



Thermal conductivity of carbon nanotube–silver composite



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Abstract: The molecular level mixing method was extended to fabricate carbon nanotube reinforced silver composite. The influence of type of carbon nanotubes (single/multiwall) reinforcement and their mode of functionalization (covalent/non-covalent) on thermal conductivity of silver composite was investigated. X-ray diffraction and electron diffraction spectroscopy (EDS) confirm the presence of silver and carbon in the composite powder. High resolution scanning electron microscopy and transmission electron microscopy ascertain embedded, anchored and homogeneously implanted carbon nanotubes in silver matrix. Effect of covalent functionalization on multiwall carbon nanotubes was monitored by Raman and Fourier transform infrared spectroscopy. These investigations confirm the addition of functional groups and structural integrity of carbon nanotubes even after covalent functionalization. Thermal conductivity of composites was measured by a laser flash technique and theoretically analyzed using an effective medium approach. The experimental results reveal that thermal conductivity decreases after incorporation of covalently functionalized multiwall nanotubes and single wall carbon nanotubes. However, non-covalently functionalized multiwall nanotube reinforcement leads to the increase in effective thermal conductivity of the composite and is in agreement with theoretical predictions derived from effective medium theory, in absence of interfacial thermal resistance.

Key words: metal matrix composites; carbon nanotubes; thermal conductivity; functionalization

1 Introduction

Silver is an attractive metal due to its high thermal and electrical conductivity. Ag and Ag-graphite composites are used as interconnect and thermal management material in integrated circuits, plug coat-ers, electric brushes and circuit breakers [1,2]. Carbon nanotubes (CNTs) are considered as versatile filler for fabrications of electrical contact and thermal management materials instead of graphite by virtue of their unique properties. They are characterized by their excellent properties: Real density similar to that of a polymer, with excellent elasticity, high aspect ratio (50–1000) and mechanical strength better than that of steel [3]. Furthermore, extraordinarily low coefficient of thermal expansion (CTE=0) and ultra-high thermal conductivity (3000–6000 W/(m·K)) in combination with the superior electrical and thermal conductivities of the silver [4], CNTs reinforced Ag matrix (CNT/Ag) composites are very attractive to meet the increasing demands for high performance thermal management

materials. CNT based metal matrix composites (MMCs) have been reviewed critically by BAKSHI and LAHIRI [5] to summarize the state-of-the-art of this field. They ascertain that the improvement in thermal properties of CNT based MMCs largely depends on the distribution of CNTs and their bonding with matrix. Hence, mode of functionalization of CNTs and processing route are two vital factors that determine thermal properties of the MMCs. Recently, novel molecular-level mixing method has been proposed for achieving a homogeneous dispersion of CNTs in MMCs [6]. This is due to covalent bonding at CNT/metal interface, as a result of strong adherence between surface-modified CNTs and metal ions at the molecular level.

Multiwall carbon nanotubes (M-CNTs) are cheaper than single wall carbon nanotubes (S-CNTs) and better suited to large scale industrial applications, but thermo-physical properties of S-CNTs are much better than those of M-CNTs due to their high effective specific surface area and aspect ratio. In order to efficiently synthesize CNT-based MMCs, it is necessary to disperse CNTs and to activate their graphene structure. CNTs can be

activated and dispersed either by covalent or non-covalent functionalization techniques. Covalent functionalization improves dispersion and interfacial bonding of CNTs with metal matrix, but at the same time degrades physical properties of CNTs. On the other hand, non-covalent functionalization of CNTs is accomplished by π stacking interactions of surfactants. This strategy does not degrade inherent properties of CNTs [7]. Therefore, the present work exhibits comparative evaluation of influence of CNT reinforcement on the thermal conductivity of Ag composite in reference to 1) S-CNT/M-CNT reinforcement and 2) covalently and non-covalently functionalized multiwall CNTs (C-CNTs/N-CNTs) reinforcement.

2 Experimental

2.1 Fabrication of CNT/Ag nanocomposite powder

Multiwall and single wall CNTs with purity of 90%–98% as per specifications given in Table 1 were purchased from Nanoshell, USA.

Table 1 Parameters of carbon nanotubes used in synthesis process

Parameter	M-CNTs	S-CNTs
Average diameter/nm	4–12	1–2
Length/ μm	15–30	1–10
Purity/%	≥ 90	≥ 98
Specific surface area/($\text{m}^2\cdot\text{g}^{-1}$)	90–350	350–450
Bulk density/($\text{g}\cdot\text{cm}^{-3}$)	0.05–0.017	0.17–0.030
Volume resistivity/($\Omega\cdot\text{cm}$)	0.1–0.15	0.1–0.15

A part of multiwall CNTs were covalently functionalized and rests of them were treated with surfactant, sodium dodecyl sulphate (SDS), known as non-covalent functionalization. In covalent functionalization, M-CNTs were treated with $V(\text{H}_2\text{SO}_4):V(\text{HNO}_3)=3:1$ solution for 2 h and subsequently washed many times with de-ionized water and dried in an oven at 120 °C. In non-covalent strategy, CNTs were sonicated in ethanol in presence of SDS for 2 h. C-CNTs and N-CNTs obtained by respective functionalization process were used in synthesis process. Metal salt was reduced chemically instead of thermal reduction as a modification in molecular level mixing method. The synthesis process is called as modified molecular level mixing method [8]. C-CNTs and N-CNTs were dispersed in 200 mL ethanol by probe sonication. AgNO_3 (Sigma Aldrich, purity 99 %) was taken as metal salt for synthesis of MMCs. 3 g silver nitrate was poured in the dispersed CNT solution with magnetic stirring for 12 h. 2 mL hydrazine hydrate (Merck, purity 99%–100%) was added to this solution as a reducing agent. All chemicals employed in synthesis

process were of analytical reagent grade and used without further purification. The resultant solution was centrifuged and precipitates were washed many times with de-ionized water to remove surfactant completely. Finally, CNT/Ag nanopowder was obtained by drying the precipitates at 50 °C on a hot plate. S-CNT/Ag nanocomposite samples were also prepared by following the same procedure.

2.2 Consolidation of CNT/Ag nanocomposite powder

The nanopowder was compacted by uniaxial molding press at a pressure of 320 MPa. Pellets of dimensions $d13\text{ mm}\times 2\text{ mm}$ of CNT/Ag nanocomposites containing 0, 3%, and 6% CNTs (volume fraction) were prepared. All fabricated nanocomposite samples were sintered in horizontal tube furnace with attached programmable temperature controller. Composite samples were placed in alumina tube by keeping them in silica crucible. Sintering temperature of 800 °C was attained at sintering rate of 5 K/min in an inert atmosphere. Sintering temperature was kept constant for 12 h. Theoretical and measured densities of samples evaluated after sintering are tabulated in Table 2.

Table 2 Relative density of CNT/Ag samples after sintering

Composition	Theoretical density/($\text{g}\cdot\text{cm}^{-3}$)	Measured density/($\text{g}\cdot\text{cm}^{-3}$)	Relative density/%
Ag	10.40	10.20	97.23
N-CNT (3%)/Ag	10.22	9.57	93.63
N-CNT (6%)/Ag	9.96	9.14	91.76
C-CNT(3%)/Ag	10.22	9.73	95.20
C-CNT(6%)/Ag	9.96	9.05	90.86
S-CNT (3%)/Ag	10.22	9.53	93.24
S-CNT (6%)/Ag	9.96	9.02	90.56

2.3 Sample characterization

X-ray diffraction patterns of powdered samples were recorded with a Panalytical 3050/60 Xpert-PRO using $\text{Cu K}\alpha$ radiation. Microstructure of the samples was studied using scanning electron microscope (SEM) FEI Quanta FEG 450 and transmission electron microscope (TEM) JEOL JEM 2100F operated at 200 kV. FTIR spectra were recorded in IR region of 1000–4000 cm^{-1} by Perkin Elmer SP-65 and Raman spectra were observed in the range of 1000–1800 cm^{-1} by Reins haw In via Raman microscope. Thermal conductivity was measured by using the following formula:

$$K_e = \alpha \rho c_p \quad (1)$$

where K_e is the effective thermal conductivity, α is the thermal diffusivity, c_p is the specific heat and ρ is the density of consolidated samples obtained at room temperature. The thermal diffusivity was measured using

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