

Thermal shock behavior of dense mullite–zirconia composites obtained by two processing routes

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Abstract

Mullite–zirconia composites were prepared using two different processing routes such as reaction sintering (RS) of alumina and zircon and direct sintering of mullite–zirconia grains. For the RS process, alumina and zircon (AZ) as raw materials were used, while in the other route mullite–zirconia-electrofused grains (MZ) previously mixed with a second ceramic phase as a binder were employed. These both types of ceramics were produced by slip casting in plaster molds and finally sintered at 1600 °C for 2 h. In this work, the microstructure and the resistance to thermal shock for both high-density mullite–zirconia composites were studied. For materials characterization, density, XRD and SEM techniques were employed. Thermal shock behavior was determined by quenching in water with ΔT of 200–1200 °C and for 1–10 repeated cycles for each ΔT used. The variation of damage with thermal shock severity was followed by measuring the dynamic elastic modulus E using the impulse excitation method.

The two processing routes resulted in ceramic composites with similar phase contents, but with microstructural differences. The dependence of elastic modulus on severity of thermal shock was in accordance with literature predictions, where below a critical quenching temperature difference (ΔT_c) no degradation in the elastic behavior was found.

The thermal shock resistance increased when zircon was used as bonding phase, rather than alumina or alumina–zircon mix.

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1. Introduction

Mullite–zirconia composites are materials with important technological applications due to their good properties such as toughness, chemical stability, and high-creep resistance. In practice they are employed in the glass industry and where a high chemical and corrosion stabilities are required. Zircon and alumina are largely employed as raw materials in their manufacture [1–6]. Composites with zirconia and mullite as the main phases can be also produced from some of the commercial types of grains MZ coming from an electrofusion process. This processing route involves the utilization of finely ground MZ powder with a bonding phase (alumina, zircon, etc.) because single electrofused grains are not easy to be sintered.

Although, mullite and zirconia are the major constituents in the composite, the physical characteristics, microstructure and properties of the resulting ceramic may be different [1–11].

The first approach to determine the thermal stresses of brittle material is a thermoelastic theory [12], which is focused in the initiation of the fracture. A second approach focuses on crack propagation for conditions of thermal shock more severe than those for crack initiation [13].

A unified theory of the thermal shock resistance considering the initiation and crack propagation in brittle ceramics is due to Hasselman [14] who presented analytical solutions for the extent of the cracks as a function of the severity of the thermal shock.

The thermal shock resistance is improved by the deliberate introduction of thermal stress concentrators in the form of microstructural inhomogeneities in the material. Thus, the dispersion of zirconia grains in a mullite matrix, improves the thermomechanical behavior as a result of microcracks formation and by the dissipation of elastic energy related to zirconia martensitic transformation [15].

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There is not known a simple and universal test to evaluate the behavior of ceramic materials to thermal shock (TS) and also capable to be extrapolated to the actual conditions in service, sample geometry, and thermal cycles. However, some experimental tests consisting in sudden heating and cooling are easy to make but they have only a comparative value between similar materials. Practical tests for evaluating the thermal shock resistance determine the variation or change of some characteristic property of the test sample. The TS can be evaluated on heating or cooling, but in most methods a sudden cooling step is used because its greater severity. A method consists in heating the test probe to a desirable temperature; followed by rapid cooling to room temperature (referred to us the quenching method), by immersion in liquids such as water, oil or alcohol, etc. [16]. A characteristic mechanical property like fracture strength or elastic modulus is measured before and after quenching. In this way the severity of the treatment can be studied by determining the relative drop in mechanical strength or elastic modulus after exposure at a given thermal cycle. Also the damage in the material can be correlated after application of repeated thermal cycles.

2. Experimental

Conventional methods to evaluate the elastic modulus E are complicated and destructive. An alternative method is the dynamic E modulus determination using the impulse excitation (sonic velocity) that is a simple and non-destructive standard test [17].

In this work, the microstructure and thermal shock behavior of mullite–zirconia ceramics were studied. Two types of composites were obtained through different processing routes: one of them using RS method and the other by sintering a commercial mullite–zirconia powder using a bonding phase as a sintering aid. Both materials were formed by slip casting in plaster molds. The probes were prismatic bars because the determination of the E modulus is easier and more reproducible employing this geometry. The sintered composites (1600 °C–2 h) were characterized in terms of microstructure and physical properties. The influence of the addition of different bonding phases was also analyzed.

2.1. Preparation of mullite–zirconia composites by reaction sintering

The mullite–zirconia composite AZ was prepared using a stoichiometric mixture of alumina and zircon. Starting powders were commercially available: alumina (α -Al₂O₃, A-16SG, Alcoa Inc., USA) and zircon (ZrSiO₄, Mahlwerke Kreutz, Mikron, Germany) with unimodal narrow particle size distributions (average particle sizes: 0.6 and 1.8 μ m; specific surface areas BET: 9.5 and 4.1 m²/g, respectively).

The alumina–zircon powder mixture was prepared with an alumina to zircon weight ratio of 45.5:54.5 (wt.%), respectively. Stabilized 48 vol.% aqueous suspension was obtained using 0.24 wt.% of Dolapix CE64 (Zschimmer and Schwartz) as a dispersant at pH 9.1. Previous studies with this mixture

Table 1

Typical chemical analysis of the MZVR from ELFUSA (www.elfusa.com.br)

Oxide	wt.%
Al ₂ O ₃	44.02
TiO ₂	0.29
SiO ₂	17.96
Fe ₂ O ₃	0.11
MgO	0.09
CaO	0.18
Na ₂ O	0.16
K ₂ O	0.04
ZrO ₂	39.13

indicated that compacts prepared from suspension in this condition had low-total volume of small pores [4,5]. Therefore bars were produced from the optimal slip condition of the mixture by slip casting in a plaster mold, dried at room temperature and then at 110 °C. Finally the prismatic 8 mm × 8 mm × 50 mm bars were sintered at 1600 °C–2 h.

2.2. Preparation of mullite–zirconia composites from an electrofused MZ powder

A commercial mullite–zirconia grains was employed as starting powder. This material (MVZR, Elfusa Ltd., Brasil) is produced from pure raw materials by an electrofusion process in electric arc furnaces. The chemical analysis is shown in Table 1. Additional information provided by the manufacturer: melting point 1850 °C, apparent density 3.71 g/cm³; apparent porosity 3.0%; true density 3.74 g/cm³; reversible thermal expansion at 1400 °C 0.68%.

A size fraction with mean particle diameter (d_{50}) lower than 10 μ m (obtained by a sedimentation process) was attrition milled to reduce d_{50} to 5 μ m. This fine powder contains mullite and monoclinic zirconia (m-ZrO₂) as crystalline phases and was used as starting material (MZ).

As bonding phases were used: α -alumina (α -Al₂O₃, d_{50} = 0.45 μ m, A-16SG, Alcoa Inc., USA) and zircon (ZrSiO₄, d_{50} = 2 μ m Mahlwerke Kreutz, Mikron, Germany).

The mixtures of MZ and the bonding additives were prepared with a proportion of 85 wt.% of MZ and 15 wt.% of sintering aids. Three compositions were prepared: (a) with α -alumina (MZA), (b) with zircon (MZz) and (c) employing as a bonding phase an stoichiometric mixture of α -Al₂O₃ and zircon (45.5/54.5 wt.%) (MZaz).

The prismatic bars 7.5 mm × 7.5 mm × 50 mm were produced from well-dispersed suspensions by slip casting in a plaster molds as previously described [5].

2.3. Characterization methods

Crystalline phases formed by sintering were analyzed with DRX equipment (Philips 3020 with Cu K α radiation in Ni filter at 40 kV–20 mA). The relative content of tetragonal ZrO₂ was determined using the Garvie and Nicholson method [18] which is based on the integrated peak areas of the phases of zirconia present.

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