

## Improvement of interface and mechanical properties in carbon nanotube reinforced Cu–Cr matrix composites

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### ARTICLE INFO

#### Article history:

Received 27 March 2012

Accepted 14 September 2012

Available online 26 September 2012

#### Keywords:

Carbon nanotubes

Metal–matrix composites

Interface

Mechanical properties

### ABSTRACT

An effective approach is utilized to establish a strong interface between the carbon nanotube (CNT) and the Cu matrix by introducing the matrix-alloying chromium (Cr) element. The interface microstructure and mechanical properties of the composites are characterized by high-resolution transmission electron microscopy (HRTEM), hardness and tensile tests, respectively. Compared to CNT/Cu composites, CNT/Cr composites show evenly improved interfacial bonding and enhanced mechanical properties, which is ascribed to the formation of thin intermediate Cr<sub>3</sub>C<sub>2</sub> transition layer between CNTs and Cu–Cr matrix.

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### 1. Introduction

Carbon nanotubes (CNTs) have superior mechanical properties with a tensile strength up to 150 GPa and an elastic modulus up to 1 TPa, as well as excellent thermal conductivity and electrical conductivity [1]. Therefore, they can be used as an excellent nanofiller to improve mechanical, electrical, thermal properties of the composites unattainable from current materials [2]. Most of investigations focus on the application of CNT in polymer composites because of the flexibility and compatibility of CNT and most polymer matrix in the composite fabrication techniques [3,4]. Only limited studies, however, have been considered CNT as reinforcement in the metal matrix composites (MMCs) [5,6], largely because of harsh fabrication conditions where the stability of the CNTs during the processing is not well maintained [7]. Furthermore, given the excellent thermal conductivity of CNT and high thermal conductivity of the metal materials, CNT/metal composites are very attractive to meet the increasing demands for high performance thermal management materials, instead of traditional SiC/Al(Cu) [8,9] composites or as a potential competitor to up-coming diamond/metal composites [10,11].

In the development of high performance of CNT/metal composites, two main issues have to be faced to improve effectively the material properties of the composites by adding CNTs as fillers. One is the proper dispersion of the individual CNTs in the matrix and the other is the effective interfacial bonding between CNTs and the matrix [12]. To solve the CNT dispersion problem, the

use of ball milling as a mechanical dispersion technique receives the most attention and has been proven to be a promising technique for dispersing CNTs in the metal matrix [13,14]. Some other new methods are also emerging in endlessly [15,16], which have been extensively investigated over the past 5 years [7,17]. Comparably, the researches on the methods to solve the interface problem of CNT/metal composites receive less attention. In typical case of CNT/Cu composites, it is known there is a very weak bonding between CNT and pure copper matrix since copper is known to be naturally non-wetting with CNT [10,18]. Attempt has been made to solve such interface problem through using metal-coatings on nanotubes, which can reduce the interface energy between copper matrix and CNTs and, in turn, will improve the load transfer ability of CNT/Cu composites. Recently, CNTs were decorated with metal coatings, such as Ni [19], Cu [18] and Mo [20] to improve the interfacial bonding of CNT/Cu composites showing enhanced mechanical properties. Nevertheless, the coating layer is easily damaged during the subsequent densification processing, which can weaken and even destroy the integration between CNTs and metal matrix and cause a significant degradation in the properties of the composites. In addition, the fabrication process of coating method always involves complicated processing steps and the costs are therefore usually high.

In CNT/Al composites, aluminum carbide (Al<sub>4</sub>C<sub>3</sub>) is a common compound formed during fabrication of the composites because of a relatively low free energy of formation [21]. It has been confirmed that the strong interfacial bonding in CNT/Al composites is due mainly to the existence of transition thin layer of Al<sub>4</sub>C<sub>3</sub> between CNTs and Al matrix [21,22]. Accordingly, a matrix-alloying approach, by the addition of matrix-alloying elements to ensure

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a certain reaction (i.e. carbide formation) at the interface, may be an effective way to greatly improve the interfacial bonding of CNT and Cu, since alloying of copper with a strong carbide forming element to promote interface wetting and bonding has been reported in other Cu–carbon materials [23,24]. Therefore, it is very necessary to evaluate the effectiveness of matrix-alloying method for the interface modification of CNT/Cu composites.

However, up to now, there is a large number of works dealing with the CNT/Cu composites by coating method, few efforts have been made to investigate the CNT/Cu composites by the use of matrix-alloy method. In the present study, the effect of the chromium applied as a matrix-alloying element is evaluated on the improvement of interfacial bonding and enhancement in mechanical–electrical properties.

## 2. Experimental details

Multi-walls CNTs (~10 nm diameter and 5–10  $\mu\text{m}$  length) and gas atomized Cu–0.76 wt.% Cr alloying powder (99% pure, 8–20  $\mu\text{m}$ ) were used as starting materials for preparing composites. Note that the selected chromium content corresponds to approximately optimum alloying content required for a strong matrix/carbon bonding in Cu/carbon system [25].

In order to obtain a high dispersion of CNTs in matrix powder, the CNTs with 5, 10 and 15 vol.% were mixed with Cu–Cr powders by a high-energy ball-milling process. Mixed powders were each placed in a steel vial and milled by a SPEX mixer under an argon atmosphere in a rotary speed of 1200 rpm for 120 min [5,26]. A ball-to-powder weight ratio of 10:1 was used, and alcohol was added as a process control agent. As-milled powders were first compacted to a green density of 75% of theoretical and then consolidated using the hot pressing. The mixed powders were put into a cylindrical graphite die with an inner diameter of 30 mm. The compact powders were sintered at 750  $^{\circ}\text{C}$  for 15 min. The heating rate was 50  $^{\circ}\text{C}/\text{min}$  and a pressure of 40 MPa was applied from the start to the end of the sintering. After sintering, the surfaces of samples were ground to remove the graphite layer. For comparison purposes, the sintered pure Cu and Cu–Cr specimens and the CNT/Cu composites without Cr addition were also fabricated under the same processing.

The microstructure of the composites was characterized by a field-emission scanning electron micrograph (SEM) and high-resolution transmission electron microscopy (HRTEM). The hardness test (ASTM E384) was measured using a Vickers tester at a load of 10 N with a holding time of 15 s. Tensile test (ASTM D638) was performed using a REGER-3010 apparatus under a crosshead speed of 0.5 mm/min at room temperature. The test sample was a dog-bone shape with a gage length of 20 mm and width of 5.5 mm. Tensile stress–strain curves for the reference and composite materials were obtained, and yield strength values were measured from the curves.

## 3. Results and discussion

### 3.1. Microstructure

Fig. 1 shows the typical morphology of mixed powders with various CNT contents. Fig. 1a illustrates that the fabricated composite powders with 10 vol.% CNTs give homogeneously dispersed CNTs and most of CNTs are tightly embedded into the Cu matrix. Nearly no CNT agglomerates can be found. This indicates that the ball-milling (BM) process applied in the present work is effective to achieve the uniform mixture of Cu powders and CNTs. In general, two competing processes are involved in a typical BM process: one is cold working of the powders that would lead to fracturing of

the particles; and the other is cold-welding of particles that tends to result in an increase of particle size [22]. Once powders are ball milled, cold-welding becomes the predominant process resulting in the agglomeration of the particles with increasing ball-milling time. Nevertheless, the addition of CNTs can maintain the equilibrium between fracturing and cold-welding of Cu particles even at the early stage of the ball milling, since the CNTs can play a role as a kind of grinding aid during the BM process by forming a network of weak interfaces between the CNT and the Cu matrix due to small size and tortuous structure of CNT [13]. This weak-interface network caused by the CNTs can act as the crack initiators to prevent agglomeration of mixture powders. Meanwhile, the aspect ratio of carbon nanotubes decreases during the BM process [27]. These could weaken van der Waals forces between nanotubes resulting in the improved dispersion of CNTs and copper powders. The uniformly dispersed CNTs in the copper powders will then ensure improved densification of the composites.

Fig. 2 shows the fracture surfaces of 10 vol.% and 15 vol.% CNT/Cu–Cr composites after the tensile test. It is obvious in Fig. 2a that the fracture surfaces of composites with 10 vol.% CNTs show a lot of dimples associated with ductile fracture. The appearance of these dimples means that the good dispersion of CNTs does not evenly impair the ductile behavior of copper matrix. However, the addition of larger amount of CNTs seems to make the dispersion of CNTs more difficult, as indicated in Fig. 2b for 15 vol.% CNT/Cu–Cr composites, in which the CNT clusters are clearly visible in some regions. The CNT clusters will have adverse effects on the microstructure and final properties of the composite products [7], as shown in the fracture surface of 15 vol.% CNT/Cu–Cr composites, in which the CNT clusters cause the significant degradation in the densification and ductile behavior of the composites.

HRTEM is used to better understanding the interfacial products of CNT/Cu–Cr composites, as shown in Fig. 3. HRTEM image shows the existence of a transition interfacial layer with the thickness of ~4 nm between CNTs and matrix, which is closely attached to the CNT surface. Careful inspection (Fig. 3 inset) indicates the interfacial layer zone displaying the lattice fringes of the  $\text{Cr}_3\text{C}_2$ -(2 1 1) face with a spacing of 0.23 nm, suggesting that the interfacial phase is  $\text{Cr}_3\text{C}_2$ . The carbides are most likely resulted from the reaction between the alloying element Cr in the Cu–Cr matrix and the amorphous carbon on the CNT surface. In general, carbide formation is restricted at the CNT/metal interface due to the absence of a prism plane in CNTs [21]. It is known that the BM process would cause some damage to the outer surface and tips of the CNTs because of repeated deformation, cold welding and fracturing processes of the mixed powders [14]. These damaged CNTs have broken carbon bonds in the prism planes of outer surface and tips, which would facilitate the formation of carbides in the presence of strong carbide forming element. In addition, the carbide formation is also feasible due to the defects present in the as-received CNTs [22]. Hence, the presence of the defects on the prismatic planes in the CNTs will serve as sites for the production of  $\text{Cr}_3\text{C}_2$  from the chemical reaction between C from the CNT and Cr from the Cu–Cr matrix.

The wetting systems in CNT/Cu and CNT/Cu–Cr composites are schematically shown in Fig. 4. In Cu–carbon system (Fig. 4a), an initial wetting angle of 145 $^{\circ}$  at 1150  $^{\circ}\text{C}$ , measured by the sessile drop method, has been reported by Standing and Nicholas [28]. This means that Cu–carbon is a typical non-reactive metal–graphite system in terms of Young–Dupre relation [29], which shows that the low wetting angle will lead to a good wettability of two different materials. In this case, the adhesion between the copper and carbon is attributed to be no more than a van der Waals force, implying the weak interfacial bonding of CNT and copper. However, as shown in Fig. 4b, the contact angle for Cu–Cr alloy on carbon substrate decreases to 45 $^{\circ}$  at 1150  $^{\circ}\text{C}$  in the presence of

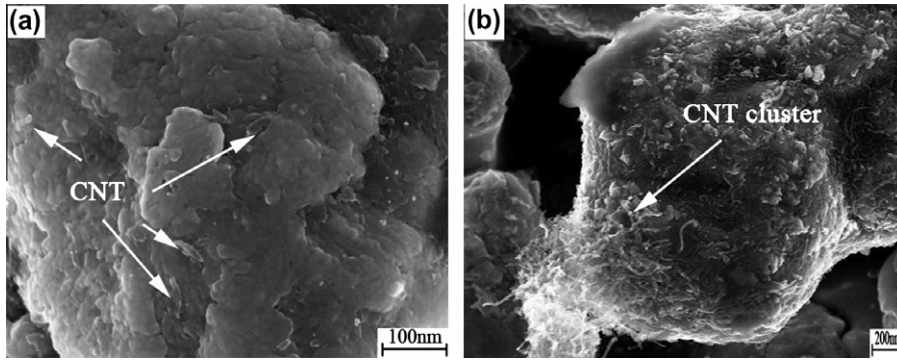


Fig. 1. SEM images of morphology of ball-milled (a) 10 vol.% and (b) 15 vol.% CNT/Cu–Cr powders.

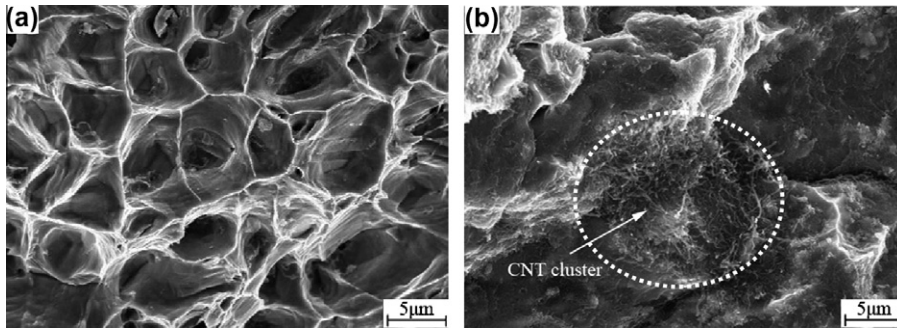


Fig. 2. Fracture surface morphologies of (a) sintered 10 vol.% CNT/Cu–Cr composite and (b) 15 vol.% CNT/Cu–Cr composite.

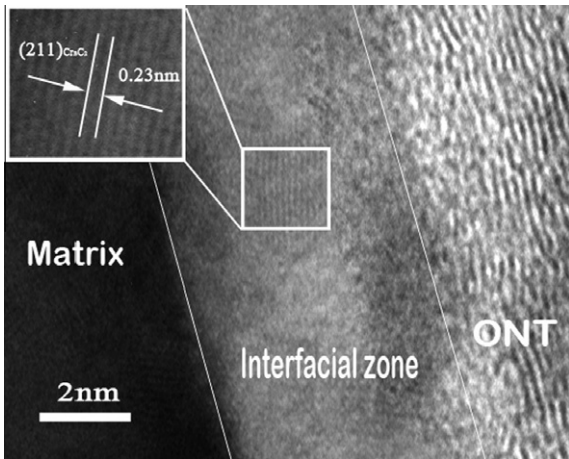


Fig. 3. HRTEM image of CNT/Cu–Cr interface (inset for the lattice fringes of  $\text{Cr}_3\text{C}_2$ ).

interfacial reaction product  $\text{Cr}_3\text{C}_2$  [25]. The low value of contact angle in the case of Cu–Cr/carbon system is attributed to the formation of interfacial carbides, which is more wetting to the carbon

substrate. Accordingly, it is concluded that the formation of interfacial  $\text{Cr}_3\text{C}_2$  transition layer is responsible for the improved wettability and interfacial bonding between the Cu matrix and CNT reinforcement.

### 3.2. Mechanical property

The hardness and yield strength in both CNT/Cu–Cr and CNT/Cu composites with different CNT contents are presented in Figs. 5 and 6. As seen, comparing with copper matrix, the hardness increases from 68 Hv to 135 Hv and the tensile strength also increases from 168 MPa to 296 MPa in 10 vol.% CNT/Cu composites. This is due basically to the fact that CNTs have a higher stiffness and strength than the copper matrix, and the CNT enhancement role is played by the homogeneous dispersion of CNTs in Cu matrix as seen in Fig. 2a. The increased strength also indicates that CNT/Cu composites are successfully fabricated by the present ball-milling process, and followed hot-press processing. The decrease in hardness and yield strength of 15 vol.% CNT/Cu composite is ascribed to the presence of CNT clusters, as shown in Fig. 2b, which can act as the points of stress concentration and result in low hardness and yield strength.

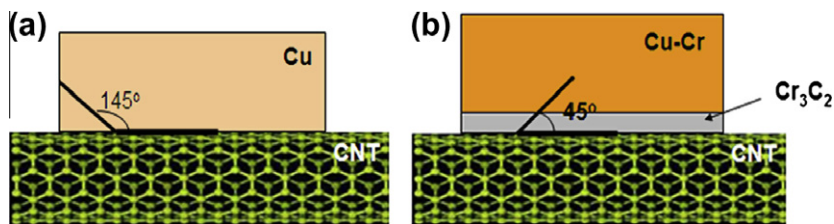


Fig. 4. Schematic illustration of the wetting systems in CNT/Cu (a) and CNT/Cu–Cr (b) composites.

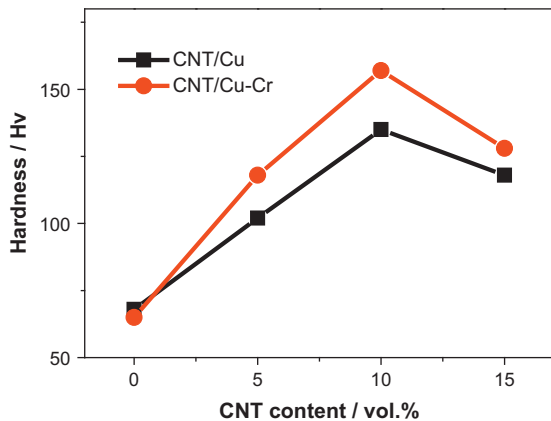


Fig. 5. Hardness of CNT/Cu–Cr and CNT/Cu composites versus CNT content.

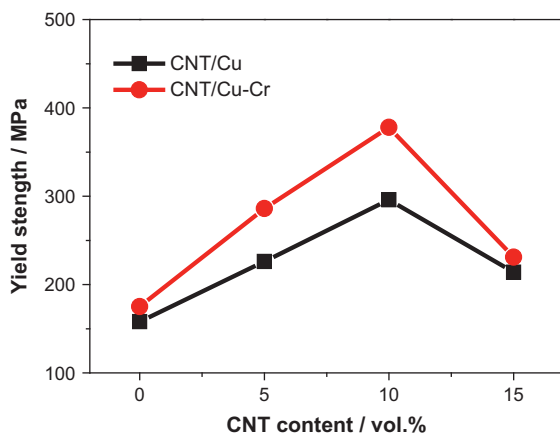


Fig. 6. Yield strength of CNT/Cu–Cr and CNT/Cu composites versus CNT content.

In addition, CNT/Cu–Cr composites display even higher hardness and yield strength than CNT/Cu composites at the same loading of CNTs. With 10 vol.% CNTs incorporated in Cr–Cu matrix, the hardness and yield strength increase to 157 Hv and 388 MPa, a increment of 128% and 135% compared to those of CNT/Cu composites, respectively. This is mainly owing to the different interface phenomena of the CNT/Cu and CNT/Cu–Cr composites. Coleman et al. [4] have proposed a model for the strength of CNT composite ( $\sigma_c$ ) related to the interfacial region, which has the following equation:

$$\sigma_c = \sigma_m + V_f \left( 1 + \frac{2b}{d} \right) \left[ \sigma_s \frac{l}{d} - \left( 1 + \frac{2b}{d} \right) \sigma_m \right] \quad (1)$$

where  $b$  is the thickness of interfacial region,  $d$  the diameter of CNT,  $l$  the length of CNT,  $\sigma_m$  the yield strength of matrix,  $\sigma_s$  the shear strength of interface and  $V_f$  the volume fraction of CNT in the composites. In the present work,  $b = 4$  nm (Fig. 3),  $d = 10$  nm,  $l = 1$   $\mu$ m (note that CNT is greatly shortened during the BM process),  $\sigma_m$  values are 168 MPa and 175 MPa for CNT/Cu and CNT/Cu–Cr composites, respectively. Given these materials parameters,  $\sigma_s$  can then be back-calculated as 10.2 MPa and 14.8 MPa for CNT/Cu and CNT/Cu–Cr composites at 10 vol.% CNT loading based on the yield strength measurements, respectively. This indicates that the interface strength of CNT/Cu–Cr composites exceeds that of CNT/Cu composites by approximately 50%. Hence, it is concluded that the formation of an intermediate  $\text{Cr}_3\text{C}_2$  transition layer between CNTs and Cu–Cr matrix greatly improves the interfacial interaction of CNT/Cu–Cr composites, which in turn helps to distribute the external load from matrix to CNTs and further enhances the strength of the compos-

ites. Moreover, the carbides formed are mostly connected to the outer surface and tips of the CNTs, which can tightly lock the CNTs inside the Cu matrix [30]. This lock effect can prevent CNT pull-out and thus improves the stress transfer efficiency during loading. Furthermore, solid solution alloying is effective in forming obstacles to motions of lattice dislocations and therefore the minor addition of alloying Cr slightly strengthens the copper matrix and in turn results in a high strength of the composites. All of these factors make the strength of CNT/Cu–Cr composites significantly higher than that of CNT/Cu composites.

For CNT/metal composites, it has been found that the formation of limited amount of carbides at the interface can largely improve the composite strength [31]. Bustamante et al. [32] obtained a 57.5% increase in the tensile strength in CNT/Al composites, which were sintered at 550 °C for 3 h followed by hot extrusion at 500 °C. They attributed the high tensile strength to the strong interfacial bonding through the formation of  $\text{Al}_4\text{C}_3$  interfacial layer of between CNTs and Al matrix, which was confirmed through X-ray diffraction. He et al. [33] achieved an extreme 184% improvement of tensile strength by 5 vol.% CNT addition in CNT/Al composites prepared by sintering at 640 °C followed by pressing and annealing at 840 °C of CVD grown CNT–Al powders. They observed thin layers of  $\text{Al}_4\text{C}_3$  formed on the surface of CNTs in the TEM images of samples. On the contrary, Goh et al. [34] reported a only 10% increase in the strength of CNT/Mg composites with addition of up to 2 wt.% CNTs. Since the free energy of formation of interfacial  $\text{MgC}_2$  and  $\text{Mg}_2\text{C}_3$  carbides is positive at their processing temperatures, they thus gave the reason for the low strength increment to the weak interfacial bonding caused by the absence of interface chemical interaction. A comparison of the interface characteristic in the present CNT/Cu composites with Al/CNT and Mg/CNT composites reported in the literature stresses the role of the formation of interfacial carbides responsible for the interfacial bonding and load transfer ability in the CNT/metal composites.

#### 4. Conclusions

In summary, the effect of matrix-alloying Cr element on the interface microstructure and mechanical properties of CNT/Cu composites is investigated. The interface microstructure analysis indicates that the good interfacial bonding can be successfully achieved through the formation of a thin transition layer of  $\text{Cr}_3\text{C}_2$  at the interface between CNTs and Cu–Cr matrix. The improved interfacial bonding makes CNT/Cu–Cr composites exhibit the increments of hardness and yield strength being 128% and 135%, respectively, in relation to CNT/Cu composites. The present study implies that a matrix-alloying method can give a successful improvement in interfacial bonding and mechanical properties of CNT/Cu composites.

#### Acknowledgments

This study was financially supported by National Natural Science Fund of China (Nos. 50971020 and 51165021), Gansu Province Science Fund for Distinguished Young Scholars (111RJDA0103) and Doctoral Start-up Scientific Research Fund (2011010431).

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