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Processing and Property Assessment of Porous Alumina and Mullite-Alumina Ceramics

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Abstract. Porous ceramics have found a wide range of applications as filters for solid, liquid, and gas separation processes. The suitability of materials for use in these types of applications depends on the microstructure (grain size, pore size and pore volume fraction) and hence the mechanical and thermal properties. In this study alumina and mullite-alumina ceramics with different levels of porosity and controlled pore sizes were fabricated. Microstructure and the mechanical properties such as elastic properties and biaxial strength were examined. Correlations between the mechanical properties and porosity were found.

Introduction

Porous ceramics are increasingly being used in applications in the chemical and power generation industries, including filters, catalyst supports and lightweight insulation [1,2]. Such applications, however, require a degree of understanding of the microstructure (grain size, pore size and pore volume fraction) and mechanical properties that is currently lacking. Porous ceramics can be produced with a wide range of porosities (0–80%) and pore sizes (10 nm to 500 μm). The addition of a fugitive agent is typically used to generate pores in ceramic structures [2]. Furthermore, the total porosity and pore size distribution can be carefully controlled by the choice of fugitive agent and the amount added. In this work, alumina and mullite-alumina ceramics with varying porosity levels and controlled pore sizes were prepared and the mechanical properties were examined.

Experimental Procedures

Materials Processing. Two material systems were selected to produce porous ceramics: alumina and mullite-alumina. To produce porous alumina ceramics, batches of alumina powder (Reynolds) with carbon of mean particle diameter of 75 μm (TIMREX KS75, TIMCAL Ltd., Switzerland) were mixed in water. To produce mullite-alumina ceramics, the same alumina and carbon powders were mixed with silica powder (Aldrich) in water. After drying at 120°C for 24 h, the powder cakes were ground with a mortar and pestle and then further de-agglomerated with a mixer/shaker. Specimens were uniaxially pressed at 5 MPa in a steel die to form disks 28.6 mm diameter x 3 mm thickness. The pressed compacts were then sintered in air at 1550°C for 5 h.

Microstructure and Mechanical Properties. The microstructure of the porous ceramics was characterised using scanning electron microscopy (SEM) on polished surfaces. Density and porosity were measured using the water-displacement technique. Dynamic Young's modulus was determined using the impulse excitation technique with a Grindo-Sonic instrument (Lemmens Pty Ltd). Biaxial strength were measured using a 'piston on three balls' biaxial flexure test. An Instron universal testing machine, Model 8562, was used to conduct the test on disk specimens, 25 mm in diameter and 2.5 mm thick. Six disks were tested at a loading rate of 10μm/sec for each type of material.

Results and Discussion

Microstructure and Phase Composition. SEM micrographs of the porous mullite-alumina (MA) ceramics are presented in Fig.1. The MA-ceramic without graphite addition (Fig.1-a) contains a significant amount of fine porosity, indicating that, under the processing conditions used in this work, the mullite-alumina system cannot be sintered to full density through reaction sintering with the silica and alumina powders. Graphite addition of 10 to 60 vol-% resulted in formation of larger, elongated pores due to burnout of graphite. At lower graphite additions of 10-20%, two types of porosity can be observed: large, elongated- and fine-pores (see Fig.1-b). For graphite amounts of 30 to 60%, large, elongated-pores become the more dominant microstructural feature. Grain size is also larger for materials with higher porosity, which is probably due to surface diffusion being more dominant for materials with higher porosity. EDS analysis of the MA-ceramic at higher magnification (Fig.1-d) identified two phases: mullite (grey) as the major phase and alumina (bright) as the minor phase. No residual SiO_2 was found, indicating that the reaction between alumina and silica is complete to form mullite.

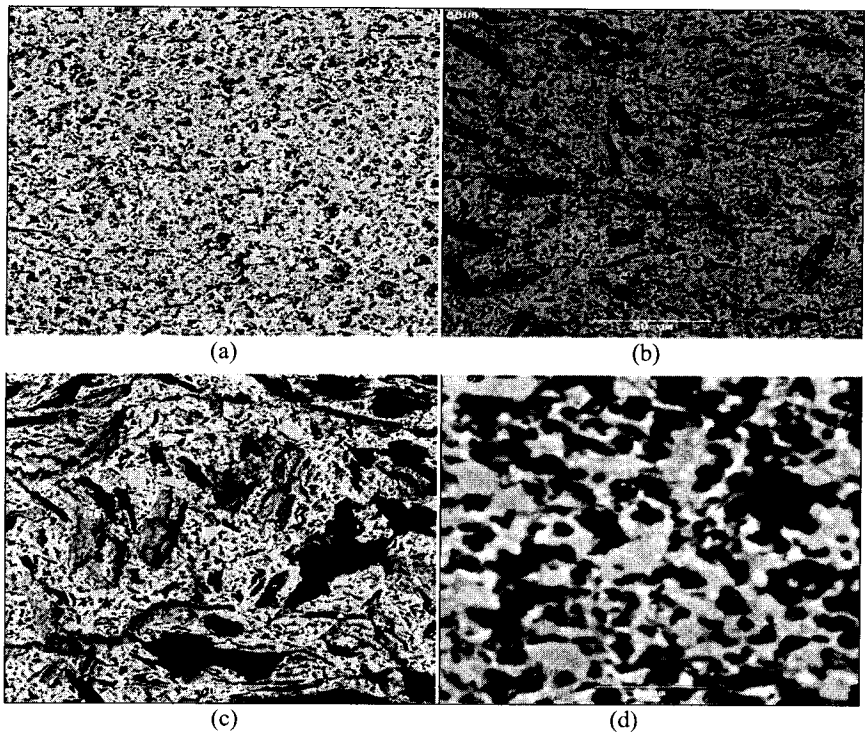

Fig. 1: SEM images showing pore size, shape and distribution of the mullite-alumina ceramics (a, b, and c). Mullite appears as grey, alumina as bright and porosity as black colour (d).

Representative SEM photos of the alumina ceramics (dense and 20.8% porosity) are shown in Fig.2. A uniform distribution of elongated pores of length 50-100 μm and diameter ≈ 10 μm can be observed. Higher magnification images revealed, that the dense material consists of fairly equiaxed alumina grains (3 μm mean grain size) with small levels of porosity mainly at grain boundary triple points. The porous materials consist of equiaxed alumina grains but slightly coarser than the dense material (5 μm mean grain size) and some larger elongated grains.

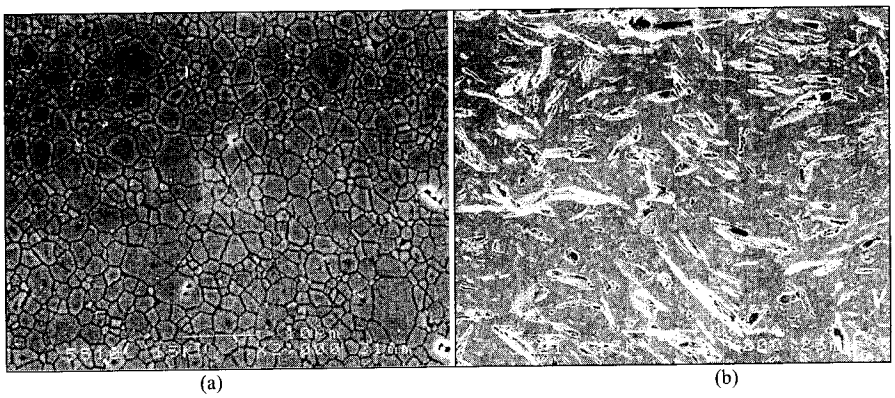

Fig.2: SEM images showing an overview of pore size, shape and distribution of Al_2O_3 with (a) 0.82% and (b) 20.8% porosity, respectively.

Porosity and Mechanical Properties. Figure 3 shows the correlation between the apparent porosity and the volume percentage of the graphite added to the systems. For both ceramic systems the porosity increases in a linear fashion with increasing graphite addition. The ratio of graphite addition to the porosity is close to 1:1 for the alumina system. For the mullite-alumina ceramic, however, the porosity starts at about 36% without graphite addition. Graphite addition of up to 60% corresponds to a porosity increase of only 24%. For this type of material system, the correlation between the overall porosity and graphite addition can be then described by the following equation:

$$P = P_o(1-V_G)+V_G = P_o+V_G(1-P_o)=0.36+0.64V_G \quad (1)$$

where P is the overall porosity, P_o the porosity without graphite addition and V_G the volume percentage of graphite addition. Linear regression of the mullite-alumina data in Fig.3 resulted in a coefficient factor of 0.41, which is much lower than 0.64. This lower than expected factor could be caused by the reaction sintering, leading to higher density of mullite than SiO_2 and Al_2O_3 and hence lower porosity.

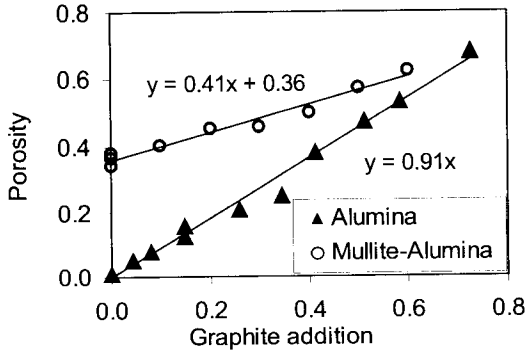

Fig.3: Correlation between the apparent porosity and graphite addition.

Figures 4 and 5 show the Young's modulus and biaxial strength of the alumina and mullite-alumina ceramics as a function of porosity, respectively. Both Young's modulus and biaxial strength are related to porosity by an exponential equation:

$$E = E_o \exp(-bP) \quad \text{and} \quad \sigma = \sigma_o \exp(-bP) \tag{2}$$

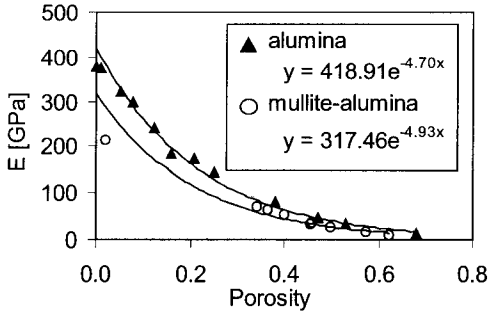

Figure 4 Young's modulus as a function of apparent porosity

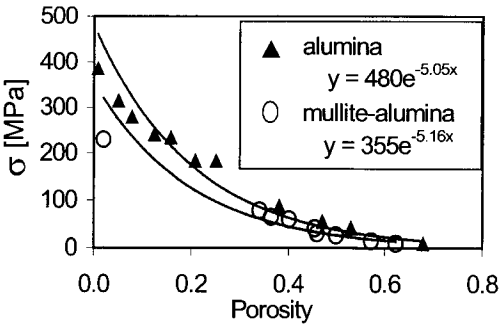

Figure 5 Biaxial strength as a function of apparent porosity

where E is Young's modulus at porosity P, σ biaxial strength at porosity P, and b experimental determined constant. E_0 and σ_o are Young's modulus and biaxial strength at zero porosity. The experimental data in this work can be described quite well by equation (2). The relationship between porosity and Young's modulus and strength of porous ceramics has been analysed extensively [3,4]. The empirical exponential relationship represents a good estimation Young's modulus and strength, with a constant of b value usually ranging between 2 and 7 for Young's modulus. The constant value b is usually in the range between 4 to 6 for tensile or bending strength and 6 to 9 for compressive strength. It is interesting to note that there is only a small difference in the Young's modulus at higher porosity range (0.4-0.7). It seems that for higher porosity materials, the amount of porosity becomes the more dominant parameter, which control the Young's modulus. Alumina ceramics exhibit slightly higher biaxial strength than the mullite-alumina ceramics.

Conclusion

In this work, porous alumina and mullite alumina ceramics were fabricated. Their microstructure and mechanical properties were characterised. The results can be summarised as follows:

- Graphite powder can be used successfully as a pore forming agent, which can be burned out before or during the sintering process.
- For both ceramic systems, the porosity increases in a linear fashion with increasing amount of graphite addition. The rate of increase, however, is different for the two systems.
- The correlation between Young's modulus, biaxial strength and porosity can be expressed by an empirical exponential equation.

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