

Effect of CNT Inclusion on Thermo-Mechanical Behavior of Cu-Ni-Zn-Sn Nanocomposite

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ABSTRACT: In recent times, there has been an increasing interest in Carbon NanoTubes (CNTs) reinforced metal matrix composites due to their promising properties such as high Young's modulus and tensile strength. Carbon NanoTubes (CNTs) are known for their extraordinary mechanical, electrical, and thermal properties. These properties make them ideal reinforcements in the metal matrix. Out of all fabrication processes, the powder metallurgy technique was frequently used due to the fewer defects, lesser chance of formation of agglomerations, and the possibility of fabricating composites with nearly net shape in powder metallurgy technique. This research work focuses on developing and analyzing the thermal and mechanical properties of Cu-CNT metal matrix composite. The experimental specimens were fabricated using powder metallurgy in which Copper was ball-milled with Nickel, Zinc, Tin, and Carbon nanotubes of varying compositions were added to the ball-milled copper composites. The resulting powder was compacted using a hydraulic press to form pellets and finally sintered using a Tubular Furnace. Thermal conductivity test and Differential Scanning Calorimetry test were conducted to study the thermal properties of the composites. Mechanical and microstructural studies were conducted on the fabricated components to explore their competency.

KEYWORDS: Metal matrix composites, Carbon nanotubes, Thermo-mechanical properties, Powder metallurgy, Sintering.

INTRODUCTION

In recent years, metal matrix components have been used in many industries and it find wide applications in many fields to produce better-performable materials [1]. Powder metallurgy is an attractive manufacturing process for producing cost-effective high-performance components with superior mechanical behavior and corrosion resistance suited for a great range of engineering applications [2]. A wide variety of materials have been used as matrix or reinforcing agents to produce different nanocomposites based on Powder Metallurgy [3]. The powder metallurgy technique is widely familiar because of intricate shapes

with precise sizes and shapes can be cost-effectively produced at a high production rate. In the powder metallurgy process, the combination of elemental or pre-alloyed powders is compressed in a die and sintered in a furnace to bond the particles [4-6]. The thermal conductivity of the composites was not enhanced by the incorporation of CNTs. Besides the effect of the sintering condition, the existence of interface thermal resistance between the CNT and the Cu matrix was considered to be the main reason for this unexpectedly low thermal conductivity. At higher temperatures above 600C,

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the composite conductivity decreased with increasing sintering temperature and holding time, and this degradation was enhanced with the increase in CNT content. The presence of CNTs segregation formed in the matrix grain rearrangement process may be a disadvantage for resulting in the reduction in thermal conductivity at higher temperatures which is proved by the previous studies conducted with CNT and different types of matrix. Dissimilar varieties of ceramic materials are widely used to reinforce aluminum alloy matrixes due to their behaviors such as refractoriness, high hardness, wear resistance, etc. Components with challenging dimensions and high strength can be easily manufactured by the powder metallurgy method [7]. The strengthening mechanisms of Aluminium matrix nanocomposites reinforced with CNTs produced by powder metallurgy were studied by many researchers. A classical powder metallurgy route was performed, using two different dispersion/mixture methods: ultrasonication and its combination with ball milling. This step of the production of nanocomposites is crucial to the dispersion of CNTs, ensuring its efficiency while obtaining a damage-free CNT structure so that the strengthening effect occurs. In this sense, the uniform dispersion of the CNTs with minimal damage is the key to improving the mechanical properties of the nanocomposites. Strain hardening and second-phase hardening are also identified since the formation of nanometric Al₄C₃ particles was observed together with a slight increase in dislocation density. Regarding grain size and texture hardening, as no significant differences were detected between the nanocomposites and the matrix, it was impossible to confirm their contributions to the Al-CNTs strengthening produced under these conditions.

EXPERIMENTAL SECTION

Materials

The materials used in the fabrication of the specimen are Copper, Carbon Nanotubes, Nickel, Zinc, and Tin. Copper exhibits excellent thermal and electrical conductivity [8]. Due to its abundance availability, copper is less expensive and hence it is commonly used for transmission of electricity and other applications. The ductility nature of copper makes it ideal for manufacturing wires and cables [9]. Copper is also well known for its excellent thermal properties; the typical thermal conductivity of pure copper is 386.00 W/(m.K) at 20 degrees Celsius.

Copper also has a high melting point of 1,085°C and hence it can be used for high-temperature applications such as bases for cooking utensils and heat exchangers in boilers. Usually, the incorporation of CNT in metal matrix often leads to the production of composites with higher mechanical, electrical, and thermal properties. Copper is used in this research in order to analyze the changes in thermal and Mechanical properties when composited with Carbon NanoTubes (CNT). Carbon NanoTubes (CNTs) are cylindrical molecules that consist of rolled-up sheets of single-layer carbon atoms (graphene). They can be single-walled with a diameter of less than 1 nanometer (nm) or multi-walled, consisting of several concentrically interlinked nanotubes, with diameters reaching more than 100 nm [10]. Carbon nanotubes can exhibit remarkable electrical conductivity. They also have excellent tensile strength and thermal conductivity because of their nanostructure and the strength of the bonds between carbon atoms. Some of the notable properties of CNT are high thermal conductivity, have high electrical conductivity, low thermal expansion coefficient, very high tensile strength, high elastic properties. Unlike traditional reinforcements such as brittle ceramics, CNTs have the added benefit of acting as highly functional materials in the surrounding metal matrix medium [11]. Nickel is a Silvery – White lustrous metal that is ferromagnetic in nature. The major use of nickel is in the preparation of alloys. Nickel alloys are characterized by high strength, more ductility, and resistance to corrosion and heat [12]. When Nickel is alloyed with copper, its properties improve, such as higher corrosion resistance, better strength, and hardness. The addition of CNT to Ni system resulted in increased hardness when compared to that of pure Nickel. It was observed that there is an overall improvement in the mechanical and thermal properties of the Ni – CNT system. Zinc has a silvery–greyish appearance and is brittle at room temperature. The major uses of zinc metal are in galvanizing iron and steel against corrosion and in making brasses and alloys for die-casting [13]. When Zinc is alloyed with copper, Brass alloys are formed. Brass can be alloyed with zinc in different proportions, which results in a material of varying mechanical, corrosion, and thermal properties. Increased amounts of zinc provide the material with improved strength and ductility. Zinc with CNT shows betterment in thermal and mechanical properties when compared to pure Zinc



Fig. 1: Experimental Set up

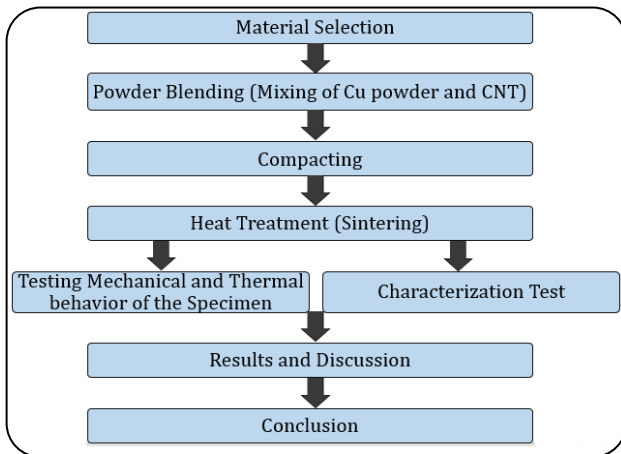


Fig. 2: Experimental Methodology

metal. Pure tin after solidifying presents a mirror-like appearance similar to most metals, Tin is widely used for plating steel cans used as food containers, metals used for bearings, and solder [14].

Methodology

The experimental specimens were fabricated using a powder metallurgy process. Copper was ball-milled with Nickel, Zinc, Ti, and Carbon nanotubes of varying compositions were added to the ball-milled copper matrix. The resulting powder was compacted using a hydraulic press to form a pellet and finally sintered using a Tubular Furnace. Thermal conductivity tests and Differential Scanning Calorimetry tests were conducted to study the thermal properties of the composite. For Studying the mechanical properties of the composites, a detailed microstructural study was conducted comprising of

Optical imaging using Metallurgical Microscope, SEM analysis, and Micro hardness test using a Vickers hardness machine and finally X-ray diffraction test was conducted. The Experimental setup built in-house is shown in Fig. 1. The Experimental methodology followed in this research work is represented in Fig. 2. Based on the previous studies conducted by many researchers, the various weight percentages of CNT are selected. The composition of the Specimen prepared is given in Table 1.

RESULTS AND DISCUSSION

Thermal conductivity

The Thermal Conductivity test was conducted on the four specimens using two Slab Guarded Hot Plate methods where the specimen was each heated to 150°C and the values T_1 to T_6 were noted at intervals 50, 75, 100, 125, and 150 °C. The obtained values were used to calculate the thermal conductivity of the specimen at the given temperature using the formula [15].

$$k = \frac{q}{2} \frac{4\Delta x}{\pi D^2} \frac{1}{(T_h - T_c)} \quad (1)$$

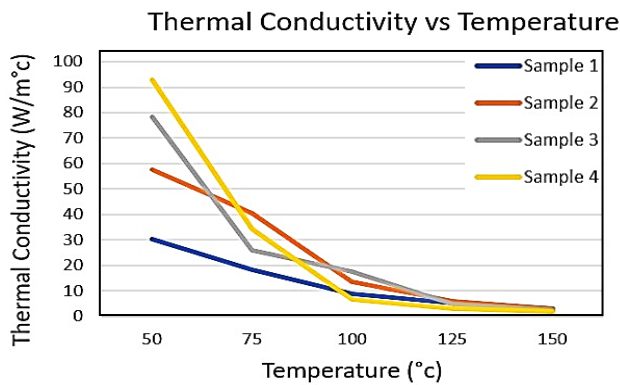
k – Thermal conductivity in $W/m^\circ C$, q – Heat rate in W/s , Δx – Thickness of sample in mm, D – Diameter of sample in mm, T_h – Temperature of upper plate in $^\circ C$, T_c – Temperature of the lower plate in $^\circ C$. Based on the calculations made, the thermal conductivity of each of the specimens at a given temperature interval was found, with which the graph was plotted. The graph was plotted from the obtained thermal conductivity values with Temperature ($^\circ C$) at X – axis and Thermal Conductivity ($W/m^\circ C$) at the Y-axis.

Table 1: Composition of Specimen

Sample	Cu	Sn	Ni	Zn	CNT	Total	
1	In %	81	8	10	1	0	100
	In grams (25gns)	20.25	2	2.5	0.25	0	25
2	In %	80.9	8	10	1	0.1	100
	In grams (25gns)	20.225	2	2.5	0.25	0.025	25
3	In %	80.8	8	10	1	0.2	100
	In grams (25gns)	20.2	2	2.5	0.25	0.05	25
4	In %	80.6	8	10	1	0.4	100
	In grams (25gns)	20.15	2	2.5	0.25	0.1	25

Table 2: Thermal Conductivity of the Samples

Temp. (°C)	Sample 1 (W/m °C)	Sample 2 (W/m °C)	Sample 3 (W/m °C)	Sample 4 (W/m °C)
50	30.317	57.752	78.412	92.801
75	18.362	40.497	32.083	34.521
100	9.031	13.635	17.556	20.754
125	5.402	5.847	4.874	3.152
150	2.978	2.944	2.632	2.079

**Fig. 3: Thermal Conductivity of the samples at various temperatures**

The values of thermal conductivity of the fabricated samples are provided in Table 2. The Graphical representation is given in the Fig. 3.

From the graph, it can be interpreted that at 50 °C, all of the specimens display higher thermal conductivity values. This was observed as the specimen was near room temperature. At 75 °C, a fall in thermal conductivity was noticed with Sample 2 consisting of 0.1% vol of CNT and Sample 4 (0.4% vol CNT) having a thermal conductivity value of 40.497 W/(m.°C) and 34.521 W/(m.°C) respectively. This can be due to the property of CNT which implies decrease in thermal Conductivity with an increase in temperature. At 100 °C, the Thermal Conductivity further decreases with Sample 2 and Sample 3 having higher Thermal Conductivity values of 13.635 and 17.556 W/(m.°C). The same is observed for the 125 °C with Sample 1 and Sample 2 sharing similar values of Thermal Conductivity (5.402 and 5.847). At 150 °C, all four samples show minimum thermal conductivity with Sample 3 consisting of 0.2 vol % CNT having a Thermal Conductivity value of 2.644. From the above results, it can be observed that all the samples show high thermal conductivity at 50 °C and

it keeps decreasing with an increase in temperature. It can also be observed that the Sample with CNT (Samples 2-4) showed better Thermal Conductivity than the sample without CNT. Therefore, it can be concluded that the addition of Carbon Nanotubes (CNT) to the Metal Matrix (Copper, Nickel, Zinc, Tin) has improved the Thermal Conductivity significantly, with Sample 2 and Sample 3 showing stable thermal only throughout the experiment.

Differential scanning calorimetry

Differential Scanning Calorimetry (DSC) is a thermal analysis technique in which the heat flow into or out of a sample is measured as a function of temperature or time, while the sample is exposed to a controlled temperature [16]. In this study, the differential scanning calorimetry is used to evaluate the properties of the Cu-CNT components such as glass transition temperature, melting point, crystallization temperature, and heat of melting. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned.

From the above Fig. 4 & Fig .5, it can be observed that, For Sample 1 and Sample 2, the glass transition temperature was observed to be around 76°C each and melting occurred at 225.8°C and 229.8°C, this may be due to the inclusion of Zinc and Tin which have low melting points. Low-temperature crystallization occurred at 269.0°C for sample 1 and 277.9°C for sample 2. Then again melting is observed at around 400°C after which recrystallization takes place for both Sample 1 and Sample 2. For Sample 3 and Sample 4 shown in Fig. 6 & Fig. 7, the glass transition temperature is 154°C for Sample 3 and 76.8°C for Sample 4. There is a deep negative melting peak for Sample 3 at 230°C. For Sample 4,

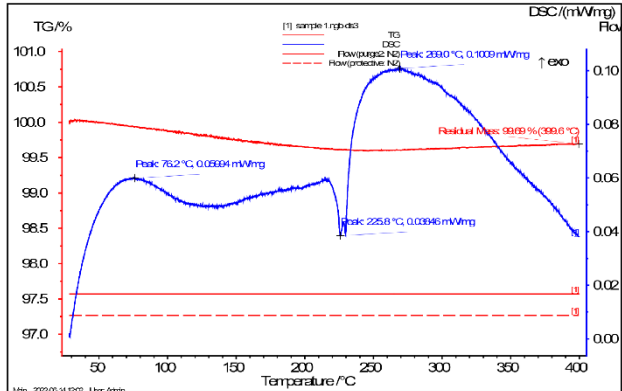


Fig. 4: Differential Scanning Calorimetry (DSC) Graph for Sample 1

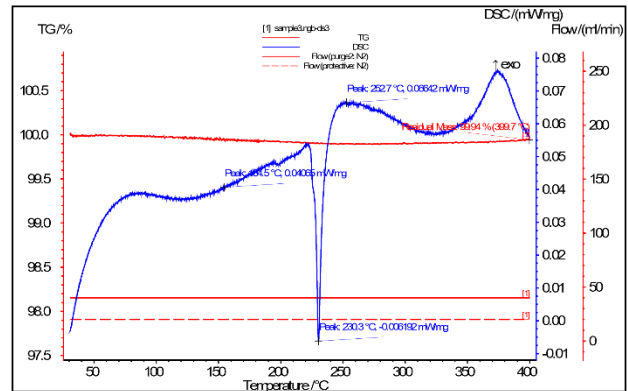


Fig. 6: Differential Scanning Calorimetry (DSC) Graph for Sample 3

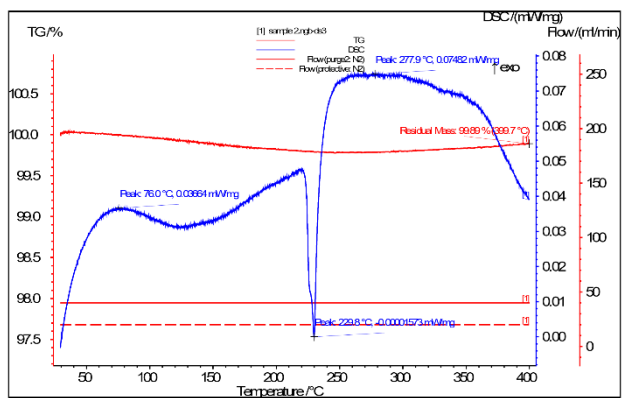


Fig. 5: Differential Scanning Calorimetry (DSC) Graph for Sample 2

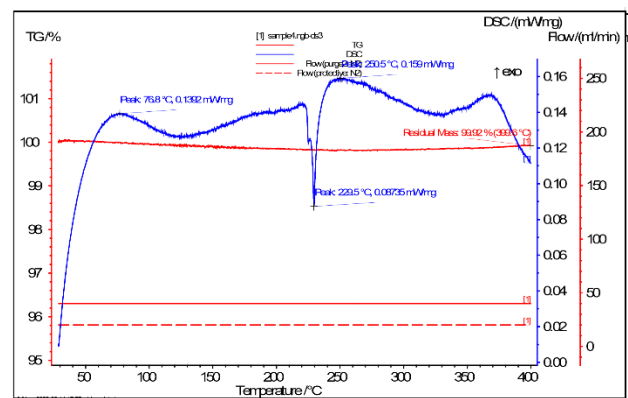


Fig. 7: Differential Scanning Calorimetry (DSC) Graph for Sample 4

the melting takes place at 229°C. The Low-temperature crystallization occurred at around 250°C for both Samples 3 and 4 and again melting takes place after which recrystallization takes place. Thus, important properties of the Cu-CNT components such as glass transition temperature, melting point, crystallization temperature, and heat of melting are observed.

Microstructural studies

A Scanning Electron Microscope (SEM) is used to study the surface topography and composition of all the samples. The four samples were molded and the surface was polished before being etched. The etchant used for the Cu-CNT composite was Ferric Chloride with 3% HCl. The SEM images were captured soon after the surface was etched.

The microstructural characteristic of Sample 1 was investigated by high resolution scanning electron microscope and is shown in Fig. 8. The scanned electron microscopic studies are conducted to confirm the excellent

interfacial bonding between the copper matrix and CNT reinforcement. Scanned electron microscopic studies are conducted to reveal the dispersion of zinc and Tin in the copper matrix at a slow melting process. It has been observed that the Zinc and Tin have been well fused with the matrix without any agglomeration due to their lower melting points. The images show traces of Nickel and Zinc on the surface of the image. From Fig. 9, it has been found that the copper and the other metals (Nickel, Zinc, Tin) are well fused. The CNTs have formed agglomerates around the surface of Sample 2. From the SEM image of sample 3 shown in Fig. 10, it is observed that the CNTs have dispersed well in the metal matrix with copper and other constituents of the specimen. There are fewer agglomerations as compared to the 2nd Sample with 0.1% vol. CNT. The SEM image of sample 4 shown in Fig. 11 with 0.4% vol. CNT, shows very good dispersion of CNT in the metal matrix. CNTs are aggregated into ropes due to strong interfacial van der Waals attraction. Thus, from

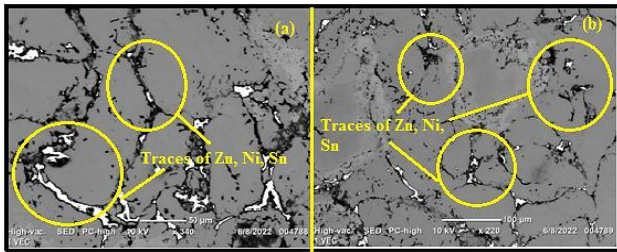


Fig. 8: SEM images of sample 1 (a): 50 μm , (b): 100 μm

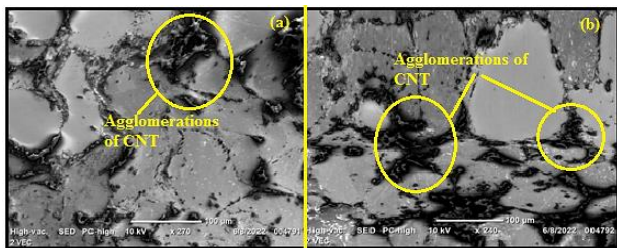


Fig. 9: SEM images of sample 2 (a): 50 μm , (b): 100 μm

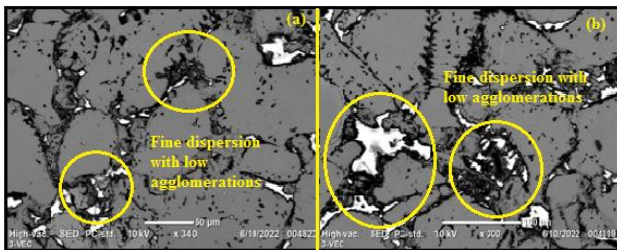


Fig. 10: SEM images of sample 2 (a): 50 μm , (b): 100 μm

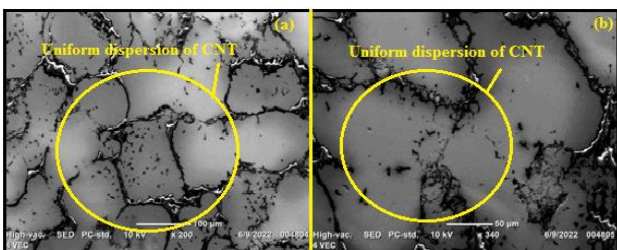


Fig. 11: SEM images of sample 2 (a): 100 μm , (b): 50 μm

the SEM analysis of the Cu-CNT composites, the microstructure was studied and it can be concluded that the CNTs have been well dispersed in the metal matrix without much agglomeration. The metals, especially Zinc and Tin have been fused well together with the other metals and components in the matrix due to their low melting points.

Optical microscopic studies

An image analyzer is used to study the cross-sections of metal targets and metallurgical mounts of the fabricated samples. The evolution patterns on the surface of the samples



Fig. 12: Molded specimen for image analyzer

can be well studied by using image analyzer [17]. The specimen mounted is represented in Fig. 12.

To study the effect of thermal loading on the surface of the fabricated nanocomposites, optical microscopic studies are conducted. For the test to be conducted, first the specimen was molded with the surface of the specimen polished and buffed. In order to view the microstructure with better contrast, it was etched with Ferric Chloride with 3% HCl. At lower magnifications, the whole of the metal matrix can be observed with Copper, Nickel, Tin, and Zinc being distinctively visible. As the surface of the specimen is magnified, each component of the composition is clearly visible i.e., at 200x, Copper, Nickel, and Zinc can be observed. At 500x, parts of Nickel and Zinc can be seen. The optical microscopic image of sample 1 is represented in Fig. 13. From the optical microscope image for Sample 2 shown in Fig. 14, it can be observed that there is considerable agglomeration of CNT. This can be clearly observed in the images with higher magnification (100x, 200x, 500x). Each of the components of the matrix are clearly visible at lower to higher magnification. For sample 3 shown in Fig. 15, the CNTs are dispersed well with the metal matrix. This can be observed well in the 100x and 200x magnification. The metals have fused well with each other. From the Optical Microscope image of Sample 4 given in Fig. 16, it can be observed that the CNT is dispersed well along with the metal matrix. All the components of the specimen especially Tin and Zinc have fused well and cannot be distinguished easily. Thus, from the microstructural analysis of Cu – CNT composites by Optical Microscope,

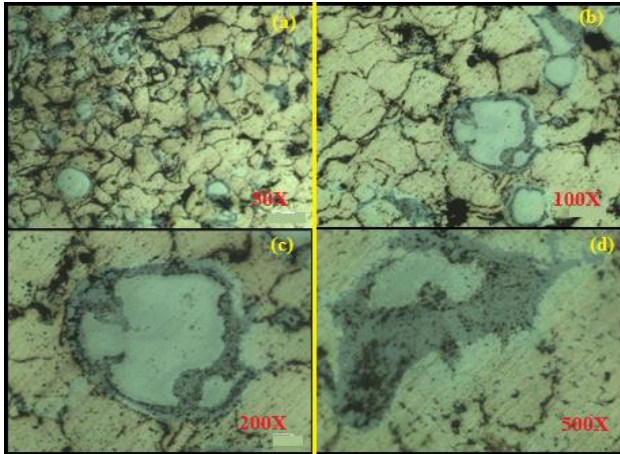


Fig. 13: Optical Microscopic image of Sample 1

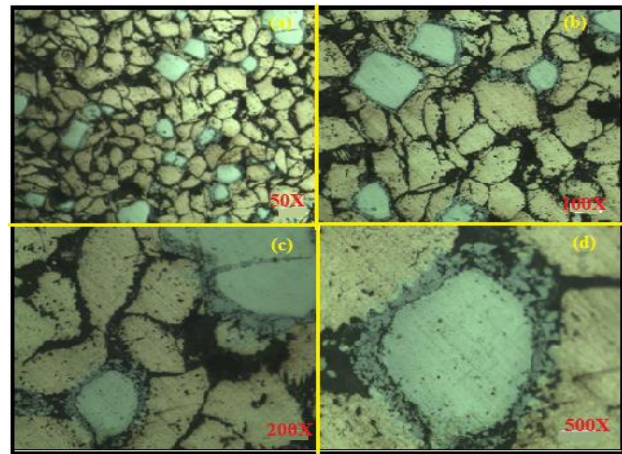


Fig. 15: Optical Microscopic image of Sample 3

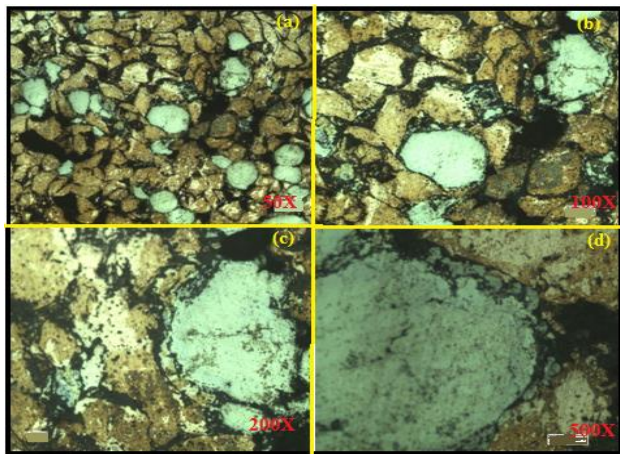


Fig. 14: Optical Microscopic image of Sample 2

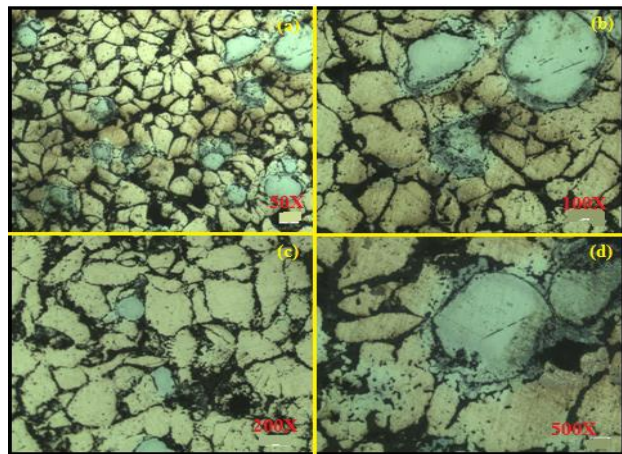


Fig. 16: Optical Microscopic image of Sample 4

it can be concluded that the CNTs have dispersed well in the metal matrix with minimal agglomeration.

Micro Vickers hardness test

The Vickers hardness test is used to find the microhardness of the fabricated four compositions of samples. The Vickers indenter is pressed into the surface to a specified force. The force is usually held for 10 seconds. After the indentation is finished, the resulting indent is analyzed optically to measure the lengths of the diagonals to determine the size of the impression on the CU-CNT samples. From the obtained values, it can be concluded that the microhardness values are improved well with the inclusion of CNT in the metal matrix. The micro-hardness values of all the samples are shown in Fig. 17. This may be attributed to the excellent load-bearing capacity of carbon nanotubes and the excellent interfacial bonding and distribution of carbon nanotubes

with the matrix material [18]. The microhardness value increased with the increase in CNT content so, 3rd and 4th samples with 0.2 and 0.4 % volume CNT show the best hardness value. The quantified values of microhardness are shown in Table 3.

CONCLUSIONS

In this research work, the powder metallurgy process is used to fabricate the metal matrix nanocomposites comprising Copper, Nickel, Tin, Zinc, and Carbon Nanotubes. The powder metallurgy process is one of the best methods to fabricate a metal matrix composite as machining work is minimized and there is less scrap loss [19-22]. Copper is well known for its good thermal and electrical conductivity. The key applications of copper nanoparticles are they act as an anti-biotic, anti-microbial, and anti-fungal agent when added to plastics, coatings, and textiles, Efficient catalysts for chemical reactions and

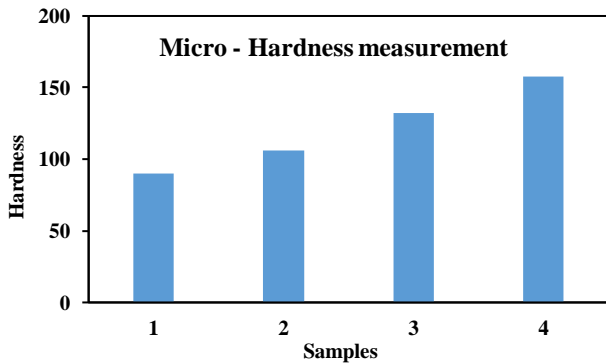


Fig. 17: Micro-Hardness measurement.

for the synthesis of methanol and glycol, as sintering additives and capacitor materials, Superficial conductive coating processing of metal and non-ferrous metal, and nanometal lubricant additives [22-31]. The CNTs have an outstanding thermal conductivity and mechanical properties [32]. At 50 °C, all of the specimens display higher thermal conductivity values with Sample 4 consisting of 0.4% vol CNT. This was observed as the specimen was near room temperature. At 75°C, we can notice a fall in Thermal Conductivity values with Sample 2 consists of 0.1% vol of CNT and Sample 4 (0.4% vol CNT) having a thermal conductivity value of 40.497 W/m°C and 34.521 W/m°C respectively. This can be due to the reason of a property of CNT which implies a decrease in Thermal Conductivity with an increase in temperature. At 100°C, the Thermal Conductivity further decreases with Sample 2 and Sample 3 having higher Thermal Conductivity values of 13.635 and 17.556 W/m°C. The same is observed for the 125 °C with Sample 1 and Sample 2 sharing similar values of Thermal Conductivity (5.402 & 5.847). At 150°C, all four samples show minimum thermal conductivity with Sample 3 consisting of 0.2 vol % CNT having a Thermal Conductivity value of 2.644. The Cu-CNT composite is synthesized by powder metallurgy process and 4 samples with variation in CNT contents were fabricated. Thermal, Mechanical, and microstructural characterization tests were conducted on the finished specimen in order to study its properties. The fabricated nanocomposite is recommended for corrosion-resistant components It was observed that the addition of CNT to the Copper metal matrix has resulted in enhanced thermal properties such as better thermal conductivity in specimens with CNT when compared with the specimen fabricated without CNT [33]. The SEM

Table 3: Vickers microhardness values for the specimens

Sample	Hardness value in HV300gm
1	90
2	106
3	132
4	158

and Optical Microscope images show well dispersion of CNT in the matrix and limited agglomeration was found [34, 35]. The mechanical property of the specimen was observed by conducting a Vickers Micro hardness test, from which it was observed that the hardness value increased with the increase in CNT content but limited to 0.4 vol% CNT after which the hardness value reduced. Hence, it can be concluded that there is good improvement in the Thermal and Mechanical properties of the Cu-CNT when the CNT content is kept to optimal levels.

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