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Mechanical properties of Copper Nanocomposites Reinforced with Uncoated and Nickel Coated Carbon Nanotubes

Carbon nanotubes have emerged as potential reinforcement material for metallic matrices since their discovery in 1991 by Japanese scientist Sumo Iijima. It is observed that carbon nanotubes were comprised of multifunctional properties and were ideal reinforcement material for metallic matrices. In the present work we report the development of carbon nanotubes with multi-walls reinforced commercial purity copper nanocomposites. The carbon nanotubes content was varied from 0.25 to 1.0 wt% in the copper matrix nanocomposites. Here, carbon nanotubes were also nickel coated for improving the interfacial bonding with the copper matrix. In order to obtain the good dispersion of carbon nanotubes in the copper matrix, both materials were subjected to ultrasonication and blending process using ball milling. Further sintered nanocomposites were subjected to upsetting forging process, which involves densification and shape change simultaneously. Microstructure studies were conducted using scanning and transmission electron microscopes to study the dispersion of carbon nanotubes. The effect of carbon nanotubes on the mechanical properties like microhardness and tensile strength of copper matrix nanocomposites is studied in detail.

Keywords: *Carbon Nanotubes, Copper, Powder Metallurgy, Mechanical Properties, Nanocomposites.*

1. INTRODUCTION

Recently the field of nanocomposites is having the attention from both academicians and industrial engineers. This interest results from the fact that the building blocks are having dimensions in the nanosize range. Nanostructured materials are characterized by a grain size or particulate size of up to about 100 nm. The high interface area of nanostructures plays an important role in enhancing or limiting the overall properties of nanocomposites. Using these nanostructured materials it is possible to design new materials with more flexibility and improvements in their mechanical, thermal, electrical, dielectric and tribological properties [1-3].

The discovery of carbon nanotubes (CNTs) has sparked a new era in the field of materials science and nanotechnology. The carbon-carbon covalent bond in the graphene sheet is the strongest bond known in nature. Since their discovery a lot of studies were conducted in evaluating mechanical properties. Out of available techniques, arc discharge is most commonly used technique for producing carbon nanotubes of high purity [4,5]. A lot of experimental and theoretical studies have been devoted to characterize the mechanical properties of carbon nanotubes [6]. The axial elastic modulus of carbon nanotubes was comparable to the in-plane elastic modulus of graphite which is about 1.04 TPa. Brief overview of (CNTs), its synthesis, mechanical properties and their use in making nanocomposites can be found in Ref [7]. To start with, Young's modulus of carbon nanotubes predicted by Overney et al. [8] in 1993 using empirical Keating Hamiltonian with parameters determined from first principles, gave him values in the range of 1.5 to 5 TPa. Lu et al. [9] conducted a thorough theoretical study of mechanical properties of both single and multiwalled CNTs using molecular dynamics. From his study Young's modulus of isolated single walled CNT was found to be 1 TPa whereas for multiwalled CNT it was 1.11 TPa. The first successful experimental measu–rement of elastic modulus was done by Treacy et al. [10]. The physical basis underlying these experiments is analysis the thermal oscillation of cantilevered multi walled CNT in transmission electron microscope (TEM) at different temperatures. Young's modulus used for individual nanotubes measured ranged from 0.4 to 4.15 TPa with a mean of 1.8 TPa. The high strength and stiffness was mainly due to the presence of carbon-carbon bond.

It is theoretically predicted and experimentally proved that CNTs have outstanding mechanical, thermal and electrical properties [4, 7]. By incorporating CNTs into appropriate matrices, it is postulated that the resulting nanocomposites will be light weight, have increased strength, stiffness and thermal conductivity. The CNT reinforced metal matrix nanocomposites have received tremendous attention in the last few years mainly for the development of light weight and high

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strength composites. These metal matrix nanocomposites are generally fabricated by liquid metallurgy, powder metallurgy, Electroless plating and Spray techniques like Cold spray, High Velocity Oxy-fuel and plasma spray. Out of all techniques, powder metallurgy is most widely used processing technique to fabricate the nanocomposites, in which Al, Cu, Mg, Ni, Ti and Ag are used as matrix materials [11]. Due to friction and wear the transport industry faces many problems like premature failure of components and high maintenance costs. In order to address these issues, high quality of lubrication is required for materials to work under harsh conditions involving high pressure and temperatures. In such cases CNTs have emerged as potential lubricants due to their lubricating, chemical and anti-wear properties [12-14].

Aluminium alloys and Copper are the most popular non-ferrous matrix materials for CNT reinforced nanocomposites. Most of the studies adopted powder metallurgy technique for processing Cu/CNT nanocomposites [15-17]. George et al. [18] fabricated Al/CNT composites by hot extrusion technique. Short milling time was adopted to ensure that no damage was done to CNTs during milling. The results showed increase in the values of yield strength and Young's modulus for Al/CNT composites. For 0.5 wt.% of MWCNT/Al composites the yield strength and modulus were 86 MPa and 74 GPa, whereas for pure aluminium process under the same conditions had 80 MPa and 70 GPa. The strengthening mechanisms involved in Al/ CNT composites were also discussed briefly. Aniruddha Ram et al. [19] processed AA6061/MWCNT nanocom–posites by hot pressing the as sintered compacts and carried out nanoindentation studies. Nanoindentation studies showed increase in the value of Young's modulus by 21% and 22% and nanohardness by 43% and 54% by addition of 1 wt% and 2 wt% to the AA 6061 alloy. Kashyap et al [20] studied the aging behavior of multiwalled carbon nanotubes (MWCNT) reinforced AA6063 nanocomposite synthesized by powder metallurgy technique. With the increase in the MWCNT content, the aging curves showed the faster kinetics owing to generation of dislocations due to coefficient of thermal mismatch. It was interesting to note that the copper coated MWCNTs reinforced AA6063 nancomposites showed greater hardness than that of uncoated MWCNTs. In their work, Tu et al. [21] fabricated CNT/Cu composites by powder metallurgy technique, in which the CNT volume fraction was varied from 0 – 16%. The composites exhibited a lower coefficient of friction and reduced wear rate with increasing volume fraction of CNTs at lower and intermediate loads. Composites with high volume fraction CNTs exhibited reduced wear resistance under high low conditions due to its high porosity. Kim et al. [22] synthesized Cu/CNT composites by spark plasma sintering and followed by cold rolling process. In this process Cu powders were fabricated by spray drying and reduction process. The CNTs and Cu powder were mixed in high energy ball mill and were pre-compacted in spark plasma sintering (SPS) system. The sintered Cu/CNT composites were subjected to cold rolling up to 50% reduction. From the hardness measurements, the yield strength for 5 and 10 vol.% Cu/CNT composites were estimated about 370 and 589 MPa, respectively, while that of pure copper region was about 190 MPa.

The present work is focussed on the development of nanocomposites by powder metallurgy technique followed by upsetting forging process. After forging the dispersion fo carbon nanotubes in copper is charac–terized using transmission electron microscope (TEM). The effect of carbon nanotubes on mechanical and triblogical process is studied.

2. EXPERIMENTATION

2.1 Materials and Methods

CNTs are available in different morphologies namely single walled, double walled and multiwalled. In the present case we have opted for multiwalled carbon nanotubes for synthesizing the nanocomposites. The specifications of procured carbon nanotubes were: length - 1 – 10 μm; outer diameter - 30 – 50 nm; inner diameter - 5 – 10 nm and purity Level - > 95%. Keeping the huge applications of copper in mind we have opted copper as matrix material for synthesis of nanocomposites. The commercial purity copper powder in electrolytic form was purchased from Loba Chemie Pvt. Ltd, India and its properties are density - 8.9 g/cm^3; size - 2 – 10 μm and purity Level - > 99.5%.

Initially, 500 ml of ethanol is taken and to it CNTs of known weight are dispersed first. Sonication is carried out for 30 minutes until all the CNTs are dispersed uniformly throughout the ethanol solution. After this the copper powder is added to the solution containing CNTs and sonication is carried out for another 30 minutes. Once the copper and CNTs are dispersed uniformly throughout the solution, the sonication is stopped and the slurry of nanocomposite powders is dried in an oven maintained at temperature of 50°C. Along with this in order improve the wettability of CNTs, a layer of nickel was deposited by following the work published in Ref [16]. CNTs were subjected to acid treatment in order to improve the surface chemistry before the electroless coating. Here dried mixture of copper powder and CNTs with varying weight percentage (0.25, 0.50, 0.75 and 1.00 wt%) obtained after ultrasonication are further subjected to blending process using ball milling process. For this purpose stainless steel vial and balls are used as milling media. The dried mixture of copper and CNTs is placed in the vial along stainless steel balls with a ball to powder ratio (B/P) of 10:1. The short time duration 60 minutes chosen for ball milling of nanocomposite powder is to avoid possible structural damage to CNTs. The blending process is carried out at 200 rpm for duration of 60 minutes in which an intermittent break of 5 minutes was given for every 15 minutes of milling. This was done to overcome from increase in temperature of powders undergoing ball milling process to avoid excessive welding of powder particles.

The blended mixtures of copper and CNTs were next subjected to compaction and sintering process. Hydra–ulic press used for compaction was having a capacity of applying 35 T load. The powders were loaded in the die (diameter: 30 mm; length: 60 mm) and using a pressure of 400 MPa for 5 minutes the compaction was carried out. In order to possible friction between the punch and die during compaction process stearic acid was used as lubricating agent. For pure copper and all nanocomposite mixture with varying CNT weight percentage the compaction was carried at the same compaction pressure of 400 MPa and holding time of 5 minutes. Once the compaction was done, all the green compacts of pure copper and nanocomposites were subjected to sintering process. The sintering was done in electric furnace consisting of alumina tube in controlled atmosphere. The sintering temperature chosen for all the green compacts was 850°C and time duration was 2 hours. The compacts of pure copper and nanocom–posites with varying CNT weigh fraction are further subjected to forging process. In this the compacts were heated to 850° once again for 120 minutes in argon protected atmosphere. As soon as the 120 minutes are completed the hot compacts are taken outside the furnace and subjected to forging process. Here in this kind of forging process lateral flow of material is observed due to the application of pressure on the top of plunger. Unlike in the literature, the forging is carried open die configuration where the lateral flow of material is not restricted. The application of pressure was gradual until a required thickness of 8 mm is obtained.

2.2 Testing & characterization

Copper and nanocomposites with varying CNT weight fraction are subjected to various characterization tech–niques to study their attributes like density, dispersion of CNTs by microscopy techniques and testing of their mechanical properties like microhardness and tensile strength.

Two types of density measurements namely theoretical and experimental density of forged copper and nanocomposites were carried out. The theoretical density of all the nanocomposites was carried using the rule of mixtures by utilizing their density and volume fraction. The experimental density of nanocomposites was calculated using Archimedes principle (ASTM B311 standard). In order to check the dispersion of CNTs in copper matrix, scanning (SEM) and transmission electron microscopy (TEM) (Philips CM 12, 100 kV, IIT Madras, India) was used. The nano–composite samples dimensions used for TEM observations were of 3 mm disc and 10 μm thick. The 3 mm disc was punched out from nanocomposite sample less than 40 μm thick, prepared by mechanical polishing using a 1200 emery paper, followed by dimpling using a dimple grinder (Model 656, Gatan). Finally the dimpled disc was ion milled using Ion beam miller (Model 691, Gatan) at room temperature.

The microhardness test was conducted on metallo–graphically polished surfaces of copper and its nanocomposites. For this purpose, digital Vickers microhardness tester (Make: Everone) was utilized. A load of 100 grams was applied on each polished sample for a dwell time of 10 seconds. For each sample total of 5 readings were taken at different locations and average of them is reported here. The tensile testing of pure copper and nanocomposites were carried out using Servo-hydraulic universal testing machine (Make: Instron 8032, Indian Institute of Science, India) at an initial strain rate of 1×10^{-4} s^{-1}. The tensile sample dimensions are cut into dog-bone shaped of sub-size of width 2.5 mm and gage length 9 mm based on ASTM E8M by using wire electrical discharge machining.

3. RESULTS & DISCUSSION

3.1 Microstructural studies

Figure 1 shows the SEM image of as received CNTs from the supplier which is in the form of clusters. As observed in the SEM images the CNTs were in the form of clusters. The CNTs tend to form clusters by interlocking between themselves to Vander Waals force acting between them. The diameter of the CNTs was in the range of 30 to 50 nm while there length was in several microns. Figure 2 shows the SEM image of CNTs taken after they were subjected to ultrasonication. In order to break the clusters the CNTs were subjected to sonication in ethanol solution for about 30 minutes using ultrasonicator. After sonication it was observed that the large clusters of CNTs were broken and individual CNTs can be seen clearly in the SEM images. It is necessary to avoid the formation of aggregates of CNTs so that during fabrication of composites they do get individually dispersed in copper matrix.

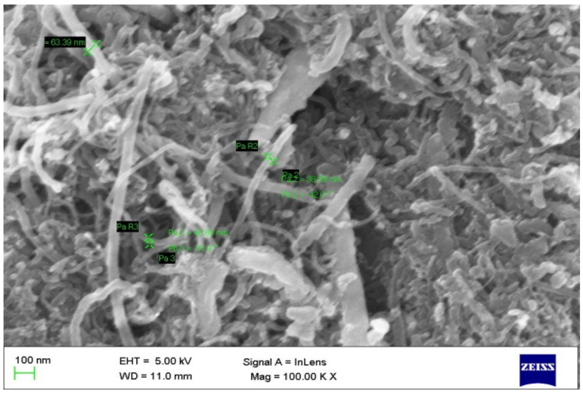

Figure 1. SEM image of as procured CNTs

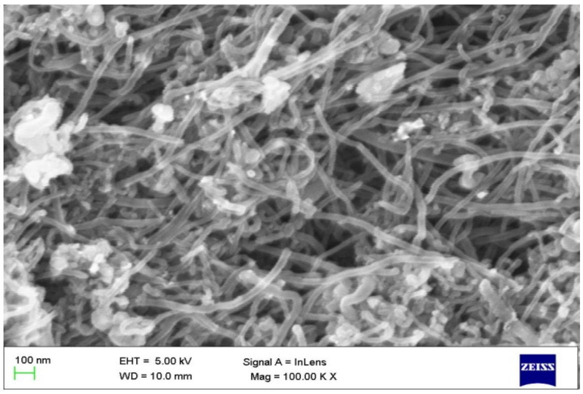

Figure 2. SEM image of CNTs after ultrasonication

Figure 3 shows the SEM image of electroless nickel coated CNTs. The nickel was found to be coated on the outer walls of the CNTs and is quite evident from SEM images. Here nickel coating of CNTs was done in order to improve the interfacial bonding between CNTs and copper matrix. Scanning electron microscope studies were conducted to check the dispersion of CNTs and as well as the effect of hot forging on densification of nanocomposites. This has to be done since all the properties of the nanocomposite depend on the dispersion and bonding of CNTs with the copper matrix. Figure 4 shows the high resolution SEM images of nanocomposite with 0.25 wt% CNT after forging process. It can be seen clearly the CNTs are dispersed uniformly in the copper matrix without forming any clusters. The dispersion of CNTs is so uniform that each CNT were found in the copper matrix. Further the bonding of CNTs was found to be very good and had clean interface with the copper grains. So it is quite evident from the SEM images that the forging process carried out at high temperature is capable of reducing the extent of porosity, improving the densification by improving the bonding between the CNTs and copper grains and breaking the CNT clusters.

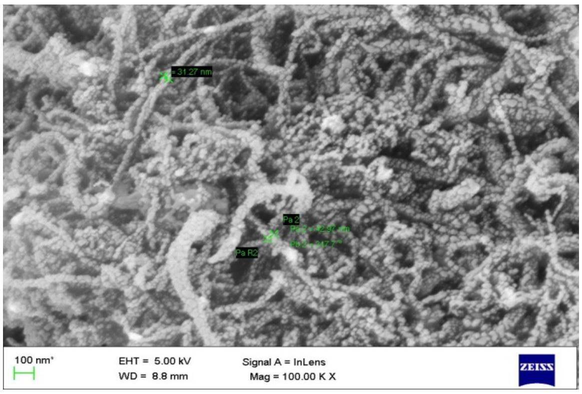

Figure 3. SEM image of CNTs after nickel coating

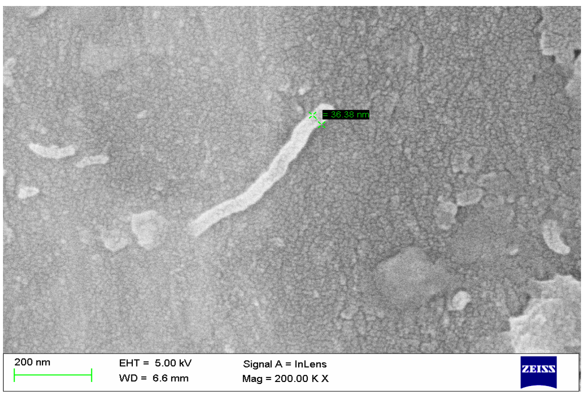

Figure 4. SEM image of Cu-0.25%CNTs after upsetting forging process.

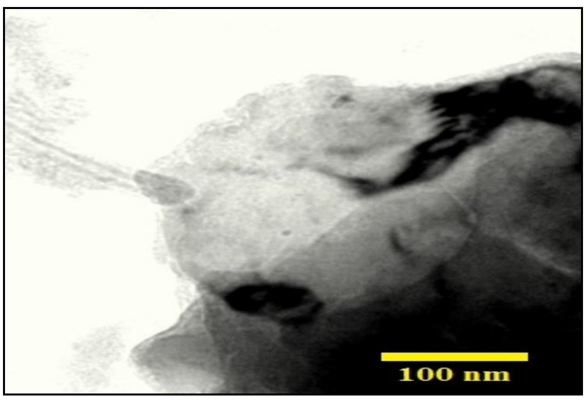

Figure 5. SEM image of Cu-0.25%CNTs after upsetting forging process.

Figure 5 shows the TEM micrograph Cu-0.25%CNT nanocomposite. CNTs are seen as thin white lines and individually dispersed in the copper matrix after hot forging without forming clusters. The dispersion of CNTs is important factor for obtaining good mechanical and physical properties of the nanocomposite. It is necessary that secondary processing technique like hot forging to be used to get dense compacts without any clusters of CNTs inside the copper matrix. The interface between CNTs and copper grains is clean and so gap is seen between them.

3.2 Density studies

The theoretical density calculated using rule of mixtures and experimental density measured using Archimedes principle for copper and its nanocomposites are shown in Table 1. In order to calculate the density of nanocomposites, the density of copper is ~8.9 g/cm^3 and CNTs is 2.1 g/cm^3 are used. The theoretical density of nanocomposites tends to decrease with the increase in CNT content. Further, the density measured using Archimedes principle for both pure copper and nanocomposites were found to be lesser than that of theoretical values.

Table 1 Density of copper and nanocomposites with uncoated CNTs

Composition	Theoretical density, g/cm^3	Measured density, g/cm^3	Relative density, (%)
Pure Cu	8.90	8.81	98.99
Cu-0.25 wt% CNT	8.83	8.71	98.64
Cu-0.50 wt% CNT	8.76	8.60	98.17
Cu-0.75 wt% CNT	8.69	8.52	98.04
Cu-1.00 wt% CNT	8.62	8.40	97.45

The experimental density of nanocomposites found to decrease in increase CNT content when compared to that of pure copper after forging process. This is mainly because the presence of low density CNTs in nano–composites tend to contribute to low density values. Overall, the relative density for pure copper was found to be highest when compared to that of nanocomposites. The highest level of density was obtained for pure copper and least for nanocomposite with 1 wt% of CNTs. Such high densification is a result of hot forging which has capability of closing the voids or minimizing the gap present in nanocomposites after compaction and sintering process. Due to the appli–cation of large amount plastic deformation via hot forging, the lateral flow of material leads to closure of pores and shearing of any oxide film if present.

3.3 Microhardness

It is well known that hardness is the resistance offered by any material to plastic deformation by an indenter. The hardness of any material is directly dependent on the microstructure, especially in case of nanocomposites were the type of reinforcing phase, dispersion, size and shape play very important role.

The effect of uncoated and nickel coated CNTs on the microhardness of copper matrix nanocomposites is shown in Fig. 6. It can be observed that the inclusion of CNTs in the copper matrix has resulted in improvement in the hardness. The increasing weight fraction of CNTs has resulted in linear increase in the microhardness values of copper nanocomposites until CNT weight percentage of 0.75%. Further increase from 0.75% to 1 wt%, the microhardness was found to decrease. The nanocomposite with 0.75 wt% nickel coated CNT had the highest microhardness value of 141 HV when compared to that of pure copper which has a microhardness value of 92 VHN. The nanocomposite with 0.75 wt% CNT had the highest microhardness value of 128 HV. The increase in the hardness value of 0.75 wt% CNT reinforced nanocomposites is about 36.73% over the pure copper. The main reason for increment in the microhardness was the presence of CNTs underneath the indenter during hardness test provides resistance to plastic deformation. In general the presence of hard reinforcing phase like CNTs present in the soft and ductile matrix like copper can significantly improve the hardness of overall nanocomposite [15,16].

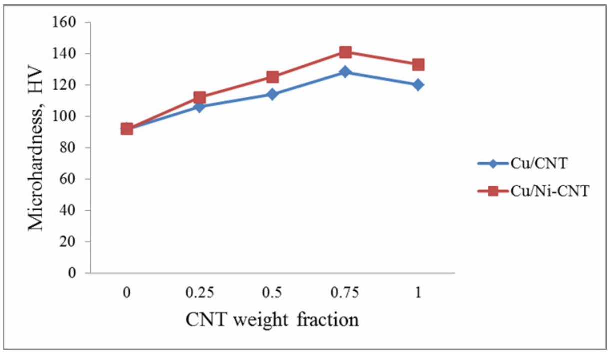

Figure 6. Microhardness of CNT based copper nanocompo–sites

The higher microhardness of CNT reinforced nanocomposites when compared to that of pure copper is attributed to following reasons,

[i] Addition of strong and stiff CNTs in the soft ductile copper matrix contributes to improvement in the microhardness of the nanocomposites.

[ii] Due to large difference in coefficient of thermal expansion between copper matrix and CNTs can lead to formation of dislocations. The dislocation density of nanocomposites increases with the increase in CNT content and acts as obstacles for plastic deformation.

[iii] Homogeneous dispersion of CNTs, good interfacial bonding between copper matrix and CNTs and grain refinement as a result of hot forging has significantly enhanced the microhardness of nanocomposites.

[iv] Presence of nickel content in the nickel coated Cu-CNT nanocomposites will enhance the hardness of nanocomposite due to high hardness of nickel when compared to that of uncoated Cu-CNT nanocom–posites

3.4 Tensile studies

Tensile property of any material is very important from structural application point of view. The effect of inclusion of new nano-sized materials and the processing techniques used can have huge influence on mechanical properties such as tensile strength.

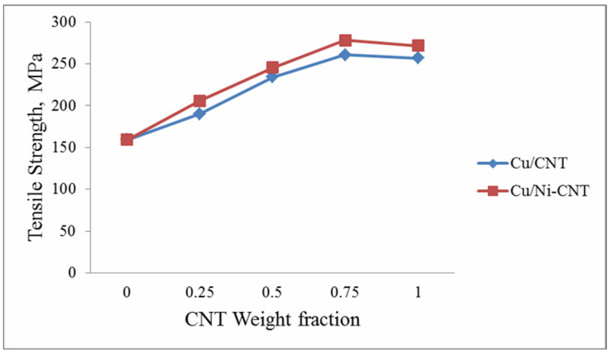

Figure 7. Tensile strength of CNT based copper nanocom–posites

The effect of uncoated and nickel coated CNTs on the tensile strength of copper matrix nanocomposites is shown in Fig. 7. It can be observed that the inclusion of CNTs in the copper matrix has resulted in increment in the tensile strength. The increasing weight fraction of CNTs has resulted in linear increase in the strength values of copper nanocomposites up to CNT weight percentage of 0.75%. The nanocomposite with 0.75 wt% nickel coated CNT had the highest tensile strength value of 277.9 MPa when compared to that of pure copper which has a tensile strength value of 159.1 MPa. For Cu-0.75%CNT nanocomposite the highest strength value of 260.7 MPa was obtained. The strengthening of copper matrix is mainly due to the uniform dispersion and good bonding of CNTs. Here uniform dispersion is attained by upsetting forging process which break downs the CNTs clusters and disperse them uniformly. The positive effect of forging on uniform dispersion of reinforcements has already been displayed in many works conducted on metal composites [23,24]. Overall the main strengthening mechanism contributing for increment in strength values are noted to dislocation strengthening and load transfer from CNTs to copper matrix. We presume that the governing mechanism for high strength in the present nanocomposites is due to dislocation-CNTs interaction which is much dependent on the dispersion of CNTs in the copper matrix. So in present case the dislocations are generated due to plastic mismatch between copper matrix and CNTs. With the increase in CNTs weight percentage in nanocomposites, the amount of dislocations generated is also increased. These dislocations interact with strong obstacles like CNTs or medium strength obstacles like dislocation forest. So with the presence of CNTs in the copper matrix, the dislocations are stopped for further movement or forced to bow between them. These CNTs not only increase the activation energy required for dislocations to overcome them but also results in higher tensile stress applied due to which we have observed higher strength values for nanocomposites. Further good bonding due to presence of nickel layer on CNTs will ensure better load transfer from copper matrix to the strong and stiff CNTs. For both uncoated and nickel coated CNTs further increment in CNT weight percentage of 1.0% has resulted in drop in tensile strength values [25,26].

3.5 Fracture surface analysis

It is very important to understand the fracture mecha–nisms of the forged copper and its nanocomposites. Failure of copper and Cu-0.75%CNT nanocomposite is shown in SEM micrographs in Fig. 8 & 9. The presence of large number of voids indicates that the pure copper as well as nanocomposites has experienced large amount of plastic deformation before final failure.

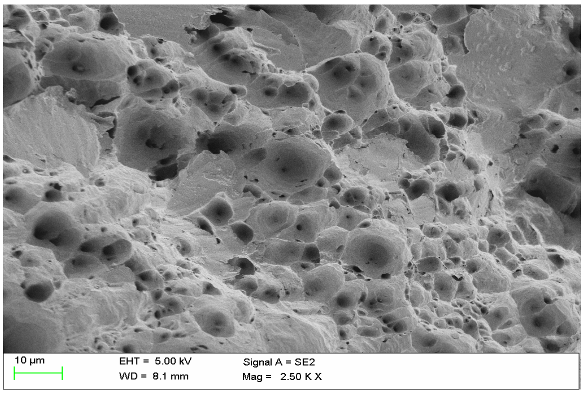

Figure 8. SEM image of fracture surface of pure copper after tensile testing

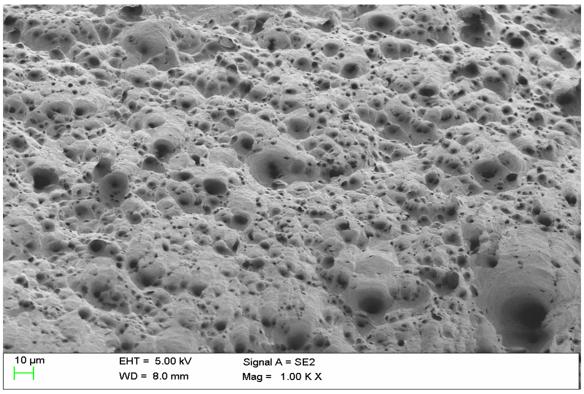

Figure 9. SEM image of fracture surface of Cu-0.75%CNT nanocomposite after tensile testing

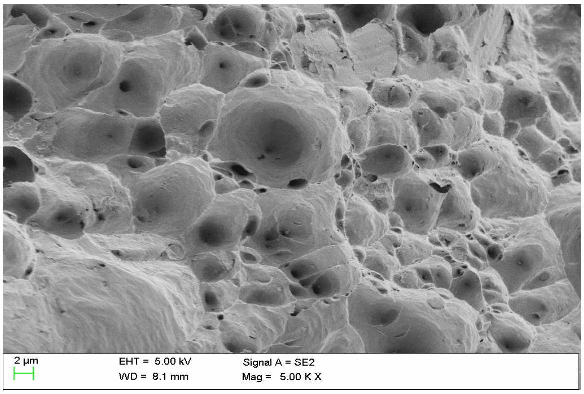

Figure 10. SEM image of fracture surface of pure copper after tensile testing in high magnification

Close examination of fracture surface of copper as shown in Fig. 10 indicates the presence of tear ridges and dimples which is typical ductile failure. In addition to this larger and deeper dimples observed in copper when compared to that of Cu-0.75%CNT nano–composite explains that it has got more ductility. Though the nanocomposite displayed brittle failure macroscopically but when we observe Fig. 11 which shows the close view of fracture surface of nano–composite with 0.75 wt% of CNT it indicated the ductile fracture microscopically. The failure observation in the nanocomposite is mainly due to cracks generated at the interface of CNTs and copper. The possibility of breaking of CNTs is very high since due localised stresses around them. Due to such high stresses ahead of pre-crack allow the voids to grow in between the damaged CNTs leading to brittle failure. These nano–composites are preferred for applications for making electrical contacts which require good hardness and tensile strength at room and moderate temperatures.

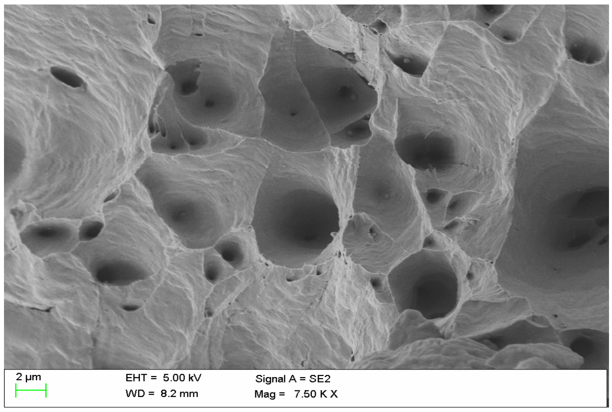

Figure 11. SEM image of fracture surface of Cu-0.75%CNT nanocomposite after tensile testing in high magnification

4. CONCLUSIONS

The following conclusions are drawn from the present work,

i. Copper based nanocomposites reinforced with CNTs in uncoated and nickel coated conditions were suc–cessfully developed by powder metallurgy technique involving ultrasonication, ball milling and upsetting forging process as secondary processing technique.
ii. Scanning and transmission electron microscopy studies showed that the developed nanocomposites showed uniform dispersion of CNTs with good bonding with the copper matrix with minimal amount of porosity after forging process.
iii. The density studies showed that the forged copper and its nanocomposites with CNTs had achieved good densification with density values close to that of theoretical values.
iv. The microhardness values of nanocomposites increased with the increase in CNT content of up to 0.75 wt%. The increase in the hardness value of 0.75 wt% uncoated CNT reinforced nanocomposites is about 36.73% over the pure copper.
v. The tensile strength values of nanocomposites increased with the increase in CNT content of up to 0.75 wt%. Compared to pure copper which had strength value of 159.1 MPa, the nanocomposite with 0.75 wt% CNT content had 260.7 MPa.

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МЕХАНИЧКА СВОЈСТВА НАНОКОМПОЗИТА НА БАЗИ БАКРА ОЈАЧАНИХ НЕОБЛОЖЕНИМ И НИКЛОМ ОБЛОЖЕНИМ УГЉЕНИЧНИМ НАНОЦЕВИМА

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Угљеничне наноцеви су се појавиле као потенцијални материјал за ојачавање металних матрица 1991. године када их је пронашао јапански научник Сумио Ијима. Угљеничне наноцеви имају мултифункционална својства и идеалан су материјал за ојачавање металних матрица. У раду се приказује развој угљеничних наноцеви са вишеслојним зидовима ојачаним нанокомпозитима на бази чистог комерцијалног бакра. Садржај угљеничних наноцеви варирао је од 0,25 до 1,0 тежинских % у нанокомпозитима на бази бакарне матрице. Наноцеви су биле обложене никлом да би се побољшало везивање са бакарном матрицом. У циљу постизања боље дисперзијенаноцеви у бакарној матрици оба материјала су била подвргнута ултрасонификацији и мешању у кугличном млину. Синтеровани нанокомпозити су затим обрађени сабијањем, што подразумева денсификацију и истовремену промену облика. У циљу проучавања дисперзије наноцеви анализирана је микроструктура помоћу електронских микроскопа за скенирање и трансмисију. Утицај угљеничних наноцеви на механичка својства као што су микротврдоћа и затезна чврстоћа нанокомпозита на базибакарне матрице проучен је у појединостима.