Mechanical Properties of Porous Alumina at High Temperature

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Porous alumina was prepared with fine alumina powder and fine PMMA particles. Porous alumina of 1.5 to 10 \( \mu \)m in diameter was obtained with up to 40\% porosity after sintering at 1600 K. After being characterized, the microstructure and air permeability, Young’s modulus, fracture strength and toughness were measured up to 1273 K. Air permeability of porous alumina increased drastically above 20\% porosity because of the formation of continuous pores. Young’s modulus and fracture strength of porous alumina was lower than that of dense alumina and decreased gradually above 1000 K. However, the rate of decrease of porous alumina was lower than that of dense alumina. A similar decrease was observed for fracture toughness. Fracture toughness also decreased above 1000 K, however, the rate of decrease was small for porous alumina with large porosity and pore size. The frontal process zone size elongated at high temperatures, contributing to the prevention of the decrease in fracture toughness at room temperature.

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1. Introduction

Porous ceramics are used in applications such as separators, catalysis supports, molecular sieves, and insulation at high temperatures due to their abrasion and chemical resistance, high temperature stability, and thermal and electrical properties. The selection of a ceramic system and pore morphology for this application depends on the chemically and thermally stable requirements simultaneously on the ease of processing and casting. Alumina is particularly useful for many of these porous products. Porous alumina can be produced at a relatively low cost and in a variety of structural configurations such as solid components. One of the methods for controlling porosity is to burn out organic particles or fibers that have been mixed with alumina slurry beforehand.\(^1\),\(^2\)

In this study, porous alumina with different porosity and pore size were prepared using organic PMMA particles as a pore-former. These porous alumina were characterized by microstructure observation, porosity and air permeability. Mechanical properties, i.e. Young’s modulus, fracture strength and fracture toughness, were measured at a temperature range of RT to 1273 K. In particular, fracture toughness was discussed with the critical frontal process zone size estimated by a SEVNB specimen.\(^3\)

2. Experimental

2.1 Preparation of porous alumina

Polycrystalline powders of alumina (TM-D, Taihei Kagaku, average particle size of 0.25 \( \mu \)m) were prepared as ceramic matrix. Commercially obtained PMMA resin particles (MX, Soken Chemical & Engineering, 1.5, 3.0 or 10.0 \( \mu \)m) were used for the pore-former. These powders were mixed at a given weight ratio with ethanol.

For the fabrication of porous alumina, the mixed slurry was dried in air for 1 day. These dried powders were cast by CIP and heated gradually up to 773 K to vaporize the organic particles. The sintering condition was 1623 K for 1 hr.

2.2 Characterization of porous alumina

The relative density and porosity of porous alumina were determined using the Archimedes method. The microstructure of the porous alumina was observed by SEM to determine the pore concentration. The gas permeability of air was measured, based on the leakage time through the porous alumina plate in a vacuum chamber.\(^4\)

2.3 Mechanical properties of porous alumina

Young’s modulus of porous alumina was at a high temperature estimated using a dynamic strain gauge extensometer (Instron, 2620–600 series), where the displacement at the center of the lower surface of the four-point bending specimen was measured directly based on JIS R 1602. Other normal measuring methods cannot be applied to porous alumina at high temperatures. The fracture strength was estimated using a four-point bending test, where the 3 \( \times \) 4 \( \times \) 40 mm specimens were lapped to mirror finish with a base with ground corners on JIS R 1601. The upper span was 10 mm and the lower was 30 mm. The cross head speed was 0.5 mm/min.

The fracture toughness was estimated by SEVNB method. The specimens similar to those for the bending test in dimensions were introduced at the lower surface with a sharp V-notch. The radius of curvature at the V-notch tip was about 20 \( \mu \)m. This curvature and wide space behind crack tip suitable for measurement in this experiment. The critical frontal process zone size, where the crack propagating energy was consumed by the formation of micro-cracks, was estimated using the fracture strength for specimens machined with a V-notch.\(^5\) Each measurement at high temperature was performed after keeping the specimen at a given temperature for 30 min.

3. Results and discussion

3.1 Microstructure and permeability of porous alumina

Porous alumina prepared with alumina and PMMA powder are shown in Fig. 1. After sintering, the grain size of alumina was constant at approximately 1 \( \mu \)m, independent of size and content of PMMA. The size of macro-pores corresponded to that of the initial PMMA particles. In this fabricated method, porous alumina with above 40\% porosity was not obtained independent of the PMMA particle size. These macro-pores were isolated below 20\% porosity and the ratio of openness to total porosity was less than 0.5. In the porous alumina with above 20\% porosity, macro-pores were continuous and the ratio of open porosity was more than 0.8. Air permeability of porous alumina is shown in Fig. 2. Permeability drastically increased above 20\% porosity, which corresponds to the pore morphology change from isolated pores to continuous pores. In particular, large pores of 10 \( \mu \)m in diameter contributed to air permeability. Therefore, for mechanical meas-
Fig. 1. Scanning electron micrographs of porous alumina prepared with different PMMA powders (1.5, 3.0, 10 μm in diameter).

Fig. 2. Air permeability of porous alumina with different pore sizes.

Fig. 3. Change in Young’s modulus for porous alumina (a), and relative values normalized that at room temperature (b) with temperature.

Fig. 4. Change in fracture strength for porous alumina (a), and relative values normalized that at room temperature (b) with temperature.

measurements, dense and porous alumina with approximately 20 or 40% porosity containing of small (1.5 μm), medium (3.0 μm) and large (10.0 μm) pores were used.

3.2 Young’s modulus and fracture strength of porous alumina at high temperatures

The changes of mechanical and thermal properties with porosity have already been discussed in previous papers.2,5 These properties were exhibited in terms of power law based on the minimum solid or area segment model. In this paper, the changes in mechanical properties are only discussed at elevated temperatures up to 1273 K. The change in Young’s modulus with temperature for dense or porous alumina is shown in Fig. 3(a) and relative Young’s modulus normalized with each value at room temperature is shown in Fig. 3(b). Young’s modulus depends only on porosity, and decreased gradually with temperature for all porous alumina. However, compared with the relative modulus for dense and porous alumina, the decreasing rate of porous alumina was minimal, particularly for large pores. The solid area in the cross section, assuming the similar grain size and fluid grain boundary, affected Young’s modulus for porous alumina significantly. The small contact area of grains for porous alumina causes the lower decrease for Young’s modulus.

The change in fracture strength with temperature for dense or porous alumina is shown in Fig. 4(a). The relative fracture strength is shown in Fig. 4(b). The fracture strength also decreased with temperature. Porous alumina with large pores (10 μm) showed lower strength than those with small or medium pores (1.5 or 3.0 μm). Large flows caused a drastic decrease of strength, however large flows resulted in a contrary effect at elevated temperatures. Condensation and healing at crack tips of large flows mainly contribute to maintaining strength for porous alumina.

In past studies, Young’s modulus and the fracture strength at room temperature were measured for porous alumina with opened pores or isolated pores.5 Young’s modulus of two types of pores was almost same at the same porosity. The fracture strength of porous alumina with isolated pores was higher than that with opened pores.

3.3 Fracture toughness of porous alumina at high temperature

Fracture toughness with temperature for dense or porous alumina is shown in Fig. 5(a) and the relative fracture toughness is shown in Fig. 5(b). The fracture toughness decreased
gradually with temperature. Toughness for porous alumina with approximately 40% porosity was independent of pore size. Toughness for porous alumina with approximately 20% porosity, however, was different with pore size at a similar temperature. These different decreasing rates with temperature were reflected remarkably in relative fracture toughness.

For dense ceramics with rising R-curve behavior, the fracture toughness consists of the fracture energy creating the critical frontal process zone size at the crack tip and the mechanical energy consumed at the bridging in the process zone wake. For porous ceramics, the former mainly contributes to fracture toughness. The critical frontal process zone sizes for dense or porous alumina at room temperature, 1073 K or 1273 K are listed in Table 1. The zone size elongated largely depending on the pore morphology and temperature. For each porous alumina, these zone sizes were sufficiently larger than each pore size, and depended on temperature, porosity and pore size in this experiment. However, the systematical changes were not observed, therefore it was difficult to discuss exactly the relationship with fracture toughness in this paper.

4. Conclusion

(1) Porous alumina was prepared with fine alumina powder and fine PMMA particles. Porous alumina with 1.5 to 10 μm in diameter was obtained with up to 40% porosity after sintering at 1600 K.

(2) Air permeability of porous alumina increased drastically above 20% porosity because of the formation of continuous pores. Porous alumina with 20 or 40% porosity was prepared for mechanical properties.

(3) Young’s modulus and fracture strength of porous alumina were lower than that of dense alumina and decreased gradually above 1000 K. However, the rate of decrease for porous alumina was lower than that of dense alumina.

(4) A similar decrease was observed for fracture toughness. Fracture toughness also decreased above 1000 K, however, the rate of decrease was small for porous alumina with large porosity and pore size. The toughness was mainly dependant on the frontal process zone size.

Table 1. Estimated Critical Size of Frontal Process Zone for Dense and Porous Alumina at Room Temperature, 1073 K, and 1273 K

<table>
<thead>
<tr>
<th>Particle size of PMMA [μm]</th>
<th>Porosity [%]</th>
<th>Critical frontal process zone size [μm]</th>
</tr>
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<tbody>
<tr>
<td>Dense alumina</td>
<td>0.7</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>1.5</td>
<td>21.1</td>
</tr>
<tr>
<td></td>
<td>3.0</td>
<td>20.5</td>
</tr>
<tr>
<td></td>
<td>10</td>
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References