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## Carbon Nanotube Reinforced Intermetallic

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# Carbon Nanotube Reinforced Intermetallic

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## Abstract

Iron aluminides ( $\text{Fe}_3\text{Al}$ ) based composite with different amounts of multi-walled carbon nanotube (MWNT) have been prepared by using spark plasma sintering. Effect of MWNTs content on the mechanical properties was investigated. The compressive yield strength and fracture toughness of the 3 vol% MWNTs– $\text{Fe}_3\text{Al}$  composite, compared with the monolithic  $\text{Fe}_3\text{Al}$ , were enhanced 73.6% and 40%, respectively. The main reinforced mechanisms include MWNTs pulling-out, crack deflection, bridging and MWNTs rupture. The defects and aligning direction of MWNTs in the matrix are also the factors that affect the mechanical properties of composites as prepared.

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## Keywords

Carbon nanotubes, composites, intermetallics, toughness mechanism

## 1. Introduction

Carbon nanotubes (CNTs) are new allotropic carbon materials with excellent mechanical, electrical, thermal and chemical stability properties. A single-wall CNT (SWNT) consists of a perfect closed cylinder, with a diameter of a few nanometers. Several concentric cylinders assembled together yield the so-called multi-wall CNTs. Both single- and multi-wall CNTs exhibit a very large aspect ratio, i.e., their length is 1000 to 10000 times their diameter. CNTs are extremely rigid; their elastic modulus has been measured to be higher than 1 TPa and they are strong, their tensile strength being in the order of 10 GPa [1, 2].

Incorporation of this one-dimensional nanostructured material into ceramics [3, 4], polymers [5, 6] and metals [7–9] has attracted research interest in recent years. Zhan *et al.* [10] successfully applied SWNTs in the reinforcement of ceramic

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composites through spark-plasma-sintering, resulting in a 194% increase in fracture toughness over pure alumina. Chen *et al.* [5], in characterizing the Epoxy/CNT nanocomposites, found that the addition CNT to Epoxy resulted in improvement in the tribological property of composite. Ti/nanotube with high hardness and Young's modulus has been fabricated by Kuzumaki *et al.*, and enhanced mechanical properties and improved electrical properties were also shown in the CNT/Al composite. Although the reinforcing of ceramics, polymers and metals with CNTs is a reasonably well-established field of research, very few works have been done considering an intermetallics matrix. Composites consisting of iron aluminide matrix and a ceramic reinforcement such as particles, whiskers, and fibers have made some progress [11, 12]. Herein, we present the results of a preliminary study on the manufacturing of multi-walled carbon nanotubes reinforced iron aluminides ( $\text{Fe}_3\text{Al}$ ) matrix composites. Although multi-walled CNTs are not the best choice in terms of mechanical properties, they were selected for this preliminary study due to their low cost in relation to single-walled CNTs.

## 2. Experimental

MWNTs were provided by Shenzhen NANO Tech. Port. Co. Ltd. They were fabricated by catalytic pyrolysis of hydrocarbon, whose purity is about 97% as claimed by the producer. The MWNTs were treated by concentrated nitric at 140°C for 6 h according to the procedure described in the literature [13].  $\text{Fe}_3\text{Al}$  powder was fabricated by mechanical alloying. The MWNTs– $\text{Fe}_3\text{Al}$  intermetallics matrix composite was sintered using spark plasma sintering (SPS) apparatus (Dr. Sinter 2080, Sumitomo Coal Mining Co., Tokyo, Japan). Four composite powders with different contents of MWNTs were prepared to fabricate intermetallics matrix composites, which were 1, 3, 5 and 7 vol%. The synthesis process of MWNTs– $\text{Fe}_3\text{Al}$  powders mixture has been reported in our previous work [14]. The annealing time, sintering temperatures, and heating rate were set to 5 min, 1000°C and 300°C/min.

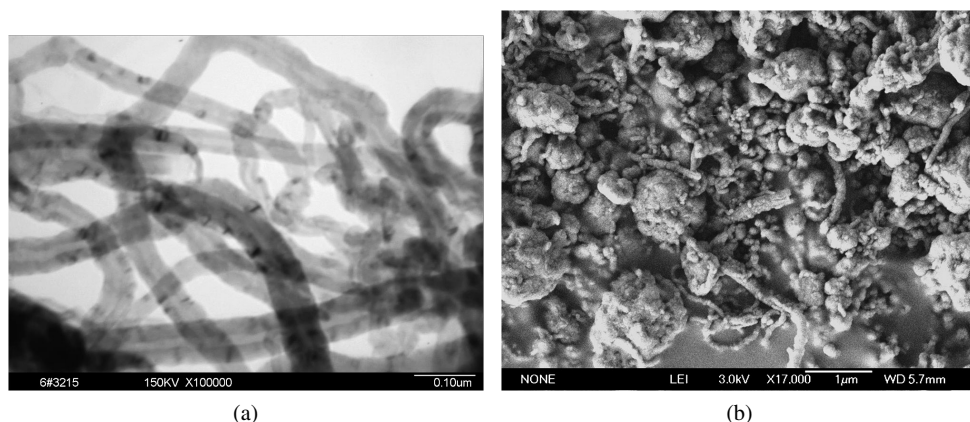
The fracture toughness was tested by three-point method with a chevron notch. Typically, five samples were tested for each sample in each way to ensure that the value of each mechanical property obtained was reasonably representative. The compressive yield strength was measured by the materials test machine with the size of  $\varnothing 4 \times 7$  mm at room temperature and at a constant cross-head displacement rate of 0.5 mm/min. The phase composition of the composites was identified by X-ray diffractometry (XRD) (Model D/max 2550V, Japan). A transmission electron microscope (TEM) (Model JEM-2010, Japan) was used to observe the micrograph of the mixture powder and MWNTs. A field emission scanning electron microscope (FESEM) (Model JSM-6700, Japan) was used to observe the fracture surface in order to confirm the integrity of MWNTs after sintering, and to detect the occurrence of possible toughening events (crack–MWNTs interactions), an observation essential to an understanding of the potential of MWNTs in reinforcing an intermetallics matrix.

### 3. Results and Discussion

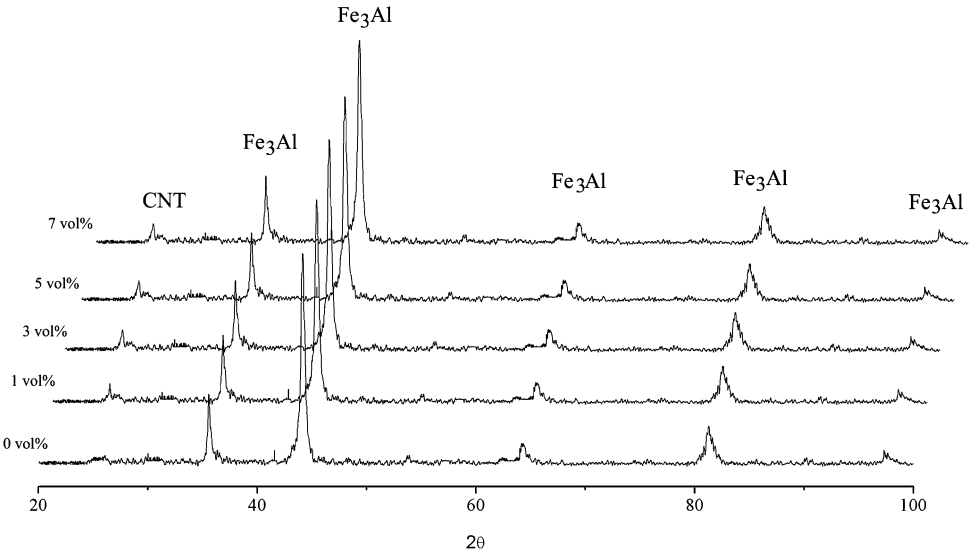
A morphological study has been performed to follow the structural changes during the preparation of MWNTs–Fe<sub>3</sub>Al composite. On Fig. 1(a) the MWNTs used in the experiment are presented. It can be seen that the MWNTs are tangled together. Their diameters are between 20 and 40 nm and length is about tens of microns (μm), the aspect ratio is up to 100–1000. Most of the MWNTs are not straight, but possess some defects and demonstrate localized kinks and bends. MWNTs introduced in the powder mixture are shown in Fig. 1(b). Note that the tubes are dispersed throughout the iron aluminized powders. The sonication and mechanical balling resulted in a successful dispersion of MWNTs in the powder mixture, due to enhance interaction and combination between MWNTs and Fe<sub>3</sub>Al particles [15].

Phase component investigation of MWNTs–Fe<sub>3</sub>Al composites with different MWNT volume content is illustrated in Fig. 2. The strongest peak of MWNTs can be observed in the sintered sample, which indicates that the structure of MWNTs has not changed after the spark plasma sintering process.

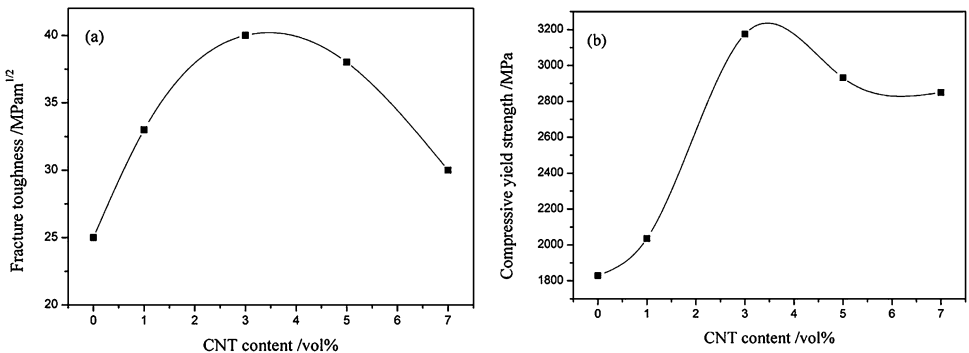
Figure 3 is the curve of the dependence of fracture toughness (a) and yield strength (b) on the MWNTs volume content. The fracture toughness and yield strength increase with the MWNTs volume increase when the volume content of MWNTs is lower than 3%. When the volume content of MWNTs is higher than 3%, the yield strength and fracture toughness decrease with the increase of MWNTs. The yield strength and fracture toughness of composites with 3 vol% MWNTs is the highest; the ratio of increment, compared with that of monolithic Fe<sub>3</sub>Al, is up to 73.6% and 40%, respectively. Two contrary factors may result in the above phenomena. First, the MWNT has large aspect ratio and excellent mechanical properties. According to the theory of short fiber reinforced composites, it can improve the mechanical properties greatly. On the other hand, MWNTs make the yield strength decrease because they can hinder the densification. With the increase



**Figure 1.** (a) TEM micrograph of MWNTs; (b) SEM micrograph of MWNTs dispersed in starting mixture.



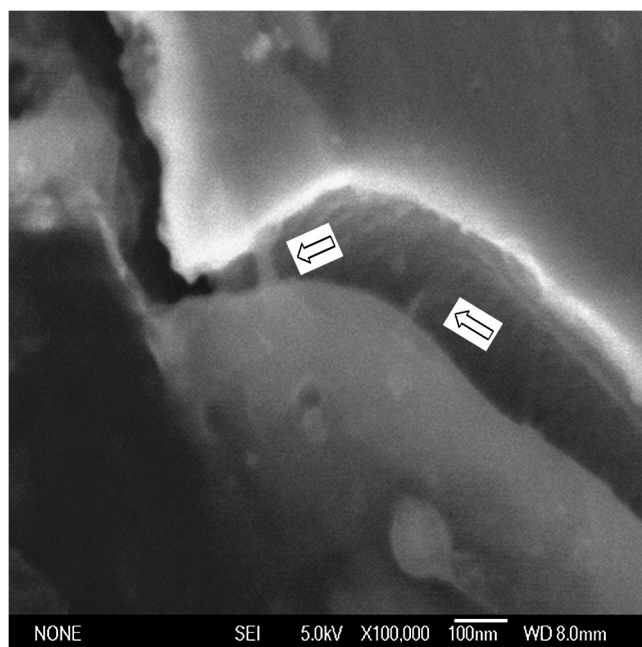
**Figure 2.** XRD patterns of  $\text{Fe}_3\text{Al}$  and MWNTs– $\text{Fe}_3\text{Al}$  composites.



**Figure 3.** Effect of CNT volume content on the fracture toughness (a) and yield strength (b) of CNT/ $\text{Fe}_3\text{Al}$  composite.

of MWNTs content, the probability of agglomeration is increased. In the agglomeration of MWNTs, the bonding is loose. When the stress transfers to the MWNTs, it is easy to separate them from the matrix, which reduced the mechanical properties.

The increase in yield strength is due to the generation of geometrically necessary dislocations in the  $\text{Fe}_3\text{Al}$  matrix around the MWNTs as a result of coefficient of thermal expansion (CTE) and elastic modulus mismatch between  $\text{Fe}_3\text{Al}$  and MWNTs. The amount of dislocations generated due to CTE mismatch is found to be proportional to the volume fraction of MWNTs and inversely proportional to the diameter of the MWNTs [16]. With higher dislocation, density can be generated, and hence higher yield strengths can be obtained. This phenomenon of increasing yield strength with higher volume fraction of MWNTs is applicable only until 3 vol% of



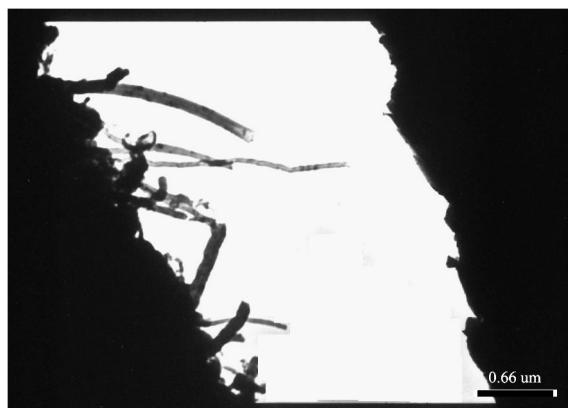
**Figure 4.** SEM micrograph of MWNTs–Fe<sub>3</sub>Al composite. A surface crack is bridged by the nanotubes.

MWNTs, above which, the yield strength starts to degenerate due to higher amount of porosity in the Fe<sub>3</sub>Al matrix.

The positive influence of carbon nanotubes on fracture toughness has already been reported [17, 18]. The toughness mechanism in carbon nanotube/iron aluminide composite fabricated by the spark plasma sintering process can be explained by a crack bridging effect of carbon nanotubes. The carbon nanotubes, which bridging the two crack surfaces as shown in Fig. 4, strongly supports the crack bridging effect during the crack propagation. Some wrapped MWNT aligns perpendicular to the crack direction and bridges a matrix crack, meaning that they carry tensile load. The fracture morphology of MWNTs–iron aluminides showed that nanotubes pulled out, indicating that the load transfer from iron aluminides to nanotubes was sufficient to fracture the nanotubes, as illustrated in Fig. 5. Given the above evidence, we can infer that a significant crack bridging effect, the bonding between MWNTs and iron aluminides matrix, and pullout of MWNTs at interface are possible mechanisms leading to the improvement of the fracture toughness.

#### 4. Conclusions

A carbon nanotube/iron aluminide composite with enhanced yield strength and fracture toughness was successfully fabricated by spark plasma sintering. The carbon nanotubes were homogeneously dispersed within the iron aluminides matrix



**Figure 5.** Fractography of MWNTs–Fe<sub>3</sub>Al composites.

in carbon nanotube/iron aluminide composite. Results of the mechanical properties show that there are simultaneous improvements in yield strength and fracture toughness up to a threshold of 3 vol% MWNTs. The strengthening mechanism is based on the load transfer between the iron aluminide matrix and carbon nanotubes; the toughening mechanism is strongly related to the crack bridging effect of carbon nanotubes during the crack propagation in carbon nanotube/iron aluminide composites.

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