CARBON NANOTUBE REINFORCED GLASS-CERAMIC NANOCOMPOSITES BY ULTRASONIC *IN SITU* SOL-GEL PROCESSING

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1 Introduction

Incorporating carbon nanotubes (CNTs) into composite materials has become a very promising topic. A problem with the composite production is that CNTs always aggregate together to form bundles; the high aspect ratio and the flexibility of CNTs lead to the physical. Therefore, the ability to disperse CNTs is the most critical factor for synthesising CNT composite materials. Although CNTs have been incorporated into different matrices, the literature describing homogeneous dispersions has been limited, especially in ceramic nanocomposites. Recently, CNT reinforced ceramic composites have been synthesized by conventional powder processing methods, such as ball-milling and hot pressing [1] and novel spark plasma sintering (SPS) [2]. However, a substantial improvement of the mechanical properties of the CNT-based ceramics has not been reported. One of the key issues is inhomogeneous dispersion of nanotubes in the matrix due to the aggregation of CNTs. In this paper, single-walled (SWNTs) and multi-walled (MWNTs) carbon nanotube reinforced aluminoborosilicate glasses were prepared by a modified sol-gel method. Composites containing various amounts of SWNTs and MWNTs were fabricated. The microstructure and mechanical properties of the composite materials were characterized by high-resolution scanning electron microscopy (HRSEM), high-resolution transmission electron microscopy (HRTEM), energy dispersion X-ray spectroscopy (EDX), Raman spectroscopy and nanoindentation. Nanoindentation results will be compared with those reported in from the literature.

2 Experimental section

2.1 Raw Materials

CVD-grown SWNTs and MWNTs were provided by Thomas SWAN Co. Ltd, U.K. The purification of both types of tube was carried out by a steam treated method which has been developed recently in the Oxford Nanotube Group [3]. In addition, the purified nanotubes were treated with a NH_4OH-alcohol solution to produce the adsorption of ethanol functional groups on the tubes. The slurry was filtered and washed thoroughly with ethanol. Finally, it was dried in air.

2.2 Preparation of Carbon Nanotube/ Glass-Ceramic Nanocomposites

The aluminoborosilicate glass was made by a modification of the ultrasonic sol-gel processing method described in [4]. Tetraethoxysilane (TEOS), aluminum sec-butoxide, trimethyl borate (TMB), and sodium acetate were used for precursors. Different weight fractions of functionalized SWNTs and MWNTs were incorporated into the sol. As-prepared CNT/glass powders were obtained and passed through a 150μm sieve. Then, the fine powders were calcined at 300°C for 4 hours to remove any organic residuals. Finally, the

composite powders were sintered by hot pressing under argon gas at 1100°C and 25MPa for 1 hour.

2.3 Characterization

The microstructure and mechanical properties of the composite materials were characterized by HRSEM (JEOL-6500F), HRTEM (JEOL 4000EX), TEM-EDX, Micro-Raman spectroscopy, XRD and nanoindentation. HRSEM of fracture surfaces and HRTEM confirmed the uniform dispersion of the nanotubes. Raman spectroscopy showed that no damage occurred to the nanotubes during the hot press process. A coating effect on both SWNTs and MWNTs was observed, induced by the *in-situ* sol-gel processing. Finally, nanoindentation results will be compared with results from the literature.

References

[1] Laurent C., Peigney A., Dumortier O. and Rousset A. "*Carbon nanotube-Fe-alumina nanocomposites: microstructure and mechanical properties of the hot-pressed composites*". Journal of European Ceramic Society, Vol. 18, pp 2005-2009, 1998.

[2] Wang X., Padture N. P. and Tanaka H. "*Contact-damage-resistant ceramic/single-wall carbon nanotubes and ceramic/graphite composites*". Nature Materials, Vol. 3, pp 539-545, 2004.

[3] Tobias G., Shao L., Salzmann G. C., Huh Y. and Green L. H. Malcolm. *"Purification and Opening of Carbon Nanotubes Using Steam"*. Journal of Physical Chemistry B, Vol. 110, pp 22318-22322.

[4] Chiou S., Hajn H. T. *"Ultrasonic sol/gel processing of aluminoborosilicate glass and its composites with carbon fibre reinforcement"*. Journal of American Society, Vol. 77, No. 1, pp 155-159, 1994.