J Mater Sci (2007) 42:5880–5882
DOI 10.1007/s10853-007-1844-y

LETTER

High temperature biaxial strength of porous mullite-alumina and mullite-zirconia ceramics

Bruno A. Latella · Edward G. Mehrtens

Received: 21 March 2007 / Accepted: 10 May 2007 / Published online: 30 May 2007

Hot gas cleaning in coal combustion and gasification in the power generation industry is achieved by using porous ceramic filters [1]. These ceramic filters are high in porosity (up to 80%), have good thermal shock resistance and adequate gas permeability. In service, the filters are subjected to repeated impacts from fine particles (8 μm or greater) at velocities of about 50 mm/s in a gas stream at temperatures ranging from 400 °C to 1,000 °C. Therefore, the ceramic filter element must be able to withstand the contact of particles, the sudden temperature increase at the face of the filter and the pressure differential due to the gas. It is well known that mullite-based ceramics exhibit excellent thermal shock resistance [2]. In this communication we report the biaxial strength of porous mullite-alumina (designated MA) and mullite-zirconia (designated MZ) ceramics over a range of temperatures up to 1,250 °C.

Standard ceramic processing methods were used to prepare the mullite-alumina and mullite-zirconia samples. Alumina (α-Al_2O_3, ≈1 μm mean particle size, Reynolds 99.7% purity), silica (Nyacol-1440, median particle size = 14 nm) and zircon (Neumann) powders were used. The processing of MA is detailed elsewhere [3, 4]. For MZ the zircon and alumina powders were mixed ($2ZrSiO_4 + 4Al_2O_3$) with 1 wt% MgO in water. Batches of MA and MZ were prepared with about 20 vol% solids loading and these were mixed by ball milling using alumina media in polyethylene containers for 24 h and then a further 2 h with differing amounts of a pore former agent ranging from 0 to 60 vol% (graphite powder, median particle size = 23 μm, TIMCAL Ltd.). Samples were die pressed or slip cast to form disks of diameter 30 mm and thickness of 5 mm. The green samples were then sintered in air: (i) MA: 1,450–1,550 °C for 5 h and (ii) MZ: 1,500–1,600°C for 5 h. The graphite burned out at <1,000 °C during the heat up cycle [4]. Density and porosity of sintered disks was determined using the boiling water method [5].

The strength of the specimens was determined using a three-point biaxial flexure test at room and high temperature. For the strength measurements at high temperature only the 43% porosity MA and the 62% porosity MZ materials were tested. Specimens were placed on an alumina jig with 3 mm alumina balls as the supports in circumference (support diameter = 24 mm) and a flat alumina punch (contact area = 4 mm). A $MoSi_2$ resistance-heated furnace attached to a universal testing machine (Instron 8561) was used. Specimens were placed centrally in the jig, held in place by thick alumina loading rods. Each test sample was then heated at 300 °C/h to the designated temperature and held for 30 min to equilibrate prior to loading to failure at 10 μm/s. Strengths were calculated from the failure loads and specimens dimensions with a minimum of five disks broken in each instance. Young's modulus was determined using the impulse excitation technique [6] and hardness, H, was determined from Vickers indentations on polished specimens using a load of 0.5 kg.

The room temperature Young's modulus and biaxial flexure strength of the MA and MZ materials as a function of apparent porosity are shown in Figs. 1 and 2, respectively. The solid lines are exponential fits to the data. It is clear that both Young's modulus and strength exhibit exponential drops with increasing apparent porosity for both MA and MZ materials. Interestingly, the

B. A. Latella (✉) · E. G. Mehrtens
Institute of Materials and Engineering Science,
Australian Nuclear Science and Technology Organisation,
Private Mail Bag 1, Menai, NSW 2234, Australia
e-mail: bal@ansto.gov.au

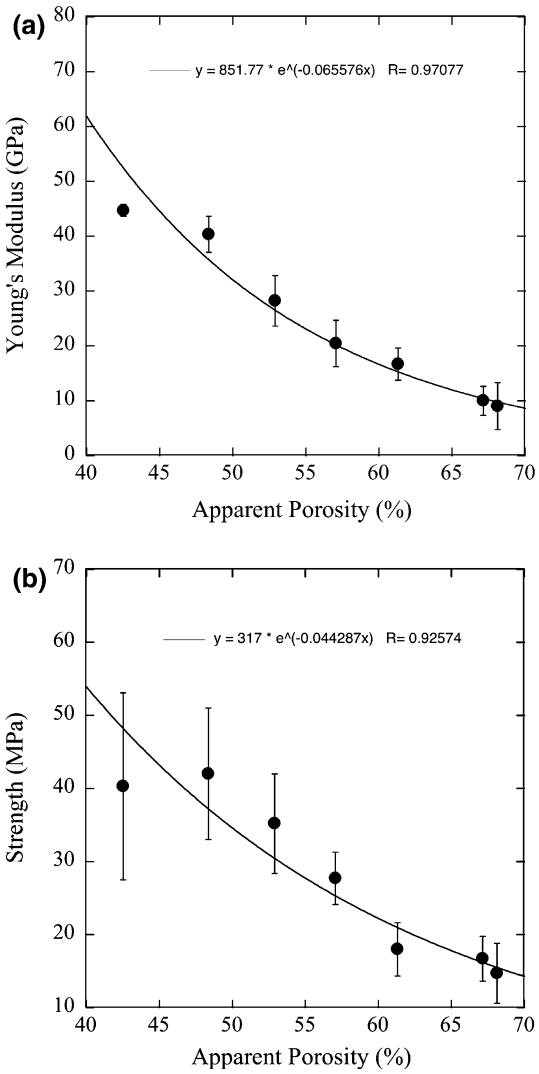

Fig. 1 Room temperature (**a**) Young's modulus and (**b**) biaxial strength of mullite-alumina (MA) as a function of apparent porosity

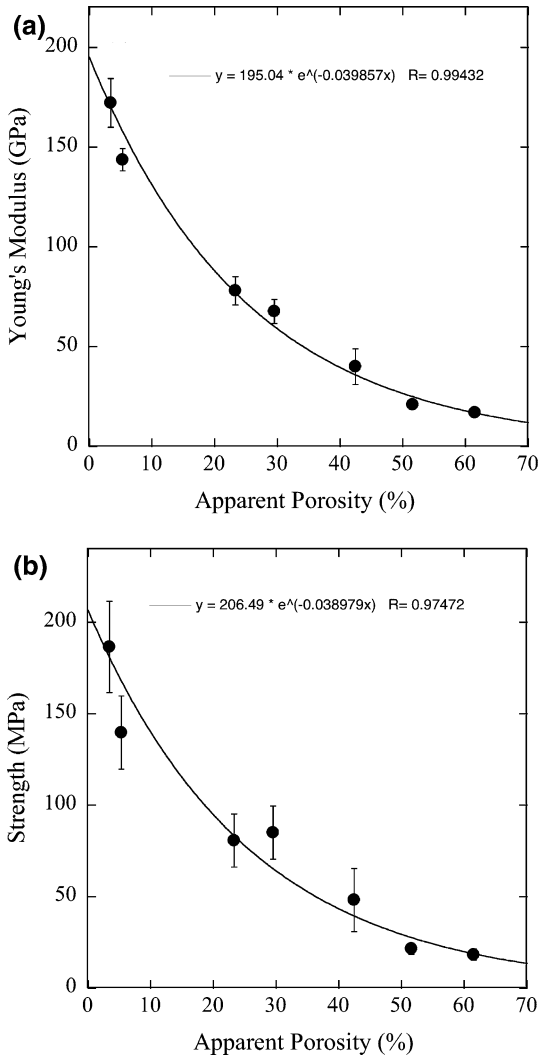

Fig. 2 Room temperature (**a**) Young's modulus and (**b**) biaxial strength of mullite-zirconia (MZ) as a function of apparent porosity

magnitudes of the properties for both materials are similar for equivalent porosity levels. There is much greater variability in the strength values of MA particularly for the samples within the 40–55% porosity range, most likely due to large variations in the critical flaw size (i.e. pore size).

Figure 3 shows the biaxial flexure strength data of the MA and MZ materials as a function of testing temperature. The data presented for each composition is at a fixed material porosity, i.e. 43% porosity for MA and 62% porosity for MZ. These porosity levels were chosen as they correspond to the lower (MA) and upper bounds (MZ) for the mullite-based porous structures fabricated in this work without and with pore formers, respectively. For both compositions there is no discernible change in the strength with temperature. As with the room temperature strength

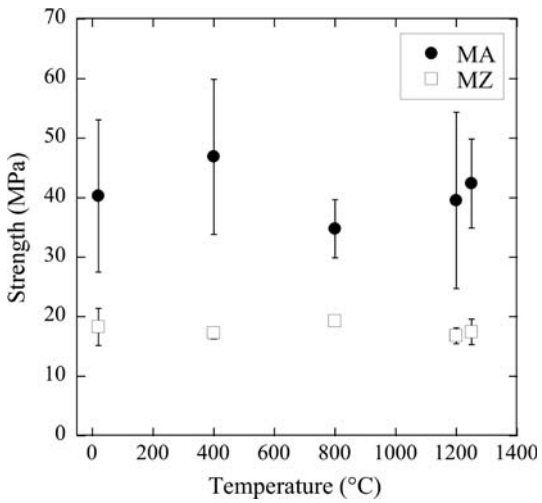

Fig. 3 Biaxial strength as a function of test temperature for mullite-alumina (MA) (43% porosity) and mullite-zirconia (MZ) (62% porosity)

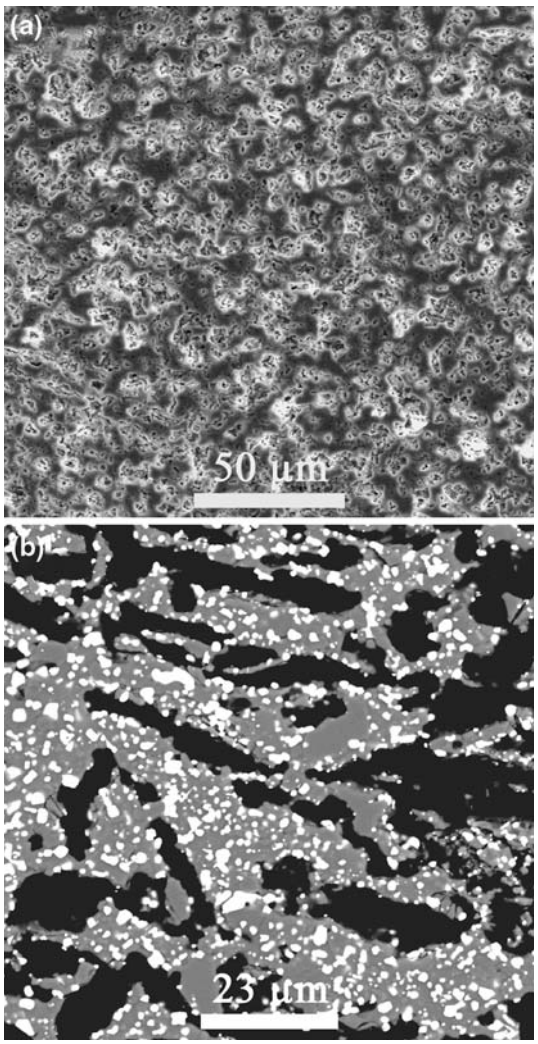

Fig. 4 Scanning electron micrographs of (**a**) mullite-alumina (MA) (43% porosity) and (**b**) mullite-zirconia (MZ) (62% porosity)

(Fig. 1a), the MA material showed wide variability in the strength values, hence the large error bars.

Previous work [4] has shown that strength of MA at high temperature exhibits the same trend with increasing porosity as with ambient testing (Fig 1b). This is also the case for MZ with different porosities tested at 1,250 °C (not presented in this study).

The microstructures of MA (43% porosity, H = 1.7 GPa) and MZ (62% porosity, H = 0.7 GPa) are shown in Fig. 4. The secondary electron image of MA shows small irregular pores (Fig. 4a) as this composition did not contain any graphite addition. The material contains relatively fine alumina and mullite grains. Figure 4b shows a backscattered image of MZ with the small white zirconia grains and the greyish alumina and mullite matrix. The pores are distinguished by the black elongated shapes scattered throughout, resulting from the graphite burnout.

Based on the above results the MA and MZ ceramics are adequate strength-wise and do not show any strength degradation when subjected to high temperatures. The processing method we have used is effective and the material microstructures obtained have fine grain size and porosity levels that can easily be controlled. Accordingly the mechanical stability at high temperatures of MA and MZ porous ceramics have potential for use in hot gas filtration environments.

References

1. Alvin MA (1996) Ind Eng Chem Res 35:3384
2. Schneider H, Okada K, Pask J (1994) Mullite and mullite ceramics. John Wiley & Sons, Chichester
3. Deng H, Latella BA, Liu T, Ke Y, Zhang L (2003) Mater Sci Forum 437–438:423
4. Latella BA, Henkel L, Mehrtens EG (2006) J Mater Sci 41:423
5. Australian Standard 1774.5 (1989) Refractories and refractory materials – physical test methods. Method 5: the determination of density, porosity and water absorption. Standards Australia, Sydney, p 1
6. Latella BA, Humphries SR (2004) Scr Mater 51:635