properties.	Experimental testing of metallurgical bonding, tensile strength, shear strength, and microstructure.	Concentrated on bimetallic joints rather than reinforced copper surface composites for multifunctional applications.
Li et al. [23]	Developed nano-TiC reinforced copper surface composites using Friction Stir Surface Processing (FSSP) and evaluated mechanical and thermal stability.	Experimental analysis of grain refinement, tensile strength, elongation, and thermal stability after heat treatment.	Electrical conductivity and hybrid reinforcement effects were not investigated.
Oliaei et al. [24]	Investigated the influence of heat input and pre-deformation on FSP processed Cu–W composites.	Experimental evaluation of hardness, tensile strength, electrical conductivity, and microstructure under different processing conditions.	Thermal conductivity and graphene-based hybrid reinforcement were not explored.
Liu et al. [25]	Reviewed fabrication techniques, strengthening mechanisms, and properties of graphene–copper composites.	Comprehensive literature review covering powder metallurgy, electro-deposition, additive manufacturing, and FSP.	Highlighted the need for improved graphene dispersion and hybrid reinforcement strategies using advanced solid-state processing.

There have been numerous studies in improving the properties of hardness, tensile strength and electrical conductivity; however, all these works were done using single reinforcement materials. Limited works were done in hybrid system of reinforcement materials with synergistic effect on the properties of hardness, electrical and thermal conductivity of the system. In order to fill these gaps in literature, the present study is conducted to develop composites of graphene and graphene–ferromolybdenum with Cu surface through friction stir processing technique and to evaluate their multi-functional performance.

METHODOLOGY

Base Plate Material and Reinforcements

The materials used in the fabrication of the surface hybrid composite consisted of commercially pure copper plates of dimensions 130 mm × 50 mm × 6 mm based on the excellent electrical and thermal conductivities of copper and its ease of working. These dimensions provided sufficient process time, good heat dissipation, and steady clamping in FSP. In this study, 2 systems of reinforcement were used, consisting of graphene nanoplatelets and hybrid system of graphene and ferromolybdenum with the

weight ratio equal to 1:1. The reinforcement materials were added to the copper surface with volume fractions of 5%, 15% and 25% to investigate the effect of reinforcement on the properties of the material. The reasons for the use of graphene include its high mechanical strength, good thermal conductivity, and ability to process and refine grains. The reason for adding ferromolybdenum was to aid in better dispersion of graphene, reduce agglomeration of particles and improve the bonding between the particles and the copper matrix. The base copper and reinforcement powders were characterized before FSP.



Figure 1. Dimensions of the commercial pure copper base plate used for friction stir processing

The pure commercial copper base plate, acting as a substrate for manufacturing graphene and graphene-ferromolybdenum surface hybrid composite through the process of friction stir processing (FSP), is illustrated in figure 1. Dimensions of the specimen are described as (a) length = 130 mm, (b) width = 50 mm, and (c) thickness = 6 mm, which are the standard dimensions employed in the experiment. This is due to the fact that the above dimensions are appropriate for providing material flow, heat generation, and stable clamping in FSP while still ensuring adequate area for the reinforcement process. The uniform dimensions of the specimen also helped in making comparative analysis of microstructural, mechanical, electrical, and thermal properties of all the manufactured composites under similar conditions.

Tool Material and Fabrication Procedure

Non-consumable WC tool was employed as FSP tool because of its high hardness, high wear resistance and high thermal stability during the processing of copper. The tool has shoulder diameter, pin diameter and pin length of 18 mm, 6 mm and 2.7 mm respectively, which has enough capability to generate sufficient frictional heat and material flow in the stir zone. The WEDM process was applied to cut out the groove on the central part of each copper plate. The depth of the groove was kept constant of 3 mm whereas groove widths of 0.27 mm, 0.81 mm and 1.35 mm were used for 5%, 15% and 25% volume fractions of reinforcement, respectively. The groove was filled with either Graphene or the mixture of Graphene and Ferromolybdenum and afterwards the groove was sealed by using a pinless tool in order to avoid loss of the powder. After that, FSP process was carried out at the spindle speed of 1000 rpm, traverse speed of 20 mm/min and axial force of 6 kN. All the specimens underwent similar processing conditions so as to ensure uniform reinforcement distribution and reliable comparison of the fabricated composites (Figure 2).

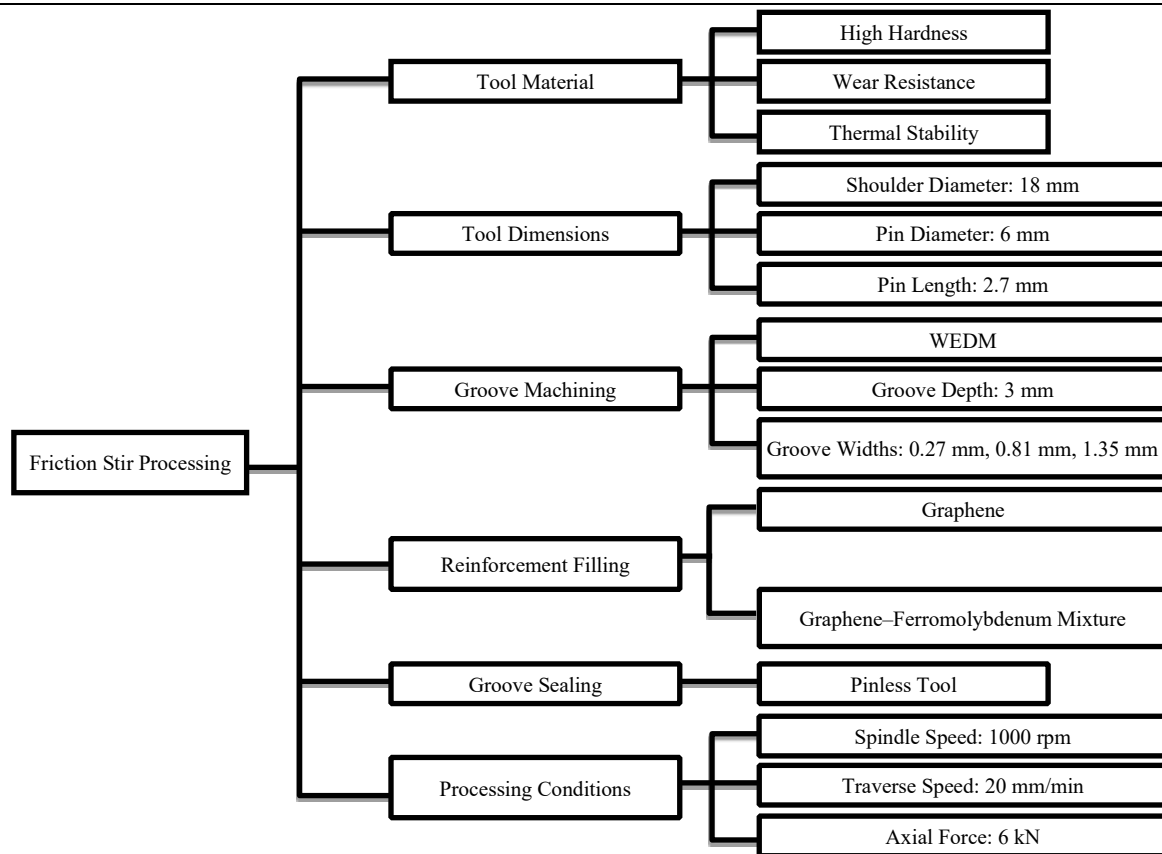


Figure 2. Schematic representation of the friction stir processing (FSP) procedure and processing parameters for fabricating copper hybrid surface composites

Characterization and Property Evaluation

Specimens were removed from the central processed area for microstructural and property assessment after friction stir processing. Prior to characterization, samples were prepared by cutting, grinding, polishing, and chemical etching. The micro-structure and distribution of reinforcement distribution were analyzed by SEM, and the elemental composition was analyzed by EDS to confirm the presence of the reinforcement particles. Microhardness testing was conducted with Vickers microhardness testing method and a load of 20 kg on a VM-50 microhardness testing machine; three indentations were made for each specimen to obtain the average hardness. The prepared cylindrical specimens were used to measure the electrical conductivity and the conductivity values were calculated from the measured resistance. The steady state heat transfer method based on heat transfer rate, specimen dimensions and temperature difference was used to determine thermal conductivity. The obtained microstructural, mechanical, electrical and thermal properties of the processed composites were compared with the unprocessed copper plate to understand how the type of reinforcement and volume fraction affects the overall performance of the fabricated copper hybrid composites.

Figure 3 represents the methodology of experimental characterization and property evaluation carried out for the performance evaluation of the processed copper hybrid surface composites. The methodology involves four stages: microstructure evaluation, mechanical property evaluation, electrical property evaluation and thermal property evaluation. Uniform distribution of reinforcement, elemental composition, and grain refinement were confirmed by the microstructure evaluation and distribution of reinforcement using SEM and EDS. Mechanical properties were evaluated using microhardness test and Vickers hardness of the surface was determined to evaluate the effect of reinforcement on the surface hardness. Electrical properties were evaluated by the measurement of electrical resistance and computation of electrical conductivity of the processed samples. Thermal properties were evaluated using heat transfer technique and applied heat, size of specimens, and temperature differences were considered for calculation of thermal conductivity. This combination of characterization techniques

provides a complete information about the microstructure, mechanical, electrical and thermal behavior of the processed copper hybrid surface composites.

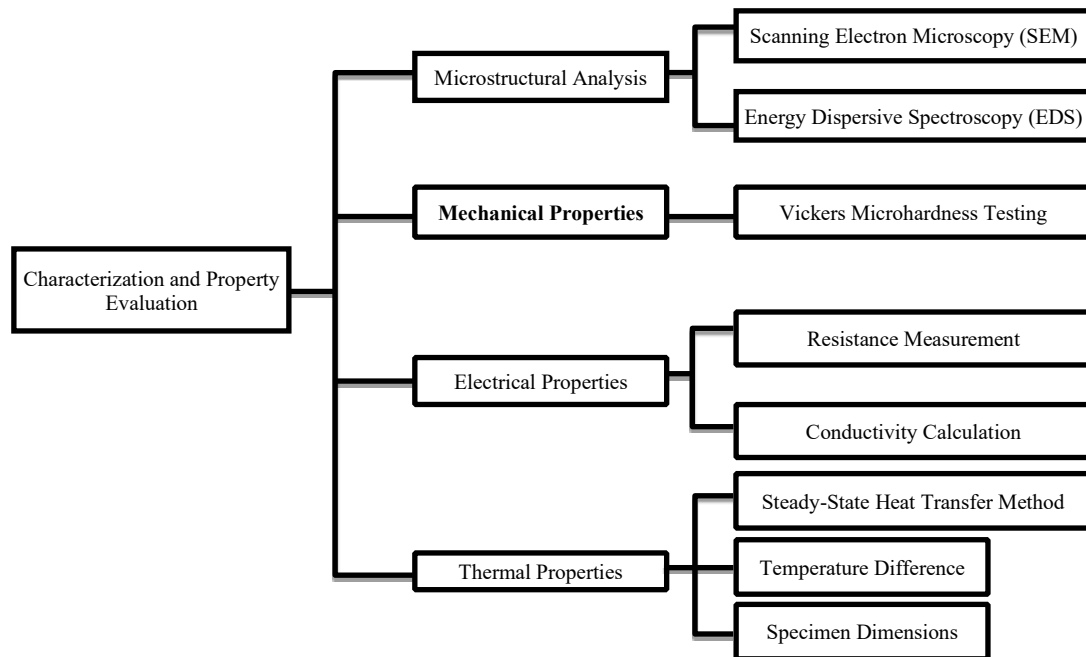


Figure 3. Characterization and property evaluation methods for friction stir processed copper hybrid surface composites

Electrical Conductivity

The electrical conductivity of the fabricated copper composites was calculated using the measured electrical resistance and specimen dimensions. The conductivity is expressed as

$$\sigma = \frac{L}{RA} \quad (1)$$

where:

- σ = Electrical conductivity (S/m)
- L = Length of specimen (m)
- R = Electrical resistance (Ω)
- A = Cross-sectional area (m^2)

The electrical conductivity of the fabricated copper composites was calculated using equation (1), which relates the specimen length, cross-sectional area, and measured electrical resistance. This equation determines the ability of the material to conduct electric current. During the experiment, the electrical resistance of each processed specimen was measured, and the conductivity was calculated using the specimen dimensions. A lower resistance value indicates a higher electrical conductivity. The calculated conductivity values were compared with those of the unprocessed copper to evaluate the influence of graphene and graphene–ferromolybdenum reinforcements. This analysis helps determine whether friction stir processing and reinforcement addition improve or preserve the electrical performance of the copper composites.

Thermal conductivity

$$k = \frac{QL}{A\Delta T} \quad (2)$$

- k = Thermal conductivity (W/m · K)

- Q = Heat transfer rate (W)
- L = Thickness of specimen (m)
- A = Cross-sectional area (m²)
- ΔT = Temperature difference (K)

The value of thermal conductivity of the produced composite materials was measured employing the heat conduction principle of Fourier's law given in equation (2). According to the equation, thermal conductivity of the material is the heat transfer rate that depends on the value of the applied heat, dimensions of the sample, and the difference between two temperatures. The thermal conductivity values were used for assessment of the heat transfer potential of the developed copper composites. The higher the value of thermal conductivity means better heat transfer ability, which is very important for electric and electronic devices.

Algorithm 1: Experimental Procedure for Fabrication and Characterization of Copper Hybrid Surface Composites

Input:

Pure copper plate

Graphene powder

Ferromolybdenum powder

FSP processing parameters

Output:

Mechanical, electrical and thermal properties

Step 1: Select commercial pure copper plate.

Step 2: Prepare graphene and graphene–ferromolybdenum reinforcement powders.

Step 3: Machine groove on the copper plate using WEDM.

Step 4: Fill the groove with reinforcement powder

(5%, 15%, and 25% volume fractions).

Step 5: Seal the groove using a pinless tool.

Step 6: Perform friction stir processing under

selected spindle speed, traverse speed,

and axial force.

Step 7: Prepare specimens from the processed region.

Step 8: Perform SEM and EDS analysis.

Step 9: Measure Vickers microhardness.

Step 10: Measure electrical conductivity.

Step 11: Measure thermal conductivity.

Step 12: Compare all specimens and identify

the optimum reinforcement condition.

End Algorithm

Algorithm 1 integrates the entire experimental procedure used in this study. It starts with the cleaning of the copper body and strengthening materials, then move on to cutting grooves and powder filling. The optimized process parameters are then applied for the friction stir processing, to obtain the hybrid surface composites. The processed specimens are then analysed by SEM and EDS analysis, and then tested for hardness, electrical conductivity and thermal conductivity. At last, the experimental results are compared and the reinforcement composition which gives the best overall results is determined.

EXPERIMENTAL RESULTS

Experimental Setup

For the experiment, the effect of graphene and graphene–Ferromolybdenum on the microstructure and properties of friction stir processed copper surface composite has been investigated as depicted in table 2. Optimized friction stir processing parameters have been used for the production of commercial pure copper that has been reinforced with graphene and graphene–ferromolybdenum with volume fractions of 5%, 15% and 25%. The specimens have been processed and cut, ground, polished and etched for microstructural analysis.

Table 2. Experimental setup and testing conditions

Parameter	Specification
Base Material	Commercial Pure Copper
Reinforcement 1	Graphene Nanoplatelets
Reinforcement 2	Graphene + Ferromolybdenum (1:1)
Reinforcement Volume Fractions	5%, 15%, and 25%
Fabrication Technique	Friction Stir Processing (FSP)
Tool Material	Tungsten Carbide (WC)
Tool Shoulder Diameter	18 mm
Tool Pin Diameter	6 mm
Tool Pin Length	2.7 mm
Spindle Speed	1000 rpm
Traverse Speed	20 mm/min
Axial Force	6 kN
Groove Depth	3 mm
Characterization Techniques	SEM and EDS
Mechanical Test	Vickers Microhardness
Electrical Test	Electrical Conductivity Measurement
Thermal Test	Thermal Conductivity Measurement

The structure and distribution of the reinforcing material have been examined by SEM, while the elemental composition has been verified by EDS. Mechanical properties have been assessed by performing Vickers microhardness tests. Electrical conductivity has been determined by measuring electrical resistance, while thermal conductivity has been calculated under steady state heat transfer condition. The results obtained through experiments have been compared with the results of unprocessed copper samples in order to find out the effects of types and volume fractions of the reinforcing materials on the performance of hybrid composites.

Microstructural Characterization

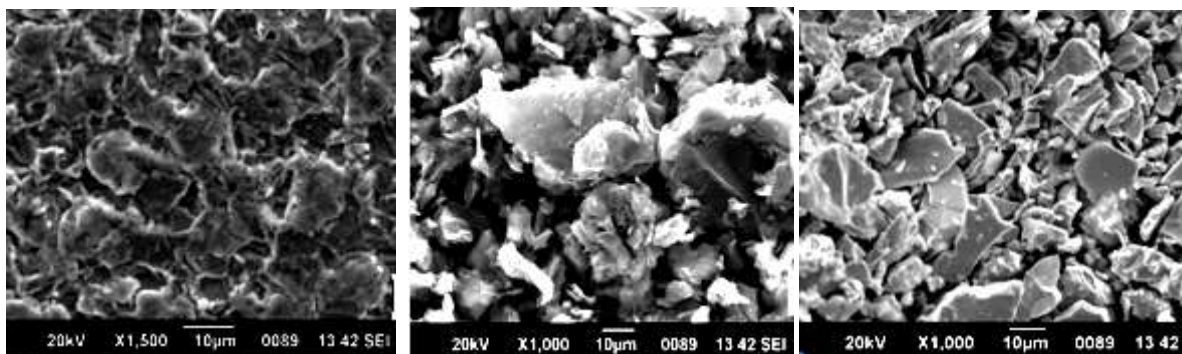
The microstructural characteristics of the prepared surface composites of copper were analyzed using the SEM and EDS. This analysis was conducted to investigate the dispersion of graphene and graphene-ferromolybdenum particles in the matrix of copper and the quality of the processed region.

Friction stir processing provided enough plastic deformation and material flow to achieve uniform dispersion of the reinforcement particles in the stir region. Microstructure analysis of the prepared samples was done based on different volume fractions of reinforcement to identify the effect of volume fraction of reinforcement on grain refinement, particle dispersion, and interface bonding. The processed composites were analyzed using the EDS technique.

Table 3. Microstructural characteristics of friction stir processed copper hybrid composites

Specimen	Reinforcement	SEM Observation	EDS Observation	Microstructural Characteristics
Base Copper	None	Coarse grains	Cu-rich composition	Uniform copper matrix
G-5	5% Graphene	Uniform particle distribution	Carbon detected	Fine grains with good bonding
G-15	15% Graphene	Slight particle clustering	Carbon detected	Refined grains with minor agglomeration
G-25	25% Graphene	Increased agglomeration	Higher carbon content	Non-uniform particle distribution
GFM-5	5% Graphene–Ferromolybdenum	Uniform dispersion	C, Fe, Mo detected	Fine grains and strong interfacial bonding
GFM-15	15% Graphene–Ferromolybdenum	Good reinforcement distribution	C, Fe, Mo detected	Refined microstructure
GFM-25	25% Graphene–Ferromolybdenum	Local particle clustering	C, Fe, Mo detected	Slight agglomeration at higher reinforcement

Summary of the fabricated copper hybrid composites is presented in table 3 showing the SEM and EDS results of the fabricated composites. The results obtained from SEM images reveal that the processing of the components using the friction stir process resulted in refining of the grain structure and distribution of the reinforcement particles in the processed area. Composites containing 5% graphene and 5% graphene-ferromolybdenum had the best uniform distribution of the particles with good bonding between the particles and the matrix with the least defects. With an increase in the content of the reinforcements to 25%, there was more agglomeration of the particles because of the increase in the number of the particles. The EDS results proved the incorporation of the reinforcements in the composites and detection of carbon, iron, and molybdenum in the hybrid composites. Therefore, it can be concluded that the optimal processing conditions in the friction stir process led to refining of the grains and uniform distribution of the reinforcement, thus enhancing their mechanical and functional properties.



(a)

(b)

(c)

Figure 4. SEM images of the reinforcement materials used for fabricating copper hybrid surface composites: (a) Graphene, (b) Ferromolybdenum, and (c) Graphene–ferromolybdenum hybrid mixture

SEM images of the reinforcement material in this research are presented in figure 4. The graphene nanoplatelets have a thin-layered structure with an irregular sheet-like structure, as shown in figure 4(a), providing a very large surface area for proper reinforcement. The Ferromolybdenum particles are presented in figure 4(b) with an irregular polygonal shape, and their size is relatively larger. Figure 4(c) presents the graphene–ferromolybdenum hybrid material, where the graphene sheets are uniformly dispersed among the ferromolybdenum particles, showing the good mixing state prior to the friction stir processing. The above morphology confirms that the selected reinforcement material has suitable particle shape to be incorporated into the copper matrix. After friction stir processing, these reinforcement powders may be useful for grain refinement in copper hybrid surface composites.

Microhardness Analysis

To find out about the effect of the reinforcements of the graphene and the graphene–ferromolybdenum on the surface hardness of the copper, microhardness analysis of Vickers was performed in regard to the copper hybrid composite. Hardness of the unprocessed copper and of the processed one with reinforcement volume fractions 5%, 15%, and 25% was determined. To make sure that the results of the experiment are repeatable and reliable, average values of hardness were obtained through numerous indentations. Obtained hardness was compared in order to see whether the hardness depends on the content and type of reinforcement in FSP. The improvements of the hardness are mainly due to the refined grains, uniform dispersion of the reinforcements and good bonding of the interfacial part during FSP.

Table 4. Comparison of vickers microhardness for friction stir processed copper hybrid composites

Specimen	Reinforcement	Microhardness (HV)
Base Copper	None	50.6
G-5	5% Graphene	62.56
G-15	15% Graphene	57.63
G-25	25% Graphene	55.34
GFM-5	5% Graphene–Ferromolybdenum	59.78
GFM-15	15% Graphene–Ferromolybdenum	56.92
GFM-25	25% Graphene–Ferromolybdenum	54.81

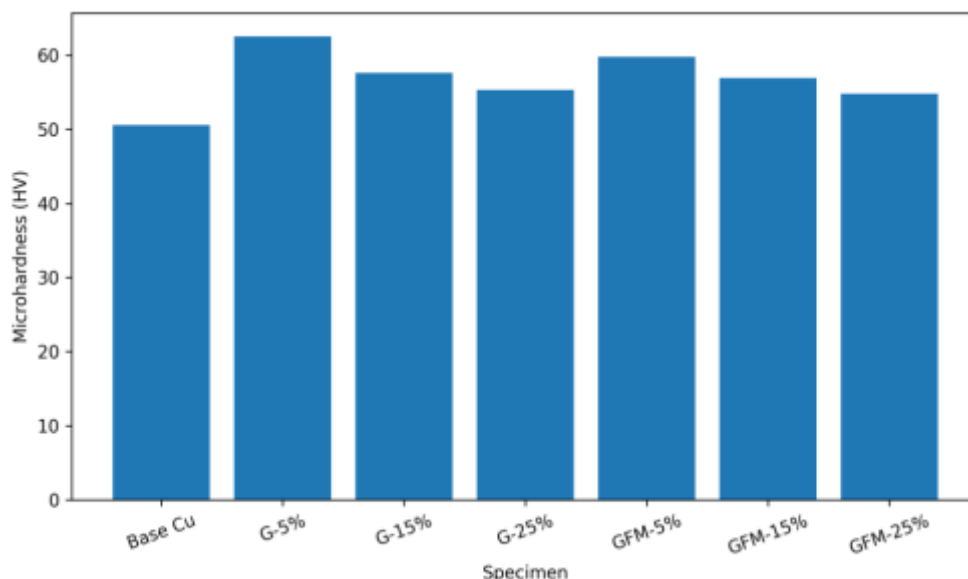


Figure 5. Comparison of vickers microhardness of friction stir processed graphene and graphene–ferromolybdenum reinforced copper hybrid composites

The Vickers hardness measurements of the developed copper hybrid composites are illustrated in table 4 and figure 5. Copper base sample showed the lowest hardness of 50.60 HV because of coarse grains

structure. All other samples with reinforcements revealed higher hardness due to the effect of grain refinement and successful particle strengthening as a result of FSP. The composite reinforced with 5% graphene exhibited the highest hardness level of 62.56 HV and it is 24% higher than that of base copper. The improvement is achieved thanks to the homogeneous distribution of graphene, dynamic recrystallization and effective load transfer within copper matrix. The hardness was higher than the one of the base materials, however, with the introduction of 15% and 25% reinforcement contents, the gradual decrease in hardness was noted due to particle agglomeration and heterogeneous distribution. The hardness was higher for the graphene-ferromolybdenum composites in comparison with the base copper, which implies an enhancement of mechanical characteristics due to hybrid reinforcement.

Electrical and Thermal Conductivity Analysis

The electrical and thermal conductivities of the FSP Cu hybrid composites were investigated in order to evaluate the influence of the presence of graphene and graphene-ferromolybdenum on the functional properties of the fabricated materials. They are highly important for the application in the cases when both electrical and thermal conductivities are required, for instance, electrical contacts, heat exchangers and thermal management systems for electronic packaging. The electrical conductivity was measured by calculating the electrical resistance while the thermal conductivity was calculated according to Fourier's law under the steady state conditions. The obtained values of the conductivity were compared in order to determine the influence of the reinforcement amount on the conductive properties of the composite material. The experimental results provide valuable information regarding the possibility of obtaining high electrical and thermal conductivities in hybrid Cu composites with improved mechanical properties using FSP.

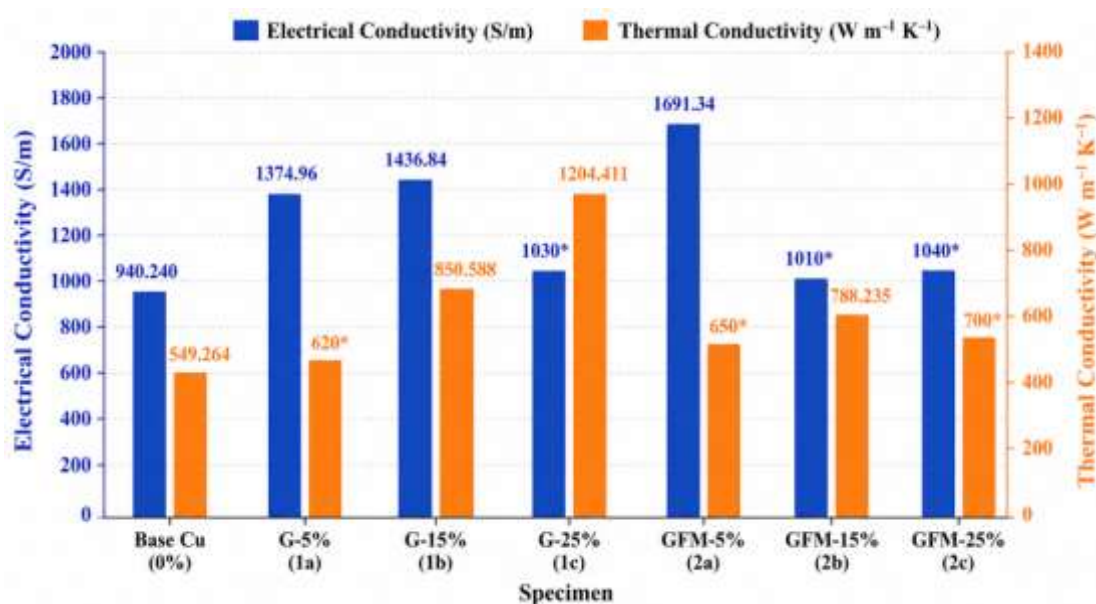


Figure 6. Comparisons of electrical and thermal conductivity

Figure 6 Electrical and Thermal Conductivity of Graphene and Graphene–Ferromolybdenum Reinforced Copper Hybrid Composites via Friction Stir Processing. In figure 6, the electrical and thermal conductivities of the base copper and manufactured composite specimens with different volume fractions of the reinforcements are shown. From the results, it is evident that the FSP process can be employed to improve the conductive behavior of the Cu matrix reinforced by graphene and graphene-ferromolybdenum.

DISCUSSION

From the experimental results, it is clear that the FSP technique is an efficient solid-state processing technique to fabricate graphene- and graphene-ferromolybdenum-reinforced copper hybrid composites with advanced mechanical and functional properties. From the SEM images, it is observed that the

microstructure obtained through the FSP technique is fine and the distribution of reinforcing particles is fairly uniform in the region. Through EDS analysis, the formation of graphene and ferromolybdenum in the copper matrix was confirmed. The microstructural improvements obtained are mainly because of the dynamic recrystallization and severe plastic deformation of the particles attained through FSP.

The micro-hardness findings revealed a significant improvement as compared to the untreated copper specimen. The composite having 5% graphene had the highest hardness, which suggests that the smaller the amount of reinforcement, the better the load-transferring capability between the reinforcement and the copper matrix, and the finer the grain sizes. Slightly lower hardness values were observed in cases where the content of reinforcement increased up to 15% and 25%. This phenomenon is mainly caused by particle agglomeration in the vicinity and the poor distribution of reinforcement in high volume fractions. It is worth noting that the hardness values of all the treated samples were higher than those of the untreated copper.

The findings regarding electrical conductivity demonstrated that the composites had good electrical conductivity after the process. The 5% Graphene–Ferromolybdenum Hybrid Composite had high electrical conductivity, indicating that hybrid reinforcement is capable of enhancing the electrical conductivity of the composites without compromising the integrity of the copper matrix. This improved electrical conductivity is caused by the dispersal of the reinforcements evenly, the creation of fewer defects, and the existence of strong interfacial bonding between the reinforcement and matrix, allowing efficient electron flow. These results are in line with the conclusion drawn regarding FSW enhancing the mechanical properties of copper without influencing its electrical properties.

Similarly, the thermal conductivity of the specimens also recorded a marked increase over the benchmark copper specimen. 25% Graphene-based composite demonstrated maximum thermal conductivity due to the excellent heat conduction property of graphene. Better thermal performance can be attributed to the intrinsic high thermal conductivity of graphene, phonon transport, and defect-free microstructure achieved via friction stir processing. It is evident that the developed composites have the potential to be used in heat dissipation applications.

Conclusion

As a result of this experimental study, the success in the manufacture of copper hybrid composites through the friction stir processing method has been proven. The copper composites have the optimal mechanical, electrical, and thermal properties. The optimum mechanical properties were achieved with 5% graphene, while the optimum electrical conductivity and thermal conductivity were found in 5% graphene-ferromolybdenum hybrid composite and 25% graphene composites, respectively. Considering the obtained results, it can be recommended that the selection of the type and volume fraction of reinforcement should be based on the engineering application. The manufactured copper hybrid composites have great applications in electrical contacts, heat exchangers, power electronic devices, conductive coatings, and thermal management systems.

CONCLUSION AND FUTURE WORK

In this study, graphene and graphene-ferromolybdenum reinforced copper hybrid surface composites have been prepared by friction stir processing (FSP), and the properties like microstructure, mechanical, electrical, and thermal properties of the prepared composites were studied. The uniform dispersion of the reinforcement particles and the fine grain structure of the material have been confirmed through SEM and EDS analysis. The highest hardness of the composite with 5% graphene was 62.56 HV, which was much higher than the hardness of pure copper (50.60HV). Electrical conductivity of the hybrid composite with 5% graphene-ferromolybdenum was measured to be 1691.34S/m which was higher compared to that of base metal (940.24S/m); on the other hand, the maximum electrical conductivity of the 25% graphene composite was 1204.41 Wm⁻¹K⁻¹, which was higher compared to that of base copper (549.264 Wm⁻¹K⁻¹). From the above results, it is seen that there has been improvement in the overall properties of the copper composites by FSP, but still electrical and thermal conductivities have remained very good, which could be useful in manufacturing electrical contacts, heat sinks, power electronics, etc.

The optimization of processing parameters, such as rotational speed of the tool, traverse speed, axial force, and tool geometrical features in friction stir processing, is to be considered as another topic for future research in order to enhance the distribution of reinforcement and composite behavior. Future studies can be carried out to explore other types of nano-reinforcements or new combinations of reinforcements in order to achieve better mechanical strength as well as electrical conductivity. Moreover, long-term testing of wear resistance, corrosion behavior, fatigue, and high-temperature stability should be conducted in order to evaluate the long-term durability of developed composites under real-life conditions. Additionally, numerical simulation and finite element modeling may be applied in order to predict the processes of heat generation, material flow, and microstructure evolution in FSP process.

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