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# Microstructure and strengthening mechanism of carbon nanotubes reinforced magnesium matrix composite

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ABSTRACT

Carbon nanotube (CNTs) reinforced Mg matrix composites were successfully fabricated by a novel approach. After hot extrusion, the influence of CNTs on microstructure and mechanical properties of Mg–6Zn matrix was investigated. The results showed that most CNTs distributed uniformly and individually in the composites, and good interfacial bonding was achieved. CNTs significantly refined the grains of Mg–6Zn matrix, and the CNTs evidently improved the ultimate tensile strength (UTS), yield strength (YS) and Young′s modulus. Grain refinement, load transfer mechanism and Orowan strengthening mechanism play important roles on the increase of the yield strength.



## 1. Introduction

Carbon nanotubes (CNTs) have attracted widespread attention as the ideal reinforcements for composites because of their extremely high elastic modulus and strength [1–6] as well as good thermal [7] and electrical properties [8]. In the past decades, the main research efforts were focused on the CNT reinforced polymer matrix composites, and works on CNT reinforced metals have been relatively rare, especially for magnesium [9,10]. Magnesium matrix composites have been extensively studied due to their low density, high specific stiffness and specific strength, high damping capacity and good dimensional stability [11–14].

Zeng [15] investigated the effects of CNTs on the microstructure of hot-extruded Mg–2.0Zn magnesium matrix composites reinforced with 1.0 wt% CNTs. However, the influence of the CNTs′ content on the microstructure had not been studied. Fukuda [16] analyzed the interface between Mg matrix and CNTs in Mg–6 wt% Al alloy matrix composites reinforced with carbon nanotubes, and found that needle-like ternary carbides of $Al_2MgC_2$ were synthesized at some interfaces between magnesium matrix and CNTs, and the other interfaces were clean without any other materials or defects. However, to the best of our knowledge, open literature reports so far suggested that rare attempt is made to analyze the interface between Mg matrix and CNTs in Mg alloys without Al element. In addition, the strengthening mechanism of the CNTs reinforced Mg matrix composite is still rare and unclear [14,17].

Accordingly, the primary aim of this paper is to investigate the influence of CNTs on microstructure and mechanical properties of magnesium matrix, and then reveal the strengthening mechanism of CNTs reinforced magnesium matrix composite. At last, the formula for calculating the yield strength of CNTs reinforced magnesium matrix composite is expected to be derived from this paper.

## 2. Materials and methods

The pure Mg ingot and pure Zn ingot were used as the starting materials. CNTs were synthesized using CVD by Chengdu Organic Chemistry Co. Ltd., China. These CNTs have an external diameter of 40–60 nm and a length around 2 μm.

The chemical dispersant TNADIS (Chengdu Organic Chemistry Co. Ltd., China) was first dissolved in ethanol in a small beaker. Then CNTs (1.0 vol% of the metal matrix, mass ratio to the organic dispersant 8:1) were added to the as-prepared solution. This mixture was put at room temperature into an ultra-sonic bath for 15 min. Then it was stirred for 30 min at 250 rpm. After adding 300 g magnesium chips, the suspension was further stirred at 250 rpm inside a fume cupboard to evaporate ethanol and homogenize the mixture.

About 640 g magnesium ingot was melted at 720 °C in $CO_2+SF_6$ protective atmosphere, and then 60 g zinc ingot was added into the crucible. And then the melt was cooled to 600 °C at

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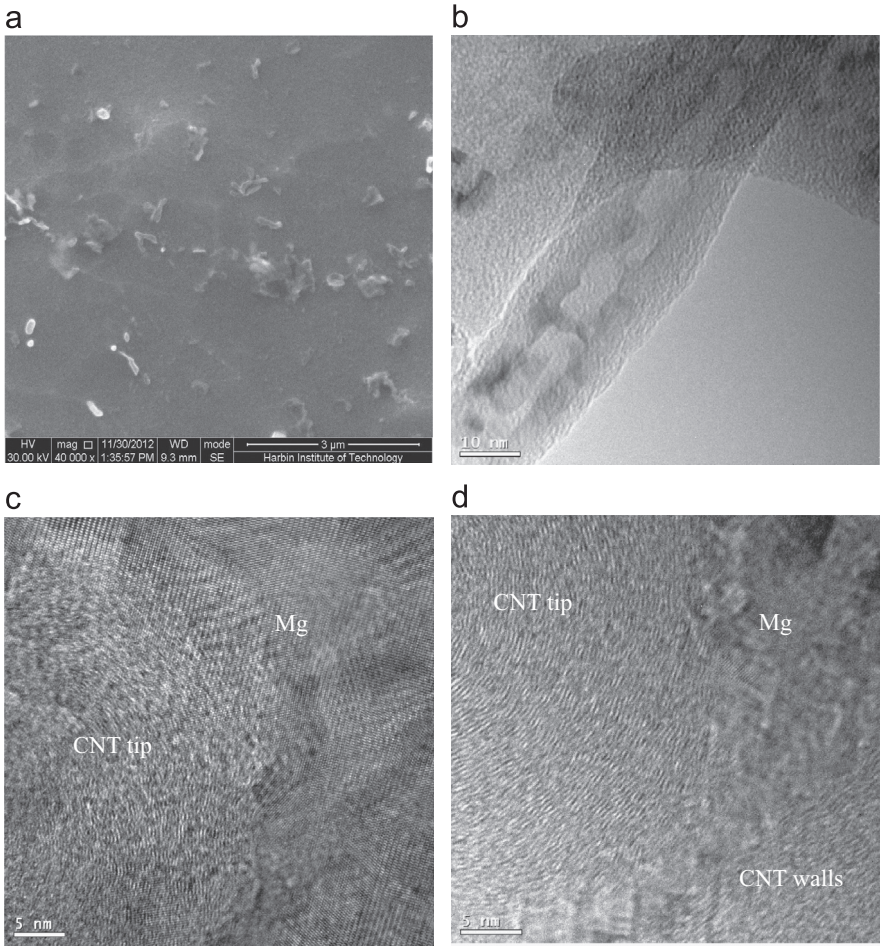


**Fig. 1.** The microstructure of the as-extruded composites. (a) SEM image for the CNT distribution; (b) TEM image of a typical CNT in composite; (c) the interface between CNT tip and Mg; and (d) for the interface between two CNTs and Mg.

which the matrix alloy was in semisolid condition. As the CNTs coated Mg chips were quickly added into the semisolid alloy the melt was stirred in the protective atmosphere of $CO_2$ and $SF_6$ to avoid burning. The stirring rate was 800–1200 r/min and the stirring time was 10 min. After semisolid stirring for 10 min, the mixture of the melt and CNTs were rapidly reheated to 690 °C, and then the melt was ultrasonically processed at 500 W power level for 20 min before the ultrasonic probe was removed from the slurry. After the ultrasonic vibration, the mixture melt of the CNTs and the matrix alloy was poured into a preheated steel mold (375 °C) and allowed to solidify under 100 MPa pressure to obtain the composite ingots without porosity. The cast billets were cut into the samples with the size of Ø 60 mm × h 35 mm and homogenized at 350 °C for 12 h before the extrusion. And then the as-cast composite was extruded at 350 °C with an extrusion ratio of 20:1. In order to identify the effects of CNTs on the matrix alloy, the monolithic Mg–6Zn alloy was also developed by the same condition.

The optical microscopy (OM) (P-3, Olympus, Japan), scanning electron microscopy (SEM) (Quanta 200FEG, FEI Co. Ltd., USA) and transmission electron microscopy (TEM) (HR-TEM, Tecnai $G^2$ F30, USA) were used to study the microstructure of the matrix and the composite. The specimens for microstructure analysis were sectioned parallel to the extrusion direction and prepared by the conventional mechanical grinding, polishing and etched in the nitric acid. The specimens for TEM tests were prepared by grinding–polishing to produce a foil of 50 μm thickness and followed ion beam thinned. For EBSD analysis, the buffed samples were electrochemically finished with the mixture of 150 ml of phosphoric acid and 90 ml of ethanol at the voltage of 5 V for 30 s. After the electrochemical finishing, the specimen surfaces were cleaned with methanol and subsequently washed in ethanol with ultrasonic vibration to completely remove the surface debris. Tensile test was carried out at a tensile rate of 0.5 mm/min by Instron-1186 tension machine. The samples of tensile tests were determined in accordance with ASTM: E8/E8M-11 standards. For each material, three samples with standard dog-bone shape were tested.

## 3. Results and discussion

### 3.1. Microstructures

Fig. 1a shows scanning electron microscopy (SEM) image of homogeneously dispersed CNTs in the as-extruded composite, obtained by the novel process described above. The CNT clusters were not observed, which indicates that CNTs distributed uniformly in the composites. Therefore, the novel process can fabricate magnesium matrix composites with uniform CNTs. Fig. 1b shows the TEM image of a typical CNT in as-extruded composite. The TEM observation further confirmed that CNTs were singly dispersed in Mg matrix. Fig. 1c shows HRTEM for interfaces between CNT tip and Mg, and Fig. 1d shows HRTEM for interface between two CNTs and Mg. As seen from Fig. 1c and d, well-graphitized multiwall CNTs were retained in the composite, which