Fabrication and Characterization of Carbon Nano Tubes reinforced Copper Nano Composites by Powder Metallurgy Technique

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ABSTRACT

The greatest challenges facing the development of metal matrix composites for wide application are the cost of nano scale reinforcements and the cost and complexity of synthesis and processing of nano composites using current methods. As with conventional metal matrix composites with micron-scale reinforcements, the mechanical properties of metal matrix composites are strongly dependent on the properties of reinforcements, distribution, and volume fraction of the reinforcement, as well as the interfacial strength between the reinforcement and the matrix. Due to their high surface area, nano size powders and nanotubes will naturally tend to agglomerate to reduce their overall surface energy, making it difficult to obtain a uniform dispersion by most conventional processing methods. In addition, due to their high surface area and surface dominant characteristics, these materials may also be highly reactive in metal matrices. The present work can be proposed to take the samples by using Powder metallurgy technique, involves the preparation of blends of powders of metal and reinforcements, followed by consolidation and sintering of the mixtures of powders to form the part. The copper as the base metal and the carbon nanotubes as reinforcements are taken as the materials for the present work. Further the work proposes to investigate mechanical characterization and Microstructural study of the composites.

Keywords – Carbon, Copper, Nano tubes, Powder metallurgy.

I.INTRODUCTION

Glass ceramics are a kind of new material formed through the controlled nucleation and crystallization of glass, which combine the advantages of glass and ceramics [1]. Powder metallurgy has a full capacity for producing a variety of particulate composites. It avoids limitations associated with the liquid phase techniques such as formation of inter metallic compounds and inhomogeneous distribution of the reinforcing materials. However, the standard powder metallurgy method, as applied for the production of particulate composites, suffers from the high costs of alloy powders and the long mixing times for obtaining a homogeneous powder mixture [2].Wei et al. [1] studied the mica/nepheline glass-ceramic prepared by melting and powder metallurgy at low temperatures and found that bulk mica glass ceramics can be obtained through sintering powder compacts at temperatures of 850°C and 900°C. The sintered density can be as high as 90% and the microstructures are well crystallized containing mainly mica and nepheline phases.Pournaderi et al. [2] studied the wear behaviour of Al 6061-Al_2O_3 composites produced by in-situ powder metallurgy (IPM) and found that in IPM, there is an interrelation between the characteristics of reinforcement powder and that of the alloy powder. The reason is that the alloy powder itself is produced by the use of reinforcement powder and characteristics of the reinforcement powder closely influence the alloy powder. Ponraj et al. [3] studied the grapheme nanosheet as reinforcement agent in copper matrix composite by using powder metallurgy method and found that slurry based mixing provides a good interfacial bonding between the Cu matrix and the GNS (Graphene nano sheet) reinforcement. The mechanical properties of Cu/GNS composites are better than the pure Cu with a compressive strength of 290 MPa, 280 Mpa at 2 wt.%and 1 wt.% addition of GNS which is 10% more than pure Cu.Bolzoni et al. [4] studied the evaluation of the mechanical properties of powder metallurgy Ti-6Al-7Nb alloy and found that irregular hybrid-dehybride powders can successfully be shaped into pressed components which can be handled without fracture. The total shrinkage increases from 7.4% to 10.7% and the porosity levels decreases from 6.2% to 4.7% with increment of the temperature. Dehestani et al. [5] studied the mechanical properties and corrosion behaviour of powder metallurgy iron-hydroxyapatite composites for biodegradable implant applications and found that Fe-HA composites have lower values of yield strength, tensile strength and ductility compared to those of pue Fe.

II.PROBLEM DEFINITION

Less work has been done on copper and carbon nanotubes by powder metallurgy technique most of the researchers are concentrated on mechanical characterization, micro-structural and wear study. The present work focuses on the development of copper as a matrix material and carbon nanotubes as a reinforcement material by using powder metallurgy technique, and study on mechanical properties, micro-structural characterization and wear behavior.

III.OBJECTIVES OF THE PRESENT WORK

The aim of the present work is to develop nanocomposites with copper as a matrix material and carbon nanotubes as a reinforcement material. Thepowder metallurgy technique is used for fabrication of samples (billets).

- Preparation of raw material.
- Fabrication of nanocomposite by powder metallurgy technique.
- The fabrication samples are investigated for various mechanical properties.
- Micro-structural study by using scanning electron microscope (SEM).
- Wear study by pin on disc wear testing machine.

Project work methodology

Powder metallurgy technique is as old as brick making animal and human figurines and objects made up of fired ceramics. In simple terms, it can be described as the fabrication components from powdered materials the normal operation would consist of pressing a calculated amount of the powder in a die of a required shape. The pressure can be applied in cold condition (cold compaction) or hot condition (hot compaction). The pressed compact has porosity and is further dandified using several processes. The classical consolidation method is sintering, in which the pressed compact is heated in a furnace at a temperature that are below the melting point of the material.Densification and change of shape result due to several processes like surface diffusion grain boundary diffusion, evaporation, and condensation at the inter particle contacts which leads to a decrease of the inter particle distances and elimination of pores.Sintered compacts can be subjected to further deformation processes like extrusion, rolling, or forging depending on the requirement. Sintering is to fully dense compact would require a long time and this could be accompanied by grain growth which could be deleterious for the application. Sometimes sintering is accompanied by application of pressure in a single direction (hot pressing) or in all directions (hot iso-static pressing).This process can achieve higher densities at lower temperatures and in less time, leading to lesser grain growth. Powder metallurgy has been used for the fabrication of composite for a long time. It is easy to disperse the two phase in powder form. The level of mixing is then governed by the size of the components added. Since there is no bulkmelting, there is no major rearrangement in the distribution of the phases in the final product.

The following powder metallurgy techniques have been used to prepare the samples,

- Blending or mixing of powder
- Compaction
- Sintering

The various tests conducted on the specimen are

- Density test
- Harness test
- Microstructure test
- Wear test

The density and hardness test have been conducted on the green compacts and sintered samples and compared the test values among green compact and sintered samples.

Material selection

In composite there are two types of material first one is matrix material and second one is reinforcement material.

Matrix material

The base material (copper) having 325 mesh and its atomic number 29. It is a ductile metal with very high thermal and electrical conductivity. Pure copper is soft and malleable a freshly exposed surface has a reddish-orange color. It is used as a conductor of heat and electricity, a building material, and a constituent of various metal alloys.

Properties of copper

- **Corrosion resistant:** Copper is low in the reactivity series. This means that it doesn't tend to corrode. Again, this is important for its use for pipes, electrical cables, saucepans and radiators. It is well suited to decorative use. Jewellery, statues and parts of buildings can be made from copper, brass or bronze and remain attractive for thousands of years.
- **Antibacterial:** Copper is a naturally hygienic metal that slows down the growth of germs such as E-coli.This is important for applications such as food preparation, hospitals, coins, door knobs and plumbing systems.
- **Easily joined:**Copper can be joined easily by soldering or brazing. This is useful for pipe work and for making sealed copper vessels.
- **Ductile:**Copper is a ductile metal. This means that it can easily be shaped into pipes and drawn into wires.
- **Tough:**Copper and copper alloys are tough. This means that they were well suited to being used for tools and weapons.
- **Non-magnetic:**Copper is non-magnetic and non-sparking. Because of this, it is used in special tools and military applications.
- **Attractive colour:**Copper and its alloys, such as brass, are used for Jewellery and ornaments. They have an attractive golden colour which varies with the copper content. They have a good resistance to tarnishing making them last a long a time.
- **Alloys easily:**Copper can be combined with other metals to make alloys. The most well-known are brass and bronze. Although copper has excellent electrical and thermal properties, it needs to be hardened and strengthened for many industrial applications.

Table-1. Properties of copper

Atomic number	29
Density of copper	8.96 g/cm^3
Melting point	1083 °C
Boiling point	2595 °C
Elastic modulus	117 Gpa
Thermal conductivity	391 W/mK

Reinforcement material

Carbon nanotubes used as a reinforcement material were procured from Redex technology, Delhi. It is a multi-walled carbon nano tubes (MWCNT), it was synthesized by chemical vapour deposition (CVD) technique.

Properties of carbon nano tubes

- The strongest and most flexible molecular material because of carbon-carbon covalent bonding and seamless hexagonal network architecture.

- Strength to weight ratio 500 times greater than for aluminium, steel and titanium.
- Maximum strain 10% much higher than any material.
- Thermal conductivity 3000 W/mK in the axial direction with small values in the radial direction.
- Very high current carrying capacity.
- Excellent field emitter, high aspect ratio and small tip radius of curvature are ideal for field emission.

Fabrication and experimental work

Cu/CNT composite is fabricate by powder metallurgy is processing technique that generally involves production of metals powders and conversion of these powders in to useful engineering structures.

Purification of carbon nanotubes

Initially the carbon nanotubes are dispersed in a solvent and sonicated for 20 min and it is been evaporated by heating the solvent .The solvent used is ethanol and sonication is done by ultra-sonic wave sonication equipment. By this process the CNTs are scattered and it can be used as reinforcement

Fabrication process

Powder metallurgy technique is used to fabrication of Cu/CNT nano composite. Powder metallurgy is an established technique for developing state of component. The process of process of powder metallurgy starts with homogeneous mixing of reinforcement (CNT) with powder matrix (copper). The mixing is followed by compaction and compacted samples which are sintered in selected environments for densification of components.

The technique used to fabricate of Cu/CNT for present work consists of three basic operations.

- Mixing of powder
- Compaction
- Sintering

Blending or mixing of powder

The matrix material (copper) and reinforce material carbon (nano tubes) are prepare accordingly by weighting them accurately in electronic weighing balance and thoroughly mixed to achieve successful result in compaction and sintering. The copper powder and carbon nano tubes are different weight fraction both mixture is fed in to the rotating container along with hardened still balls the ball diameter are 10 to 12mm, at a speed of 200 rpm for a duration of 5 minute the short duration and slower milling speed were selected to ensure that the reinforcement remain intact without breakage during mixing.

Table-2. Weight percentage for Cu/CNT

CNT (%)	Copper (gm)	CNT (gm)	Total weight (gm)
0	30	0	30
0	60	0	60
0.5	29.85	0.15	30
0.5	59.7	0.30	60
1	29.7	0.30	30
1	59.4	0.60	60
1.5	29.55	0.45	30
1.5	59.1	0.90	60

Die design

The die is made up of EN-19 steel and operations carried turning, boring and grinding. Die consists of three parts.

- Punch
- Body
- Bottom plate

All dimensions are in mm

Front view

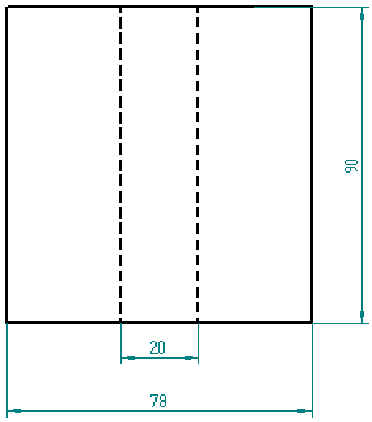

Fig 1. Front view of die

Top view

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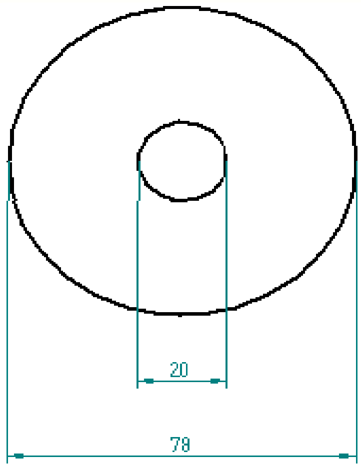

Fig 2. Top view of die

Front view of bottom plate

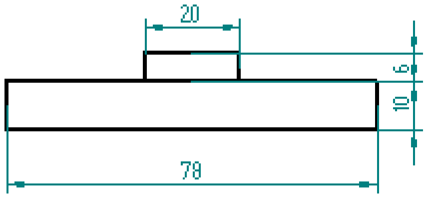

Fig 3. Front view of bottom plate

Top view of bottom plate

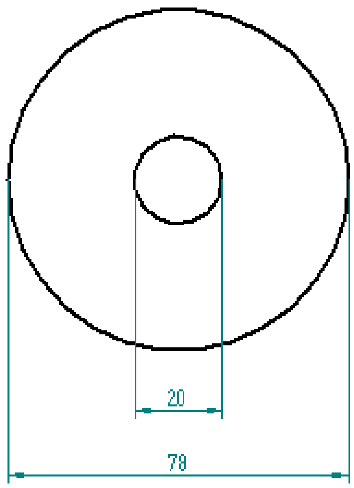

Fig 4. Top view of bottom plate

Front view of plunger

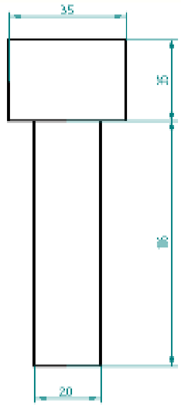

Fig 5. Front view of plunger

Top view of plunger

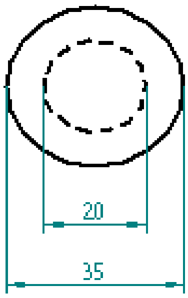

Fig 6. Top view of plunger

Assembly of die

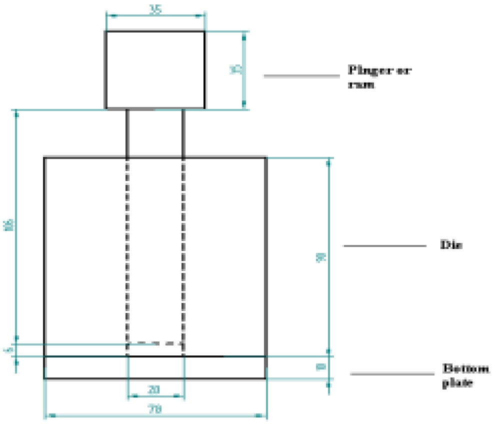

Fig 7. Assembly of Die design

Compaction

The compaction process involves pressing a calculated amount of powder by universal testing machine in a circular die to produce a disc shape of solid green samples at a room temperature. Green samples means yet not fully processed samples when the load is increase the particles are plastically deformed which leads to a decrease of the inter particle distances and elimination of pores and the applied load is 147 KN.

Sintering

Sintering is a heat treatment process in which compacted samples or known as green samples must be sintered to achieve their required mechanical properties (to increase strength and hardness). The green samples are heated in a controlled atmosphere. The temperature between 0.7 to 0.9 times of melting point of the base metal (copper) time duration is 45 min. sintering process is done by tube furnace.

Fig 8.Sintered samples

Hardness test

Hardness is defined as the resistance of a material to penetration. Methods to characterize hardness can be divided in to three primary categories.

- Scratch test
- Rebound test
- Indentation test

Indentation test

This test actually produces a permanent impression in the surface of the material. The force and size of the impression can be related to a quantity (hardness) which can be objectively related to the resistance of the material to permanent penetration. Because the hardness is a function of the force and size of the impression, the pressure used to create the impression can be related to both the yield and ultimate strength of materials.

Brinell hardness test: The Brinell hardness method consists of indenting the test material 10 mm hardened steel ball subjected to a load of 250 Kg, the load is normally applied for 30 seconds. The diameter of the indentation left in the test material is measured with a low powdered microscope. The Brinell hardness number is calculated by dividing the load applied by the surface area of the indentation.

$$BHN = \frac{2P}{\pi D\left[D - \sqrt{D^2 - d^2}\right]}$$

Where

BHN:Brinell hardness number

P:Load in Kg

D: Ball diameter in mm

d:Impression in mm

Wear test

Wear study is defined as the loss of material due to abrasion, adhesion, erosion or other types of wear mechanism is a fundamental phenomenon occurring between two surfaces in a relative motion on each other. The wear test is conducted using pin on disc wear testing machine. A typical pin is cylindrical in shape with diameter equal to 8 mm and length equal to 20 mm. A typical disc has diameter of 180 mm and thickness of 12 mm. The disc is ground to get a surface roughness of 0.8 micrometer. The disc is made up of highly polished EN-25 steel. During wear testing wear loss experienced by the pin specimen is measured in microns. Measurement of wear loss of the pin was used to evaluate the wear loss during wear test.

Micro structural study

It is a study of the crystal structure of a material, their size, composition, orientation, formation, interaction and ultimately their effect on the macroscopic behavior in terms of physical properties such as strength, toughness, ductility, hardness, corrosion resistance.

Scanning electron microscope

It is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, produces various signals that can be detected and that contain information about samples surface topography and composition.The electron beam generally scanned in a raster scan pattern, and the beam position is combined with the detected signal to produce an image. Scanning

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electron microscope can achieve resolution better than one nanometer specimens can be observed in a high vacuum, low vacuum and in environment.

IV.RESULTS AND DISCUSSION

Density

The green compact density and sintered density of the sample with carbon nanotubes and without carbon nanotubes were measured and compared. The density of both compacted and sintered samples increases as the percentage of reinforcements (carbon nanotube) increases. It is also observed that sintered density values are higher than the compacted density values. This indicates the presence of porosity in the green compact. The variation of green compacted and sintered densities shown in below table.

Table-3.Density of Cu/CNT samples for compacted and sintered samples

Composition	Compacted density (g/cm^3)	Sintered density (g/cm^3)
0%	8.948	8.956
0.5%	8.882	8.920
1%	8.914	8.812
1.5%	8.205	8.749

Hardness test

A considerable improvement of hardness is observed by addition of carbon nanotubes in the copper matrix. The hardness increases almost linearly with increasing carbon nanotubes so it is confirmed that such remarkable improvement of hardness by carbon nanotube (CNT) reinforcement is originated from the high interfacial strength at CNT/Cu interface, the homogeneous distribution of CNTs within the copper matrix and attained at high relative densities based on this result it can be shown that the improvement of mechanical properties carbon nanotubes reinforced nanocomposites is expected when the external load can be shared by homogeneously distributed carbon nanotubes.

Table-4.Hardness numbers of Cu/CNT samples

Percentages (%)	Hardness of compacted samples in BHN	Hardness of sintered samples in BHN
0	77	79
0.5	80	84
1	84	89
1.5	87	93

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Weight loss

The effect of the applied load on the weight loss for carbon nanotube reinforced copper matrix composite shows that increasing the load, weight loss also increases, small effect of weight loss on carbon nanotube reinforced copper matrix as compared to pure copper material.With increase in sliding distance, there is higher weight loss for the matrix material the weight loss of the composites was much lower when compared with the matrix material and weight loss reduces with increased content of carbon nanotubes in the matrix material.

Table-5. Weight loss with load at constant speed 100 rpm

Load in kg	**Weight loss in gms**			
	0%	**0.5%**	**1%**	**1.5%**
5	0.0163	0.0147	0.0113	0.0105
7	0.0307	0.0286	0.0254	0.0224
9	0.0354	0.0328	0.0318	0.0261

Table-6. Weight loss with time at constant speed 100 rpm

Time (min)	**Weight loss (gms)**			
	0%	**0.5%**	**1%**	**1.5%**
5	0.0158	0.0139	0.0102	0.0098
10	0.0162	0.0148	0.0113	0.0104

V.CONCLUSIONS

With the observed results following conclusions are made:

- Carbon nanotube reinforced copper matrixes composite weresuccessfully developed through powder metallurgy technique.
- With about 147 KN sufficient to enough to produce near full density specimens and sintering at a temperature of 750°C in the argon gas atmosphere is sufficient to produce good sintered specimen at the 45 minute sintered time.
- The reinforcement of the carbon nanotubes proves to enhance of the copper composite such asdensity. The hardness and wear resistance of carbon nanotube reinforced copper matrix composite were significantly increased with increasing the volume fraction of carbon nanotubes.
- The remarkable enhancement of hardness is originated from the homogeneous distributed of carbon nanotubes in the copper matrix.

- The dispersed carbon nanotube in the copper matrix composite provides considerably enhanced wear resistance.

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