International Journal of Innovative Technology and Exploring Engineering (IJITEE)
ISSN: 2278-3075 (Online), Volume-8 Issue-5, March 2019

Preparation of Carbon Nanotubes for Copper Coating and Characterizing

Raghuvaran P, Fameen Sha M, Deepakh K, Aravinth Athithya S, Aadham Irfan S

Abstract: ***Composites have gained the interest of research scholars over conventional materials due to their productive advantage. Specifically Metal Matrix Composites (MMCs), which have comparatively higher strength are widely preferred than other available matrices. Reinforcements have certain properties that are expected to be attained in the composites. Carbon Nano Tubes (CNTs) with paramount mechanical properties is one such reinforcement. Based on our observations from literature collected earlier, we have used Electroless Coating Technique to coat copper on Carbon Nano Tubes. The presence of copper coating was assured by the photographic images of Scanning Electron Microscope (SEM). These copper coated Carbon Nano Tubes were later used as reinforcement materials in Metal Matrix Composites preparation in which Aluminium alloy 7075 (Al 7075) was used as the base matrix. Stir casting method was chosen for fabrication of composites.***

Index Terms: Carbon Nanotube, Electroless coating, Metal matrix Composites, Stir casting.

I. INTRODUCTION

One of the key aspects of material science and engineering design is to obtain and ascertain advanced materials exhibiting comparatively good properties. Composite materials have been developed for such needs. Composites are classified based on the chemical and physical nature of matrix phases, e.g., metal matrix, ceramic matrix and polymer matrix composite.

Carbon NanoTubes was first discovered by Iijima in 1991 [1], by rolling a graphene sheet onto itself forming tubular structures. The Young's modulus of CNT is 1 TPa, which makes them better suited reinforcements for composites [2]. On comparison with base metal, CNT reinforced MMC is characterized by improved strength to weight ratio, a higher modulus to density ratio, better fatigue resistance, improved high temperature mechanical properties, lower coefficient of thermal efficiency, and improved wear resistance. Researchers used CNT as reinforcements for aluminium [3], magnesium [4], copper [5], nickel[6], titanium [7] and other conventional materials or their alloys for improving the mechanical [8], electrical [5], tribological [5] and thermal [9] properties. The dispersion quality of CNT within the matrix [10] decides the final mechanical property of the produced material.

We have fabricated and characterized carbon nanotubes with electroless coated copper particles in this study. The electroless coating process involves various steps as demonstrated by the authors in literature [11]. The samples obtained form coating process was further given for SEM analysis to ascertain the coating of copper. These samples were used as reinforcements for Al 7075 composites in fabrication later. The variations in properties of prepared samples were compared to the pure aluminium obtained and tested at same conditions.

Manuscript published on 30 March 2019.
***Correspondence Author(s)**
Raghuvaran P, Assistant Professor, Department of Mechanical Engineering, Sri Krishna College of Engineering and Technology, Coimbatore, Tamilnadu, India-641008.
FameenSha M, UG Scholar, Department of Mechanical Engineering, Sri Krishna College of Engineering and Technology, Coimbatore, Tamilnadu, India-641008.
Deepakh K, UG Scholar, Department of Mechanical Engineering, Sri Krishna College of Engineering and Technology, Coimbatore, Tamilnadu, India-641008.
Aravinth Athithya S, UG Scholar, Department of Mechanical Engineering, Sri Krishna College of Engineering and Technology, Coimbatore, Tamilnadu, India-641008.
Aadham Irfan S, UG Scholar, Department of Mechanical Engineering, Sri Krishna College of Engineering and Technology, Coimbatore, Tamilnadu, India-641008.

II. EXPERIMENTATION

The process chosen for coating copper on CNT is electroless coating method. Coating of CNTs resulted in improved strength of samples and wettability with the matrix. The experimentation was carried out based on the literature.

A. Electroless Coating

Electroless process is one of the techniques used for coating in which chemicals are used to perform various simultaneous reactions resulting in coating without electricity. Compared to electrocoating this method has many advantages like cost effective and simple process procedures.

The copper coating process on the CNTs is shown in figure 1.

B. Steps involved in coating of copper on CNT's

The following steps are to be done for CNTs before subjecting to electroless coating:

a. Oxidation,
b. Pre-activation and Sensatization,
c. Electroless coating

Oxidation of CNTs:

A solution of 200 ml containing 3:1 mixture of HNO_3 and H_2SO_4 is mixed with 2g of CNT maintained at 120°C for 10 hours.

Pre-activation and Sensatization:

Published By:
Blue Eyes Intelligence Engineering and Sciences Publication (BEIESP)

Oxidised Carbon NanoTubes were immersed in a solution of $SnCl_2$ (0.1 mol./l) – HCl (0.1 mol./l) for about 30 minutes, which was held at room temperature for about 72 hours and is shown in figure 2. The Carbon NanoTubes were then transferred to a solution consisting $PdCl_2$ (0.0014 mol./l)-HCl (0.25 mol./l) and was held for 30 minutes. Both were initially mixed using magnetic stirrer before introducing CNT as shown in figure 3. Stirring was prolonged at 500 rpm until complete dissolving of $PdCl_2$ in the HCl solution was observed.

Electroless coating:

*T*he Carbon NanoTubess were then introduced to electroless bath having the solutions as listed in table 1. The temperature and pH value of the bath was shown in the table. CNT was immersed in the solution for about 2 minutes.

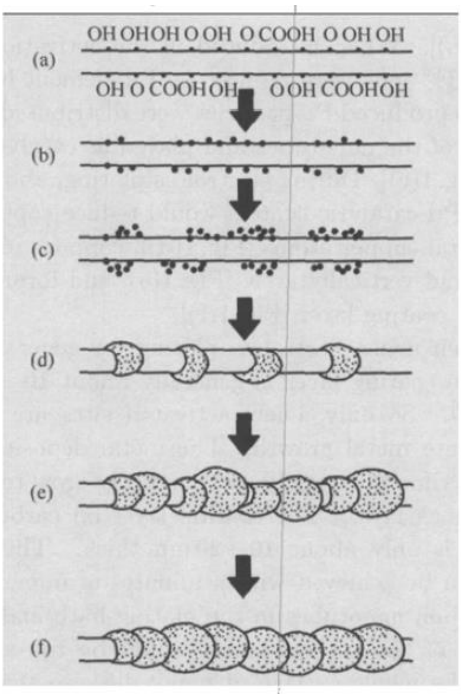

Fig. 1 Process of coating copper on CNTs [11]
a. Oxidation, b. Sensatization, c. Activation, d. Copper particles depositing, e. Copper particles growing, f. Copper particles filming.

TABLE I
Electoless bath solutions

S.No:	Bathing solution	Amount of chemicals
1.	$CuSO_4.5H_2O$	6.25 g/L
2.	$KNaC_2H_6O_6.4H_2O$	10 g/L
3.	HCHO(added later)	3 ml/L
4.	$NiCL_2.6H_2O$	0.5 g/L
5.	Polyethylene glycol 6000	10 mg/L
6.	pH (adjusted with NAOH)	11.5
7.	temperature	30 ^{0}C

The CNTs were washed with distilled water after the completion of each process to prevent foreign particles. Then they were separated from the medium by centrifugation process. Photographic images CNTs before and after centrifugation were displayed in figures 4 and 5. Filtration was also tried instead of centrifugation, but it did not obtain desired results. The reason for failure of filtration process is the fact that CNTs were nano sized particles. They blocked the pores of filters, which prevented the flow of distilled water.

Captured images during processing: The obtained copper coated samples were confirmed with the presence of copper coating by characterization using SEM images

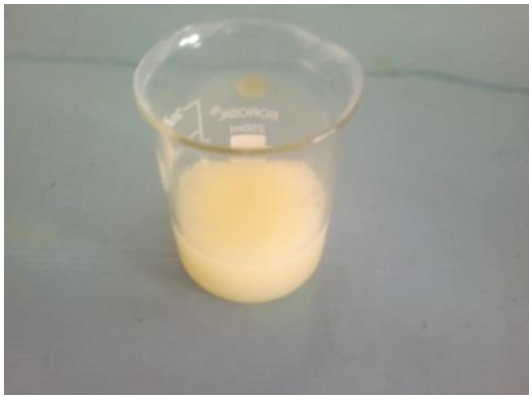

Fig. 2 $SnCl_2$ and HCl solutions after 72 hours of aging

Fig. 3 Magnetic stirring of $PdCl_2$ and HCl

Fig. 4 CNTs in a test tube before centrifugation

Published By:
Blue Eyes Intelligence Engineering and Sciences Publication (BEIESP)

Fig. 5 CNTs in a test tube after centrifugation

III. RESULT AND DISCUSSION

The morphlogical structure of Carbon Nano Tubes before and after coating of copper was analyzed and presented using SEM images. Carbon Nano Tubes before oxidation were long and their surface had impurities. The SEM image shown in figure 6 indicates that there were longer lengths with diameter in nano scale, indicating that they have large length to diameter aspect ratio.

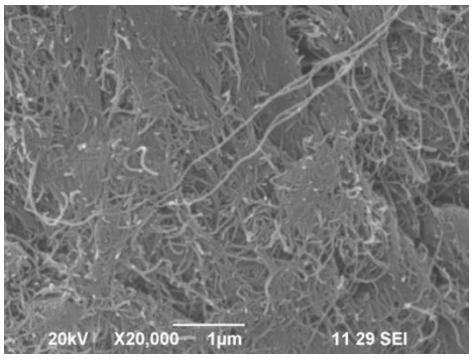

Fig. 6 SEM image of CNTs before oxidation

The aspect ratio of these CNTs was reduced after oxidation which was suitable for coating effectively. This was further confirmed by the SEM image shown in figure 7.

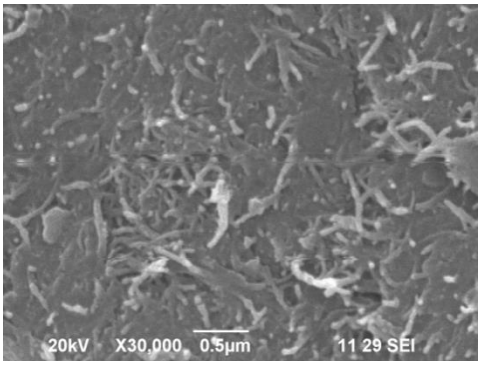

Fig. 7 Oxidised CNTs – SEM image

Wettability of CNTs gets improved in addition to removal of surface impurities on oxidising of CNT [12]. Hence, oxidised CNTs fetch better results, if added as composite reinforcements. In order to achieve a uniform coating layer on CNT surface, sensatization and activation are done before coating. To speed up the process $PdCl_2$ is used as catalyst while conducting experiment.

Copper gets coated on the CNTs during electroless coating. They were washed in distilled water and separated by centrifugation. The coating was confirmed by SEM image shown in figure 8.

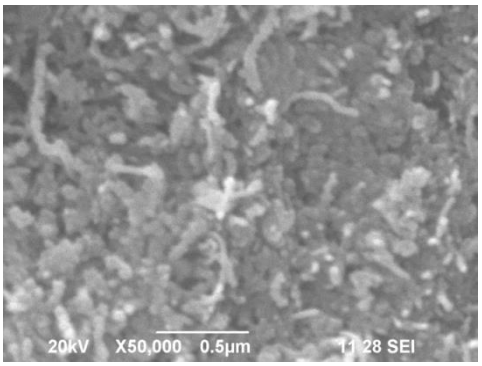

Fig. 8 SEM image of CNTs coated with copper

In presence of $PdCl_2$, the CNTs were dipped for a few minutes in electroless bath. Thickness of the coating can be controlled by controlling the pH value of solution and the temperature. Coating may not be even on the surface of CNTs. Anyhow, non uniformity in coating doesn't affects the properties of composites casted.

IV. CONCLUSIONS

SEM micrograph reveals the coat of copper on to surface of CNTs successively. The method helps to improve the interfacial adhesion of reinforcement with that of matrix. The method moreover was found to be economical and simple. It's a partial work as per the references of already did researches. The coated CNTs will be used by us in future for casting of aluminium composites

REFERENCES

1. Bakshi S R, Lahiri D and Agarwal A, Carbon nanotube reinforced metal matrix composites-a review, International Materials Reviews, 2010, 55(1), 41-64.
2. George R, Kashyap K T, Rahul R and Yamdagni S, Strengthening in carbon nanotube/aluminium (CNT/Al) composites, Scripta Materialia, 2005, 53(10), 1159-1163.
3. Julien Stein, Blanka Lenczowski, Nicole Frety and Eric Anglaret, Mechanical reinforcement of a high-performance aluminium alloy AA5083 with homogeneously dispersed multi-walled carbon nanotubes, Carbon, 2012, 50(6), 2264-2272.
4. Goh C S, Wei J, Lee L C and Gupta M, Development of novel carbon nanotube reinforced magnesium nanocomposites using the powder metallurgy technique, Nanotechnology, 2006, 17(1), 7.
5. Tu J P, Yang Y Z, Wang L Y, Ma X C and Zhang X B, Tribological properties of carbon-nanotube-reinforced copper composites, Tribology Letters, 2001, 10(4), 225-228.
6. Borkar T and Harimkar S, Microstructure and wear behaviour of pulse electrodeposited Ni-CNT composite coatings, Surface Engineering, 2011, 27(7), 524-530C.
7. Mizuhisa Nihei, Akio Kawabata1, Daiyu Kondo, Masahiro Horibe, Shintaro Sato and Yuji Awano, Electrical properties of carbon nanotube bundles for future via interconnects, Japanese journal of applied physics, 2005, 44(4A), 1626-1628
8. Choi H.J, Shin J H and Bae D H, The effect of milling conditions on microstructures and mechanical properties of Al/MWCNT composites, Composites Part A: Applied Science and Manufacturing, 2012, 43(7), 1061-1072.

Published By:
Blue Eyes Intelligence Engineering and Sciences Publication (BEIESP)

9. Seungchan Cho, Keiko Kikuchi, Takamichi Miyazaki, Kenta Takagi, Akira Kawasaki and Takayuki Tsukada, Multiwalled carbon nanotubes as a contributing reinforcement phase for the improvement of thermal conductivity in copper matrix composites, Scripta Materialia, 2010, 63(4), 375-378.
10. Esawi A and Morsi K, Dispersion of carbon nanotubes (CNTs) in aluminum powder, Composites Part A: Applied Science and Manufacturinjg, 2007, 38(2), 646-650.
11. Feng Yi and Yuan Hailong, Electroless plating of carbon nanotubes with copper. Chinese Journal of Chemical Engineering, 2004. 12(5): p. 728-731.

AUTHORS PROFILE

Raghuvaran is working as Assistant Professor in Sri Krishna College of Engineering and Technology since June 2014. He completed his master's degree (M.E - Engineering Design) with distinction at Kongu Engineering College, Erode, in 2014 and bachelor's degree (B.E - Mechanical Engineering) with distinction at Kongunadu College of Engineering and Technology, Trichy with University 11th rank. He completed his diploma degree (Mechanical Engineering) at Kongu Polytechnic College, Erode. His areas of interest are Design of Machine Elements, Engineering Mechanics and Engineering Graphics. He is currently pursuing his part time PhD degree in the area of Composite Materials.

Fameen Sha is currently pursuing his final year Mechanical Engineering at Sri Krishna College of Engineering and Technology, Coimbatore.

Deepakh is currently pursuing his final year Mechanical Engineering at Sri Krishna College of Engineering and Technology, Coimbatore.

Aravinth Athithya is currently pursuing his final year Mechanical Engineering at Sri Krishna College of Engineering and Technology, Coimbatore.

Aadham Irfan is currently pursuing his final year Mechanical Engineering at Sri Krishna College of Engineering and Technology, Coimbatore.

Published By:
Blue Eyes Intelligence Engineering and Sciences Publication (BEIESP)