



Tape casting of alumina/zirconia suspensions containing graphene oxide

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Abstract

The introduction of carbon derivatives (nanotubes, graphene, etc.) as a second phase in ceramic matrices has limitations arising from their difficult processing. This paper studies the colloidal stability and the rheological behaviour of concentrated suspensions of alumina with 5 vol.% Y-TZP (AZ) and the effect of the addition of 2 vol.% of graphene oxide (AZGO) on the suspension stability, rheological behaviour and tape casting performance. The colloidal stability was studied using zeta potential measurements in terms of concentration of deflocculants and pH and homogenisation was optimised adjusting the sonication mode and time. The best results were obtained for pulsed mode. The optimum rheological properties were obtained for solid loadings of 53 vol.% and 40 vol.% for AZ and AZGO. Homogeneous, flexible tapes with thickness of $\sim 120 \mu\text{m}$ were obtained reaching densities of $>60\%$ of theoretical density in which secondary phases are well dispersed.

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1. Introduction

Carbon derivatives such as carbon nanofibers (CNFs),¹ carbon nanotubes (CNTs)^{2,3} and graphene⁴ are receiving increased attention in several technologies owing to their unique combination of electrical and mechanical properties that make them excellent candidates to be used either in functional applications^{5–7} or as structural reinforcement in the production of composite materials.^{8–11} However, the manufacture of ceramic composites containing those carbon derivatives as a dispersed phase has important limitations due to the difficulties to obtain a uniform dispersion of the carbonaceous phase inside the ceramic matrix, on one hand, and the degradation of their properties after sintering, on the other hand. Therefore, these two main drawbacks need to be studied in-depth to allow an easy preparation of the composites with conventional ceramic processing techniques.

The dispersion of the carbonaceous reinforcing phase into a ceramic matrix is complicated due to the strong tendency of these compounds to agglomerate as a consequence of their inherent

hydrophobicity and their high specific surface areas; however, achieving a good dispersion in the matrix is crucial for obtaining materials with uniform microstructures and enhanced properties. To overcome this problem different strategies have been followed.^{12,13}

In first place, the dispersion of graphene can be achieved by wet milling the ceramic powders and graphite in ethanol, obtaining in just one step the exfoliation of the graphite into graphene and the uniform mixing of both powders; however, using this preparation technique some graphite agglomerates remain in the final material and the graphene flakes produced are of poor quality.¹⁴ This mechanical milling approach has been also used replacing the initial graphite by a solution of graphene platelets in dimethylformamide, leading to better dispersion results.¹⁵

In second place, the colloidal process has proved to be a most effective method to get the correct dispersion of the nanofibers in ceramic matrices, allowing control over all the stages of the process thus leading to materials with better properties.^{16,17}

A third approach used successfully to prepare carbon-reinforced ceramics has been the heterocoagulation route^{18,19} which is suitable to promote the formation of a coating of the dispersed phase onto the surfaces of the particles constituting the matrix by core–shell reactions or simply by electrostatic interactions. The heterocoagulation is very effective when matrix and

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secondary phase are quite different in shape or size. Otherwise, heterocoagulation of similar particles leads to agglomerates formation.

The use of graphene oxide (GO) is presented as an alternative to reduced graphene for obtaining stable suspensions,²⁰ due to the presence of functional groups on their surface; the GO has a polar character that favours the electrostatic repulsion between the particles and makes it to readily disperse in polar solvents such as water. However, the surface oxidation helps to create structural defects in the graphene sheets so that their properties are worse than those presented by reduced graphene. Hence, materials obtained with GO must be reduced in a further step, normally by using sintering cycles under reducing atmosphere.²¹

Most papers dealing with the processing of GO–ceramic composites make use at some stage of a colloidal dispersion; however, in most cases the suspension/solution is dried and the resulting powder mixture is then compacted by pressing methods. Axial pressing can lead to the introduction of defects in the material, and limits the shaping capabilities. Colloidal shaping techniques allow the manufacture of complex shaped parts and tailored microarchitectures while maintaining a high reliability and microstructural uniformity. In particular, tape casting appears as an excellent option to design composites with such complex structures. This technique has been largely used in the manufacture of electroceramics, but tape casting is very useful for the production of structural materials too, as the green tapes can be easily laminated, machined and shaped, for the production of coatings and laminates.^{22,23} However, to the best of our knowledge, the production of substrates of GO-reinforced ceramics by conventional ceramic processing has not been yet described.

The final objective of this investigation is to study the mechanical performance of GO-reinforced alumina–zirconia multilayers. For such purpose it is necessary to obtain the individual tapes of the composition constituting the layers of the composite. In this context this paper describes the early stages of their manufacture by an aqueous tape casting process. As ceramic material a mixture of alumina with 5 vol.% Y-TZP (referred to as AZ) was used, and GO was used as nanodispersoid phase. One common strategy to produce materials with enhanced properties is by designing laminates with strong interfaces, the most well-known case study being that formed by alumina–zirconia layers. However, the effect of GO on the processability and mechanical response of those laminates is unknown. The most important issue is to demonstrate the capability to produce homogeneous tapes of alumina–zirconia–GO as an example of single layer with complex composition, thus providing the potential to investigate different multilayers and investigate the properties.

The tape casting performance was studied measuring the rheological behaviour of concentrated suspensions of AZ and AZGO as a function of different parameters, including dispersing conditions (content of deflocculant and pH), solids loading, homogenisation procedure (continuous and pulsed sonication), etc. Further, the effect of tape casting additives (binder and defoamer) was also studied in order to produce homogeneous tapes without large macro-defects and easy to handle.

2. Experimental

Two commercial ceramic powders were used as starting materials: an α -alumina powder (Ceralox, Condea HPA05, USA), with an average particle size of 0.35 μm and a specific surface area of 9.5 $\text{m}^2 \text{g}^{-1}$, and a nanosized zirconia doped with 3 mol% of Y_2O_3 (TZ-3YE, Tosoh, Japan) with an average particle size of 100 nm and a specific surface area of 14.5 $\text{m}^2 \text{g}^{-1}$. A monolayer graphene oxide (Nanoinnova Technologies, Spain), simply referred to as GO, with average lengths and thicknesses in the range of 1–4 μm and 0.7 to 1.2 nm, respectively, and a surface area of $\sim 103 \text{m}^2 \text{g}^{-1}$ was also used as a secondary reinforcing phase.

Suspensions of these powders and their mixtures were prepared in deionised water and stabilised adding as a deflocculant an ammonium salt of poly(acrylic acid), PAA (Duramax D3005, Rohm & Haas, Philadelphia, USA) with low molecular weight (2400 g mol^{-1}) available as a 35 wt% aqueous solution which provides electrosteric stabilisation at moderate pH values.

Colloidal stability was evaluated through zeta potential measurements that were performed using a Zetasizer NanoZS instrument (Malvern, UK), based in the laser Doppler velocimetry technique. The suspensions were prepared to a solid content of 10^{-1}g l^{-1} using 10^{-2}M KCl as an inert electrolyte in order to maintain the ionic strength. Suspensions containing different dispersant concentrations were tested. Given concentrations are referred to as wt% on a dry solid basis.

The rheological behaviour of a concentrated alumina suspension containing zirconia as secondary phase (simply referred to as AZ) was firstly studied. When the properties of this one were suitable for tape casting, alumina–zirconia suspensions containing graphene oxide as a dispersed phase (referred to as AZGO) were then prepared by considering the same rheological parameters. In all cases the relative volume ratio of alumina to zirconia was 95:5 vol. (92.5:7.5 wt). In the AZGO suspensions graphene oxide was added to a concentration of 2 vol.% (0.5 wt%) referred to dry ceramic powders and maintaining constant the A/Z ratio.

The AZ suspensions were prepared to solid loadings of 47, 50, 53 and 55 vol.% (78, 80, 82 and 84 wt%, respectively), whereas in the AZGO suspension the solid loadings studied were 40 and 50 vol.% (71.5 and 80.1 wt%, respectively) since the incorporation of GO led to significantly higher viscosities thus limiting the final solids loadings.

The suspensions were prepared under continuous mechanical stirring with helices, using the following protocol of sequential addition. Firstly, the zirconia powder was added to deionised water and it was homogenised by sonication for 1 min. The required amount of PAA required to stabilise the zirconia particles (1.0 wt% PAA in relation to the zirconia content) was then added and the mixture was maintained under mechanical stirring for 10 min to allow the deflocculant to adsorb onto the particle surfaces. Afterwards, the relative amount of PAA needed to disperse the submicron-sized alumina particles was added (0.8 wt% PAA in relation to the alumina content), followed by incorporation of the alumina powder and the mixture was maintained under mechanical stirring for 30 min.

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