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Effect of titania addition on the properties of freeze-cast alumina samples

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Abstract

This work investigated the behavior of TiO_2-containing α-Al_2O_3 samples prepared by the freeze-casting technique. Camphene and liquid nitrogen were used as the solvent and cooling fluid, respectively. Camphene resulted in the formation of dendritic pores, in the direction of the freeze-casting cold front during sample preparation. The formation of β-Al_2TiO_5 phase occurred at 1300 °C, and became more evident as the sintering temperatures reached 1500 °C. The TiO_2 loading did not affect the sample porosity at a given temperature, but it was detrimental in the case of mechanical properties under certain conditions. For instance, the flexural strength slightly improved with increasing the TiO_2 loading and sintering temperature from 1100 to 1300 °C. This effect was attributed to the occurrence of a more effective sintering of alumina. However, as the heat treatment temperature was raised from 1300 to 1500 °C, the flexural strength did not increase as a function of the TiO_2 loading, even though the densification occurred with loss of porosity. The loss of mechanical strength was found to be associated with the formation of microcracks which stemmed from the formation of β-Al_2TiO_5 phase for TiO_2 loadings in excess of 4 wt% at these high sintering temperatures.

Keywords: B. Porosity; D. Al_2O_3; D. TiO_2; D. Al_2TiO_5

1. Introduction

A number of processing techniques have been developed for decades for preparing porous ceramic materials. These include, among others, the replication of polymer foams by impregnation [1,2], foaming of aqueous powder suspensions [3,4], pyrolysis of pre-ceramic precursors [5,6], and heating of powder compacts with pore-forming sacrificial templates [7,8]. However, these methods cannot completely satisfy a controlled level of interconnected porosity combined with an outstanding mechanical strength. An alternative approach is to use the freeze-casting technique to obtain porous ceramic samples.

Freeze-casting is a versatile route for preparing ceramic materials with tailored pore structures [9]. It is an environmentally friendly, cost effective, and easy to scale-up method. This preparation technique consists of a stable colloidal suspension of ceramic particles which is poured into a mold, freezing the suspension, removing the dispersing medium, and sintering the obtained material. In this methodology the slurry particles are pushed away by the solidification front and trapped between the growing crystals [10]. Freeze-cast samples usually show an improved mechanical strength and a highly interconnected three-dimensional pore network [11]. This behavior can provide to these materials an enhanced fluid permeability and chemical stability [12,13]. Freeze-cast materials have been used in a range of applications, including biomaterials [14,15], sensors [16,17], drug delivery systems [18,19], photocatalysis [20,21], and gas purification [22–24].

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The addition of titania into alumina has been widely investigated for a variety of applications, including catalysts and mechanically reliable ceramics [25–28]. It is well established that the formation of an aluminum titanate (Al_2TiO_5) phase can increase the thermal shock resistance and mechanical strength of alumina [29–31]. In this work we investigate TiO_2-containing α-Al_2O_3 samples prepared by the freeze-casting technique. As far as we know, this is the first attempt to incorporate titania into freeze-cast alumina, thus allowing the examination of novel tailored pore structures coupled with mechanical properties. This study is supported by a series of experimental tests including X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Archimedes method, X-ray microtomography (μ-CT), and mechanical tests using a stage available in the μ-CT system.

2. Materials and methods

2.1. Synthesis

Fig. 1 depicts a schematic of the steps used in this work for samples preparation. Initially a solution of Texaphor 963 (Cognis, Southampton Hampshire, UK) and camphene (Aldrich/ ≥ 95%) was prepared at 60 °C. CT3000SG alumina particles (Almatis, Brazil) with a mean diameter of 1 μm and 99.8% purity were added under vigorous stirring to the solution. The as-prepared slurry was kept under sonication for 15 min. Titania (Sigma/325 mesh anatase/ ≥ 99%) was then added to the slurry. The titania loading in the prepared slurries ranged from 1 to 14 wt% of the solid concentration. Blank samples containing pure alumina were also prepared for comparison purposes. The Texaphor 963 loading was kept at 1.8 wt% of the alumina concentration. The ceramic concentration in the obtained slurries was kept at 25 vol%. Subsequently, the slurries were poured into Polytetrafluorethylene (PTFE) molds as shown in Fig. 1. The molds were placed on a high thermal conductivity copper plate. The top side of the mold was open so that the slurry surface was kept under atmospheric conditions. As a result, the camphene crystals obtained during the freezing step were stimulated to grow in the vertical orientation from the bottom to the top side. Liquid nitrogen was used as the cooling fluid. The sublimation of camphene was performed by keeping samples in air at room temperature for at least 3 days. Then, the materials were heat treated in air at 1100, 1300 or 1500 °C using a temperature-controlled Thermolab furnace (Pt30%Rh/Pt6%Rh-thermocouple) at a heating rate of 2 °C min^{-1}. The sintering time was kept constant at 2 h.

2.2. Characterization

The porosity of the sintered samples was assessed by the Archimedes method. The samples shrinkage was evaluated taking into account their diameter before and after the heat treatment step. XRD was carried out using a PHILIPS-PANALYTICAL PW 1710 diffractometer, with Cu Kα radiation and operating at 40 kV and 30 mA. XRD patterns were taken in the range of 10–80° (2θ), using a step size of 0.06°. The identification of the crystalline phases was performed using the XPert Plus software. FTIR was performed using samples prepared as pellets with KBr and examined in a PERKIN-ELMER Frontier spectrometer. The spectra were taken from 4000 to 400 cm^{-1}, with a resolution of 4 cm^{-1}

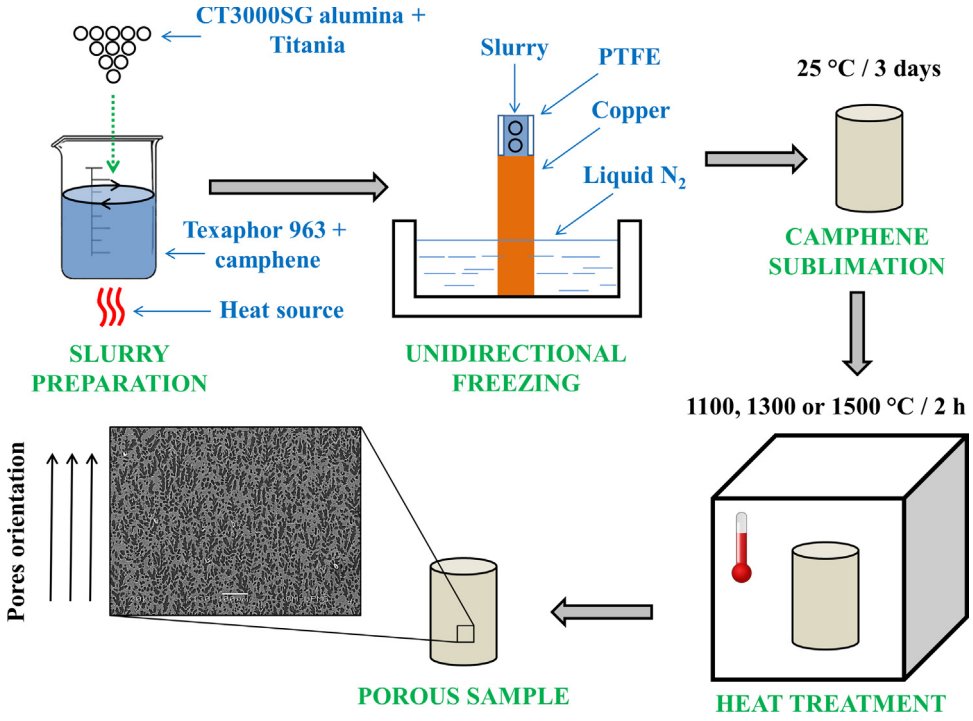

Fig. 1. Schematic of the steps used in this work for samples preparation.