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Mechanical properties of SiC composites incorporating SiC-coated multi-walled carbon nanotubes

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Abstract

Multi-walled carbon nanotubes (MWCNTs) were coated with a nanometer-sized SiC polycrystalline layer by the reaction of SiO(g) and CO(g). Nanometer-sized SiC powders were mixed with 1–5 vol% SiC-coated MWCNTs and successfully sintered at 1800 °C by means of pulsed electric current sintering. These composites showed superior microhardness of 30.6 GPa and toughness of 5.4 MPa $m^{1/2}$ compared to monolithic SiC ceramics due to the improvement of adhesion between MWCNTs and the SiC matrix by the SiC coating. The microhardness and toughness of the monolithic SiC ceramics are 25.5 GPa and 4.8 MPa $m^{1/2}$, respectively. The SiC-coated MWCNTs/SiC composites showed an interesting behavior with the elastic recovery when undergoing an indentation test.

Keywords: Carbon nanotube; SiC coating; Ceramic matrix composite; Hardness; Toughness

1. Introduction

Multi-walled carbon nanotubes (MWCNTs) have been tested as reinforcements for various matrices [1–3] because of the unique mechanical [4–9] and physical properties [10–14]. For example, such reinforcements can improve the strength of matrices due to the extremely high tensile strength of MWCNTs (~60 GPa). However, higher performance composites based on MWCNTs have not been obtained. It is suggested that the weak adhesion between MWCNTs and the matrix reduced the original effect of MWCNTs as nano-reinforcement and limits their applications.

We have developed a new and simple process for coating carbon materials, such as fine diamond particles and MWCNTs, with SiC [15–18]. The MWCNTs were completely and uniformly coated with a nanometer-sized β-SiC polycrystalline layer. The SiC coating protects MWCNTs from molten cobalt and provides dense SiC-coated MWCNTs/cemented carbide composites, which can be fabricated by pulsed electric current sintering (PECS) [19]. The SiC coating is expected to improve the weak adhesion between MWCNTs and various matrixes.

In this study, the SiC-coated MWCNTs were used as nano-reinforcements for SiC ceramics. SiC has high heat and oxidation resistance [20,21]. Therefore, various applications related to space developments and efficient power generators are expected. However, the low reliability due to the brittle nature of SiC is a critical problem. MWCNTs could be one of the best candidates to reinforce the SiC matrix if the original strength of MWCNTs appears. Monolithic SiC ceramics, MWCNTs/SiC composites, and

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SiC-coated MWCNTs/SiC composites were fabricated by PECS. The microstructure and mechanical properties were studied to evaluate the effect of the SiC coating on MWCNTs.

2. Experimental procedure

2.1. SiC Coating on MWCNTs

MWCNTs (CNT20, CNRI Inc., Tokyo, Japan) synthesized from hydrocarbon were used for the reinforcement of SiC due to their high yield and uniform diameter (20–50 nm). Their length is ~60 μm. The SiC coating on MWCNTs is very simple and convenient to produce. Commercial SiO powders (99.9% pure, Nacalai Tesque Co., Kyoto, Japan) were used as the silicon source. The SiO powders were set on the bottom in an alumina crucible and MWCNTs were placed on a carbon felt, which was then set upon the SiO powders. This apparatus was covered with an alumina lid to keep the SiO gas pressure inside the crucible, and heated at 1150 °C or 1250 °C in vacuum at about 0.03 Pa. A heating temperature of 1150 °C is necessary to vaporize SiO.

After the coating treatment, crystalline phases of MWCNTs were characterized by X-ray diffraction (XRD, CuKα, JEOL JDX-3530M, Tokyo, Japan). The surface morphology and microstructure were observed using transmission electron microscopy (TEM, 300 kV, HITACHI H-800SS, Tokyo, Japan).

2.2. Sintering and mechanical properties of SiC-coated MWCNTs/SiC composite

The SiC-coated MWCNTs or uncoated MWCNTs were dispersed in isopropyl alcohol (IPA) using ultrasonic vibration for 5 min. The SiC-coated MWCNTs or uncoated MWCNTs were then mixed with nanometer-sized SiC (mean diameter of 30 nm) and B_4C (mean diameter of 240 nm) powders in IPA using ultrasonic vibration for 10 min. The B_4C was added at 2 wt% as a sintering aid. After the mixing, the IPA solution was evaporated and the mixed powders were dried at 100 °C. The MWCNTs were uniformly dispersed without aggregation. The mixed powders were sintered at 1800 °C, 40 MPa for 5 min under a vacuum by means of PECS (Dr. Sinter, Sumitomo Coal Mining Co. Ltd., Tokyo, Japan). The content of the SiC-coated MWCNTs was changed between 1–5 vol%. The Vickers hardness was measured under 9.8 N and 19.6 N loads. The fracture toughness was evaluated under the same load using the indentation fracture method [22]. The Vickers hardness and fracture toughness were measured at 10 points for each sample. The obtained results were compared with the monolithic SiC ceramics, which were sintered under the same conditions. The microstructure and indentation of the samples were observed by using 3D-SEM (Elionix ERA-8800FE, Tokyo, Japan).

3. Results and discussion

3.1. SiC-coated MWCNTs

X-ray diffraction peaks of β-SiC and MWCNTs are seen in all samples. The peak intensity of MWCNTs for the coating samples decreases with an increase in coating temperature. We reported that the SiC layer is formed by two steps: (1) conversion of the MWCNTs surface to SiC; and (2) deposition of nanometer-sized SiC on the thin SiC layer. At higher coating temperatures, the SiC layer formed by the first conversion step may be thicker according to the following reaction:

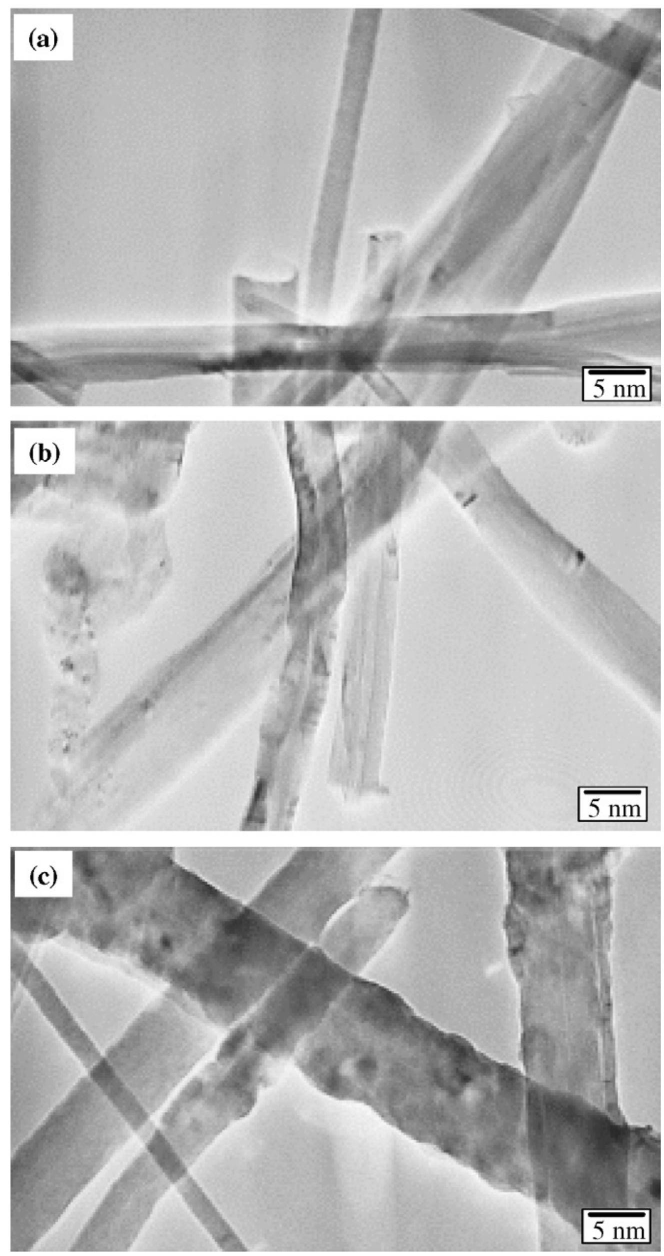

Fig. 1. TEM images of as-received MWCNTs and the SiC-coated MWCNTs. (a): As-received MWCNTs, (b): MWCNTs coated at 1150 °C, (c): MWCNTs coated at 1250 °C.