International Journal of Modern Engineering Research (IJMER)

www.ijmer.com Vol.2, Issue.3, May-June 2012 pp-1167-1170 ISSN: 2249-6645

Processing and Characterization of Aluminium-Silver Coated MWCNT Composite Made By Vacuum Hot Pressing

UNNIKRISHNAN. R[1], RENJITH. M. S[2], ANEESH KUMAR. J[3], T. KRISHNAKUMAR[4]

1. DEPARTMENT OF MECHANICAL ENGINEERING, GOVERNMENT ENGINEERING COLLEGE, THRISSUR, INDIA,
2. DEPARTMENT OF NANO SCIENCE AND TECHNOLOGY, ANNA UNIVERSITY, CHENNAI, INDIA.
3. DEPARTMENT OF MECHANICAL ENGINEERING, LOURDE MATHA COLLEGE OF SCIENCE AND TECHNOLOGY, TRIVANDRUM, INDIA.
4. DEPARTMENT OF MECHANICAL ENGINEERING, GOVERNMENT ENGINEERING COLLEGE, THRISSUR, INDIA.

ABSTRACT

In this research work, Al - 1wt% silver coated multi wall carbon nanotube (MWCNT) composite have been processed using powder metallurgy technique. In order to distribute CNT uniformly in the aluminum matrix high energy ball milling is required. . It was found that excessive milling can lead to carbide and alumina formation. So to reduce the milling time, the surface of CNT is modified by coating it with silver. In order to obtain surface modification of carbon nanotubes, electroless coating of silver has been given to carbon nanotubes after liquid phase oxidation, sensitization and activation. Since Al / 1wt% MWCNT could not be sintered, Silicon has been added as sintering agent. Al along with 1wt% silver coated MWCNT and 9wt% of Silicon is milled together in a planetary ball mill for 4 hour. The composite powders were compacted using Vacuum Hot Pressing (VHP). CNT dispersion, phase analysis and powder morphology of the composite is investigated using XRD, SEM and optical microscopy. Density of the composite is measured using Archimedes density principle. Hardness of the composite is measured using Brinell hardness tester.

Keywords: Activation, Carbon nanotube, Electroless coating, Sensitization, Vacuum hot pressing.

1. INTRODUCTION

Nanostructured materials have attracted many researchers due to their outstanding mechanical and physical properties. One example is Carbon Nanotube (CNT) reinforced composites. Most of the researchers have focused on using CNTs to reinforce polymeric and ceramic matrices. A single-wall carbon nanotube, i.e., a nanotube made of only one graphite sheet rolled-up in cylinder, has a young's modulus as high as 1.8TPa and a tensile strength as high as 63GPa, which is one to two orders of magnitude superior to best known steels. Multi-wall nanotubes display lower but still exceptional mechanical properties, and they are easier to synthesize. These superior mechanical properties combined with low density generate several outlets for a composite reinforced by CNTs. CNT-reinforced metallic composites are quickly emerging as attractive materials combining light weight with superior strength and stiffness. Potential application includes automobile and aerospace industry where the decrease of fuel consumption by weight reduction is a priority.

In this research work powder metallurgy techniques were employed to produce a CNT composite with unique mechanical properties. 99.7% purity Aluminium (Al) powder along with multi-wall carbon nanotube of purity greater than 90% having diameter of 5nm and length 20 – 40 microns is used. CNT have a strong tendency to form bundles and agglomerate. So the composite powder where ball milled, where particles are repeatedly fractured and welded and CNT is uniformly dispersed with in Al matrix. The excessive milling of Al / CNT composite powder can lead to oxidation of Al and formation of aluminium carbide which can hinder the mechanical properties. So to avoid this, the surface of CNT is coated with silver using electroless technique. . Since CNT is non-reactive some pre-treatments such as liquid-phase oxidation, sensitization and activation were done on the surface of CNT before coating it with silver. Since Al / 1wt% silver coated CNT milled powder could not be sintered, Silicon is added as the sintering agent. Al along with silver coated MWCNT and Si is milled together for 4hrs in a planetary ball mill. The composite powders were compacted using vacuum hot pressing.

2. EXPERIMENTAL PROCEDURE

2.1 ELECTROLESS COATING OF MWCNT WITH SILVER

For surface modification of carbon nanotubes, electroless coating of silver has been given to carbon nanotubes surface. Like graphite, the surface of carbon nanotube has low chemical reactivity and does not act as catalyst for the deposition of silver and therefore no metal coating take place. Therefore some pretreatments are required to improve chemical reactivity of CNT which includes liquid phase oxidation, sensitization and activation [1]. Liquid phase oxidation is done by immersing raw MWCNT in a mixture of H_2SO_4 and HNO_3 at 1:3 ratios. During sensitization oxidized MWCNT is immersed in a mixture of aqueous solution of 0.1M $SnCl_2$ and 0.1M HCl. During activation the sensitized MWCNT is immersed in a mixture of aqueous solution of

www.ijmer.com Vol.2, Issue.3, May-June 2012 pp-1167-1170 ISSN: 2249-6645

0.0014M $PdCl_2$ and 0.25M HCl. After each step the mixture is agitated using ultrasonic method for one hour. The agitated mixture is washed and then filtered using distilled water. The filtered MWCNT is then heated at 100^0C in an oven.

Finally, the activated carbon nanotubes were introduced in to the electroless bath kept in an alumina crucible. The electroless bath was prepared by mixing the solution I, II and III given in table I. The pH of the electroless bath is 10. The composition of the solution I, II and III and reaction condition for silver coating is given in Table 1. Nanotubes kept in the electroless bath was sonicated at room temperature for 3 hours and kept at room temperature for 30 hours. After coating treatment nanotubes were washed with distilled water and filtered and dried at 100^0C for 3 hours. All the process parameters used in this work were optimized by surface coating of graphite. During electroless plating, the Pd catalytic center would reduce silver ions present in the solution to neutral silver atoms, which get coated on the surface of CNT. The silver particles grew laterally and vertically and form a continuous layer on the surface of CNT.

Table 1 Bath composition for electroless plating

Solution	Composition	Time
Solution I	Sugar - 4-6 Gm Tartaric acid - 0.6gm C_2H_5OH-10 - 15 cc	
Solution II	$AgNO_3$ - 4-6 gm NH_4NO_3 - 6-8gm H_2O - 120cc	
Solution III	NaOH - 10-12 gm H_2O - 120 cc	
Electroless Ag deposition	Solution I + Solution II + Solution III.	4 -5 hrs

2.2 PREPARATION AND COMPACTION OF COMPOSITE POWDERS.

According to Esawi and Morsi [2] aluminium particles kept on welding together with time forming spherical balls. These large particles would pose processing problems during compaction and extrusion. Also CNT has a tendency to form bundles and to get agglomerated in the metal matrix. So high energy ball milling was done so that particles are repeatedly fractured and also to disperse CNT uniformly in the metal matrix. Here Al along with 1wt% silver coated MWCNT is used. 110gm of Al containing 1wt% silver coated MWCNT and 10gm of silicon (to achieve a weight fraction of 9wt%) as sintering agent is milled together using planetary ball mill at 120 rpm for 4 hours in a cylindrical vial containing tungsten carbide balls with ball-to-powder ratio 10:1. The composite powders were compacted using vacuum hot pressing. Vacuum hot pressing is done using an industrial vacuum hot press having a maximum load capacity of 250T and temperature upto 2000^0C. The powders were compacted in graphite cylindrical dies having bore diameter 30mm. The compaction was done at a load of 10T and sintering temperature of 600^0C. The entire process is done at very high vacuum of 10^{-5} mbar.

2.3 CHARACTERIZATION AND TESTING

In order to investigate the properties and the quality of produced sample, the physical, mechanical, and microstructural properties had to be investigated. The physical property was determined by measuring the density of the composites and comparing it with theoretical density. Hardness was used to evaluate the mechanical property. The microstructure was investigated by employing optical microscopy and XRD. XRD is done using panalytical X-Pro diffractometer. It is mainly used for phase analysis and to find out the dispersion of CNT in aluminium matrix. The optical microscope observation of compacted samples was made under Olympus microscope. Hardness of the compacted specimen was measured using universal hardness tester. The diameter of ball indenter used is 2.5mm at a load of 31.25kgf. The dwell time was 15 seconds. From each specimen five indentations are made at different sites and the average is taken as final result. The actual density of composite is measured using Archimedes density principle. It is compared with theoretical density to find out relative density of the composite.

3. RESULT AND DISCUSSION

3.1 MICROSTRUCTURE ANALYSIS

Figure 1 shows the microstructure images of Al / 1wt% silver coated MWCNT / 9wt% Si compacted using vacuum hot pressing at 600^0C. The grey phase denotes the presence of aluminium and dark phase denote the presence of CNT welded to aluminium matrix. Comparing with the microstructure of compact made using uncoated CNT, the uniform distribution of CNT is evidently

www.ijmer.com Vol.2, Issue.3, May-June 2012 pp-1167-1170 ISSN: 2249-6645

seen in compact made using silver coated CNT. It also reveals that CNT's are embedded in the aluminium particle. The microstructure reveals a typical sintered aluminium structure. From the microstructure it is evident that CNT is distributed along the grain boundaries. The microstructure also reveals that milling time can be reduced by using silver coated CNT because Silver coated CNT disperse more uniformly in less milling time which reduces alumina and carbide formation. Some porosities are also present which is clearly revealed at microstructure images taken at higher magnification.

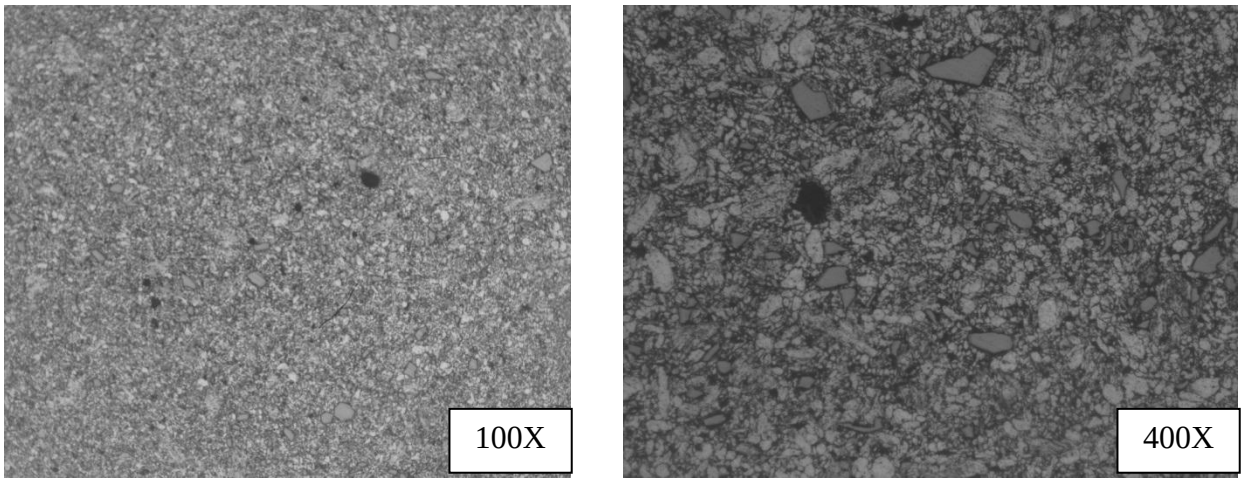

Fig.1 Microstructure images of Al / 1wt% silver coated MWCNT / 9wt% Si compact made by Vacuum hot pressing at 600^0C

3.2 XRD ANALYSIS

Figure 5.25 reveals the XRD pattern for Al / 1wt% silver coated MWCNT / 9wt% Si compact. There is no characteristic peak related to CNT or silver at $2\theta = 26^0$. The major reason is that 4hrs milling in planetary ball mill is sufficient for uniform dispersion of silver coated CNT. The peak appears when the CNT is clustered and diminishes with dispersion of CNT. Another reason for absence of CNT peak is due to the small weight percentage of CNT used, unfavorable strain or CNT deformation and amorphization of CNT. The XRD pattern contains peak corresponding to aluminium and silicon [3].

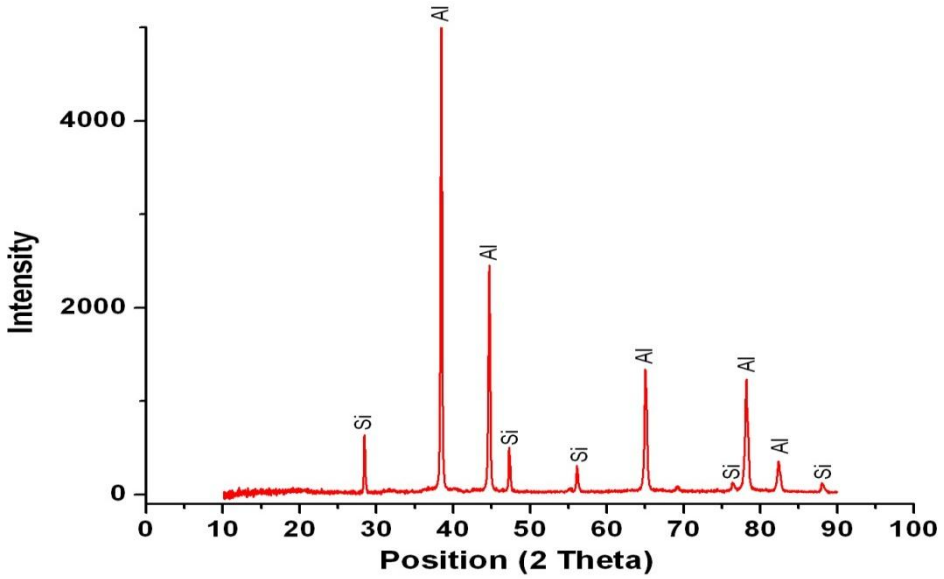

Fig.2 XRD pattern of Al / 1wt% Ag coated MWCNT / 9wt% Si compact

3.3 DENSITY AND HARDNESS MEASUREMENT

The compacted sample has a very high relative density. Sample compacted using 1wt% silver coated CNT has almost density equal to sample compacted using 4wt% CNT [4]. This reveals that silver coated CNT disperse well in aluminium matrix which reduce porosity and increases the relative density.

www.ijmer.com Vol.2, Issue.3, May-June 2012 pp-1167-1170 ISSN: 2249-6645

The compacted sample gives an hardness of 75 BHN, i.e. sample made using 1wt% of silver coated CNT gives hardness value near to sample compacted using 4wt% of uncoated CNT [4]. This shows the uniform dispersion of silver coated CNT in aluminium matrix. Silver coated CNT gives better hardness because it is distributed more uniformly in the composites leading to dispersion strengthening, and fills up the voids resulting in increase of the relative density of the composites. The uniformly dispersed CNT restrain the growth of aluminium grains during fabrication of the composites bringing on grain refinement strengthening.

4. CONCLUSION

In summary, silver coating is one of the best methods to improve the dispersion of CNT in Al matrix. Using silver coated CNT milling duration can be reduced to $1/4^{th}$ of milling time taken for uncoated CNT. For silver coated CNT 4hrs of milling in planetary ball mill is sufficient for uniform distribution. Porosity is also less observed in composite made using silver coated CNT. Even small weight percentage of silver coated CNT gives better relative density and hardness value. The composite made using 1wt% of silver coated CNT gives relative density and hardness almost equal to composite made using 4wt% of uncoated CNT. This reveals the uniform distribution of silver coated CNT in aluminium matrix. Hardness of aluminium composites made using CNT is significantly higher compared to other aluminium composite compacted at similar conditions. This reveals the strengthening effect of CNT.

REFERENCE

1. Yeng, Hailong Yuan: "Electroless plating of carbon nanotube with silver", *Material Science, 2004, Vol. 39, pp. 3241-3243.*
2. R P´erez-Bustamante, I Estrada-Guel, C D G'omez-Esparza, R Mart´ınez-S´anchez, M Miki-Yoshida: "Microstructural and mechanical characterization of Al – MWCNT composite produced by mechanical milling", *Material Science and Engineering, 2009, Vol. 502(A), pp. 159-163.*
3. H J Choi, G B Kwon, G Y Lee, D H Bae: "Reinforcement with carbon nanotubes in aluminium matrix composites", *Scripta Materialia, 2009, Vol. 59, pp. 360-363.*
4. C F Deng, D Z Wang, X X Zhang, A B Li: "Processing and properties of carbon nanotubes reinforced aluminium composites", *Materials Science and Engineering, 2007, Vol. 444(A), pp. 138-145.*
5. C Deng, Xue Xi Zhang, Dezun Wang, Q Lin, "Preparation and characterization of carbon nanotubes / aluminium matrix composites", *Material Letters, 2007, Vol. 61, PP. 1725-1728.*
6. A M K Esawi, K Morsi, A Sayed, A Abdel Gawad, P Borah: "Fabrication and properties of dispersed CNT – aluminium composites", *Material Science and Engineering, 2009, Vol. 508(A), pp. 167-173.*
7. S R Bakshi, Anup K Keshri, Virendra Singh, Sudipta Seal, Arvind Agrawal: "Interface in CNT reinforced aluminium silicon composites: Thermodynamic analysis and experimental verification", *Journal of Alloy and Compounds, 2009.*