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Carbon nanofibers replacing graphene oxide in ceramic composites as a reinforcing-phase: Is it feasible?

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ABSTRACT

In recent years, the interest of graphene and graphene-oxide has increased extraordinarily due to the outstanding properties concurring in this material. In ceramic science, the possibility of combining excellent electrical conductivities together with an enhancement of mechanical properties has motivated the research in fabrication of graphene oxide-reinforced ceramic composites despite the intrinsic difficulties for sintering. In this work a comparison is made between graphene oxide-reinforced alumina composites and carbon nanofiber-reinforced alumina ones. It will be concluded that the improvement of mechanical properties is scarce, if any. Since carbon nanofibers have also a good electrical conductivity their importance for future applications as a replacement of more sophisticated but expensive graphene-based ceramic composites will be stressed.

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1. Introduction

Advanced ceramics, such as alumina (Al_2O_3), have been used in materials industry and have potential applications covering high speed cutting tools, dental implants, chemical and electrical insulators, wear resistance parts and some coatings. These applications are due to its excellent properties: high hardness, chemical inertness and high electrical and thermal insulation properties [1].

A usual strategy to improve the properties and minimize their main drawback, i.e. their brittleness, is the addition of a second phase, i.e. by fabrication of 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 ($2 \times 10^5\text{ cm}^2\text{ v}^{-1}\text{ s}^{-1}$) [2,3], high Young modulus ($\approx 1.0\text{ TPa}$) [4], a fracture strength of 130 GPa [5] thermal conductivity ($\approx 5000\text{ Wm}^{-1}\text{ K}^{-1}$) [6], high optical transmittance ($\approx 97.7\%$) and therefore it is fine for applications such as transparent conductive electrodes [7,8]. Chemically modified graphene

(CMG) has been studied in the context of many applications, such as polymer composites, energy-related materials, sensors, 'paper-like' materials, field-effect transistors (FET), and biomedical applications [9,10]. Graphene platelets (GPLs) are formed by several layers of graphene with thickness of up to 100 nm [11] which are called graphene nanosheets (GNS), multilayer graphene nanosheets (MGN) or graphene nanoplatelets (GNPs).

Carbon-based materials, like carbon fibers or carbon nanotubes (CNTs) have been used in the last decade to improve the mechanical properties of a variety of materials. CNTs, with high tensile strength and stiffness, good flexibility, and low density have been used to this purpose. A large number of studies have been reported. Since the discovery of carbon nanotubes by Iijima in 1991 [12], this material has also promising properties such as high tensile strength (2.7 GPa), a Young modulus of 230 GPa and an electrical resistivity of $1 \times 10^{-3}\text{ }\Omega\text{ m}$ [13,14]. Due to these properties, carbon nanotubes have found many applications as electrode materials in lithium ion secondary batteries, electrode materials for supercapacitors, and supporting materials for metal nanoparticles [15]. Many studies have been devoted to the improvement of the fracture toughness of Al_2O_3 by CNTs additions. The results show a large scatter in the values of the hardness and fracture toughness, probably due to the wide range of types of nanotubes, the very different degrees of CNTs dispersion and the extreme difficulty to achieve a fully

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homogeneous microstructure. This fact opens several doubts on the reproducibility of the mechanical response of CNT-based composites. A review on the mechanical properties of CNTs-reinforced ceramics is reported by Zapata-Solvas et al. [16].

Another way to improve the mechanical properties of advanced ceramics materials is by addition of fibers (NF) in a so-called fiber-reinforced ceramic composite, which show significant improvements in toughness over monolithic ceramics. Fibers 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 [17]. Ostertag et al. [18] fabricated SiC-fiber reinforced alumina matrix composites using slip casting and achieved a 30% improvement of fracture toughness over pure material. Hansson et al. [19] sintered SiC-whisker reinforced alumina composites by hot-pressing and this material exhibited a 100% increase in fracture toughness. In the case of carbon nanofibers, their applications are numerous because of their outstanding electrical, mechanical and catalytic properties. A review on the matter is provided elsewhere [20]. In the field of ceramic science, carbon nanofibers have been considered as a reinforcement phase in potential systems such as AlN because of their superior advantages compared with single-walled or multi-walled carbon nanotubes; i.e. their low price and their good dispersibility [21]. Carbon nanofiber-reinforced alumina composites have been the object of study and a few results are reported in literature. Maensiriet al. [22] fabricated these composites by hot-pressing at 1450 °C achieving a mean grain size $\sim 1 \mu\text{m}$. The fracture toughness improvement of $\sim 13\%$ is found for a 2.5 vol% CNF-reinforced alumina composite, although the hardness decreased slightly to 16.2 GPa (with respect to a hardness in pure alumina equal to 19.1 GPa).

In the case of graphene there are several studies in this field: Yazdani et al. [23] reported a 63% increase in fracture toughness by using a hybrid mixture of multiwalled nanotubes and graphene platelets as reinforcement phase in Al_2O_3 matrix. The role of GPLs and CNTs has been investigated separately. Wang et al. [24] prepared graphene nanosheets/ Al_2O_3 composites by SPS and reported an increase of 53% in their fracture toughness. Porwalet et al. [25] fabricated graphene-reinforced alumina nanocomposites by SPS also and found that toughness increased in all cases compared to that pure alumina. The highest value of toughness was reported in composites of $\text{Al}_2\text{O}_3 + 0.8 \text{ vol}\%$ graphene ($3.90 \pm 0.13 \text{ MPa m}^{1/2}$) although in detrimental of the hardness. Kim et al. [26] studied graphene/alumina nanocomposites and reported an enhancement of fracture toughness of 75% ($5.60 \text{ MPa m}^{1/2}$) compared with that in pure Al_2O_3 . Yazdani et al. [23] found that the average fracture toughness of the nanocomposites reached up to $5.7 \text{ MPa m}^{1/2}$, against $3.5 \text{ MPa m}^{1/2}$ of the pure alumina. Centeno et al. [27] reported that the addition of 2 wt% of graphene in the alumina composite gave rise to a fracture toughness increase up to values as high as $7 \text{ MPa m}^{1/2}$. Rincon et al. [28] 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 tiny increment of the fracture toughness. On the contrary, the electrical conductivity increases remarkably, reaching the maximum value for 2 vol% graphene-oxide concentration.

The main goal of this study is the analysis of the mechanical enhancement of hardness and fracture toughness in graphene-oxide reinforced alumina ceramics and compared the results with those found in carbon-fiber-reinforced alumina composites. All the composites are fabricated by spark plasma sintering. A critical analysis of the comparison can be useful to decide whether it is worth fabricating graphene-reinforced ceramic composites commercially despite their much higher economical cost.

2. Experimental procedure

2.1. Starting materials

α -alumina powder (Celarox, Conclea HPA05), with an average particle size of $0.35 \mu\text{m}$ and a surface area of $9.5 \text{ m}^2/\text{g}$, was used in this study. Graphene oxide (Nanoinnova Technologies), with a thickness of $1\text{--}4 \mu\text{m}$, a length of $0.7\text{--}1.2 \text{ nm}$ and a surface area of $1030 \text{ m}^2/\text{g}$ was used as a secondary phase. On the other hand, carbon nanofibers (Grupo Antolin) with an outer diameter of $20\text{--}80 \text{ nm}$, a length of more than $30 \mu\text{m}$ and a surface area of $1500\text{--}2000 \text{ m}^2/\text{g}$ were also used as a secondary phase in another alumina-reinforced composite. A simple calculation shows that the critical volume concentration to cover all the grain boundaries with graphene is given by $\left(1 + \frac{\rho d \sigma}{6}\right)^{-1}$ with ρ , σ and d standing for the graphene density, its surface area and the average grain size of alumina respectively. Assuming that the density of graphene is 2.2 g cm^{-3} , the surface area given above and $d \cong 500 \text{ nm}$, the optimal ratio for covering all the grain boundaries with graphene would be approximately 0.5 vol% graphene versus 99.5 vol% alumina. Assuming a similar density for the carbon fibers, a similar ratio was considered. However, in the case of fibers, the aspect ratio of the fibers and their topology around the grains cannot permit to assess a full overlapping of the boundaries. To the purpose of fabricating a composite with the optimal mechanical properties, a ratio close to the theoretical one is chosen: 2 vol% graphene. This is consistent with the achievements reported by Maensiri et al. commented previously [22].

2.2. Powder mixture preparation

Firstly, graphene oxide was dispersed in water and was further sonicated for 2 min and alumina was also sonicated using different times to this end. Solution had a pH of 10 and it was constant during this process. The best conditions to prepare the solution was 45% vol and for 5 min of ultrasonication. The final solution was lyophilized. The resultant mixture of powders was 98 vol% α -alumina (A) and 2 vol% graphene oxide (GO). This mixture is denoted in this study A-GO.

In the case of powder nanofibers the powder mixture consisted of 98 vol% α -alumina (A) and 2 vol% carbon nanofibers (CNF) and they were prepared similarly. This mixture is denoted in this study A-CNF.

Pure alumina was prepared from the as-received commercial powders for the sake of comparison. This powder is denoted as A.

2.3. Spark plasma sintering and characterization

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 A). The powder mixtures were poured into a graphite die of 15 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 $1300 \text{ }^\circ\text{C}$ at a rate of $100 \text{ }^\circ\text{C}/\text{min}$. 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 sintered samples were grounded and polished to $0.5 \mu\text{m}$ using SiC paper and diamond suspension. The bulk density of the samples was measured by the Archimedes method with distilled water as the immersion medium using 3.98, 2.1 and 2.1 g/cm^3 as densities of alumina, graphene oxide and car-

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