

Preparation and Characterization of Electroless Copper Coated Carbon Nanotubes

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ABSTRACT: Composites are gaining interest among researchers due the advantages yielded by them compared to conventional materials. Metal matrix composites (MMCs) are majorly preferred than the other available matrices due to maximum strength. Carbon nanotubes (CNTs), with superior mechanical properties, are a part of reinforcements used in MMCs. In our study, with previously collected literatures, we have coated copper on the CNTs using electroless coating technique. The coating was confirmed by the images of Scanning Electron Microscope (SEM). These coated CNTs in future was used as reinforcements for metal matrix composites, were Aluminium alloy 7075 (Al 7075) was used as matrix. The method of fabrication of composites chosen was stir casting.

KEYWORDS: Composites, Metal matrix, Electroless coating, Carbon nanotube, Stir casting

I. INTRODUCTION

Great interest in engineering design is to search for advanced materials exhibiting good properties. Composite material has been developing such materials and it is classified based on the physical or chemical nature of matrix phases, e.g., metal matrix, ceramic matrix and polymer matrix composite.

CNT was first discovered by Iijima in 1991 [1], and they recognize tubes are made by rolling a graphene sheet onto itself. They have a Young's modulus of 1TPa, which make them better suited as composite reinforcements [2]. Compared to metal alone CNT reinforced MMC is characterized by a higher strength to weight ratio, a higher modulus to density ratio, better fatigue resistance, better high temperature mechanical properties, a lower coefficient of thermal efficiency, and better 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 [9], tribological [5] and thermal [10] properties. Anyhow, the final mechanical property of the produced material depends on the dispersion quality of CNT within the matrix [11].

In this paper we have fabricated and characterized carbon nanotubes with electroless coated copper particles. The process of coating involves several steps as told in literature [12]. The samples were further given for SEM analysis for confirming the coating of copper. These samples were used as reinforcements for Al 7075 composites in future. The property variations of as prepared samples were compared to that of the pure matrix made and tested at same conditions.

II. EXPERIMENTATION

The method of processing chosen for coating is electroless method. Coating of CNTs results in improved strength of samples and wettability with the matrix. The experimentation was done as per the literature.

A. Electroless coating

Electroless process is one of the techniques used for coating of copper on CNTs. Compared to electro coating or electroplating this method has many advantages like no need of electricity, cost effective and easiness in processing. The process of coating of copper on the CNTs was shown as in figure 1.

B. Steps involved in Electroless coating of copper on CNTs:

It involves the following steps to be done for CNTs prior to electroless coating:

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

Oxidation:

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

Pre-activation and Sensatization:

Oxidised CNTs were immersed in a solution of SnCl₂ (0.1 mol./l) – HCl (0.1 mol./l) for 30 minutes, which was aged at room temperature for 72 hours and is shown in figure 2. The CNT was then transferred to solution having PdCl₂ (0.0014 mol./l)- HCl (0.25 mol./l) and kept for 30 minutes. They both were initially mixed together using a magnetic stirrer before introducing CNT as shown in figure 3. Stirring was done at 500 rpm until complete dissolving of PdCl₂ in the HCl solution.

Electroless coating:

The CNTs were then transferred to electroless bath having the solutions as listed in table 1. The temperature and pH value of the bath was adjusted as shown in the table. CNTs solution was kept in the solution for 2 minutes.

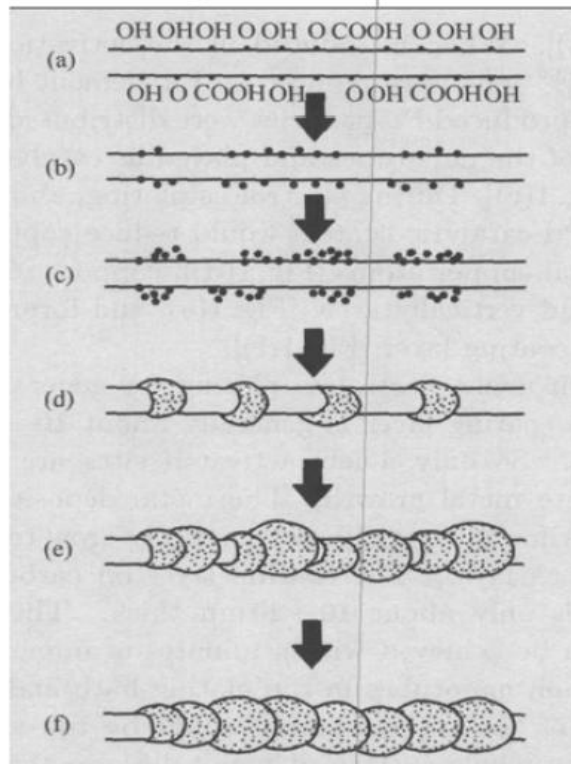


Fig. 1 Process of coating copper on CNTs [12]

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 ₂ O	6.25 g/L
2.	KNaC ₂ H ₆ O ₆ .4H ₂ O	10 g/L
3.	HCHO(added later)	3 ml/L
4.	NiCL ₂ .6H ₂ 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 and separated from the medium by centrifugation. CNTs before and after centrifugation were shown in figures 4 and 5. Filtration was also tried instead of centrifugation but it does not gave good results. The reason for which is, since the CNTs were of nano size, they got occupied on the pores of filters, which prevented the flow of distilled water through the pores.

Captured images during processing:



Fig. 2 SnCl₂ and HCl solutions after 72 hours of aging



Fig. 3 Magnetic stirring of PdCl₂ and HCl



Fig. 4 CNTs in a test tube before centrifugation



Fig. 5 CNTs in a test tube after centrifugation

The as coated samples were confirmed for the presence of coating by characterization using SEM images.

III. RESULT AND DISCUSSION

The morphology before and after coating of CNT was analysed using SEM images. CNTs before oxidation were long and their surface may have impurities. The SEM image of it was shown in figure 6, which indicates that they were of longer lengths with diameter in nano scale, indicating that they have large aspect ratio.

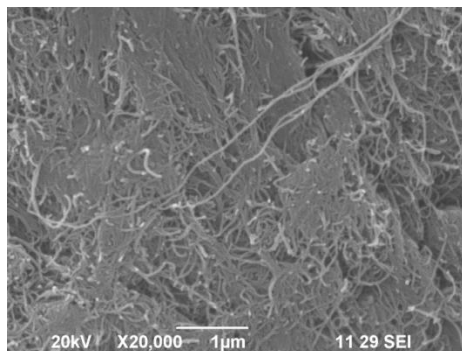


Fig. 6 SEM image of CNTs before oxidation

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

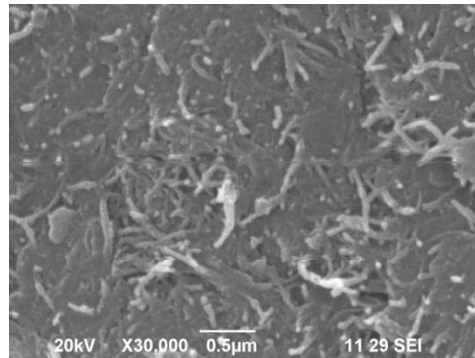


Fig. 7 SEM image of oxidised CNTs

Oxidation of CNT not only cleans the surface by removing the impurities on its layer but also improves its wettability [12]. Thus the oxidised CNTs if used without coating in a composite may also expected to have improved strength than the non-oxidised one.

Sensitization and activation are done before coating to achieve a uniform layer of coating on all surface of CNT. PdCl₂ used in this process acts as a catalyst during coating.

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.

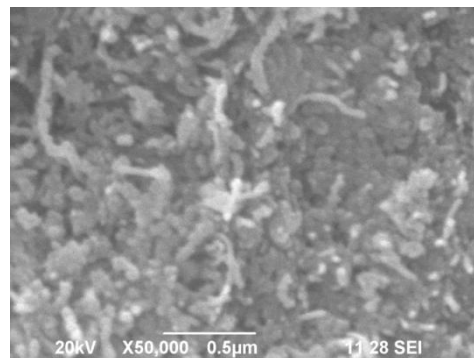


Fig. 8 SEM image of copper coated CNTs

The CNTs were immersed in the electroless bath for just a few minutes because of the presence of PdCl₂. The temperature and pH value of the solution plays a major role in thickness of copper coated on CNT. It may result in non uniform coating thickness, even though it does not lower the performance if used in composite.

IV. CONCLUSIONS

CNTs were successfully coated on the surface of CNTs, which were confirmed by SEM images. The method of coating was simple and inexpensive. It helps in improving the interfacial adhesion of the matrix and the reinforcement. It's a part of our work as per the previous literatures. These coated CNTs were used as reinforcements in a metal matrix composite with matrix as Al 7075 with the aim of improving the mechanical property of conventionally available Al 7075 alloy, which was our ongoing work.

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