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Investigation of yttria-doped alumina nanocomposites reinforced by multi-walled carbon nanotubes

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Abstract

Nanocomposites of Y_2O_3 doped Al_2O_3 reinforced with 2 wt% MWCNTs were fabricated by hot-pressing. The influence of a small Y_2O_3 addition (300 ppm) on the microstructure and mechanical properties of CNT-reinforced Al_2O_3 was investigated. The resulting Y_2O_3 doped Al_2O_3-CNT nanocomposites showed near theoretical densities (> 99%) with an astounding 5 times finer grain size and substantial improvements in the fracture toughness (40%), flexural strength (20%) and hardness (18%) than pure Al_2O_3, prepared under identical experimental conditions. The Y_2O_3 addition improved the density of the matrix, eliminated the residual flaws and formed YAG (yttrium–aluminium–garnet) particles at the grain boundaries in the nanocomposites. The submicron YAG particles, together with the CNTs, promoted the Al_2O_3 matrix to form fine-grained microstructures and altered the fracture mode, thus leading to higher toughness and other mechanical properties. The improved mechanical properties allows for the Y_2O_3 doped Al_2O_3-CNT nanocomposites to be used for applications in load bearing structures.

Keywords: B. Nanocomposites; C. Mechanical properties; C. Toughness and toughening; Microstructure

1. Introduction

Among structural ceramics, alumina (Al_2O_3) is appreciated for high stiffness, excellent thermal stability and relatively low density, but its extreme brittleness restricts this promising ceramic from many advanced structural applications, such as in military armour systems, as aircraft engine parts and in space engineering [1–6]. In order to overcome the brittleness issue, carbon nanotubes (CNTs) with extremely high strength and exceptional flexibility have inspired researchers to investigate the possibility of incorporating them into Al_2O_3. Several reports on the CNTs-reinforced Al_2O_3 have demonstrated large enhancements in fracture toughness, hardness and other mechanical properties [7–13]. After years of rigorous research, the role of CNTs in Al_2O_3 ceramics has been well-documented, such as hampering the densification process, refining the matrix by grain pinning, increasing the fracture toughness by crack bridging/crack deflection, improving the wear resistance by sliding/rolling, and improving the electrical/thermal properties [2–15]. In literatures, issues such as the homogenous dispersion of CNTs, achieving higher densities of the nanocomposites, understanding the toughening mechanism and Al_2O_3-CNT interfacial bonding have been intensively discussed, whilst the microstructural tuning is hardly attempted [7–15]. Tailoring the microstructures of pure Al_2O_3 and Al_2O_3 based matrices with metal oxide (MgO, Ce_2O_3, Li_2O, Y_2O_3) has been an eminent practice [16–24]. For an example, the addition of a small amount of Y_2O_3 (< 1000 ppm) has upgraded the monolithic Al_2O_3 to a material with higher densities (99%), finer grains and higher mechanical properties. The role of Y_2O_3 in improving the densification, microstructures and mechanical properties of the ZrO_2-reinforced Al_2O_3 nanocomposites has been discussed

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[20–24]. Built on top of these successes, the study of potential advantages of Y_2O_3 in CNT-reinforced Al_2O_3 ceramics is thus an interesting subject.

This paper describes the fabrication and characterisation of Y_2O_3-doped Al_2O_3 matrix nanocomposites reinforced by CNTs, aiming to show the influence of a small amount of Y_2O_3 on the structure (densities, grain size, residual porosity), mechanical properties (fracture toughness, flexural strength, hardness) and the fracture mode of the Al_2O_3-CNT nanocomposites.

2. Experimental procedures

2.1. Materials and manufacturing process

Well-mixed powders of 300 ppm Y_2O_3 and pure Al_2O_3 reinforced with 2 wt% of CNTs were hot-pressed to fabricate the nanocomposites, as listed in Table 1. Fig. 1a shows the transmission electron microscopy (TEM) image of the multi-walled CNTs used in this study, supplied by Tsinghua University, Beijing, China, and the CNTs exhibited an average diameter $\varnothing < 40$ nm. We first suspended the CNTs into distilled water by adopting a colloidal chemistry technique, as described earlier [12]. Al_2O_3 nanoparticles having a mean particle diameter of ~40 nm (Sigma-Aldrich, UK) were separately suspended into distilled water with the aid of 60 min sonication, then the desired amounts of Y_2O_3 nanoparticles (∅25–30 nm, Sigma-Aldrich, UK) were slowly added to them. The suspension was sonicated for another 60 min, in order to obtain an even distribution for better results. Subsequently, the well-dispersed CNT/water suspension was poured into the Al_2O_3 and Y_2O_3 mixture water suspension, and the combined suspension was again sonicated for 120 min to assure a thorough mixing of the nanocomposite constituents. The mixture was dried at 130–150 °C to acquire well-mixed powders. A typical example of well-dispersed ceramic nanoparticles covering a single CNT, as revealed by TEM, is shown in Fig. 1b. The dry powders were then hot-pressed in the form of discs of ∅ 32 ± 0.5 and 3 ± 0.5 mm thick, at the University of Mining and Technology, Beijing, China. Hot-pressing was carried out at 1600 °C for 60 min, under a constant pressure of 40 MPa during the whole sintering process under vacuum of 6.2×10^{-2} Pa. Pure and Y_2O_3-doped Al_2O_3 reference samples, without CNTs, were also fabricated.

Table 1
Mechanical properties of Y_2O_3 doped CNT-reinforced Al_2O_3 nanocomposites.

Sample designation	Matrix material	CNT contents (wt%)	Y_2O_3 contents (ppm)	Vickers hardness (*HV*) GPa	Flexural strength (σy) MPa	Fracture toughness (K_{IC}) MPa $m^{1/2}$
A0	Al_2O_3	–	0	16.0 ± 0.3	357 ± 27	3.0 ± 0.2
A3Y	Al_2O_3	–	300	16.5 ± 0.2	359 ± 22	3.2 ± 0.3
A2C	Al_2O_3	2	0	18.2 ± 0.2	367 ± 18	4.3 ± 0.3
A2C3Y	Al_2O_3	2	300	19.4 ± 0.2	442 ± 15	5.0 ± 0.1

2.2. Density and structural characterisation

Apparent densities of all sintered samples were measured by the water buoyancy (Archimedes) method using distilled water. Prior to measurements, surface cleaning of all samples was performed by using fine SiC paper. Theoretical densities for Al_2O_3, Y_2O_3 and CNTs of 3.99 g/cm^3, 5.01 g/cm^3 and 1.85 g/cm^3 respectively, were used for relative densities calculations in this work [10,25].

Phases of sintered samples were acquired using a Cu K_α radiation from a X-ray diffraction machine (D8 Advanced X-ray Differactometer manufactured by Bruker Corporation), and were identified with the help of Bruker Advanced X-ray Solutions computer software. Fracture surfaces of selected samples were studied using a Philips/FEI scanning electron microscopy (SEM). For detailed grain boundaries study and accurate determination of the grain size, all solid samples were finely polished up to 1 μm and then thermally etched in a tube furnace under an Ar atmosphere at 1400 °C for 15 min. Prior to SEM observation, all samples were coated with a thin layer of Au. An interesting and convenient chemical etching technique was also adopted to prepare samples for TEM analyses. Before chemical etching, the solid nanocomposite sample was milled to fine size and immersed in NaOH solution for several weeks then rinsed repeatedly with distilled water. The residue was finally transferred to a carbon coated lacey copper grid for subsequent investigation using a JEOL 2000 FX TEM.

2.3. Mechanical properties evaluation

All sintered discs were sliced into rectangular bars of 25 ± 1.5 mm (length) × 2 ± 0.15 mm (breadth) × 2.5 ± 0.20 mm (height) using a diamond cutting disc, which were subjected to three-point bending test using a 20 mm bending span and 0.50 mm/min testing speed. Eq. (1) was used for the flexural strength (σ_f) assessment, where F is the load at the fracture point, L the span length, b the sample breath and d is the sample thickness [12].

$$\sigma_f = \frac{3FL}{2bd^2} \tag{1}$$

Small samples of 10 ± 1.0 (length) × 10 ± 1.0 (breadth) × 4 ± 0.25 (thickness) mm were also prepared for hardness evaluation. Prior to testing, all samples were cold mounted using epoxy resin, and then ground on diamond pads of 120 and 220 grit and polished to 6 μm and 1 μm by using DP-suspension on polishing cloths. 9.8 N loads for 15 s was selected on a M-400 hardness tester (LECO, Japan) for the microhardness testing and the Vickers hardness number (*HV*) were further converted to GPa [6]. Young's modulus of all samples was calculated by applying Eq. (2). In which E is Young's modulus, ρ is the bulk density of the sample, V_c is the compression velocity and V_s the shear velocity [26].

$$E = \frac{V_s{}^2\rho(3V_c^2 - 4V_s^2)}{V_c^2 - V_s^2} \tag{2}$$