



Formation mechanism of nano titanium carbide on multi-walled carbon nanotube and influence of the nanocarbitides on the load-bearing contribution of the nanotubes inner-walls in aluminum-matrix composites



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ABSTRACT

Carbon nanotubes (CNTs), generally do not completely impart their exceptional properties in aluminum-matrix composites because of the poor interfacial bonding between these nanofillers and the matrix. In the present study, surface modification of CNTs with nanocrystalline titanium carbide (TiC) was performed through a pressureless-spark plasma sintering. Mechanisms for the formation of nano TiC during the applied method were proposed. It was found that single nanocrystals of TiC were epitaxially grown on the surface of raw CNTs with the typical orientation relationship of TiC (200)//CNT (002) at the interface. The shapes of carbides were mostly nanolumps and nanoblocks relating to the reacted carbon sources. Different preferential nucleation sites for TiC, such as bamboo-like defects, radially unzipped sites were also investigated in details. Thermal stability of the modified-CNTs, analyzed by thermogravimetric analysis, showed a significant increment in thermal properties compared to raw CNTs. It was shown that TiC-modified CNT hybrid structure is helpful to improve the inner-wall bonding conditions, leading to the effective load transfer between the walls. It was revealed that compressive yield strength of Al-1.5 wt% modified-CNTs with the TiC:CNT mixing ratio of 4:2 is the highest compared with other composites with different amounts of CNTs.

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1. Introduction

Since the discovery of carbon nanotubes (CNTs) by Sumio Iijima in 1991 [1], CNTs have attracted much more attention in different fields. Many techniques for producing CNTs have been reported in the literature such as arc discharge [2], laser ablation [3] and chemical vapor deposition (CVD) [4,5]. The remarkable properties of CNTs including high Young's modulus (~1 TPa), significant strength (~200 GPa) [6–8], thermal and chemical stability as well as extraordinary flexibility [9–11] and nano-scale one dimensional structure [7,8], make them exclusive candidate for reinforcing many kinds of materials. They have been particularly considered as ideal nano-reinforcements in multifunctional composites, specially metal-matrix composites, due to their outstanding properties

[12–14]. The interface between CNTs and metal matrix plays a critical role in controlling the overall properties of the composites [14]. Matrix needs to strongly adhere to the reinforcements so that the load can be transferred from matrix to CNTs, preventing the interface slipping [15]. Moreover, the exceptional strength of inner-graphene-walls in multi-walled carbon nanotubes (MWCNTs) cannot be exploited owing to extremely weak inner-wall shear resistance [16]. This important issue causes that just the outermost wall of MWCNTs carry the applied load. Specific method causing load transfer to the inner-walls of MWCNTs has not yet been experimentally proposed by carbide decorating nanotubes. Peng et al. [17] reported that the inter-wall load transfer can be improved with increasing the defect density. However, the net defective surface of MWCNTs may deteriorate the other properties of resultant composites.

There are many studies regarding filling, decorating and coating of CNTs with various kinds of materials such as metals, carbides, and oxides [18–22]. Coating of CNTs may shield them from reactive

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environments at elevated temperatures, improving the CNTs-matrix interfacial properties [23–26]. So et al. showed that silicon carbide, which highly wets Al and its alloys, can be successfully coated on the surface of CNTs to promote interfacial strength between reinforcement and matrix [27]. However, tensile tests did not exhibit a prospective increase in the strength results, maybe because the CNT structures were severely damaged during the coating process [14].

A simple, effective and novel three-stage method for modifying the surface of carbon nanotubes by nanocrystalline TiC was introduced and primarily investigated in our previous study [28]. However, the formation mechanism of the nano-TiC on the surface of CNTs in this novel approach still needs further investigations. In addition, knowledge about the preferential nucleation sites of TiC nanoparticles may elucidate formation mechanism of the carbide layers and also can be useful for controlling the heterostructure of TiC-CNT with the well-defined crystalline interface. Therefore, the present study aims to understand the formation mechanism and preferential nucleation sites of nanocrystalline TiC on the surface of MWCNTs, as well as its effect on Al-matrix composites. It is of great importance for a deep understanding of the formation mechanism of TiC on the surface of CNTs, as well as the effect on Al-matrix composites with improved load bearing contribution of the inner walls. This study is the first example, in which the inner walls of the MWCNT are linked by nanocrystalline titanium carbides.

2. Experimental

MWCNTs (10–20 nm in diameter, length > 5–15 μm) and Ti powders (99.9% purity, 300 mesh) were manufactured by Shenzhen Nanotech and Xingye Metallic Materials, respectively. The proposed modifying method included three stages. At the first stage, 1:1 atomic ratio (4:1 weight ratio) of Ti to CNTs was mixed by using 1 h of ultrasonication followed by wet milling (QM-3SP2) with hexane protection for 20 h at BPR and RPM of 20:1 and 250, respectively. At the second stage, additional raw CNT (2 g) was added to the crushed mixture and thereafter the mixture was wet milled for one hour with the same parameters as the previous stage. Different mixing ratios of the crushed mixture to the raw CNTs, namely Ti:CNT, were prepared. According to the previous study [28], pressureless-SPS technique (SPS, FCT-HP-D5) was performed at 1050 $^{\circ}\text{C}$ for a holding time of just 5 min. In order to achieve pressureless conditions during SPS process, some modifications were made to the conventional die assembly. T-shaped punches were fabricated from the same graphite material used for the die. There was a working space above the sample which could produce no external load condition during processing. The working space was filled by several graphite foils between the sample and upper punch of the graphite mold to ensure the pulsed current can pass through the powders. A minimum pressure was applied in order to supply sufficient contact resistance through the punch exterior and die wall interior.

After the modification of the tubes, different amounts of CNTs were added into Al matrix (0.5, 1, 1.5, 2 and 5 wt%) through ultrasonication and wet milled (for 1 h to prevent damage to the structure of CNTs, BPR; 20:1, rpm; 250) and then sintered at 520 $^{\circ}\text{C}$, with a holding time of 10 min, a heating rate of 50 $^{\circ}\text{C}/\text{min}$, and a pressure of 50 MPa by SPS process. Reference samples including a pure aluminum and Al+1.5 wt% unmodified-CNTs were prepared by the same route. In addition, Al-1.5 wt% [pure TiC (Sigma-Aldrich-325 mesh)+CNT] was also prepared with the mixing ratio of 4:2 through the same conditions and methods, as a comparison. Morphology of the modified-CNTs was studied by a high-resolution transmission electron microscope (HRTEM, Tecnai, FEI). In addition, thermogravimetric analysis (TGA, Netzsch) was also carried out in

air condition with a heating rate of 20 $^{\circ}\text{C}/\text{min}$ in the temperature range of 25–1000 $^{\circ}\text{C}$ for the modified- and unmodified-CNTs. Raman spectroscopy (Thermo Scientific DXR) was performed by using a 532 nm argon laser as the excitation source to evaluate the structural integrity of CNTs. The compressive strength of the composites was measured by a mechanical tester (MTS-SANS-CMT5305) according to the standard DIN 50106 with a 0.5 mm/min crosshead speed and height ($h_0 = 10$ mm) to diameter ($d_0 = 10$ mm) sample ratio (h_0/d_0) of 1.

3. Results and discussion

3.1. Formation mechanisms of nanocrystalline TiC

Knowledge about the nature of the formed nanocrystalline TiC and carbon sources in the modification procedure can elucidate the formation mechanism of the coated layer. Fig. 1a schematically illustrates the different types of carbon sources involved in the modification process along with the corresponding TEM observations of TiC formed on the surface of CNTs (Fig. 1b–e).

In the applied modification process, there are two sources of carbon for producing a carbide layer; carbon materials mostly provided from the sidewall of the raw CNTs (the area marked with 1 in Fig. 1a) and carbon mainly obtained from crushed nanotubes

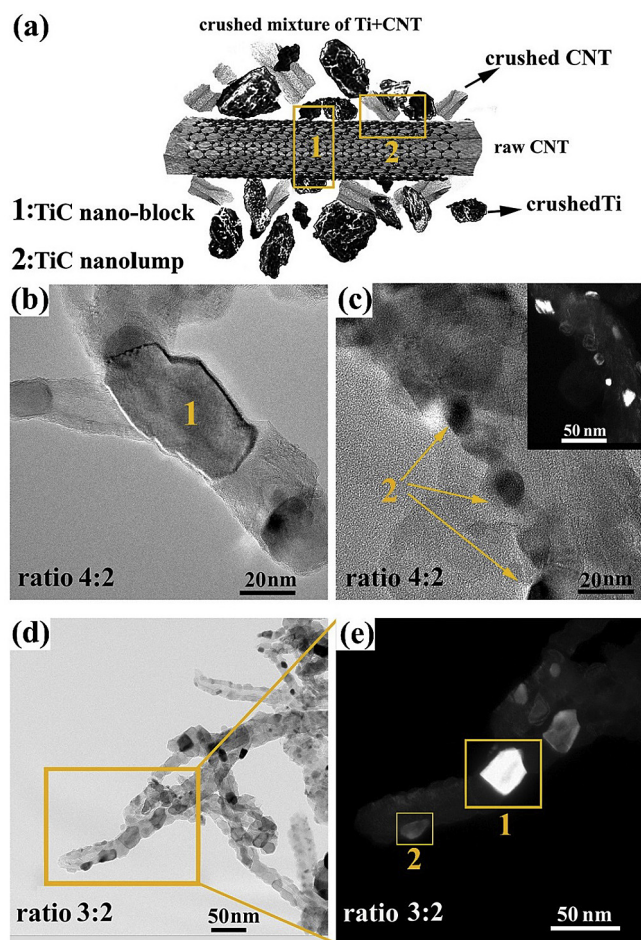


Fig. 1. (a) Schematic illustration of a raw CNT coated with crushed mixture powder showing different carbon sources. (b–e) TEM observations of TiC nanoblocks (regions marked by 1) and nanolumps (regions marked by 2) produced mainly from the sidewall of the raw CNTs and crushed CNTs, respectively. (A colour version of this figure can be viewed online.)

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