Processing and Microstructural Characterization of Porous Alumina-Zirconia Ceramic Using CMC and PVC

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Processing and Microstructural Characterization of Porous Alumina-Zirconia Ceramic Using Cmc And PVC

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Abstract. Porous materials are of significant interest due to their wide application in catalysis, separation, lightweight structural materials, biomaterials and other areas. Porous ceramics are produced within a wide range of porosities and pore sizes depending on the application intended. Porosity and pore size distribution can be carefully controlled by the choice of organic composite and the amount added. The material may have two types of pores: open and closed pores. The open pores, also called interconnected pores, are those which are in contact with the external surface of the material, being very useful for the manufacture of ceramic filters. A high number of closed pores are important for the manufacturing of materials used in thermal applications. There are many methods for obtaining porous ceramics, in general consisting in adding to the ceramic matrix organic particles, which volatilize during the first heat-up. The objective of this study was to produce ceramic composite nanostructure of alumina and yttria stabilized zirconia (Y-TZP) with micrometric pore sizes. The effects of ZrO_2 additions in the mechanical properties of Al_2O_3 have been intensively investigated, due to the possible increase of the mechanical strength of this material. The organic particles used to create the pores were CMC and PVC. The microstructure of the porous ceramic samples obtained was evaluated considering the degree of sinterization of the nanoparticles, pores formation, porosity, specific surface of the pores and the distribution of the interconnecting pores.

Introduction

Porous ceramics have been used as thermal insulate, filters, membranes, gas burners and materials for bone implants, just to mention some.

Different techniques are used for creating porous ceramic, such as sacrificial template, replica, and direct foaming methods [1]. The choice of the technique depends on the desired structure and pore size.

The sacrificial template technique usually consists of the preparation of a biphasic composite, comprising a continuous matrix of ceramic particles or ceramic precursors and a dispersed sacrificial phase that is initially homogeneously distributed throughout the matrix and is ultimately extracted to generate pores within the microstructure. This method leads to porous materials displaying a negative replica of the original sacrificial template, as opposed to the positive morphology obtained from the replica technique described below.

The replica method is based on the impregnation of a cellular structure with a ceramic suspension or precursor solution in order to produce a macroporous ceramic exhibiting the same morphology as the original porous material.

In direct foaming methods, porous materials are produced by incorporating air into a suspension or liquid media, which is subsequently set in order to keep the structure of air bubbles created. In most cases, the consolidated foams are afterwards sintered at high temperatures to obtain high-strength porous ceramics [2]. The total porosity of directly foamed ceramics is proportional to the amount of gas incorporated into the suspension or liquid medium during the foaming process. The pore size, on the other hand, is determined by the stability of the wet foam before setting takes place [3].

Among the several techniques employed in the production of these materials, the addition of organic particles, which volatilize during the first heat-up, to the ceramic matrix is one of the most promising techniques. This procedure can be optimized in order to attain a better control over the porosity using the heterocoagulation of the inorganic and organic parts of a suspension.

The objective of this study was to produce ceramic composite nanostructure of alumina with 3% yttria stabilized zirconia (Y-TZP) with micrometric pore sizes.

The effects of ZrO_2 additions on the mechanical properties of Al_2O_3 have been intensively investigated, due to the possible increase of the mechanical strength of this material [4]. The organic particles used to create the pores were CMC (carboxmetilcelulose) and PVC.

Experimental procedures

Materials. Commercially available powders used in this work were the α Al_2O_3 (Op1000 - Alcan-USA) and ZrO_2 with 3% mol yttria stabilized of Shandong Zhongshun Sci. & Tech. Dev. CO. Ltd, with average size of particles between 20 and 30 nm, characterized by an equipment of the mark CILAS.

Table 1 – Chemical characteristics analysis of Alumina Op 1000 produced by Alcan-USA.

Composition [oxides]	% [weight]
Al_2O_3	> 99,50
TiO_2	0,06
Na_2O	0,05
Fe_2O_3	0,03

Table 2 – Chemical composition of 3% yttria stabilized zirconia (ZS2) produced by Shandong Zhongshun Sci. & Tech. Dev. CO. Ltd.

Componente	% m/m
$Zr(Hf)O_2$	94,8
Y_2O_3	5,2
SiO_2	0,005
Al_2O_3	0,02
Fe_2O_3	0,006
TiO_2	0,002
Cl^{-1}	0,005

The material was prepared with mixture of oxides of 82% Al_2O_3 and 18% ZrO_2. To attain the compositions, mechanical milling was performed in isopropilic alcohol with 50wt% of solids content for 5 hours. After milling, the powder mixtures were dried in a heater at 80^{o}C for 12 hours and then, deagglomerated.

Samples made of composite Al_2O_3-ZrO_2 and organic powder mixtures were compacted and sintered at 1400^{o}C.

Different compositions have been prepared varying the type and amount of organic material.

Table 2.1.3 – Type and quantity of organic materials used in the mixtures

Composite	% [weight] of organic material
$Al_2O_3 + ZrO_2$	20% CMC
$Al_2O_3 + ZrO_2$	25% CMC
$Al_2O_3 + ZrO_2$	20% CMC-PVC

Results and discussion

Figure 1 shows SEM images of fracture surface of the ceramic bodies compacted and sintered at 1400^{o}C studied in this work, with 20% and 25% of CMC and 20% PVC-CMC. The microstructure of the porous ceramic samples obtained was evaluated considering size distribution of the interconnected pores. It can be observed that the pores show some shapes similar to needles, which is typical of the presence of CMC. Figure 1c presents some bigger pores probably formed due to merging of CMC to PVC. Bigger interconnected pores are also observed due to release of gases formed during burning of organic material.

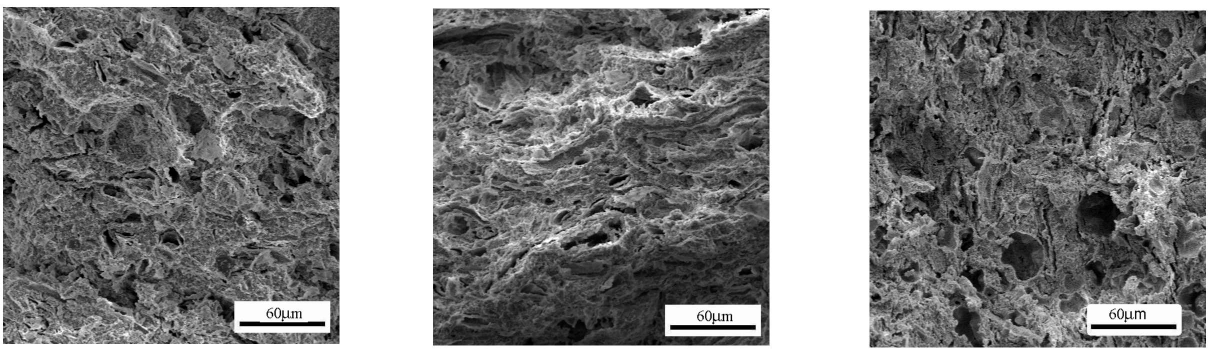

Fig. 1 - (a) SEM imagem of sintered Al_2O_3 obtained using 20% CMC (b) SEM imagem of sintered Al_2O_3, obtained using 25% CMC (c) SEM imagem of sintered Al_2O_3 obtained using 20% CMC-PVC, sintered at 1400^{o}C.

Figure 2 shows pore size distributions, obtained by Hg porosimetry of the sintered materials obtained using the modified organic particles and it displays how the pore volume and the pore size increased with the mixture of the PVC and CMC.

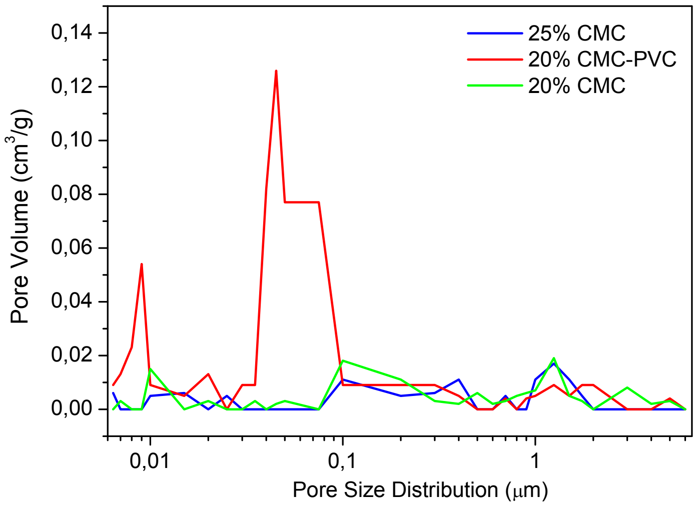

Fig. 2 – Pore size disribution in sintered ZrO_2-Al_2O_3 samples, obtained using different amounts of organic particles, sintered at 1400°C.

Conclusion

It can be concluded that with the sacrificial template techniques used so far, it is not yet possible to control the porosity and the microstructural homogeneity of the material produced. This technique is being improved to overcome this need.

In this study the best results regarding porosity and pore size distribution were obtained in the material produced with 20% CMC-PVC.

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