

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

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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 from 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.

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:

- Oxidation,
- Pre-activation and Sensatization,
- Electroless coating

Oxidation of CNTs:

A solution of 200 ml containing 3:1 mixture of HNO₃ and H₂SO₄ is mixed with 2g of CNT maintained at 120°C for 10 hours.

Pre-activation and Sensatization:



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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:

The Carbon NanoTubes 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.

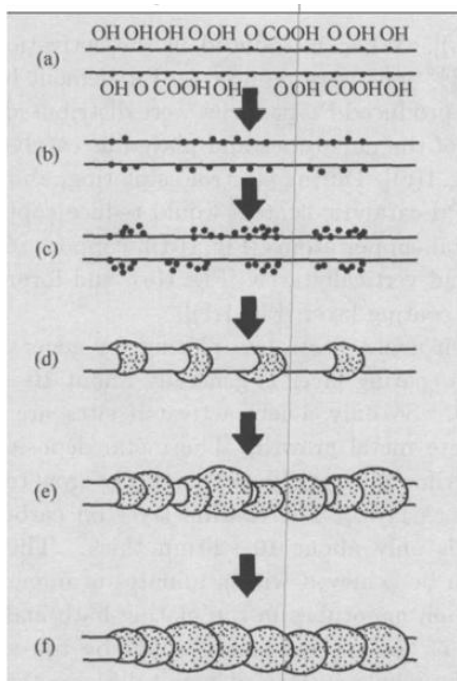


Fig. 1 Process of coating copper on CNTs [11]

a. Oxidation, b. Sensitization, c. Activation, d. Copper particles depositing, e. Copper particles growing, f. Copper particles filming.

TABLE I
Electroless bath solutions

S.No:	Bathing solution	Amount of chemicals
1.	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	6.25 g/L
2.	$\text{KNaC}_2\text{H}_6\text{O}_6 \cdot 4\text{H}_2\text{O}$	10 g/L
3.	HCHO(added later)	3 ml/L
4.	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	0.5 g/L
5.	Polyethylene glycol 6000	10 mg/L
6.	pH (adjusted with NaOH)	11.5
7.	temperature	30 °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



Fig. 5 CNTs in a test tube after centrifugation

III. RESULT AND DISCUSSION

The morphological 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.

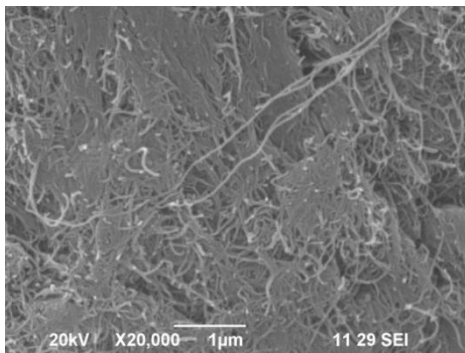


Fig. 6 SEM image of CNTs before oxidation

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.

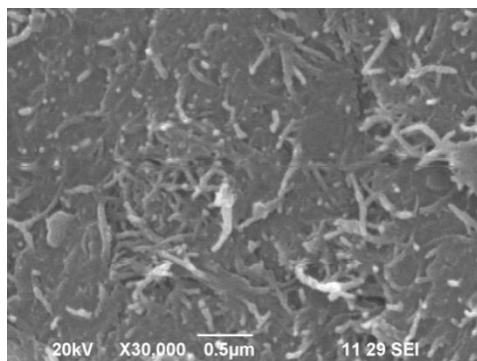


Fig. 7
Oxidised
CNTs –

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, sensitization 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.

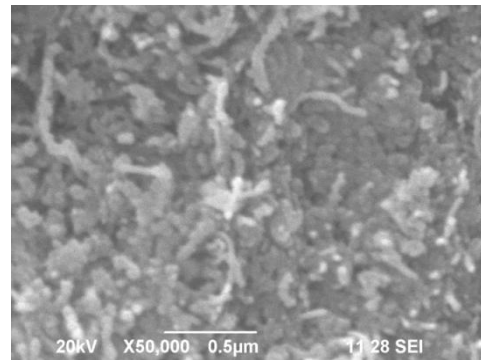


Fig. 8 SEM image of CNTs coated with copper

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

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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.



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