

Microstructure and Microhardness of Carbon Nanotube-Silicon Carbide/Copper Hybrid Nanocomposite Developed by Powder Metallurgy

H. M. Mallikarjuna^{1,6*}, C. S. Ramesh², Praveennath G. Koppad³,
K. T. Kashyap⁴ and R. Keshavamurthy⁵

¹Advanced Composites Research Centre (ACRC), PES Institute of Technology, Bangalore - 560085, Karnataka, India;
mallikarjuna.hm@gmail.com

²Department of Mechanical Engineering, Alliance College of Engineering and Design, Anekal, Bangalore - 562106
Karnataka, India

³Research and Development, Rapsri Engineering Products Company Ltd, Ramanagar, Ramanagar - 562112,
Karnataka, India

⁴Department of Mechanical Engineering, Atria Institute of Technology, Bangalore - 560024,
Karnataka, India;

⁵Department of Mechanical Engineering, Dayananda Sagar College of Engineering, Bangalore - 560078,
Karnataka, India

⁶Department of Mechanical Engineering, Government Engineering College,
K. R. Pet- 571426, Karnataka, India

Abstract

The main objective of current work is to improve the mechanical properties of copper without losing their inherent electrical conductivity. In this regard, we have reported the effect Multiwalled Carbon Nanotubes (MWCNTs) and Silicon Carbide (SiC) reinforcement addition on microstructure, microhardness and electrical properties of copper-based hybrid nanocomposite. Hybrid nanocomposites have been synthesised by powder metallurgy technique which involves blending of composite powder, compaction, sintering followed by hot pressing. Microstructural characterization of prepared hybrid nanocomposites were carried out using optical and Scanning Electron Microscope (SEM). Developed nanocomposites were also subjected to density measurement, microhardness test and electrical conductivity studies. Optical and SEM images reveal that SiC particles and MWCNTs were dispersed uniformly within the copper matrix. The densities of nanocomposites were significantly reduced owing to low densities of MWCNTs and SiC. An improvement in hardness of about 1.5 times has been observed for nanocomposites when compared with pure copper prepared under similar conditions. However, reduction in electrical conductivity of nanocomposites was observed due to grain refinement caused by multiple reinforcements. The developed hybrid nanocomposites have the potential as material candidates for self-lubricating bearings and bushes.

Keywords: Carbon Nanotubes, Copper, Hybrid Nanocomposites, Microhardness, Powder Metallurgy, Silicon carbide

1. Introduction

Carbon nanotube (CNT), a new allotrope of carbon was discovered by a Japanese scientist¹. Carbon nanotubes possess superior thermal (3000 W/m K) and electrical

conductivity, high Young's modulus up to 2 TPa and strength up to 63 GPa. In addition to this, low density and almost zero coefficient of thermal expansion allow for them to be added to other matrix materials. Since then, carbon nanotubes have emerged as one of the important

* Author for correspondence

material as a reinforcement for metal matrix composites owing to their extraordinary mechanical, thermal and electrical characteristics². Copper is ductile, soft, and highly conducting metal. Copper is widely used for making electrical cables and heat dissipation components in electronic circuit board due to their high thermal and electrical conductivity when compared with other metals and alloys. Due to low strength and wear resistance, several attempts have been made by many research groups across the world to develop copper based composites with better mechanical properties without sacrificing its natural thermal and electrical characteristics. Various reinforcements like SiC, Al₂O₃, TiB₂, graphite, diamond, carbon fiber and carbon nanotubes are being used to enhance the mechanical, wear resistance and thermal properties of monolithic copper³⁻⁷.

Many research works have been published on carbon nanotubes reinforced into plastic, ceramic and metal matrix composites. Especially vast number of research papers can be found on CNT reinforced polymer nanocomposites⁸⁻¹⁰. Most of the papers have reported that CNTs reinforced composite do exhibit better mechanical and tribological properties. Carbon nanotubes reinforced composites have been processed by various techniques such as conventional powder metallurgy, cold and Hot Isostatic Pressing (HIP), Spark Plasma Sintering (SPS), Equal Channel Angular Pressing (ECAP), and planar shock-wave compaction^{2,11}. Copper-based nanocomposites reinforced with 12-15 vol % CNTs were developed by well-established powder metallurgy to study microhardness and tribological properties. The microhardness of composite was found to be in the range of 100-115 HV. Microhardness was increased with increase in carbon nanotube content till 15 vol.% and then start reducing due to agglomeration and porosity. Significant reduction in friction and wear loss was observed with increase in carbon nanotube due to its lubrication behavior and tribolayer formed between composite and counter surface¹². Carbon nanotube reinforced copper nanocomposites were consolidated by High Ratio Differential Speed Rolling (HRDSR) process to study the tensile strength. The developed composite showed significantly improved in uniform dispersion and alignment of CNTs. Prepared nanocomposite exhibited better mechanical property compared with both pure copper and conventionally produced composites. It was observed that significant decrease in grain size of nanocomposite with 3 vol.% CNT (0.6 μm) when

compared with grain size of copper is 1.9 μm . Increase in ultimate tensile strength of nanocomposite to 500 MPa whereas for copper it was 280MPa. The increase in tensile strength was attributed to large amount of redundant shear strain induced on nanocomposite during HRDSR¹³. Copper based composite reinforced with carbon nanotube were developed by powder metallurgy technique followed by hot forging to investigate microhardness. The developed nanocomposites showed increase in microhardness from 70 for pure copper to ~145 HV for 4 wt.% CNT reinforced copper nanocomposites¹⁴. CNT reinforced copper and copper alloy composite were fabricated by hot press sintering to study the effect of size and shape on mechanical properties. The particle size of copper metallic powder was varied between 3 to 45 μm and also different shapes of copper powder (dendrite and spherical) were used to investigate the behavior of composite. It was found that hardness of composite with 0.5 wt.% CNTs decreases with increase in particles size of copper. However, hardness of pure copper increases with increase in particle size up to 10 μm . Further, hardness remains stable with increase in particle size. BHN of composite reduces with increase in wt.% of carbon nanotube in composite with particle size of 45 μm . However, BHN of composite increases with increase in content of carbon nanotube with particle size of 3 μm and electrical conductivity decreases with increase in wt.% of CNTs in comparison with pure copper due to agglomeration and porosity¹⁵. Copper-carbon nanotube nanocomposites were developed by spark plasma sintering to investigate the hardness of composite. CNTs content was varied from 5-20 vol. % for copper matrix composite. The micro hardness of CNT/Cu nanocomposite is 1.4 GPa which is almost 2.1 times higher than pure copper. Yield strength of composite is increased to 2.85 times higher than that of pure copper. The electrical conductivity reduced as increase in content of carbon nanotube due to interfacial resistance between matrix and reinforcements¹⁶.

There are many challenges and issues are involved in developing nanocomposites with better mechanical, thermal, and electrical property. These include homogeneous distribution, maintain good bonding between reinforcement and matrix, reducing porosity and orientation of reinforcement along the loading direction especially in case of carbon fiber and CNTs. In order to achieve uniform distribution of reinforcements in matrix especially nanomaterials like CNTs and nanoparticulates, most of researchers have adopted ultra-sonication followed

by mechanical alloying technique. The non-wetting nature of reinforcements with copper is addressed by electroless coating of copper or nickel^{17,18}. Therefore, to improve the interfacial bonding strength between carbon nanotubes and silicon carbide with copper matrix, electroless coating method is adopted to coat the reinforcements. Porosity of the nanocomposite is minimized by optimizing process parameters such as compaction pressure, sintering temperature, time and application of secondary process like extrusion, forging or rolling.

In recent years, the demand for materials with multifunctional characteristics like high thermal and electrical conductivity coupled with good mechanical and tribological properties is increasing as they are extremely important for engineering components which are simultaneously subjected to mechanical, thermal and dynamic loads. However, the information on research works about hybrid nanocomposite which involves two reinforcement having nano and micron sized are limited. Nano-sized SiC and carbon nanotubes reinforced copper composites were produced via mechanical milling followed by hot pressing technique. It is found to be observed that decrease in grain growth rate with increase in content of carbon nanotube leading to drastic improvement in mechanical property. An improvement of hardness and compression strength of composite was attributed to three important mechanisms such as thermal mismatch, hall petch and orowan looping. The measured grain size of hybrid nanocomposite (Cu/2SiC-6CNT) was 828.8 nm while that of pure copper was 1350 nm. It is observed that the microhardness and modulus of pure copper 63 HV and 105.69 GPa when compared with hybrid composite 108.5 HV and 140.23 GPa, respectively, indicating strengthening by both carbon nanotubes and nano SiC¹⁹.

In the present work, an attempt has been made to develop copper based hybrid nanocomposite with SiC and MWCNTs as reinforcements using powder metallurgy technique. MWCNTs have been chosen owing to their outstanding mechanical and physical properties and SiC was used as secondary reinforcement due to its high temperature thermal stability, low density, and better wear resistance behavior.

2. Experimental Procedure

2.1 Materials Selection

Electrolytic pure copper powder (Purity: 99.5 %, grain size: 2-20 μm , density: 8.94 g/cm³, supplier: Loba chemie

Pvt. Ltd., India) was used as matrix material to fabricate composite. Multiwalled carbon nanotube (purity: 99%, nanotube size: 20-40 nm, length: 1-10 μm , density: 2.1 g/cm³) and silicon carbide (purity: 99.5 %, size 20-30 μm , density: 3.21 g/cm³) were used as reinforcements to synthesis the copper and its hybrid nanocomposites.

2.2 Copper Coating on MWCNTs and SiC by Electroless Technique

MWCNTs and SiC are non-wetting reinforcements with most of the metals and alloys. The mechanical and thermal properties of composites are influenced by interracial bonding strength between matrix and reinforcement. Thus, it is essential to enhance bonding strength by coating either same matrix metal or nickel on reinforcements that improves wetting property. Electroless coating method was employed to coat copper on reinforcements. Pretreated reinforcements (MWCNTs and SiC) are introduced into electroless copper bath for Cu coating on reinforcements^{18,20}.

2.3 Mechanical Alloying of Nanocomposite Powders

The nanocomposite powders were prepared by using planetary ball mill. The weight fractions of copper coated MWCNTs was varied from 1 to 4 wt.% while that of SiC fixed to 2 wt.%. All the raw materials and stainless steel balls with diameters of 10-30 mm were filled into vial of planetary ball mill. The ball to powder ratio 8:1 was maintained for all composition. In order to avoid oxidation of starting powders, few drops of ethanol was added as process controlling agent. The mixed powders were blended for about 3 hours at 300 rpm for all composition.

2.4 Consolidation of Copper and Nanocomposites

The prepared nanocomposite powder was heated upto 80^o C to remove any moisture content. Figure1 shows the photographs of die and punch used to consolidate nanocomposite samples. The inside diameter of die is 30 mm and height is 60 mm. A load of 400 MPa was applied using hydraulic press to compact nanocomposite powder. Before compaction, the wall surface of punch and die were coated with solid lubricant to facilitate easy removal of compacted specimens. All compacted specimens were sintered at 900^o C in an electric furnace under argon

protected atmosphere for about 1 hour. Further, composite samples were resintered for about 4 h to improve the bonding strength between the particles and then repressed at same temperature in hydraulic press to enhance relative density. Again the samples were resintered at same temperature for 4 h and repressed at same condition to achieve full density and reduce the porosity.

2.5 Characterization of Copper and Nanocomposites

The prepared copper and nanocomposites were characterized by using different techniques. The theoretical densities were calculated by using rule of mixture, where as actual density was determined using Archimedes principle. The microhardness was determined by Shimadzu Micro hardness tester. Before measuring the hardness, the surfaces of the samples were polished with emery paper from rough (100) to smooth (1200) using double disc polisher. The hardness of samples was determined by applying load of 500 g on the polished samples for a dwell time of 10 sec. The mean hardness was calculated by average of eight indentations at different locations on the polished specimen. Electrical conductivity of the copper and nanocomposites were measured by using Autosigma-3000 electrical conductivity meter. The electrical conductivity values are expressed in % of IACS (International Annealed Copper Standard). The morphology of initial powder used for preparing composite powder were characterized by using SEM equipped with EDS.

Figure 1(a) shows metallic punch and die was used to fabricate copper and composites samples and prepared hybrid nanocomposites samples with varying the content of carbon nanotubes (1-4 wt.%) keeping SiC being fixed at 2 wt.% for all samples is shown in Figure 1(b).

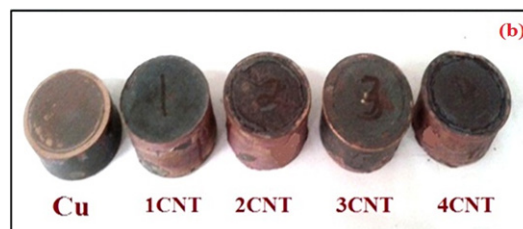
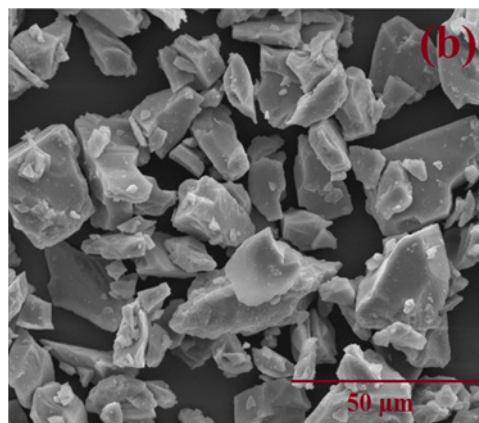
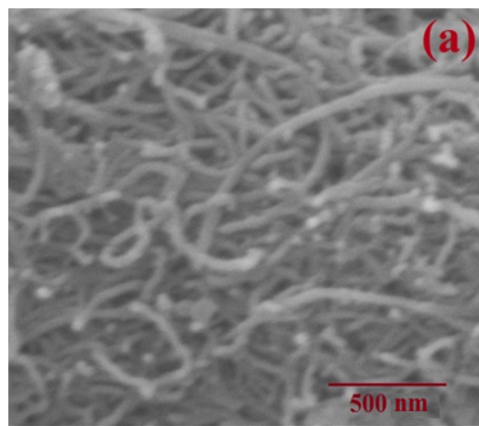


Figure 1. Photograph of (a) Metallic Punch and die (b) copper and hybrid nanocomposite samples prepared by hot pressing.

3. Results and Discussion

3.1 Morphology of Raw Materials and Nanocomposite Powders

Figure 2(a) shows SEM micrograph of Multiwalled Carbon Nanotubes (MWCNTs). It is observed that MWCNTs are in the form of bundles and their diameter in range of 20-40 nm with length of 1-10 μm . SEM image of silicon carbide (SiC) is shown in Figure 2(b). SEM image reveals that silicon carbide has flakey morphology with sharp edges and their size is in the range of 20-30 μm . Figure 2(c) shows SEM of copper powder has dendritic morphology with sub-rounded. SEM of copper shows the size of particles varied between 1-5 μm . In order to assess the purity of copper powder, EDS analysis of copper was carried out that clearly reveals three peaks of copper present and no other element peaks present (as shown in Figure 2(d)).



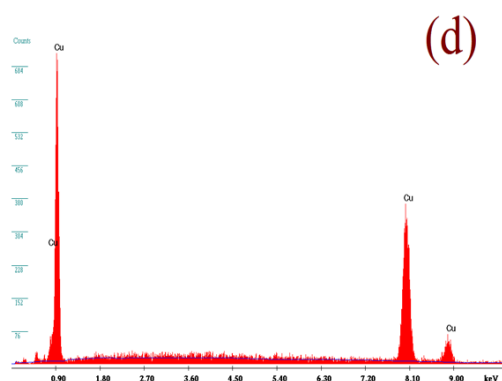
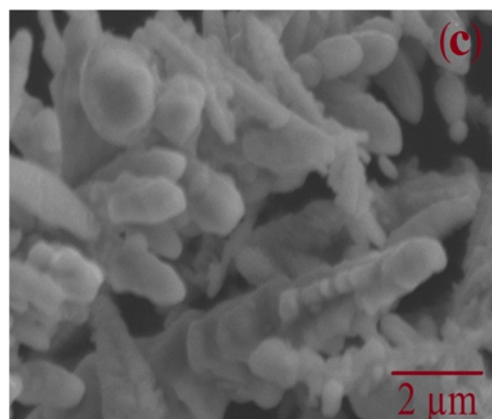
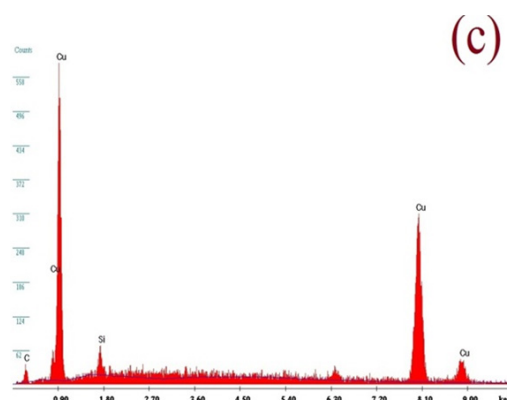
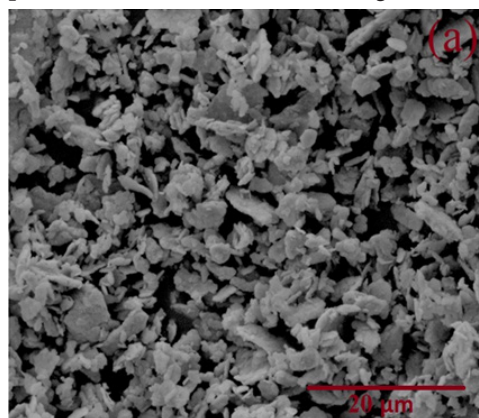


Figure 2. SEM micrograph of (a) Multiwalled carbon nanotubes (MWCNTs) (b) Silicon carbide (SiC) (c) Copper and EDS of (d) copper (Cu).

Figure 3(a) and (b) shows SEM micrograph of Cu/2SiC-1CNTs and Cu/2SiC-4CNTs hybrid nanocomposite powder. It is observed that composite powder contains copper, carbon nanotubes and silicon carbide. The mixed composite was milled in planetary ball mill for about 3 h at 300 rpm. Initially stage of ball milling, the particles of nanocomposite powder are appeared to be flattened in shape. During ball milling, MWCNTs and SiC are embedded inside the copper particles due to cold welding between individual flattened copper particles. SEM of nanocomposite powders shows the size is in the range of 5-30 μm with fine to coarse. It is observed that silicon carbide particles are distributed homogeneously in copper matrix. Figure 3(c) and (d) shows EDS spectrum of nanocomposite

powder with varying content of carbon nanotube. EDS of nanocomposite powder confirms the presence of copper, silicon and carbon element in the nanocomposite powder. Absence of foreign elements such as iron in the nanocomposite powder shows the effectiveness ball milling parameters without contaminating it.



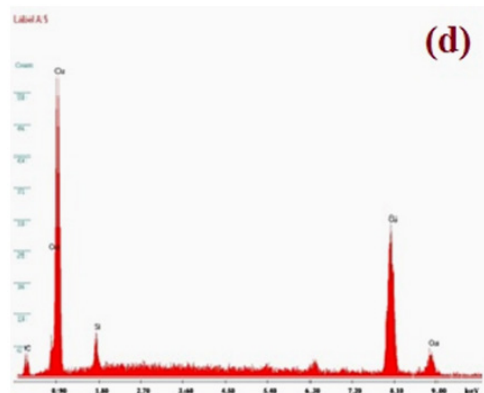


Figure 3. SEM micrograph of (a) Cu/2SiC-1CNTs (b) Cu/2SiC-4CNTs composite powder and EDS spectra of (c) Cu/2SiC-1CNTs (d) Cu/2SiC-4CNTs nanocomposite powder.

3.2 Density Studies on Copper and Hybrid Nanocomposites

Table 1 reports theoretical and actual density of developed copper and hybrid nanocomposites. Hot pressing was adopted to ensure that prepared samples have low density with minimum porosity. Theoretical density of nanocomposite decreases with increase in content of carbon nanotubes with SiC being fixed at 2 wt.% due to low density of carbon nanotubes (2.1 g/cm^3) and silicon carbide (3.21 g/cm^3) when compared with density of pure copper (8.94 g/cm^3). Samples of copper and nanocomposites were subjected to hot pressing at 900°C temperature after sintering in order to achieve fully dense copper and nanocomposites and to reduce the porosity level.

$$\text{Density of composite } (\rho_c) = (\rho v)_{\text{Cu}} + (\rho v)_{\text{CNTs}} + (\rho v)_{\text{SiC}}$$

Where, ρ and v are the density and volume fractions of copper, Carbon nanotube and silicon carbide, respectively.

Actual density of copper and nanocomposites were determined by Archimedes principle. Calculated values indicate that actual density of nanocomposites decreases with increase in weight percent of carbon nanotube. Table 1 shows the level of porosity of composite is minimum and slightly increases with increase in content of carbon nanotubes with SiC being fixed at 2 wt.%. Hot pressing was carried out after sintering to reduce the porosity of copper and its nanocomposites. However, porosity slightly increases with increase in content of carbon

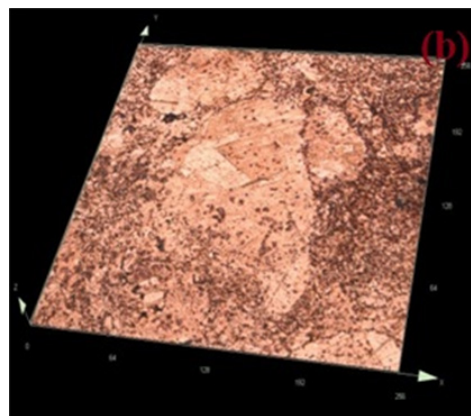
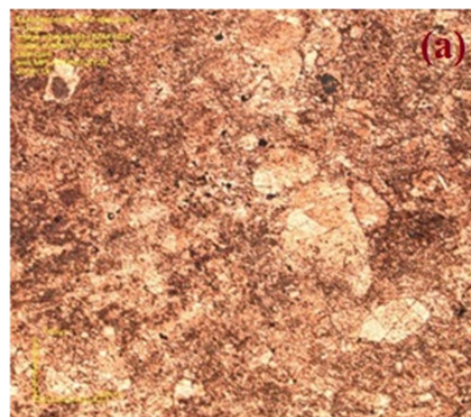
nanotubes may be due to agglomeration and micropores.

Table 1. Density of copper and nanocomposites

Sl. No.	Particulars	Theoretical density (g/cm^3)	Actual density (g/cm^3)
1	Copper	8.94	8.89
2	Cu-2 SiC-1CNTs	8.36	8.26
3	Cu-2 SiC-2CNTs	8.12	7.98
4	Cu-2 SiC-3CNTs	7.88	7.71
5	Cu-2 SiC-4CNTs	7.66	7.46

3.3 Microstructure of Copper and Nanocomposites

Figure 4(a-d) show confocal image of copper and hybrid nanocomposites. It is observed that porosity of copper is appeared to be minimum due hot pressing and grain size varied from $16\text{--}18 \mu\text{m}$. Hybrid nanocomposites reveals that SiC particles are clearly visible and distributed homogeneously in the copper matrix. The size of SiC varies between $5\text{--}25 \mu\text{m}$. The gap between reinforcements and matrix is minimal suggesting good bonding. The morphology of SiC has not changed after ball milling for 3 h. MWCNTs are not seen in the image due to nanosize and low magnification and are expected to be implanted into copper grains.



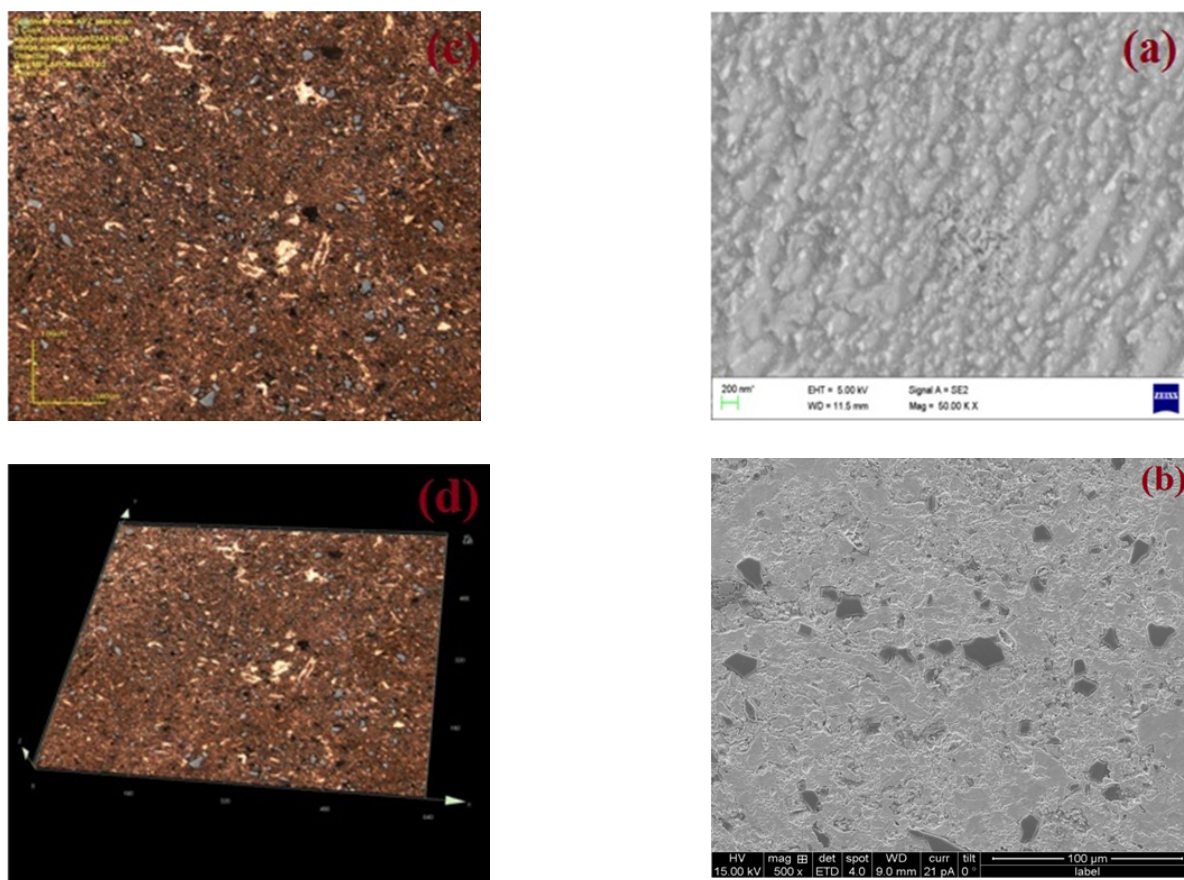
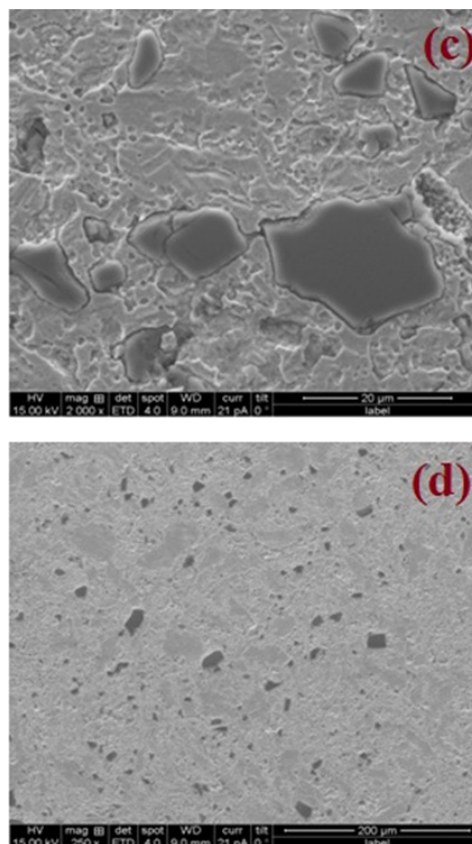


Figure 4. Confocal micrograph of (a) Copper (b) Isometric view of copper (c) Cu/2SiC-3CNTs and (d) Isometric view of Cu/2SiC-3CNTs hybrid nanocomposite.

Figure 5(a-e) show SEM micrographs of copper and its hybrid nanocomposites. It is observed that SEM of copper possesses fine grains and minimum porosity as shown in Figure 5(a). SEM of hybrid nanocomposite with carbon nanotube varied from 1-4 wt.% in step of 1wt.% and SiC being fixed at 2 wt.% is shown in Figure 5(b-e). Both silicon carbide and carbon nanotube distributed uniformly in copper matrix is being observed. The SEM images reveals that the porosity of nanocomposite increases slightly with increase in weight percent of carbon nanotubes. Hybrid nanocomposites showed good bonding between matrix and reinforcements. Hot pressing was effective method to reduce the porosity of nanocomposite. The copper coating on SiC and MWCNTs has proven useful in achieving good bonding between reinforcement and matrix.

Fig. 5(f) shows the EDS spectra of Cu/2SiC-4CNTs nanocomposite. EDS analysis reveals that both matrix and reinforcement elements are present in the prepared nanocomposite and high purity level.



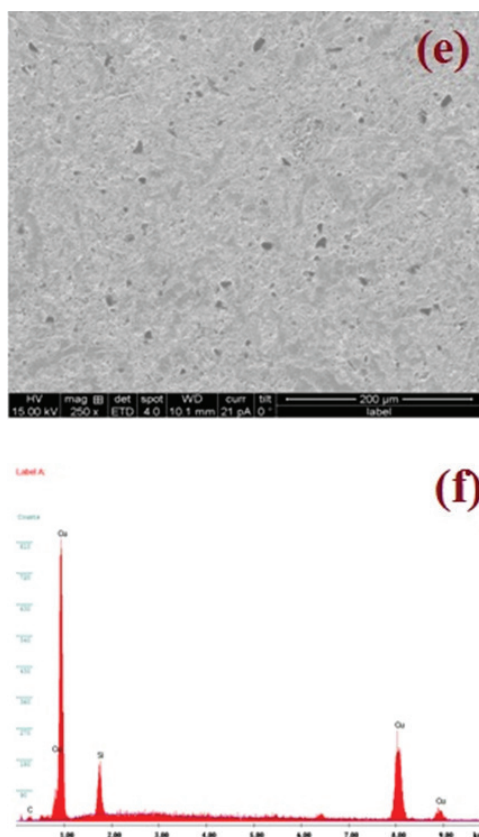


Figure 5. SEM micrographs of (a) Pure copper (b) Cu/2SiC-1CNTs (c) Cu/2SiC-2CNTs and (d) Cu/2SiC-3CNTs and (e) Cu/2SiC-4CNTs hybrid nanocomposite and EDS spectra of (f) Cu/2SiC-4CNTs nanocomposite.

3.4 Microhardness of Copper and Nanocomposites

Figure 6 illustrates variation of microhardness of nanocomposite with increase in content of carbon nanotubes. It is observed that microhardness of developed hybrid nanocomposite with 3 wt.% carbon nanotube is 143 HV in comparison with pure copper (96 HV). An improvement in hardness of nanocomposites can be attributed to high hardness of silicon carbide and high strength of carbon nanotube. Silicon carbide is being a hard ceramic particle that mainly contributes in increasing the hardness of composite. Thermal mismatch between matrix and reinforcements is another factor which effectively contributes to increase in hardness. The coefficient of thermal expansion of copper, SiC and MWCNTs are $17 \times 10^{-6} \text{K}^{-1}$, $2.7 \times 10^{-6} \text{K}^{-1}$ and $2 \times 10^{-5} \text{K}^{-1}$, respectively. MWCNTs and SiC possess low coefficient of thermal expansion and less than pure

copper. The dislocations are generated around the both the reinforcements due to this thermal mismatch. So increase in dislocation density will lead to improvement in hardness^{20,21,25}. If carbon content is increased beyond 3wt.%, microhardness start reducing from 143 HV to 131 HV. This can be due to agglomeration of MWCNTs inside copper matrix. These agglomerates will act as pores rather than reinforcement.

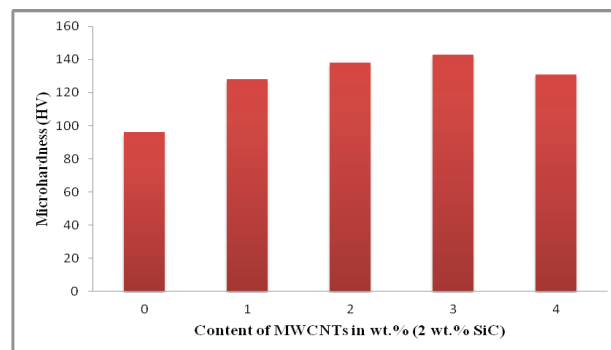


Figure 6. Variation of microhardness with CNTs content and SiC (2 wt.% SiC).

3.5 Electrical Conductivity

Table 2 reports electrical conductivity of copper and hybrid nanocomposites. It is observed that electrical conductivity of pure copper is 95.2 %IACS and nanocomposite with 4 wt.% CNT is 19.8 %IACS. The drop in electrical conductivity is mainly due to grain refinement caused by multiple reinforcements (MWCNTs & SiC) which means increase in grain boundaries volume. The electron scattering taking place at grain boundaries and copper-multiple reinforcement significantly decrease the electrical conductivity of hybrid nanocomposites²²⁻²⁴.

Table 2. Electrical conductivity of copper and nanocomposites

Compositions	Electrical Conductivity, %IACS
Copper (Cu)	95.2
Cu/2SiC-1CNTs	28.3
Cu/2SiC-2CNTs	25.8
Cu/2SiC-3CNTs	23.4
Cu/2SiC-4CNTs	19.8

4. Conclusions

- Carbon Nanotubes (CNTs) and silicon carbide (SiC) reinforced copper hybrid nanocomposite was successfully developed by powder metallurgy route followed by hot pressing.

2. SEM of composite powder and hybrid nanocomposites reveal that both silicon carbide and carbon nanotube homogeneously dispersed in copper matrix.
3. The grain size of composite decrease with increase in the content of carbon nanotubes.
4. The microhardness of hybrid composite significantly improved when compared with pure copper.
5. Electrical conductivity of pure copper is 95.2 %IACS in comparison with nanocomposite with Cu/2SiC-4CNTs(19.8 %IACS). This can be attributed to the grain refinement caused by multiple reinforcements and micropores which increase with increase in content of carbon nanotube.

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