Evaluation of Fracture Toughness of Porous Ceramics*

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The indentation fracture (IF) method, the single-edge precracked beam (SEPB) method, and the single-edge V-notch beam (SEVNB) method were applied to evaluate the fracture toughness of four kinds of porous ceramics of SiC, Al₂O₃ and Mg₃Al₂Si₃O₁₄ with porosity ranging from 37 to 43%. The microstructures of these materials were composed of ceramics grains, glassy grain boundaries and pores. Each grain was joined together with the glassy grain boundary phase. The IF and SEPB methods were not applicable because both precracks and indenter traces were not visible. On the other hand, the SEVNB method was applicable because the V-notch could be easily machined by grinding. In the case of the SEVNB method, the applied load versus back-face strain plots under four-point bending showed nonlinearity prior to the maximum load. The R-curve behavior was estimated from the compliance change of specimens. The fracture toughness of porous ceramics was smaller than that of dense ceramics, and increased with increasing crack extension. Since the stable crack predominantly propagated along glassy grain boundaries, the R-curve behavior depended on the loading rate and matrix grain size. The increment of the R-curve by grain bridging became larger for coarser-grain sized ceramics.

Key Words: Porous Ceramics, Fracture Toughness, Indentation Fracture Method, Single-edge Precracked Beam Method, Single-edge V-notch Beam Method, R-curve Behavior

1. Introduction

Recently, porous ceramics of about 40% porosity are noticed as dedusting filters for high-temperature turbines(129). However, there are few research studies on the mechanical properties and fracture behavior of porous ceramics(32)(44). Therefore, in the present study, the conventional testing methods for fracture toughness of dense ceramics such as the IF method (Indentation Fracture method), the SEPB method (Single-Edge Precracked Beam method) and the SEVNB method (Single-Edge V-notch Beam method)(5)

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were first examined for application to typical porous ceramics with porosity ranging from 37 to 43%. Next, the R-curve behavior of porous ceramics was measured by the compliance method using back-face strains of the SEVNB specimen(3), and then the effect of the microstructure of porous ceramics on fracture toughness and behavior was examined.

2. Experimental Procedure

2.1 Materials

The materials used in the present study were the sum total of four types of porous ceramics of one silicon carbide (KD-DSC T10-20, manufactured by Schumacher U & T in Germany(49)), two kinds of alumina (FA10 and FA4, manufactured by Kyocera Co. in Japan) and one corderite (manufactured by Kyocera Co. in Japan). They are simply designated as SiC, Al₂O₃(A), Al₂O₃(B) and Mg₃Al₂Si₃O₁₄. The mean pore size, porosity, density and bending strength of each material are summarized in Table 1. Figure 1 shows the SEM microstructures of porous ceramics. The porosity of each material is around 40%, while the microstructures are significant different depending
Table 1 Properties of porous ceramics

<table>
<thead>
<tr>
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<th>Mean pore size (μm)</th>
<th>Porosity (%)</th>
<th>Density (g/cm³)</th>
<th>Bending strength (MPa)</th>
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<td>41</td>
<td>37</td>
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<td>25</td>
<td>43</td>
<td>1.61</td>
<td>13.0</td>
</tr>
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Fig. 1 Microstructures of porous ceramics

Fig. 2 Distribution in grain diameter of porous ceramics

2.2 Specimens and measurement of fracture toughness and R-curve behavior

The IF, SEPB and SEVNB methods were first applied to porous ceramics for evaluating fracture toughness. In the case of the IF and SEPB methods, the bend-bar specimens with a width of 3 mm, a thickness of 4 mm, and a length of 40 mm were used in conformity with JIS R 1607. For the IF method, the testing surface of the bend-bar specimen was mechanically polished with diamond slurry to be as fine as possible, and then was indented using a Vickers indenter. For the SEPB method, three Vickers indentations were introduced at the center of the specimen at a normal load, \( P = 98 \) N, as a starter of the precrack, and then the pop-in precrack was introduced by the bridge-indentation (BI) method using an acoustic emission (AE) sensor cemented on the precracker.

In the case of the SEVNB method, the single-edge V-notch beam specimens with a width of 5 mm, a thickness of 7 mm, and a length of 45 mm were used. V-notches were machined by grinding using a V-shaped diamond wheel, as shown in Fig. 3. The range of V-notch wheel was from 0.67 to 4.93 mm. The radius of the notch root curvature was about 20 μm. The SEVNB method was carried out in either air or water at room temperature under four-point bending, as shown in Fig. 4. The inner and outer spans were 20 and 40 mm, respectively. During loading, the bending load and the back-face strain were monitored using a strain gauge cemented on the SEVNB specimens. The crosshead speed, \( CHS \), ranged from 0.005 to 0.5 mm/min.
Fig. 3 V-notch in the SEVNB specimen of porous SiC

Fig. 4 Schema of the SEVNB method in water

Fig. 5 Micrograph of Vickers indent in porous SiC

Fig. 6 Micrograph of Vickers indent in porous Al$_2$O$_3$ (A)

Fig. 7 Micrograph of Vickers indent in porous Al$_2$O$_3$ (A)

3. Experimental Results and Discussion

3.1 IF method

Using SiC and Al$_2$O$_3$ (A) samples in which mean pore sizes were maximum and minimum in four types of porous ceramics, the IF method was carried out for evaluating fracture toughness.

To begin with, a Vickers indenter was impressed on the surface of the SiC specimen at $P=9.8$ and 4.9 N. As the result, near the impressed area, almost all the pores were squashed and SiC particles sank significantly, so that both the indentation trace and Vickers-induced surface cracks were not visible. Then, the Vickers indenter was directly impressed on a SiC particle with a small indentation load to measure the fracture toughness of the matrix SiC grain. Figure 5 shows the SEM micrograph of the indentation in SiC particle at $P=2.9$ N. In order to evaluate the fracture toughness of the matrix ceramic grain, the radial/median crack lengths that arose from the four corners of the indentation trace must be measured. Using a scanning electron microscope, only the indentation trace could be identified, while the Vickers-induced surface cracks could not be discriminated. Furthermore, the subsurface particle shape was obscure so that the flaw shape could not be specified. Therefore, the fracture toughness of the matrix grain in porous ceramics could not always be evaluated, even if the Vickers-induced surface cracks could be observed.

Next, the Vickers indenter was impressed on the surface of the Al$_2$O$_3$ (A) specimen at $P=98$ and 9.8 N. Figure 6 shows the SEM micrographs around the indentation at $P=98$ N. The indentation trace was more distinct in comparison with that of the SiC sample. However, Vickers-induced surface cracks were not visible (see the detailed photograph of A in Fig. 6). Figure 7 shows the SEM micrographs around the indentation at $P=9.8$ N. Under the condition of decreasing the indentation load, the indentation crack was still invisible.

It was concluded that the fracture toughness testing using the IF technique was inapplicable to
evaluate the fracture toughness for porous ceramics.

3.2 SEPB method

In the case of the SEPB method, using either a machined straight-through notch or indentation cracks as a starter of the precrack, the pop-in precracks are usually introduced by the bridge-indentation (BI) method. Therefore, both introduction and identification of the pop-in precrack are key technologies for evaluating the fracture toughness using the SEPB technique.

Whether the pop-in precrack was introduced in the SiC specimen with the maximum mean pore size was first examined. After the precracking procedure, oily ink soaked into the entire specimen to say nothing of precrack by capillarity, because pores of about 41 μm was enough size to permeate oily ink into the entire material. Therefore, it is impossible to dye the precrack alone in the SiC specimen.

Next, the precrack introduction testing using the BI technique was carried out for the Al2O3(A) specimen with the minimum pore size. The pop-in signal could not be detected regardless of the testing load, because the event level of AE was very low during testing. The pop-in crack could not be observed at all from the testpiece side. In the present study, after compressive loading to a constant load level on the precracker, oily ink was made to soak from the test-piece side. Figure 8 shows one example of stained SEPB specimens with red ink. The red ink permeated into the entire specimen as well as into the SiC sample, although the mean pore size of the Al2O3(A) specimen was very small of 1.3 μm. The existence and crack length of precrack could not be measured.

It was concluded that the fracture toughness testing using the SEPB technique was also inapplicable to evaluate the fracture toughness for porous ceramics.

3.3 SEVNB method

In the case of the SEVNB method, it is possible to use the notch as a precrack, if the V-notch can be machined as shown in Fig. 3. Furthermore, the notch depth can be easily measured using a profile projector. Therefore, the SEVNB method would be applicable to evaluate the fracture toughness for porous ceramics having high porosity and low fracture strength such as the present materials.

Figures 9 and 10 show typical applied load versus displacement and applied load versus back-face strain plots during loading for the SiC sample. Both relationships showed nonlinearity before reaching the maximum load. The SEVNB specimens were not broken unstably after reaching the maximum load. According to the in-situ observation of the same material reported by Song and Takeda(6), microcracks were generated from the V-notch tip before reaching the maximum load, and propagated stably. Consequently, the applied load versus back-face strain plots seem to become nonlinear. Furthermore, the broken pieces of the SEVNB specimen were still undivided after unloading, even if the main crack propagated to the compression side. The fracture surfaces clung to each other. Figure 11(a) shows the SEM micrograph of the side-view of broken pieces after the fracture toughness test. The main crack that arose from the notch tip propagated along the interface between matrix SiC grains, and was bridged by some grains across the crack faces. Figure 11(b) shows the fracture surface near the V-notch tip that was divided compulsorily. In this figure, there were few grains fractured transgranularly. It was found that fracture surface of porous ceramics was formed as a result of selective fracture of the glassy grain bound-
Fig. 11 Fracture surface of porous SiC

Fig. 12 $R$-curve behavior for porous SiC

Fig. 13 $R$-curve behavior for porous SiC under several crosshead speeds

For dense ceramics in which the microcrack stably propagated from the notch tip, the fracture toughness characteristic could be directly evaluated as $R$-curve behavior from the side-view observation of the crack tip using an optical microscope. However, in the case of porous ceramics, it is difficult to directly observe the crack tip from the side-view of the specimen because of high porosity. In the present study, crack extension was estimated by the compliance method using back-face strain, and then $R$-curve behavior was evaluated. The details are described in the appendix.

3.4 $R$-curve behavior of each material

3.4.1 $R$-curve behavior of SiC sample Figure 12 shows $R$-curves, $K_\alpha$, of SiC specimens at $a_0/W = 0.41$ together with the applied stress intensity factor, $K_{app}$-curve, at the maximum load. In this figure, the $R$-curves were estimated from the results on the condition that $CHS = 0.5$ mm/min in air at room temperature, where $a_0$ is initial notch depth and $W$ is thickness of the SEVNB specimen. $R$-curves overlapped one another. The average stress intensity factor, $K_\alpha$, at the point where the $K_{app}$-curve at the maximum load came in contact with the $K_\alpha$-curve was 0.55 MPa $\sqrt{m}$. This value was much smaller than the fracture toughness of a dense silicon carbide. The $K_\alpha$-$\Delta a$ relationship shows rising $R$-curve behavior, where $\Delta a$ is a crack extension from the notch tip. The average amount of crack extension was 0.69 mm until the applied load reached a maximum. Figure 13 shows the $R$-curve behavior under several crosshead speeds. The initial stress intensity factor $K_\alpha$ at $\Delta a = 0$ and the amount of crack extension at the maximum load, $P_{max}$, were nearly constant regardless of the crosshead speed. While, the increment of $K_\alpha$ for $\Delta a$ on the slowest condition of $CHS = 0.005$ mm/min was lower than that of others. The dependence of cross-
water seemed to overlap with that in air. Therefore, the loading condition of fracture toughness test for porous ceramics should be carefully determined because the SCC effect directly influences $R$-curve behavior. In the present study, the following fracture toughness tests were all carried out in air under $CHS = 0.05 \text{ mm/min}$. 

### 3.4.2 $R$-curve of Al$_2$O$_3$(A) sample

Figure 16 shows the relationship between the applied load and back-face strain of the Al$_2$O$_3$(A) sample, in which both pore and grain sizes were the smallest. In this figure, the result of the specimen with a different notch depth, $a_0$, is summarized. These curves, as well as those for the SiC sample, showed nonlinearity before reaching the maximum load. However, the change of the compliance of each specimen was slight, and it tended to unstably fracture immediately after reaching the maximum load.

Figure 17 shows $R$-curves, $K_r$, and $K_{iso}$-curves at the maximum load. $R$-curves were estimated from the applied load versus back-face strain plots of Fig. 16. Although the compliance, $\lambda$, and its change differ by the notch depth, $R$-curves almost agreed with one another. The average $K_r$ value, at which the $K_{iso}$ curve met with the $R$-curve, was 0.52 MPa $\sqrt{\text{m}}$, and was also much smaller than the fracture toughness of dense alumina. The $K_r$-$\Delta\alpha$ relation showed rising $R$-curve behavior in the same manner as the SiC sample. In this case, the amount of crack extension until the maximum load was about from 0.24 to 0.45 mm, and then the average value was 0.32 mm.

Figure 18 shows the SEM micrographs of the side-view and fracture surface in the vicinity of the notch tip after the test. From a macroscopic point of view, the main crack that arose from the notch tip propagated straightly. The main crack microscopically propagated along the interface between matrix Al$_2$O$_3$ grains, and the fracture surface near the notch tip was entirely composed of intergranular fracture surfaces. It was found that the fracture surface was formed as a result of the fracture of the glassy phase that connected Al$_2$O$_3$ grains. In this material, the
stress shielding effect of the crack tip due to grain bridging was relatively small because the matrix Al₂O₃ particle was very small with the mean grain size of 4.7 μm.

### 3.4.3 R-curves of Al₂O₃(B) sample

Figure 19 shows the applied load versus back-face strain plots of the Al₂O₃(B) sample in comparison with the typical result of the Al₂O₃(A) sample. These curves for the Al₂O₃(B) sample showed nonlinearity before reaching the maximum load. Specimens were not broken unstably immediately after reaching the maximum load. The mean grain size of the Al₂O₃(B) sample was 72 μm, and was about 15 times larger than that of the Al₂O₃(A) sample. Furthermore, the maximum load was about 1.8 times higher than that of the Al₂O₃(A) sample although the initial compliance without crack growth, λ, was about 0.8 times higher. This is because of its slightly lower porosity of about 38% (see Table 1).

Figure 20 shows R-curves, $K_R$, and $K_{app}$-curves at the maximum load. R-curves were also estimated from the applied load versus back-face strain curves in Fig. 19, and were compared to the R-curve for the Al₂O₃(A) sample. R-curves overlapped one another, and the $K_R$-$\Delta a$ relations showed rising R-curve behavior. The initial stress intensity factor $K_R$ at $\Delta a=0$ coincided with that of the Al₂O₃(A) sample. While the average value of $K_R$ at which the $K_{app}$-curve met with the R-curve was 1.15 MPa√m, and the average amount of crack extension before reaching the maximum load was about 0.59 mm. From the above experimental results, it was found that increment of $K_R$ for $\Delta a$ was larger than that of the Al₂O₃(A) sample.

Figure 21 shows the SEM micrographs of the side-view and fracture surface in the vicinity of the notch tip after the test. The main crack that arose from the notch tip propagated along grain boundaries between Al₂O₃ grains, and was bridged by some grains across the crack faces similar to the fracture of the SiC sample. It was found that this grain-bridging contributed to the rising of the R-curve. The fracture surface was also formed from intergranular fracture, though the morphologies of the matrix grain and pore of the Al₂O₃(B) sample were significantly different from the Al₂O₃(A) sample.
3.4.4 $R$-curve of Mg$_2$Al$_4$Si$_5$O$_{18}$ Sample

Figure 22 shows the applied load versus back-face strain plots of the Mg$_2$Al$_4$Si$_5$O$_{18}$ sample. The mean grain and pore sizes of secondary grains in the Mg$_2$Al$_4$Si$_5$O$_{18}$ sample were 143 and 25 $\mu$m, respectively. The secondary grain of the Mg$_2$Al$_4$Si$_5$O$_{18}$ sample was the second largest in the sample materials. Therefore, the load vs. back-face strain plots showed nonlinearity before reaching the maximum load, and specimens were not broken unstably immediately after reaching the maximum load as well as the case of the SiC sample.

Figure 23 shows $R$-curves and $K_{spp}$-curves at the maximum load. $R$-curves of this material were also estimated from the applied load versus back-face strain curves in Fig. 22. The $K_s$-$\Delta a$ relation showed rising $R$-curve behavior such as shown by other porous materials. The average value of $K_s$ at which the $K_{spp}$-curve met with the $R$-curve was 0.56 MPa $\sqrt{m}$, and the average amount of crack extension before reaching the maximum load was about 0.55 mm.

Figure 24 shows the SEM micrographs of the side-view and fracture surface in the vicinity of the notch tip after the test. The main crack that arose from the notch tip propagated along grain boundaries between the secondary grains in the Mg$_2$Al$_4$Si$_5$O$_{18}$ sample, and was bridged by some grains. It was found that the grain bridging of secondary grains contributed to the rising of the $R$-curve. As mentioned above in section 2.1, the main pore that controls the permeability of the ceramic filter was made up of spaces between secondary grains, and the bonding strength of its grain boundary phase was very weak. Therefore, the main crack predominately propagated along the grain boundaries between secondary grains. Transgranular fracture of secondary grains was observed in a few areas since the fracture toughness of secondary grains was also weak.

From the above experimental results, it was concluded that $R$-curve behavior of porous ceramics could be evaluated by the compliance method using the applied load vs. back-face strain plot obtained from the fracture toughness test using the SEVNB technique.

3.5 Effect of Microstructure on $R$-Curve Behavior

Figure 25 shows the relationship between mean grain size, $d$, and amount of crack extension, $\Delta a$, until the applied load reached the maximum for all samples. The $\Delta a$ of the Al$_2$O$_3$ (A) sample in which the matrix grain size is the smallest among the four types of porous ceramics was very short from 0.24 to 0.45 mm, and specimens were broken brittlely in a short time after reaching the maximum load. On the other hand, the $\Delta a$ of the remaining three samples were slightly long from 0.55 to 0.69 mm. The main crack tended to propagate stably after reaching the maximum load, and specimens were difficult to break unstably. The fracture of porous ceramics propagated as a result of selective microfracture of the glassy
grain boundaries with a weakest bonding-strength. However, in the case of porous ceramics having very small matrix grains such as the Al₂O₃ (A) sample, the main crack propagated straightly, and then did not contribute significantly to the rise in the R-curve. On the other hand, in the case of porous ceramics having very large matrix grains such as SiC, Al₂O₃ (B) and Mg₂Al₄Si₂O₁₁ samples, the main crack made a detour around matrix grains, and was grain-bridged across the propagated crack faces. As a result, the R-curve rose with an increase in crack extension. It can be concluded that at the initiation of fracture in porous ceramics, the stable crack easily grows by the fracture of glassy grain boundaries since the bonding strength between matrix grains is very weak. R-curve behavior after crack growth is greatly dependent on the matrix grain size. It is found that the increment of the R-curve by grain bridging becomes larger for coarser-grain sized porous ceramics.

In the present study, the R-curve was estimated on the assumption that only one crack propagated straightly from the initial notch tip and crack extension was determined from the compliance change using back-face strain. Therefore, the amount of crack extension, Δa, might be slightly different from actual crack extension, when the crack detoured around large matrix grains. The effects of crack deflection and grain-bridging on either the compliance or R-curve behavior will be examined in the future.

4. Conclusions

The fracture toughness methods for porous ceramics were examined, and R-curve behavior of four kinds of porous ceramics was evaluated using the SEVNB technique. The results are summarized as follows:

(1) The IF and SEPB methods were inapplicable because it was difficult to introduce both indentation crack and pop-in crack in porous ceramics.

(2) The SEVNB method was applicable because the V-notch could be easily machined by grinding. R-curve behavior could be evaluated from the compliance change using the applied load versus back-face strain plot.

(3) Although the Kc value of porous ceramics at the maximum load was much smaller than the fracture toughness of dense ceramics, the Kc-Δa relationship showed rising R-curve behavior.

(4) The fracture of porous ceramics was controlled by the microfracture of glassy grain boundaries between matrix grains, so that the fracture propagated at the lower Kc value, the loading rate of fracture toughness test was slower.

(5) R-curve behavior after crack growth was greatly dependent on the matrix grain size. The increment of the R-curve by grain bridging was found to become larger for coarser-grain sized porous ceramics.

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Appendix: Evaluation of R-curve Behavior by Compliance Method Using Back-Face Strain

The compliance, λ, of the SEVNB specimen is expressed as

$$λ = \varepsilon_b/P$$  (1)

where $\varepsilon_b$ is back-face strain and $P$ is bending load. The amount of crack extension, $\Delta a$, can be estimated from the above compliance change, if the relation of compliance, λ, for arbitrary crack length, $a$, can be obtained. In this study, the following equation that was derived by Tanaka et al. from the finite element analysis was used

$$\lambda_0 = 1.000 + 0.04596 + 3.4148d^2 + 94.986a^2 - 816.22a^2 + 3433.3d^2 - 8003a^2 + 10740a^2$$

$$- 7801.4a^2 + 2451.2a^2$$  (2)

where $\lambda_0$ is compliance without notch, $a = a/W$ is dimensionless crack length, $a$ is crack length and $W$ is thickness of the SEVNB specimen. The $\lambda_0$ is given by

$$\lambda_0 = \frac{3(S_1 - S_0)}{2EBW}$$  (3)

where $E$ is Young's modulus, $B$ is width of specimen, $S_1$ and $S_0$ are outer and inner spans, respectively.

Figure 26 shows the relationship between $\lambda_0\Delta a$ and $a$, as defined by Eq. (2). In this figure, the experimental data calculated from the applied load vs. back-face strain curve of each sample were plotted together. The experimental data agree very well with
the results of the finite element analysis. It was found that the amount of crack extension could be estimated from the change of back-face strain.

In the experiment, the initial compliance, $\lambda^*$, was first calculated from the slope of the applied load vs. back-face strain curve without crack growth. The compliance, $\lambda^*$, was determined by substituting $a_0$ and $\lambda$ into Eq. (2), where $a_0$ is initial notch depth. Next, the compliance, $\lambda$, during crack growth was calculated from each data point of the applied load vs. back-face strain curve. Substituting $\lambda$ into Eq. (2) again, the change of crack length, $a$, for the applied load, $P$, was obtained. Subsequently, the stress intensity factor, $K_s$, for the crack length, $a$, was calculated by the following equation (9), and then $K$-curve behavior was derived from the relationship between the calculated $K_s$ and the amount of crack extension, $\Delta a (= a - a_0)$:

$$K_s = \frac{3P(S_1 - S_0)}{2BW^2} - F(a) \sqrt{\pi a}$$ (4)

where $F(a)$ is dimensionless geometric factor. $F(a)$ is given by

$$F(a) = \sqrt{\frac{2}{\pi a}} \tan \left( \frac{\pi a}{2} \right) \frac{0.923 + 0.199 (1 - \sin \frac{\pi a}{2})^4}{\cos \frac{\pi a}{2}}.$$ (5)

In the present study, only one crack was assumed to propagate straightly from the notch tip, even though the crack did not always propagate straightly. The compliance change was assumed to be entirely caused by the stable crack growth (9).

References