Electromagnetic Containerless Processing of Single-walled Nanotube Reinforced Metal-Matrix Composites

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ABSTRACT

Single-walled carbon nanotube (SWNT) reinforced copper (Cu) matrix composites were produced by powder metallurgy (PM) and electromagnetic levitation methods. The nanotubes were first decorated with a nickel coating and then dispersed by ball milling. The optimization of factors such as cold pressing pressure, powder grain size and sintering temperature for full densification were essential in the preform stage of PM in order to produce a sample suitable for levitation. Scanning electron microscopy (SEM) observations have shown that the well-dispersed nanotubes in the composites are not damaged during the high-temperature composite preparation. The preforms were then melted by electromagnetic levitation in an argon environment. SEM observations showed that the nanotubes survived the high-temperature melting. Hardness testing demonstrated that the strength of the composites is increased with increasing volume fraction of SWNTs.

1 INTRODUCTION

Since Iijima's discovery of carbon nanotubes [1], these structures have been recognized as fascinating materials with nanometer dimensions promising exciting new areas of application. It has been demonstrated by both theoretical calculations and experimental measurements that due to their unique structure individual SWNTs and SWNT ropes display remarkable electrical, thermal, and mechanical properties [2]. An ideal-single walled carbon nanotube consists of a rolled graphene layer with a cylindrical hexagonal lattice structure capped by half a fullerene molecule at both ends. Treacy et al. [3] estimated the Young's modulus of individual multi-walled nanaotubes to be in the range of 1 TPa. A number of theoretical studies have suggested that the smallest single-walled nanotubes might have a Young's modulus as high as 5 TPa [4]. Those features combined with high aspect ratios on the order of 1000 or more makes the SWNTs excellent additives for composites [5-8].

2 EXPERIMENTAL PROCEDURE

Cu is widely used in aerospace sciences and numerous industries due to its high electrical conductivity and corrosion resistance. A size range (5-40µm) and spherical morphology has been selected to achieve the best dispersion and flowability of powders during powder metallurgy. The purified single walled carbon nanotubes were supplied by Carbon Nanotechnology, Inc. SWNTs were produced by a High-Pressure CO (HiPCO) process and fabricated into millimeter-sized BuckyPearl pellets. This commercial material contains about 13 wt% Fe catalyst. The pelletized SWNTs were grounded to a finer level. The Fe catalyst was further reduced by refluxing the SWNTs in HCl at 80 °C for 5 hours, and then washed with methanol. These purified SWNTs were nickel coated via the electroless plating method [9-12] and then a mixture of copper powder (5 µm) along with 0-1 wt% of these tubes was mixed by 10 min sonication, followed by 10 hours of ball milling [13,14]. No damage of the SWNTs was observed after the ball milling process by SEM.

The result was a dark powder of uniform dispersion. This well-mixed final sample was slowly cold pressed under a pressure of roughly 100 MPa for 10 minutes [15]. The resulting sample preform was a cylinder 0.25 inches in diameter and 0.25 inches in height weighing roughly 0.75 grams. The samples were placed in a hydrogen furnace for 5 hours at a temperature of 400^0C to help remove any unwanted oxidation that could have formed before or during the cold pressing process. The samples were then sintered in an argon atmosphere at 800^0C for 15 hours [15-17].

SEM was used to analyze the microstructure following the sintering process. After that, the samples were then ready to be levitated and melted using a Radyne EI-40 model radio frequency generator. This generator produces 400-600 A of current at a single frequency of roughly 400 kHz. The large current and frequency create an electromagnetic field strong enough to levitate the copper sample while simultaneously inducing a heating eddy current sufficient enough to melt metals [18]. The samples were melted and mixed in the levitation process in an argon atmosphere for roughly 30 seconds. The surface was then cracked and SEM was again applied to analyze the fractured surface microstructure.

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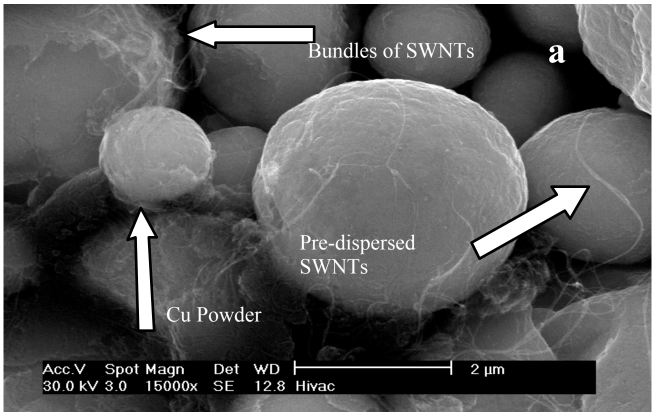
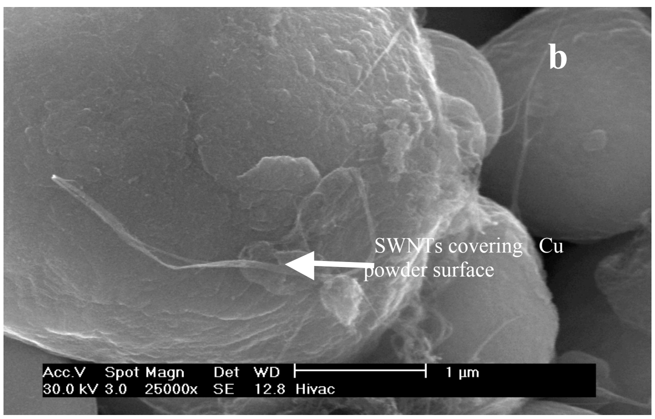
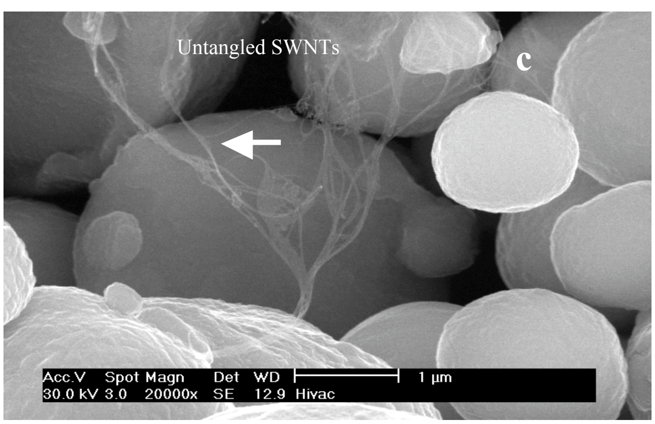
3 RESULTS AND DISCUSSION







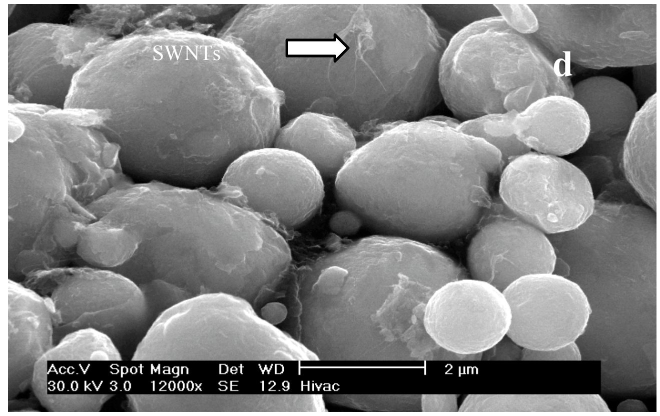


Figure 1(a-d). SEM image of SWNT/Cu particles after ball milling.

The SEM pictures in Fig.1 illustrate the microstructure of the premixed Cu/SWNT sample. It shows that the SWNTs were successfully pre-dispersed among the Cu particles (Fig. 1a) and some SWNTs were already untangled from bundles and attached on the particles surface after 10 hrs ball milling (Fig. 1b-c) without any surface modification or damage. However, some SWNTs were still entangled to each other forming a thin sheet covering the Cu particles surface (Fig. 1d).

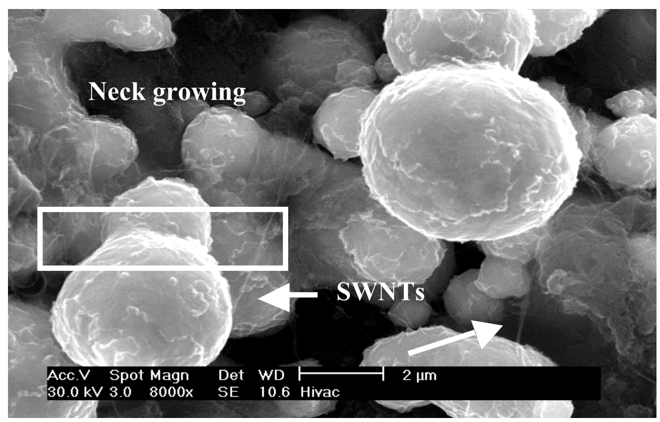
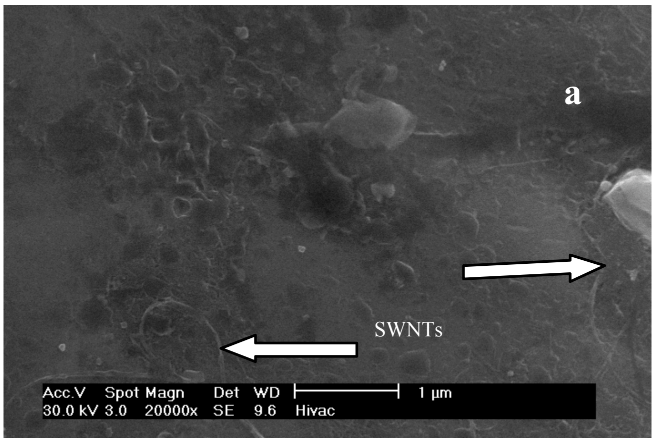


Figure 2. SEM image of fractured surface of the SWNTs/Cu after PM process and 800 °C sintering treatment.

Figure 2 shows the microstructure of the composite sample after hydrogen treatment and high-temperature sintering. It is observed by SEM that there is neck-like growth between particles; therefore the distance between copper particle centers decreases. The principal driving force for the shrinkage is surface tension which causes transport of material from surfaces with a small radius of curvature to those with a larger radius of curvature. These forces also cause a decrease in the total surface of the pores and thereby shrinkage of the compact preform. The SWNTs in the SEM image of the composite samples sintered at 800°C are similar to those of the premixed material as shown in Fig. 1. No significant change either in morphology or in microtexture is observed.

Figure 3(a) shows the surface microstructure of the SWNT reinforced copper composite sample after the levitation process. It is noticed that there are SWNTs which still survive even after the high temperature electromagnetic processing. It is observed by SEM that some SWNTs appear on the melted copper composite surface (Fig. 3a). Pulling-out and bridging occurs on the fractographs (Fig. 3b), and the fractures propagate along the direction of the initial cracks. Therefore, by SEM observation, the main fracture mechanism in regard to the SWNTs is pulling-out of the fibers.



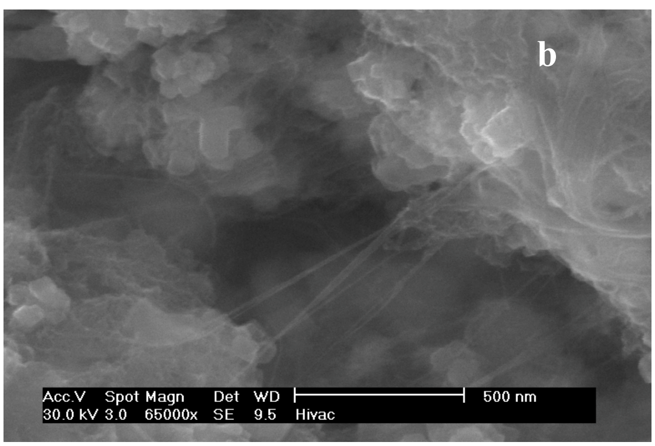


Figure 3. SEM image of SWNT reinforced copper composites (a) the surface of composite microstructure; and (b) the fracture surface of the composite.

Electromagnetic levitation has been discussed in the literature [18-22] in theory and in practice, and for that reason has not been included in this paper. Vickers hardness was used for comparison with [14]. The hardness results of the current study can be seen in Figure 4.

These indicate that increasing hardness is achieved with an increase in percentage of SWNTs. While in other methods adding more SWNTs might lead to an even greater increase in hardness, the electromagnetic levitation process is the factor which dictates how much SWNT reinforcement can be added in the current study. Poor levitation and heating was a result of increasing the SWNT content above 2% wt. The reason is that the nickel-coated nanotubes, because of their surface area to volume ratio, tend to have a negative effect on the internal heat generation and levitating force in the copper. Since nickel is not as electrically conductive as copper, it would follow that the addition of more SWNTs, covered in nickel, would impede the overall electrical properties of the composite. This lowered electrical conductivity causes a significant decrease in the heat generation in the levitation process. It is for this reason that the focus of the present research was on compositions of 1%wt SWNTs and below. Fig. 3 (a) also shows that the Cu powder was completely melted into a relatively smooth surface indicating the SWNTs seen in the figure survived at least the melting point of copper (~1083^{0}C) [23].

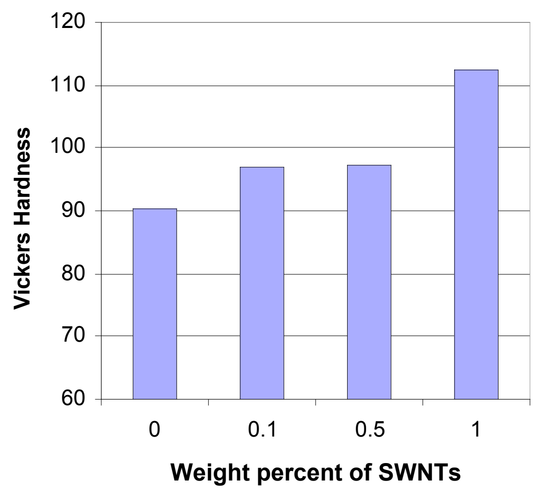


Figure 4. Vickers Hardness vs. Weight percent of SWNTs in the Cu/SWNT matrix showing a 24.6% increase in hardness with the addition of 1 wt% of SWNTs.

4 CONCLUSIONS

The powder metallurgy process along with electroless plating was used to premix nanotubes and copper powder. The powder was then cold pressed, hydrogen treated, sintered, and finally electromagnetically levitated. The result was a copper/SWNT metal-matrix composite with improved hardness. This process is usually capable of producing samples approximately 0.5 – 0.75 grams in mass, based on the frequency used for levitation.

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