Microstructure characteristics of Ce-ZTA ceramic composites obtained by liquid phase sintering

H. Beck(1), M.C.A. Nono(1), F. Piorino(2)

(1) National Institute for Space Research - INPE
Av. dos Astronautas, 1758 - CP 515
12201-970 - São José dos Campos – SP - Brazil

(2) CTA-IAE-AMR
haine@las.inpe.br

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Abstract: Zirconia-toughened alumina (ZTA) ceramics with Ce-TZP (tetragonal zirconia polycrystalline doped with ceria) volume fraction on 33% were prepared with the addition of. The influence of glass infiltrated method was investigated. Coprecipitated Zr and Ce hydroxide mixture was obtained from $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ and $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$ aqueous solution. $\text{CeO}_2\text{-ZrO}_2$ calcinated powder was compacted and the compacted samples were sintered at 1180°C . Powder samples was characterized by scanning electronic microscopy (SEM), The volume fraction of each phase, the grains size and shapes, and the porosity were investigated with SEM. The relative density and shrinkage was investigate too. The results showed that the crystalline matrix was composed by $\text{SiO}_2\text{-B}_2\text{O}_3\text{-La}_2\text{O}_3\text{-Al}_2\text{O}_3\text{-Ce-TZP}$ and revealed the important role played the glassy phase in the densification of this ceramic composite.

Introduction

Zirconia-toughened alumina ceramics are attractive to the science/industry because they provide higher strength, abrasion resistance and better biocompatibility, as compared with other types of materials [1, 2 and 3]. On the other hand, previous studies show that $\text{Al}_2\text{O}_3/\text{ZrO}_2$ powder require a higher sintering temperature ($>1550^\circ\text{C}$) to achieve a comparable density relative to their constituents because the densification is significantly retarded by the presence of ZrO_2 inclusions.

Conversely, the densifications of polycrystalline matrices containing a liquid phase are much less affected by the second-phase inclusions [8]. The spreading liquid penetrates the solid-solid interfaces, which can disintegrate the solid grain structure (agglomerates) in some extent [9]. Furthermore, the lower sintering temperature by the presence of liquid phase can save the fabrication cost. However, liquid-phase sintering of zirconia-toughened alumina has received little attention.

Recently was developed a new method for making ceramics by liquid phase sintering called glass-infiltration processing [9 and 10]. The alumina-glass ceramics obtained by this method have low shrinkage, exceptional mechanical properties and homogeneous microstructure [4, 5, 6, 7, 8 and 11].

The objective of this work was prepared 12Ce-TZP/ Al_2O_3 /glass ceramics by glass-infiltration processing and investigated your microestructure and physical properties.

2. Experimental procedure

2.1. Glass composition

The composition of the powder that was made for the lanthanum borosilicate glass is given in Table 1. The glass was obtained from comercial source (Vita Zahfabrik, Germany).

Oxide	Composition (wt.%)
SiO_2	19
Al_2O_3	17
B_2O_3	12.5
TiO_2	3.7
CaO	1
La_2O_3	38
CeO_2	4.8
NaO_2	4

Table 1. Glass compositions for infiltration.

2.2. 12Ce-TZP/ Al_2O_3 matrix preparation

The Al_2O_3 powder was obtained from a commercial source (ALCOA, Brazil) and the purity of the powder was 99.98% α - Al_2O_3 . The t- ZrO_2 was obtained from our laboratory using 12 mol% CeO_2 as stabilizers. Coprecipitated Zr and Ce hydroxide mixture was obtained from $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ and $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$ aqueous solution. CeO_2 - ZrO_2 powder was calcinated at 700°C .

67 vol.% of α - Al_2O_3 powder with 33 vol. % of 12Ce-TZP powder were ball-milled to reduce agglomeration and to enhance mixing, respectively. The average particle size of Al_2O_3 and ZrO_2 were 2.6 and 0.3 μm , respectively. 12Ce-TZP/ Al_2O_3 bars (4,0 x 4,0 x 30,0 mm) were formed via slip casting and then partially sintered at 1180°C for 2 h.

2.3. Ceramics preparation

The lanthanum borosilicate glass was infiltrated into the porous Ce-TZP/ Al_2O_3 matrixes at 1140°C for 2,5h. The process of glass application and high-temperature infiltration was repeated three times in order that glass could reach the bottom of the samples. The extra glass was removed by grinding and the ceramics were ground flat with a diamond wheel. Density of the composites was measured by the Archimedes method. Shrinkage from infiltrating was measured using calipers. The bulk density of the matrix after partially sintering was measured by the Archimedes principle.

2.4. Microstructure characterization

To investigate infiltration depth and microstructure of samples before and after infiltrated, fracture surfaces were prepared and examined by scanning electron microscopy (SEM) (JSM-5310, JEOL).

3. Results and discussion

3.1. SEM images

Some short rodlike Al_2O_3 particles and very small ZrO_2 particles are observed in the fractographies of the ceramics without glass infiltrated. Ceramics microstructures consist of fused Al_2O_3 and ZrO_2 particles with many residual pores (Fig. 1). After infiltrated, the ceramics are well densified. Nonplanar fracture surface and the appearance of porous are also observed (Fig.2).

Fig. 1. Fracture surface of Ce-TZP/ Al_2O_3 (Magnification of 5000x).

Fig.2. Fracture surface of Ce-TZP/ Al_2O_3 /glass (Magnification of 5000x).

3.2. Density and Shrinkage

The shrinkage and volume density are given in Tabel 2. The sintering causes low density while infiltrating causes relatively large density. The data in Table 2 show shrinkage of ceramics decreases with the glass infiltrated. Matrixes with no glass infiltrated have a large shrinkage during sintering, resulting in the inefficient density. On the other hand, partially sintering temperature are favorable to glass infiltrated process because some open pores transform to closed pores, resulting in the stable dimension and effective infiltration

Sample	Shrinkage (%)	Density (g/cm ³)
Ce-TZP/Al ₂ O ₃	10.23	3.25
Ce-TZP/Al ₂ O ₃ /glass	0.25	4.5

Table 2. Shrinkage and density of ceramics (mean of 10 samples).

The temperature required for fully densification of the ceramics is higher than 1500 °C in the literature. Thus only porous skeletons were formed after partially sintering. Under this situation, the densification of the compacts mainly results from surface and volume diffusion improving the interparticles contacting. Enhancing sintering temperature is beneficial for surface and volume diffusion, so the strength of skeleton and ceramic can maybe increases. Bending strength and fractural toughness of ceramics will be reports in the future.

4. Conclusions

The glass infiltration process of lanthanum borosilicate glass show it was possible to make Ce-TZP/Al₂O₃ ceramics with good density and low shrinkage of the ceramics prepared by a melt-infiltration method. Dimensional control of the samples was possible too.

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