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Original Article

Graphene or carbon nanofiber-reinforced zirconia composites: Are they really worthwhile for structural applications?

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ABSTRACT

The use of allotropic phases of carbon (i.e. nanotubes, graphene or carbon nanofibers) as second phases to design ceramic composites is a hot topic at present. Researchers try to provide a remarkable improvement of the parent ceramic assuming that some of the outstanding mechanical properties of these phases migrate to the resultant composite. This reasonable idea has been questioned severely in the case of nanotubes addition but there is not any analysis for the other two phases cited previously. To elucidate this question, zirconia was selected as a model ceramic. This paper reports the mechanical properties of zirconia composites reinforced either with graphene or carbon nanofibers, with special emphasis on the high-temperature plasticity.

1. Introduction

Zirconia (3Y-TZP) and/or zirconia-based composites are interesting multifunctional materials that have been used for many applications, such as solid-oxide fuel cells, oxygen sensors and ceramic membranes, due to their good high-temperature stability, high breakdown electrical field or large energy bandgap [1]. These applications are due to its excellent properties: biocompatibility, high fracture toughness, high strength and low wear rates [2]. Monolithic 3YTZP has been extensively studied since last decades, and grain-boundary sliding GBS is recognized as being the primary deformation mechanism responsible for the high-temperature superplastic behavior accommodated by diffusion [3,4].

A usual strategy to improve the properties and minimize their main drawback, i.e. their brittleness [5,6] is through the design of ceramic-ceramic composites either by tailoring complex microarchitectures (as in the case of laminates and FGMs) or by reinforcing the matrix with the addition of a second phase [7].

There are several strategies to improve the mechanical properties of zirconia ceramics with a reinforcing second phase. For instance, doping with another cation: Bernardi-Martín et al. [8,9] studied the high-temperature plasticity of ceria-doped zirconia nanostructured polycrystals. Another approach has been the design of composites:

Calderón-Moreno et al. [10] studied the high temperature creep of alumina-zirconia composites. The use of nanocrystalline-based specimens was the strategy of Lorenzo-Martín et al. [11]. They studied the creep of nanocrystalline YTZP ceramics with a glassy phase and found grain boundary sliding as the deformation mechanism. They found that the mechanical behavior depends critically on the glassy phase content [12]. Another way to improve the mechanical properties of advanced ceramics materials is by addition of nanofibers (NF) in the so-called fiber-reinforced ceramic composites, which show significant improvements in toughness over monolithic ceramics. Fibers/whiskers usually have diameters in-between tens of nanometers to tens of micrometers and lengths around several micrometers to hundreds of micrometers, which are embedded inside a fine-grained ceramic matrix [13]. To this purpose, whiskers of a harder material such as SiC have been used traditionally. Calderón-Moreno et al. [14] studied the high-temperature deformation of $ZrO_2-Al_2O_3$ / SiC whisker composites.

In recent years graphene, the two dimensional array of a one-atom thick layer of carbon atoms arranged in a honeycomb lattice, is a promising candidate. Graphene has a large specific surface area ($2630\text{ m}^2\text{ g}^{-1}$), high intrinsic electron mobility ($2\cdot 10^5\text{ cm}^2\text{ v}^{-1}\text{ s}^{-1}$) [15,16], high Young modulus ($\approx 1.0\text{ TPa}$) [17], a fracture strength of 130 GPa [18], thermal conductivity ($\approx 5000\text{ W m}^{-1}\text{ K}^{-1}$) [19], high optical transmittance ($\approx 97.7\%$) and therefore it is fine for applications

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such as transparent conductive electrodes [20,21]. Chemically modified graphene has been studied in the context of many applications, such as polymer composites, energy-related materials, sensors, ‘paper-like’ materials, field-effect transistors, and biomedical applications [22,23]. Graphene platelets (GPLs) are formed by several layers of graphene with thickness of up to 100 nm [24] and called as graphene nanosheets, multilayer graphene nanosheets or graphene nanoplatelets (GNPs). Rincón et al. [25] have reported the mechanical properties and electrical conductivities of alumina-zirconia composites reinforced with either graphene oxide or carbon nanofibers. Their mechanical properties changed modestly, with slight decrease of hardness and a tiny increment of the fracture toughness. On the contrary, the electrical conductivity increases remarkably, reaching the maximum value for 2 vol% graphene oxide concentration. Chen et al. [26] fabricated graphene-reinforced zirconia ceramics using field-assisted sintering and reported a value of fracture toughness of $15.3 \text{ MPa m}^{1/2}$, as tested by the single-edge notched beam method, which reflects an increase of 61% compared to GNPs-free 3Y-TZP. On the other hand, with higher GNPs content, the improvement in fracture toughness is limited. There is a tiny increment of hardness compared to the monolithic material. Shin et al. [27] sintered reduced graphene oxide reinforced yttria-stabilized zirconia composite ceramics by spark plasma sintering and reported that the hardness decreased slightly with reduced graphene oxide (RGO) addition, whereas the fracture toughness slowly increased from 4.4 to $5.9 \text{ MPa m}^{1/2}$. The RGO pull-out and crack bridging contributed to the improved fracture toughness. Liu et al. [28] sintered zirconia toughened alumina (ZTA) adding GNPs as the secondary phase (0.8 vol %) by SPS and obtained a value of fracture toughness of $9.05 \text{ MPa m}^{1/2}$ (40% of increase respect to material without GNPs). On the other hand, the hardness decreased from 17.5 GPa to 11.1 GPa in the worst case. Rincón et al. [29] fabricated fully stabilized zirconia laminates comprising graphene oxide-enriched (GO) layers by colloidal processing and found that the presence of GO-rich layers stops the crack without deflection or bifurcation. The hardness was 17.6 GPa for all layers.

Other carbon phases have been considered in literature with the aim of improving the mechanical properties of zirconia. Regarding carbon nanotubes (CNTs), a review reports the main achievements and limitations of this approach [30].

In the field of ceramic science, carbon nanofibers (CNFs) have been considered as a reinforcement phase in potential systems such as AlN [31] or in Al_2O_3 matrix ceramics at room temperature [32] and at high temperature [33] because of their superior advantages compared with single-walled or multi-walled carbon nanotubes; i.e. their low price and their good dispersibility. Carbon nanofiber-reinforced zirconia composites (ZrO_2 -CNF) have been the object of study and few results are reported in literature. Duszová et al. [34] sintered ZrO_2 -CNF and found a decrease in the values of Vickers hardness and fracture toughness as compared to monolithic ZrO_2 . Dusza et al. [35] achieved a decrease of hardness and fracture toughness for ZrO_2 -CNF sintered by hot pressing and spark plasma sintering with 2.0 and 3.3 vol.% of CNFs respect to pure zirconia.

Until now, there is not a systematic study on the high-temperature plasticity of these composites. This work is aimed at covering this lack of information. A comparison between room-temperature performance and high-temperature creep is carried out, with special emphasis on the deformation mechanisms.

2. Experimental procedure

2.1. Starting materials

Tetragonal zirconia powder stabilized with 3% mol of Y_2O_3 (TZ-3YSE, Tosoh, Japan) with an average particle size of 90 nm and a specific surface area of $6.7 \text{ m}^2 \text{ g}^{-1}$, was used in this study. GO (Nanoinnova Technologies, Spain), with a length of 1–4 μm , a thickness of 0.7–1.2 nm and a surface area of $103 \text{ m}^2/\text{g}$ was used as secondary

phase. CNFs (GANF – Grupo Antolin, Spain) with an outer diameter of 20–80 nm, a length of more than 30 μm and a surface area of $150\text{--}200 \text{ m}^2 \text{ g}^{-1}$ were also used as secondary phase. For more details on the materials features, see [36,37].

2.2. Powder processing methods

The colloidal stability of zirconia powder was studied measuring the zeta potential of diluted suspensions (10^{-1} g l^{-1}) prepared in deionized water as a function of deflocculant content, using an ammonium salt of polyacrylic acid (PAA, Duramax D3005, Rohm & Haas, USA) as a deflocculant. For the dispersion of CNFs in water a copolymer of polyacrylic acid with polyethyleneglycol (Hypermer KD 7, Uniqema, UK) was used. GO readily disperses in water without the addition of any deflocculant [36].

Zeta potential measurements were performed by laser Doppler velocimetry (Zetasizer NanoZS, Malvern, UK). $\text{KCl } 10^{-2} \text{ M}$ was used as inert electrolyte to maintain the ionic strength. Suspensions for zeta potential measurements were prepared by mechanical agitation during 20 min followed by 1 min sonication (dr Hielscher, UP400S, Germany). pH was maintained at pH 9–10, which is the natural pH of zirconia suspensions, while in the case of CNFs that pH was reached adding tetramethylammonium hydroxide (TMAH).

Concentrated suspensions of zirconia with GO and with CNFs were prepared to a solids loading of 35 vol.% using the optimized contents of deflocculant and different sonication times. The suspensions were prepared by dispersing first the CNFs or the GO in water with 2 min sonication. In a second step zirconia was added and sonicated for different times in order to determine the optimum sonication time. Rheological measurements were performed using a rotational rheometer (MARS, Thermo Haake, Karlsruhe, Germany) under controlled shear rate conditions at 25°C and using a double-cone and plate measuring system.

The optimized suspensions were frozen in a rotatory chamber using liquid nitrogen as refrigerant (-196°C). The frozen suspensions were introduced in a freeze-dryer (CRYODOS-50, Telstar, Spain) for 24 h. The condenser temperature was -50°C , and the conditions of the storage camera were 20°C and 0.050 mbar [36]. The resultant mixtures of powders contained 98 vol.% of zirconia (Z) and 2 vol.% of either GO or CNFs (e.g. 0.5 wt%). Those mixtures are denoted in this study as Z-GO and Z-CNF, respectively.

As a reference material the as-received pure zirconia commercial powders without any treatment were used. This powder is denoted as Z.

2.3. Sintering

Bulk composite samples were sintered using a SPS process (Dr. Sinter Lab Inc., Model 515 S, Kanagawa, Japan (pulsed high DC current 20 V, 0–1500 composite samples were sintered using a SPS A)). The powder mixtures were poured into a graphite die of 20 mm in diameter. A sheet of graphitic paper was placed between the punch and the powder and between the die and the powder for easy removal of the sintered sample. The sintering process was conducted under a vacuum of 4–6 Pa. A uniaxial pressure of 75 MPa was applied throughout the sintering cycle. The sintering temperature was increased to 1300°C at a rate of $100^\circ\text{C}/\text{min}$. The holding time was 5 min. The temperature was measured and controlled using an optical pyrometer. Shrinkage, displacement, heating current, and voltage were also recorded during the sintering process. The bulk density of the samples was measured by the Archimedes method with distilled water as the immersion medium using 6.10, 2.1 and $2.1 \text{ g}/\text{cm}^3$ as densities of zirconia, GO and CNFs, respectively.

2.4. Microstructural characterization

Raman spectroscopy (Model LabRAM Horiba Jobin Yvon, Horiba

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