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Short communication

Failure investigation of carbon nanotube/3Y-TZP nanocomposites

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Abstract

3Y-TZP matrix composites containing 0.1–1 wt.% of multi-wall (MWCNT) and single-wall (SWCNT) carbon nanotubes were fabricated by spark plasma sintering technique. The sintered composites reached full density. Hardness and fracture toughness were measured using Vickers indention method. The hardness of the composite decreased with increasing weight content of the MWNT. The fracture toughness was 5.52 MPa $m^{0.5}$ when the amount of MWCNTs was 0.5 wt.%, however it decreased to 4.5 MPa $m^{0.5}$ when the content was raised to 1.0 wt.%. The composite containing 0.5 wt.% SWCNTs showed similar fracture toughness as that of matrix. The incorporation of CNTs into 3Y-TZP matrix led to no prominent improvement on the mechanical properties. The failure mechanism was analyzed finally.

Keywords: B. Composites; C. Fracture; C. Mechanical properties; D. ZrO_2; Carbon nanotubes

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1. Introduction

In recent years, there has been a steadily increasing interest in the development of CNTs reinforced composites, the aim being to transfer the exceptional mechanical properties of CNTs to the bulk engineering materials. Particularly CNTs have been considered as reinforcements in nanocomposites, due to their uniquely attractive mechanical and physical properties [1]. In this light, ceramics [2,3], polymer [4,5] and metal [6,7] are favorably considered as a matrix material. It has been demonstrated that adding an appropriate amount of nanotubes to ceramic-base materials can improve the mechanical properties. Ma et al. [8] prepared carbon nanotube/SiC composite powder via mixing nanosize SiC particles with 10% carbon nanotubes and then hot press it. They reported a 10% enhancement on both the strength and the fracture toughness as compared to the monolithic ceramics. Chang et al. [9] fabricated alumina matrix composites containing 5–20 vol.% of multi-wall carbon nanotubes. An improvement of 24% on fracture toughness compared with that of the single-phase alumina was found. Recently, Zhan et al. [10] fabricated fully dense nanocomposites of single-wall carbon nanotubes with nanocrystalline alumina matrix at sintering temperatures as low as 1150 °C by spark-plasma sintering (SPS). A fracture toughness of 9.7 MPa $m^{1/2}$, nearly three times that of nanocrystalline alumina, could be achieved. Hence, to expand the potential of CNTs for applications in nanocomposites, a wider investigation of CNTs and other ceramic matrix remains to be achieved. In this paper, 3 mol% yttria-doped zirconia matrix composites containing 0.1–1 wt.% of multi-wall (MWCNTs) and single-wall (SWCNTs) carbon nanotubes were fabricated by SPS. Hardness and fracture toughness were measured using Vickers indention method. The microstructure was observed by scanning electron microscope (SEM). The failure mechanism was analyzed from the viewpoint of the microstructure.

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2. Experimental procedure

2.1. Preparation of the powder

Shenzhen NanoPort Company kindly provided carbon nanotubes, whose purity is about 95% as claimed by the producer. Colloidal processing route [3] was adopted to make the distribution of CNTs more homogenously in matrix. 0.5 wt.% CNTs were dispersed into diluted solutions with 2 wt.% SDS (sodium dodecyl sulfate) as dispersant [11]. 1 vol.% 3Y-TZP powders (Tosoh Corporation) were prepared as diluted suspensions with 2wt.% PEI (polyethyleneamine, Mw 50,000, BDH Laboratory) as dispersant [12]. The diluted 3Y-TZP suspensions were added dropwise into the vigorously stirred as-prepared carbon nanotube suspension with SDS. After mixing, the surface of CNTs was coated by zirconia particles due to the heterogeneous coagulation reaction. The coated CNTs were subsequently ball mixed into a concentrated 3Y-TZP suspension of about 50 wt.% in ethanol; the CNTs-3Y-TZP composite powder with content of MWCNTs as 0.1, 0.5 and 1.0 wt.% were obtained after drying and grinding. For comparison, 0.5 wt.% SWCNTs-3Y-TZP composite powders were also prepared followed the same procedure.

2.2. Measurement of mechanical properties and the microstructure observation

CNTs-3Y-TZP composite powder sintered by SPS was carried out in Ar atmosphere using Dr Sinter 1020 SPS apparatus (Sumitomo Coal Mining Co., Ltd., Japan). The prepared composite powder was carefully placed into a 20 mm diameter graphite die, and heated to the sintering temperatures at 1300 °C for 5 min. A pressure of approximately 20 MPa was applied from the beginning of sintering and relaxed after the temperature below 800 °C. The bulk densities of the sintered samples were measured by the Archimedes method. Both hardness and fracture toughness were measured using a Vickers indenter (Akashi-A). The fracture toughness (K_{IC}) was calculated from the crack half-length and hardness [13]. The morphology of the composite powders was observed by TEM (JEOL, JEM-200CX), while the fractured surfaces were examined by scanning electron microscopy (SEM, INSTRUMENT JSM 6700F).

3. Results and discussion

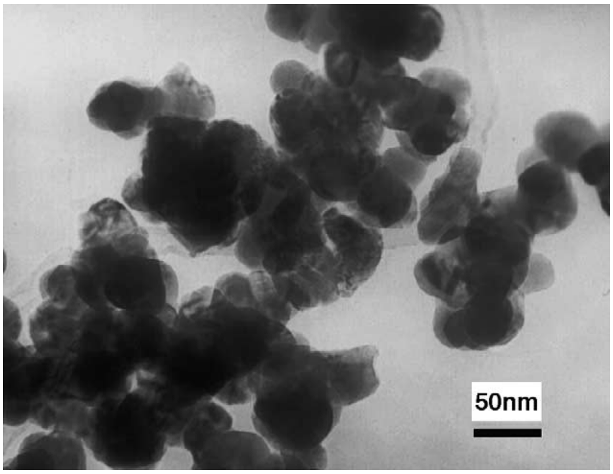

Fig. 1. TEM image of CNTs-3Y-TZP composite powders.

Fig. 1 shows TEM micrograph of CNTs-3Y-TZP composite powders, the surface of CNTs was coated by zirconia particles due to the heterogeneous coagulation reaction. Table 1 shows the density, hardness (HV_{10}) and fracture toughness (K_{IC}) of the five samples. It can be found that the hardness decreased with the addition of carbon nanotubes. The addition of CNTs as low as 0.1 wt.% almost has no effect on fracture toughness value. The fracture toughness increased from 5.28 to 5.52 MPa m$^{0.5}$ when the MWCNTs content is 0.5 wt.%, but decreased to 4.47 MPa m$^{0.5}$ when the content of MWCNTs increased to 1.0 wt.%. It can be concluded that the addition of CNTs does not result in enhanced hardness and fracture toughness. Instead, the hardness and fracture toughness decreased by 27% and 15% as the weight fraction of MWCNTs is increased to 1.0%. When CNTs were incorporated as reinforcements, it was proposed that during load transfer to multiwall nanotubes, only the outer layers are stressed in tension whereas all the layers respond in compression [14]. From this viewpoint, SWCNTs seem more effective as reinforcement than MWCNTs. Zhan et al.'s result [10] also showed that single-wall carbon nanotubes could increase the fracture toughness of alumina matrix as high as 9.7 MPa m$^{1/2}$. For comparison, 0.5 wt.% SWCNTs-3Y-TZP composites were sintered by SPS under the same condition. The hardness and fracture toughness value is almost as the same as that of specimen contained 0.5 wt.% multi-wall nanotubes. From our results, it can be deduced that single-wall nanotubes be no help in improving the mechanical properties of 3Y-TZP either.

It is generally accepted that the mechanical properties of the composites are dominated not only by the reinforcement

Table 1
HV_{10} and K_{IC} of pure 3Y-TZP and CNTs/3Y-TZP composites

Specimens	Relative density (%)	HV_{10} (GPa)	K_{IC} (MPa m$^{0.5}$)
3Y-TZP	100	13.24 ± 0.25	5.28 ± 0.05
3Y-TZP + 0.1 wt.% MWCNTs	100	13.18 ± 0.84	5.24 ± 0.09
3Y-TZP + 0.5 wt.% MWCNTs	99.1	11.89 ± 0.36	5.52 ± 0.07
3Y-TZP + 0.5 wt.% SWCNTs	99.3	10.59 ± 0.21	5.41 ± 0.10
3Y-TZP + 1.0 wt.% MWCNTs	99.1	9.71 ± 0.17	4.47 ± 0.15

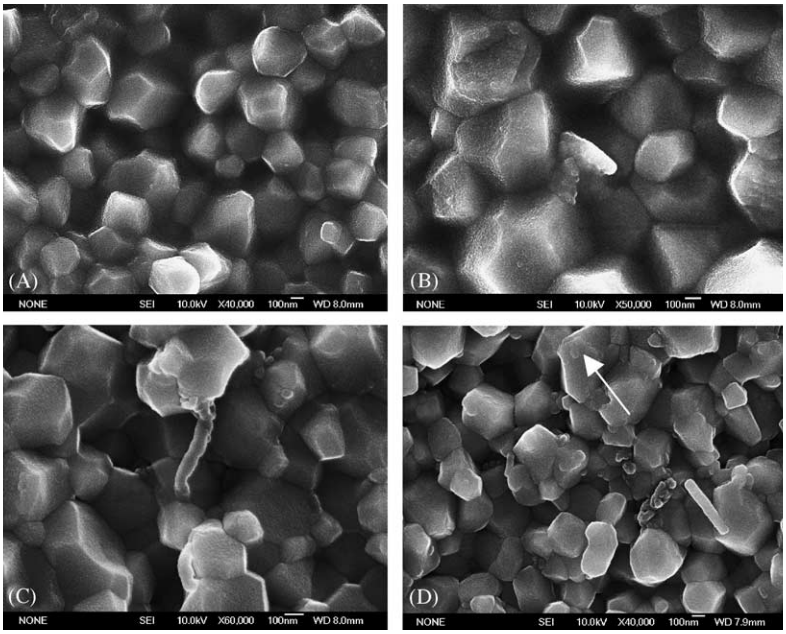

Fig. 2. SEM images of the fractured surface: (A) 3Y-TZP, (B) 0.1 wt.% MWCNTs, (B) 0.5 wt.% MWCNTs, and (D) 1.0 wt.% MWCNTs.

and the matrix but also by the interfacial bonding status between them [15,16]. It is necessary to study the materials fracture behavior to understand the load transfer between the matrix and reinforcements. Fractured surfaces were examined by scanning electron microscopy as shown in Figs. 2 and 3. Fig. 2 is the fracture surfaces of pure 3Y-TZP and composite containing MWCNTs. It is scarcely to find CNTs in sample B due to the low content as only 0.1 wt.%. With more addition of CNTs, in sample C and D, most of MWCNTs are located at the zirconia grain boundary. The fracture surface exhibited predominantly intergranular fracture. In sample D, few CNTs were found located inside the zirconia grains (as shown by arrows), but failed surviving from broking.

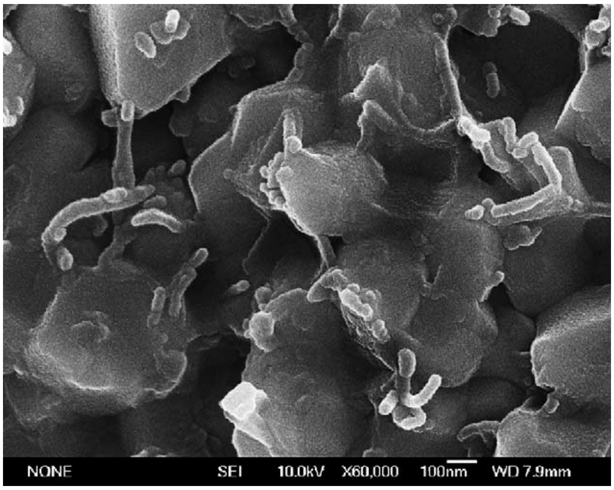

Fig. 3. SEM image of the fractured surface of specimen containing 0.5 wt.% SWCNTs.

Fig. 3 shows the fractured surface of sample contained 0.5 wt.% SWCNTs. Though the wt.% is as the same as that of sample C in Fig. 2, more quantity of SWCNTs can be found due to its lower density. Both intergranular and intragranular fracture modes existed, but more pores could be found between the grains. These pores might be closed end type and cannot be detected by Archimedes method. On the one hand, more quantity of SWCNTs impede the densification process and lead to the decrease in mechanical properties; on the other hand, some SWCNTs located within the zirconia grain and formed net-like work to prevent the fracture.

Improvements in the strength of CNTs/ceramic are largely attributable to sufficient load transfer from the matrix to CNTs through the interface. To ensure a good load transfer, the composite must maintain a medium strong interfacial bonding. From Fig. 2, we can see that the bonding between CNTs and 3Y-TZP matrix is very weak. So, the load transfer efficiency is very low. In addition, we cannot exclude the existence of the agglomerated CNTs in the grain boundary, which not only impede the densification of the specimens, but also become the defect source. The lack of reinforcement was attributed to poor carbon nanotube and zirconia matrix bonding; the addition of CNTs did not result in pronounced enhanced mechanical properties.

4. Conclusions

CNTs-3Y-TZP composites were sintered by SPS technique with the content of carbon nanotubes ranged from 0.1 to 1 wt.%. The hardness values decreased prominently with the addition of CNTs. The fracture toughness kept almost the same value as that of the matrix when the amount of MWCNTs was kept 0.5 wt.%, however it decreased to 4.47 MPa $m^{0.5}$ when the MWCNTs content was raised to 1.0 wt.%. 0.5 wt.% SWCNTs-3Y-TZP composites showed no improvement on the mechanical properties of 3Y-TZP either. The existence of the agglomerated CNTs in the grain boundary and the weak bonding between carbon nanotube and zirconia matrix are reasons that led to the failure in reinforcement.

Acknowledgements

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