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CNT Reinforced Silver Nanocomposites: Effect of Sintering on their Mechanical Behavior

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Abstract

In the present study, two important mechanical properties namely hardness and elastic modulus of nanocomposite are tailored by reinforcing CNTs into silver matrix using physical mixing method. Microstructure studies revealed that nanotubes are homogenously dispersed into silver matrix. Fabricated samples are then subjected to sintering for 12 hrs at 800ºC. Effect of sintering on mechanical properties of CNT based crystalline nano-silver composite is analyzed by measuring their hardness and elastic modulus before & after sintering. Experimental results indicate that both mechanical properties of the nanocomposite increased appreciably upon sintering.

Keyword: Metal matrix composites, Sintering, Hardness, Elastic modulus

INTRODUCTION

Metallic nanoparticles have attracted the interest of the researchers in the last few years. Nanoparticles possess far superior properties than their bulk counterparts. Silver is an important noble metal due to its excellent thermal & electrical conductivities. However, in pure state, it is a soft metal which restricts its use as an interconnect in high end electronic materials. Silver-graphite composites are generally used as interconnect in ultra large scale integrated circuits (ULSI), circuit breakers & electric brushes [1-3]. Carbon nanotubes (CNTs) possess elastic modulus as high as 1

TPa and hardness as high as 150 GPa [4-5]. Their current carrying capacity can go up to 10^9 A/cm^2. These extraordinary properties can make them ideal reinforcements to improve the properties of silver matrix. Most of the work done in this regard has been done on Cu and Al matrices [6-8]. Some reports are also available on CNT/Ag composites. Recently, Dausch et. al. have reported improvement in hardness and elongation properties upto 7.5 volume percentage of MWCNTs [9]. Feng et. al. have reported improvement in hardness, electrical resistivity and density of MWCNT reinforced silver composites up to 8 volume percentage of CNTs [10]. But, poor dispersion of nanotubes into metallic matrices remains a challenge. Nanotubes have agglomeration tendencies due to van der Walls interactions between individual tubes. Recently, Ultrasonication has succeeded in overcoming these challenges [11]. Two major types of CNTs are known: Single wall CNTs and Multi wall CNTs. SWCNTs are basically a graphene sheet rolled into a seamless cylinder whereas MWCNTs can be considered as a collection of concentric SWCNTs with increasing diameters arranged in a nested fashion. Although, SWCNTs are relatively expansive but they possess superior thermo-physical properties such as higher aspect ratio & larger specific surface area than MWCNTs. Most of the studies involving nanotubes have utilized MWCNTs due to their economic viability. It is also understood that sintering of materials tends to influence their mechanical properties.

In the present work, we have reinforced silver matrices with both type of CNTs (single wall as well as multi wall) using physical mixing method. Mechanical properties namely hardness and elastic modulus of the composite has been measured before and after sintering to elucidate the effect of sintering.

EXPERIMENTAL DETAILS:

Fabrication of CNT/Ag nanopowder:

Carbon Nanotube (0, 1.5, 3.0 & 4.5 Volume %) reinforced silver nanocomposites were fabricated using modified physical mixing method [12]. Functionalized nanotubes (SWCNTs & MWCNTs) & silver nanopowder were purchased from Nanoshel, USA. Weighed amounts of as received CNTs were soaked in 100 ml ethanol & sonicated in a probe sonicator for 2hrs. Then required amounts of silver nanopowder were added to the sonicated solution & simultaneous magnetic stirring & sonication was carried out further for 2 hrs. All the chemicals employed in the fabrication process were of analytical grade & were used without further purification. The mixture was then dried at 50oC on a hot plate to obtain the CNT/Ag nanopowder. Both MWCNT/Ag and SWCNT/Ag nanocomposites were prepared separately using this method.

Consolidation of CNT/Ag nanopowder:

Samples were compacted at a pressure of 320 MPa using a hydraulic press into pallets of size 13mm x 2mm. sintering of the pallets was carried out in a horizontal tube furnace equipped with a temperature controller at 800^{o}C for 12hrs. XRD patterns of samples were recorded on a panalytical 3050/60 Xpert- PRO using a Cu Kα radiation. Morphology of the samples was analyzed using a scanning electron microscope (SEM) FEI Quanta FEG 450 operated at 15 KV. Hardness & modulus of elasticity of the samples were measured using a CETR- Apex nanoindenter equipped with a berkovich diamond tip.

RESULTS AND DISCUSSION

Microstructure Characterization:

Structural characterization of the fabricated CNT/Ag samples has been carried out using X-Ray Diffraction, Electron Dispersive Spectroscopy and Scanning Electron Microscopy. XRD patterns of CNT/Ag nanocomposite samples have been displayed in figure 1.

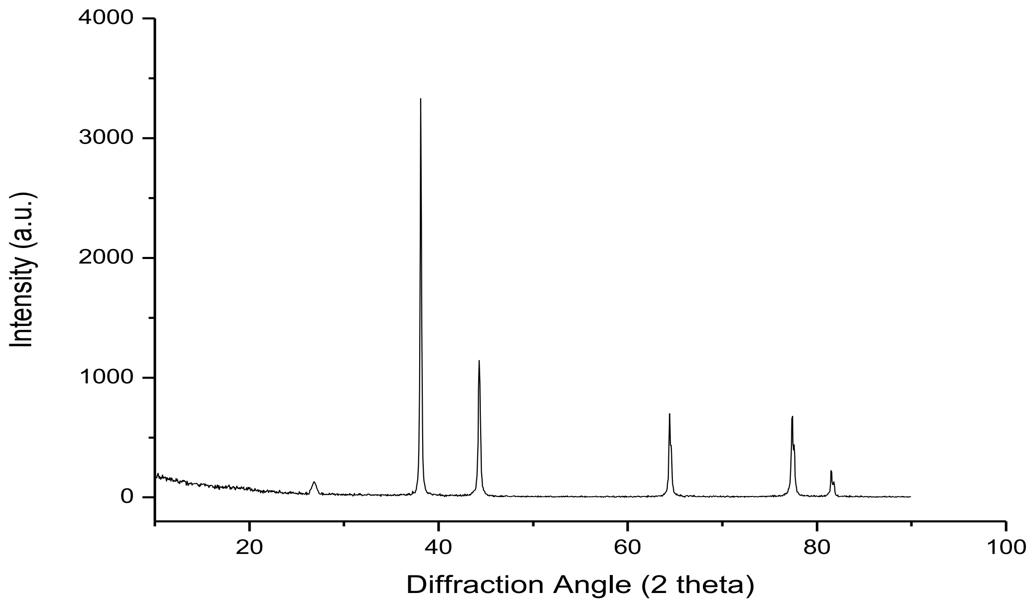


Figure 1. XRD pattern of CNT/Ag nanocomposite

Crystalline nature of the samples is indicated by peaks at 2θ values of 38.9°, 45.1°, 65.1° & 78.2° which correspond to (111), (200), (220) & (311) reflections of FCC phase of silver. A slight hump at 2θ values between 20-30° indicate the presence of CNT's in the sample. Elemental composition of the CNT/Ag nanocomposite samples has been determined using electron dispersive spectroscopy. Figure 2 confirms the presence of CNTs in the sample. Binding energies of silver correspond to peaks at 3.0, 3.2 and 3.4 KeV whereas binding energy of carbon corresponds to peak at 0.25 KeV.

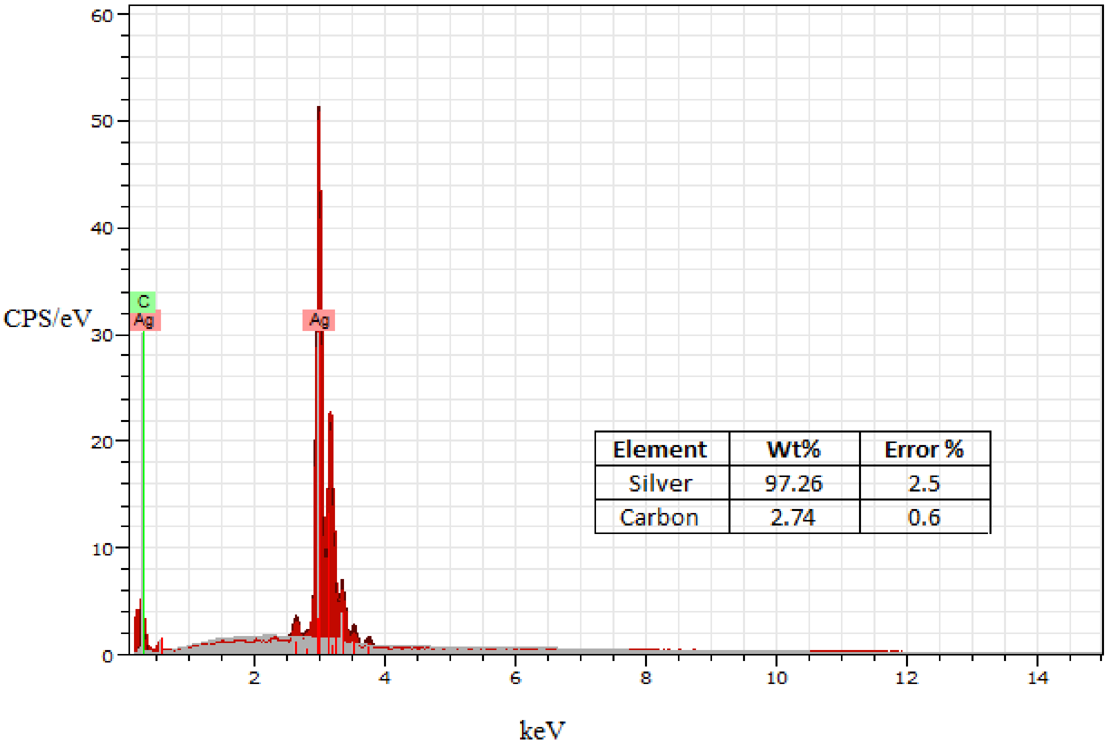
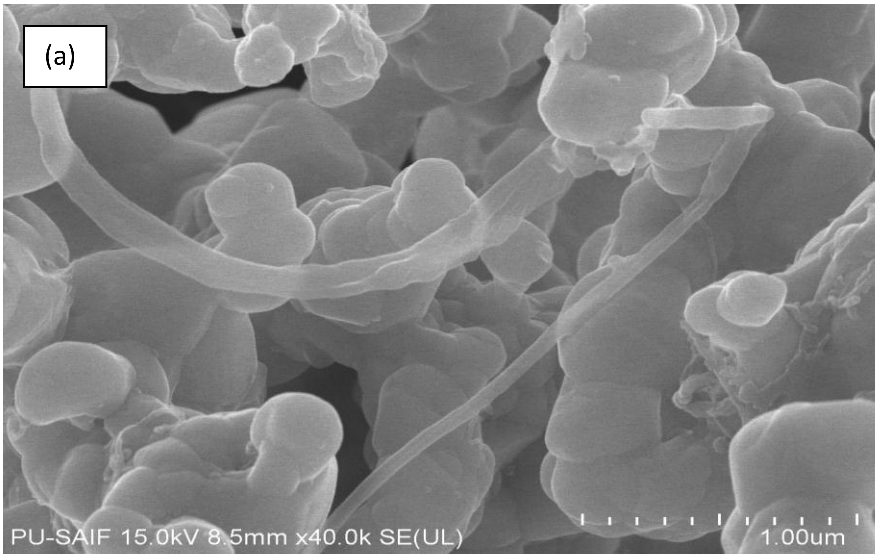
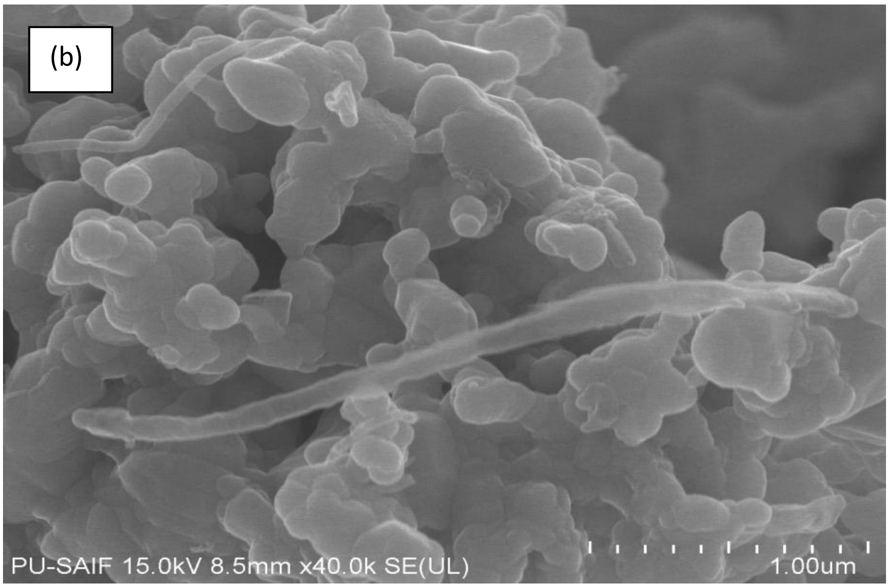


Figure 2. EDS characteristic profile of CNT/Ag Nanocomposite

Morphological studies of the synthesized CNT/Ag composite samples have been carried out using scanning electron microscopy. SEM images of the samples are displayed in figure 3 a-c.





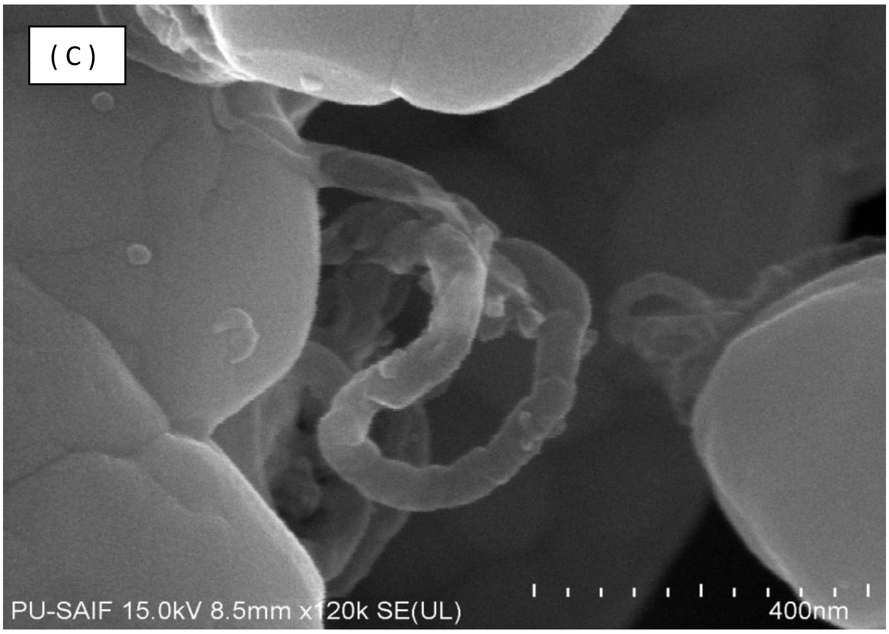


Figure 3. SEM micrographs of (a) CNT (1.5 vol. %) /Ag, (b) CNT (3 vol. %) /Ag and (c) CNT (4.5 vol. %) /Ag

SEM micrographs disclose that CNTs are embedded, anchored and uniformly distributed into the silver matrix. Physical mixing method has managed to achieve debundling of nanotubes to a large extent. However, some agglomerates can be seen in sample reinforced with 4.5 Volume % of carbon nanotubes.

3.2 Evaluation of Mechaical Properties : Both mechanical properties namely hardness and elastic modulus of the samples has been measured using nanoindentation. Experiment was performed under constant displacement mode. Effect of heterogenioty of the sample was twarted by taking average of five observations. Hardness and elastic modulus of CNT/Ag composite samples are measured against CNT (single wall as well as multi wall) concentarion. Results of nanoindetation experiment performed before sintering are displayed in figure 4. Both mechanical parameters increase with increase in CNT content in the matrix which can be attributed to higher stiffness and strength of the CNTs as compared to the silver matrix. SEM images reveal that CNTs are bridged between silver particles. This brdging helps in creating a strong interfacial adhestion between CNTs and silver matrix which is cruicial in transfererence of load from matrix to nanotubes. Increase is more pronounced in case of SWCNT/Ag nanocomposites. This is expected due to superior aspect ratio and specific surface area of SWCNTs as compared to MWCNTs. These superior properties helps in creating a larger and stronger interfacial adhesion between silver and single wall nanotubes. Hardness and elastic modulus increase from .63 GPa and 52.5 GPa to 1.29 GPa and 77 GPa at 4.5 volume of SWCNTs. Compacted nano silver is processed into bulk material by sintering it for 12hrs at 800^{o}C under inert atmosphere. Both hardness and elastic modulus are then again measured after sintering.

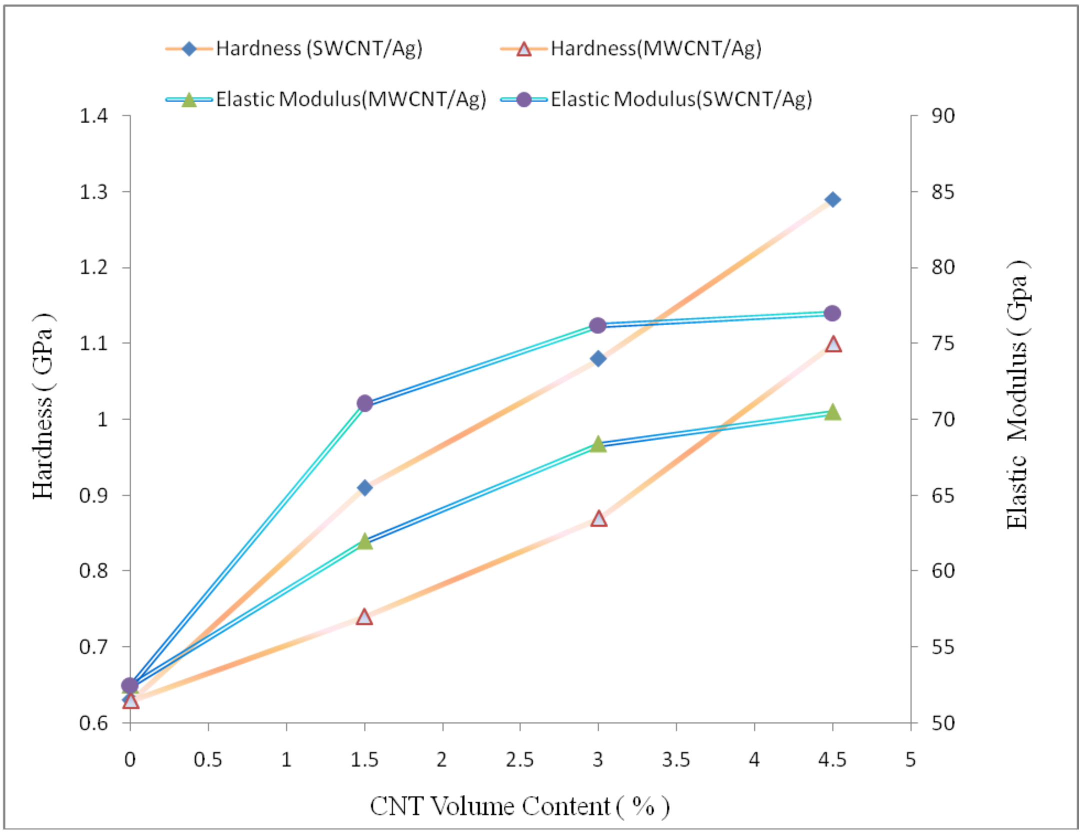


Figure 4. Variation of Hardness and Elastic Modulus of CNT/Ag nanocomposite with variation in CNT volume Content before sintering.

Figure 5 displays the nanoindentation data of hardness and elastic modulus measured after sintering. It can be seen from the figure that both mechaical parameters improve upon sintering. Hardness increased from 0.63-1.3 GPa to 1.05-1.75 GPa upon sintering whereas elastic modulus increased from 52-77 GPa to 78-97 GPa. Both mechanical parametrs are improved by 25-35% on sintering. Sintering brings the silver particles closer to each other. This coalescence of silver particles reduces the porosity of sample [13]. Reduction in porosity along with residual stress removal is responsible for improvement in hardness and elastic modulus of the CNT/Ag composite upon sintering.

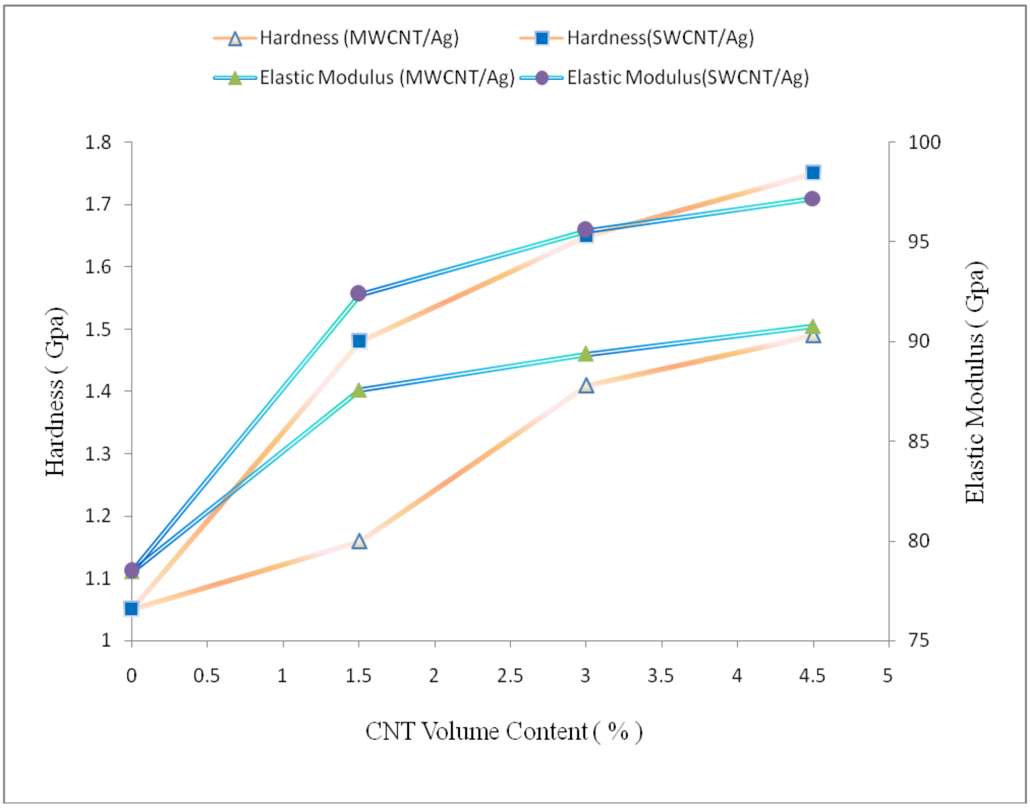


Figure 5. Variation of Hardness and Elastic Modulus of CNT/Ag nanocomposite with variation in CNT volume Content after sintering.

CONCLUSIONS

CNT (single wall as well as multiwall) reinforced silver nanocomposites were fabricated using physical mixing method. Structural studies revealed that CNTs are uniformly distributed into silver matrix. Effect of sintering on mechanical and electrical properties was evaluated. Both mechanical properties viz. hardness and elastic modulus improved by 25-35 % upon sintering. These improvements in mechanical properties could be attributed to increase in density of the samples upon sintering.

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