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Crack-healing behavior and resultant strength properties of silicon carbide ceramic

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Abstract

Silicon carbide (SiC) has good high temperature strength and resistance to radioactivity. However, it has poor fracture toughness. To overcome this weakness, a crack-healing ability is very desirable. This study focuses on the crack-healing behavior of commercial SiC ceramic. The crack-healing behaviors of SiC ceramic were investigated systematically, as a function of crack-healing temperature, time, crack size and temperature dependence of the resultant bending strength. Three-point bending specimens were made and a semi-elliptical crack was introduced on the specimen by a Vickers indenter. Pre-cracked specimens were healed at various conditions. All fracture tests were performed on a three-point loading system with a 16 mm bending span. The main conclusions obtained were: (1) optimized crack-healing condition is; temperature: 1773 K, 1 h in air. (2) The maximum crack size that can be healed completely under the optimized condition was semi-elliptical surface crack of 450 μm in diameter. (3) Limiting temperature for bending strength of crack-healed zone for bending strength was about 873 K.

Keywords: SiC; Crack healing; Strength; Annealing

1. Introduction

Silicon carbide (SiC) has good high temperature strength and is resistant to radioactivity. It is a leading candidate material for future generation gas turbines and the inner containment of nuclear fusion reactors.[1,2] The demand is for the highest structural integrity. To meet this demand, the following three technologies are very useful: (a) non-destructive inspection with very high ability, (b) increase fracture toughness by fiber-reinforcement and decrease the sensitivity to crack, (c) introduce crack-healing ability.

A critical semi-circular crack diameter of SiC is about 20–50 μm, the strength is reduced to about 50% by this crack, thus it is almost impossible to detect the crack with high reliability. Then the application of the above (a) technology to SiC structure is almost impossible. There are much useful researches related to the above (b) technology. However, a crack-healing ability is assumed to be the pre-eminent technology similar to (b) above. It is well known that SiC exhibits a very interesting crack-healing ability.[3–7] However, very few studies have been made on the crack-healing behavior systematically, as a function of crack-healing temperature and time, crack-healing environment and chemical composition of SiC, and also very few studies have been made on the fracture behavior of crack-healed components, such as bending strength and fatigue strength at elevated temperature.

Recently, one of the authors developed silicon nitride,[8–10] mullite[11,12] and alumina[13,14] with very high crack-healing ability. If a SiC were able to exhibit high crack-healing ability similar to the above ceramics, it would be very desirable for structural integrity. As a phase 1 study, crack-healing behavior of three commercial SiCs was investigated systematically.[6] As a phase 2 study, the SiC that showed the best crack-healing behavior among them was selected as a sample, and its crack-healing behavior was investigated

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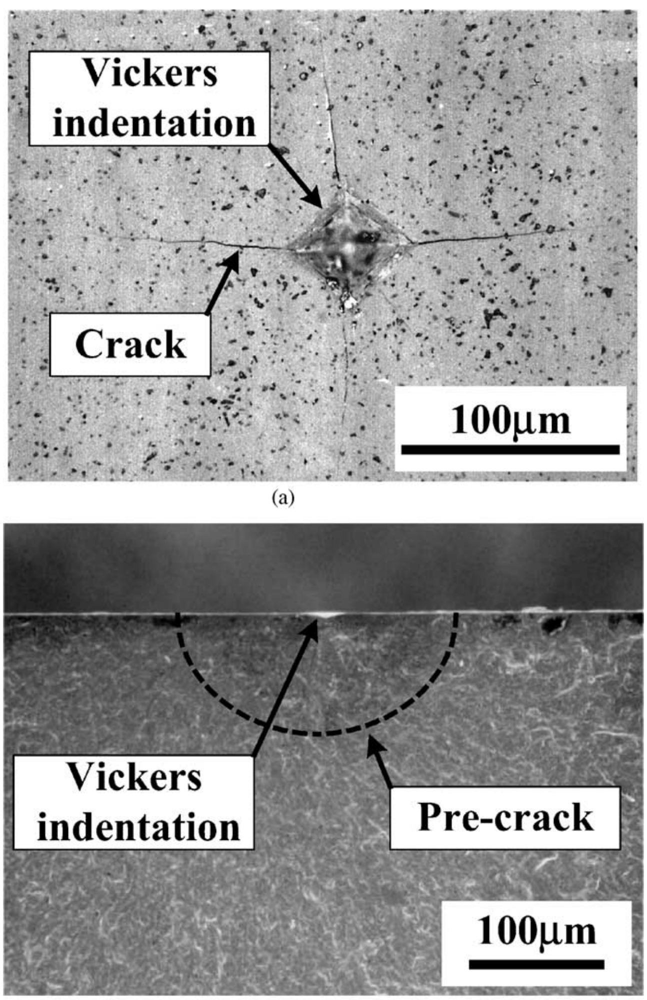
systematically, as a function of crack-healing temperature, time, crack size and temperature dependence of resultant bending strength.

2. Material, specimen and test method

The test sample is SiC ceramic which exhibited the best crack-healing ability among three commercial SiC ceramics.[6] The sintered plates were cut into specimens with 3 mm × 4 mm × 23 mm. A tensile surface of the test specimen was mirror finished. A semi-elliptical surface crack was made at the center of the tensile surface of the test specimen using a Vickers indenter at a load of 4.9–196 N. By this method, semi-elliptical cracks of 50–700 μm in surface crack length were made as shown in Fig. 1(a). The ratio of depth (a) to half surface length (c) of crack (aspect ratio) was $a/c \approx 0.8$–0.9. A micrograph of crack profile observed by scanning electron microscopy (SEM) is shown in Fig. 1(b).



(a)

(b)

Fig. 1. SEM micrographs of (a) indentation and cracks, (b) pre-crack shape.

Three kinds of the crack-healing processes are conducted to this study. Pre-cracked specimens were healed at 1573–1773 K for 40 min to 100 h in air environment, as healing process I. The healing processes II and III were selected to investigate the effect of crack-healing conditions on the bending strength at 1073 K. The healing process II is that pre-cracked specimens were healed at 1773 K for 40 min followed by crack-healing at 1473 K for 5, 20 or 100 h, respectively. The healing process III is that pre-cracked specimens were healed at 1773 K for 40 min followed by crack-healing at 1473 K for 5 h five times. The crack-healing treatments under cyclic heating are called “multi crack-healing” in this paper.

The bending test of a crack-healed specimen was carried out at room temperature ∼1673 K. All fracture tests were performed on a three-point loading system with a 16 mm bending span. The cross-head speed in the bending tests was 0.5 mm/min.

The specimen surfaces were analyzed by electron probe microanalysis (EPMA) and X-ray diffraction. And the fracture surfaces were analyzed by scanning electron microscopy (SEM).

3. Test results and discussion

3.1. Effect of crack-healing temperature and time on the bending strength of crack-healed specimen

Fig. 2 shows the effect of crack-healing time on the bending strength (σ_B) of the crack-healed specimens at RT Fig. 2(a) shows the test results on specimens crack healed at 1773 K. The average σ_B of smooth specimen (○) is ≈560 MPa, and the σ_B of standard pre-cracked specimen ($2c \approx 200$ μm, △) is ≈180 MPa. In the case of crack-healing time 40 and 60 min, the average σ_B of crack-healed specimen recovered up to 585 MPa. The crack-healed specimen (▲) showed a little higher bending strength than that of the smooth specimen. Of course, this increase in the σ_B of the smooth specimen was attained by healing the small surface cracks. The specimens with (※) show that fracture occurred outside of the crack-healed zone indicating that the pre-crack was healed completely. In Fig. 2(a), two of seven samples crack healed for 40 and 60 min fractured outside the crack-healed zone, and also the σ_B exhibited a little scatter, thus it can be concluded that crack healing was complete. Nevertheless, the heat treated smooth specimen (●) exhibited the same level of bending strength up to 40 h, the crack-healed specimen (▲) over 2 h exhibited considerably lower bending strength. This decrease in bending strength means that some degradation occurred during long term crack healing. Kim and Moorhead[15] observed significant reductions in strength of silicon-based ceramics as a result of material loss through the formation of volatile species such as SiO(g). Moreover, previous investigators[16,17] observed bubbles on polycrystalline SiC and thought that they

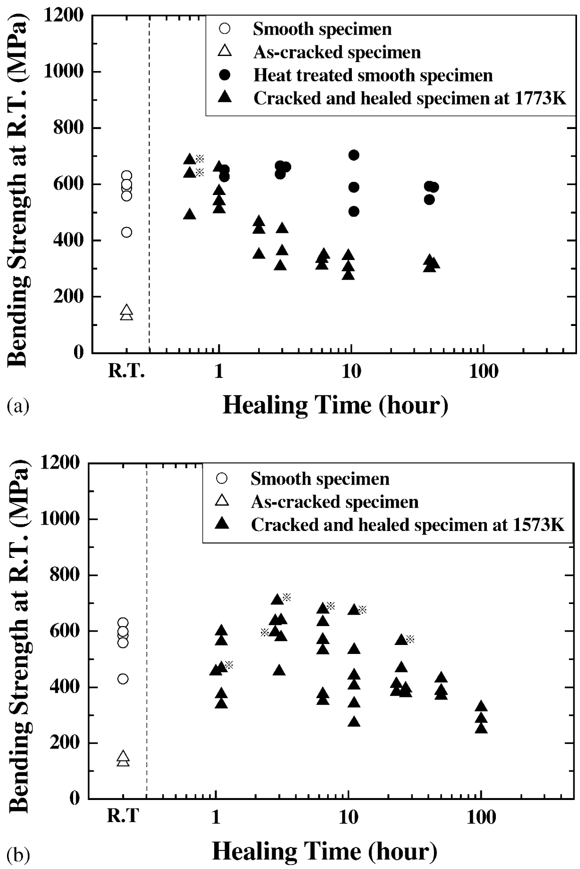


Fig. 2. Relationship between crack-healing time and bending strength at room temperature. Data marked with (※) indicate that fracture occurred outside the crack-healed zone: (a) crack healed at 1773 K, (b) crack healed at 1573 K.

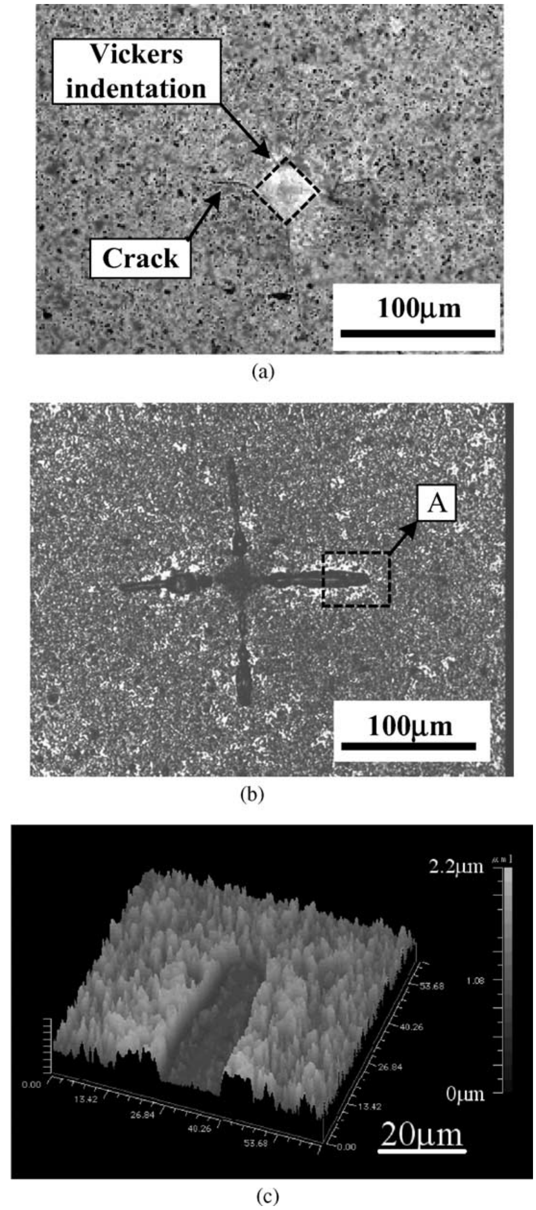


Fig. 3. Photograph of surface condition after crack healing: surface crack length (2*c*) is 200 µm: (a) crack-healing condition: 1773 K, 40 min, (b) crack-healing condition: 1773 K, 10 h, (c) detail of crack-healed zone (A) by laser microscope.

resulted from evolution of CO at the SiC/SiO_2 interface during oxidation. Therefore, it was assumed that the degradation of this SiC was occurred by the formation of volatile SiO and CO(CO_2) gas.

Fig. 2(b) shows the test results on specimens crack healed at 1573 K. In the case of crack-healing time from 1 to 25 h, the σ_B of crack-healed specimens exhibited large scatter. Six of the 30 samples fractured outside the crack-healed zone, however nine specimens exhibited lower σ_B than 400 MPa indicating that crack healing was incomplete. The crack-healed specimen (▲) over 50 h exhibited considerably lower bending strength showing a little scatter in the σ_B. This decrease in σ_B also means that some degradation occurred during long term healing. The initiation time of this degradation is defined as t_D, then from Fig. 2(a) and (b), t_D can be determined as 2 and 50 h, respectively. Based on the σ_B versus crack-healing time at 1573–1773 K, the optimized crack-healing condition was decided as 1773 K, 1 h in air.

Fig. 3 shows the surface condition of a crack-healed specimen. The surface condition of a 40 min crack-healed specimen was shown in Fig. 3(a), cracks can be seen faintly, however strength recovered completely as shown in Fig. 2 and no special degradation such as a groove cannot be found. On the other hand, wide and deep groove can be seen on the 10 h crack-healed specimen as shown in Fig. 3(b) and (c). The true depth of the groove cannot be measured because of the scale limit of the laser microscope. These grooves acted as stress concentrator, thus bending strength was reduced considerably.

To investigate the crack-healing material, the elemental mapping images of Si, C and O on the specimen surface were investigated by EPMA, as shown in Fig. 4. On the smooth specimen which was not subjected to crack-healing treatment, a large amount of Si and C and very small amount of O were detected as shown in Fig. 4(a–c). By contrast,

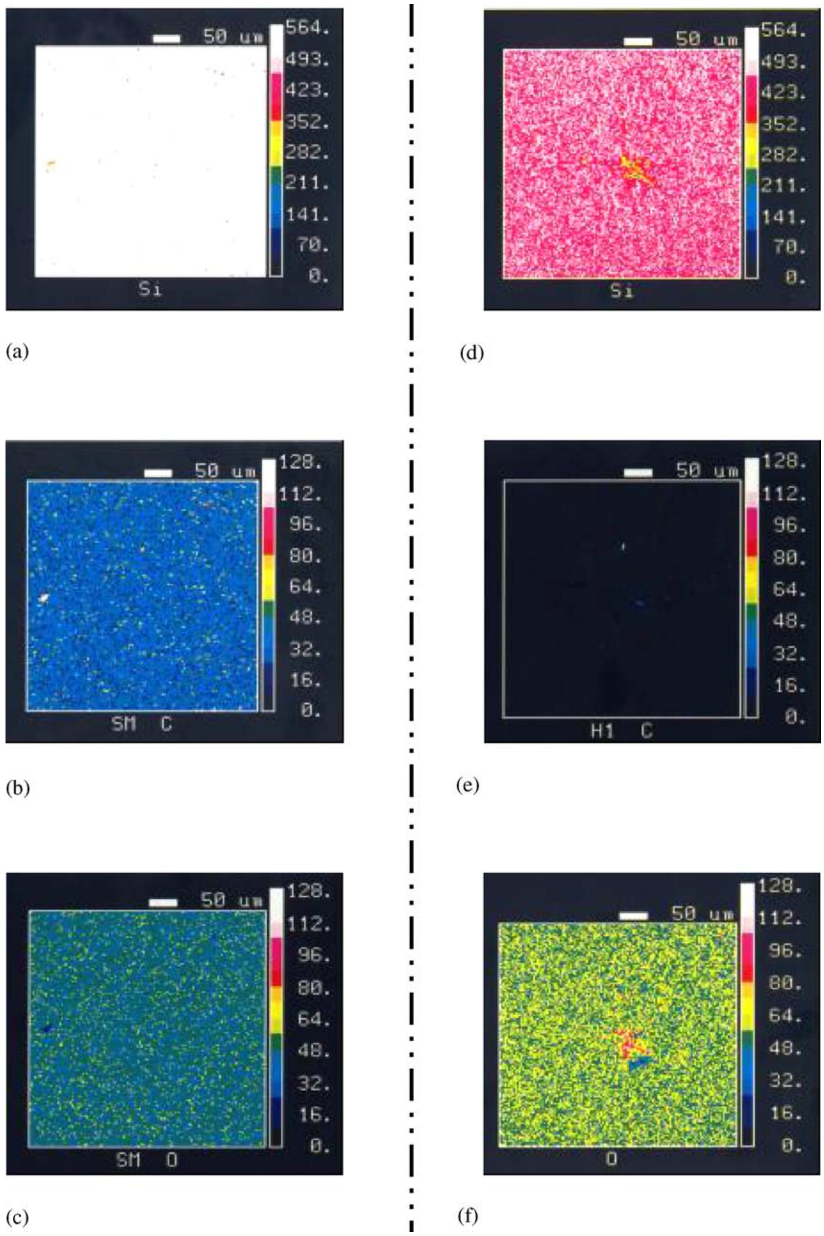


Fig. 4. The elemental mapping of surface before and after crack healing by EPMA: (a) smooth specimen—Si, (b) smooth specimen—C, (c) smooth specimen—O, (d) crack healed at 1773 K for 1 h—Si, (e) crack healed at 1773 K for 1 h—C, (f) crack healed at 1773 K for 1 h—O.

on the crack-healed specimen at 1773 K, 1 h in air, a large amount of Si and O, and little C were detected as shown in Fig. 4(d–f). Thus, it was assumed that Si oxide was produced on the specimen surface by the crack-healing treatment. The crack-healing reaction of this specimen was estimated as follows:

$$SiC + \frac{3}{2}O_2 \rightarrow SiO_2 + CO(CO_2) \quad (1)$$

The SiO_2 has two phases; a glassy and a crystal phase. Fig. 5 shows the examples of X-ray diffraction from the surface oxide layer. The amount of crystallized SiO_2 was dependent on the crack-healing time. The 1 h crack-healed specimen had a very small amount of crystallized SiO_2 compared to 40 h crack-healed specimen. Therefore, it can be concluded that this SiC ceramic recovered its bending strength by means of glassy phase SiO_2. And, based on above facts, it was assumed that the groove in Fig. 3(b) was caused by the vaporization of glassy SiO_2.

In a previous study,[18] activation energies for the crack healing were evaluated, however, in this case, the σ_B of crack-healed specimens healed at 1723–1473 K exhibited large scatter as shown in Fig. 2(b), thus activation energy for the crack healing could not be evaluated. However, the degradation behavior during crack healing was observed clearly as shown in Fig. 2(a) and (b), then $1/t_D$ versus healing temperature (T_H) was plotted in Arrhenius curve as shown in

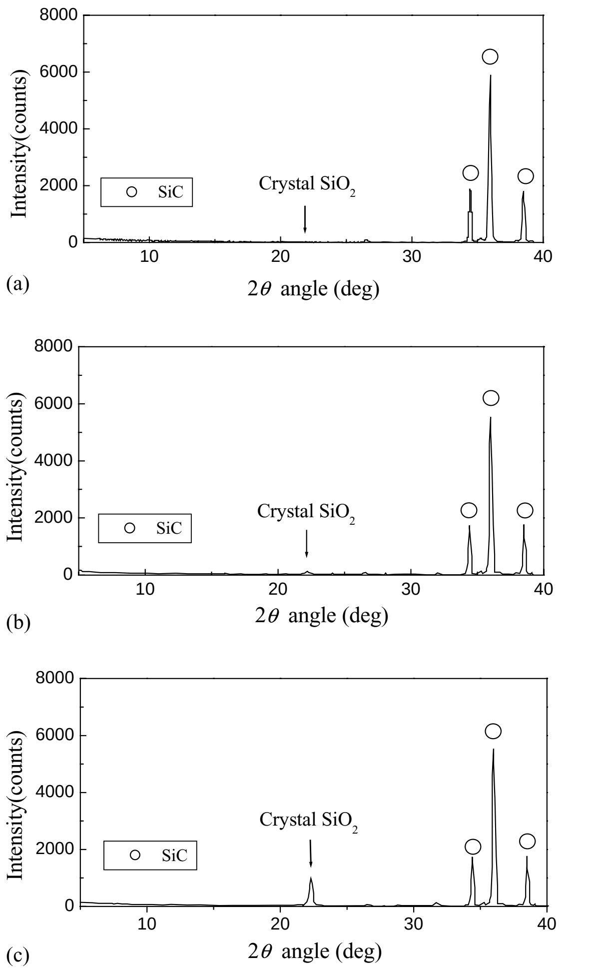


Fig. 5. Results of X-ray diffraction from sample surfaces: (a) smooth specimen subjected to no crack-healing treatment, (b) crack healed at 1773 K, 1 h, (c) crack healed at 1773 K, 40 h.

Fig. 6. From this Fig. 6, activation energy for degradation is evaluated as ≈255 kJ/mol. In early studies of the oxidation of pure SiC,[19–22] activation energies for forming reaction of SiO_2 in oxygen within the temperature range of interest were obtained. The activation energies for forming reaction of SiO_2 from SiC were measured as 142–293 kJ/mol and 128–213 kJ/mol by Costello and Tressler,[19] and Filipuzzi et al.,[20] respectively. However, these values were obtained by the experiment employed short exposure times (=24 h). Ogbuji and Opila,[21] and Fox[22] studied the oxidation behavior of SiC films made by CVD from 1473 to 1773 K for 100 h. The activation energies were measured as 118 and 190 kJ/mol, respectively. Therefore, the activation energy for degradation was higher than the literature value for some SiO_2-former.

3.2. Effect of pre-crack size on crack-healing behavior

Fig. 7 shows the effect of pre-crack length (2*c*) on the crack-healing behavior at 1773 and 1573 K. In Fig. 7(a), the σ_B of cracked specimens (△) decreases with increasing 2*c*.

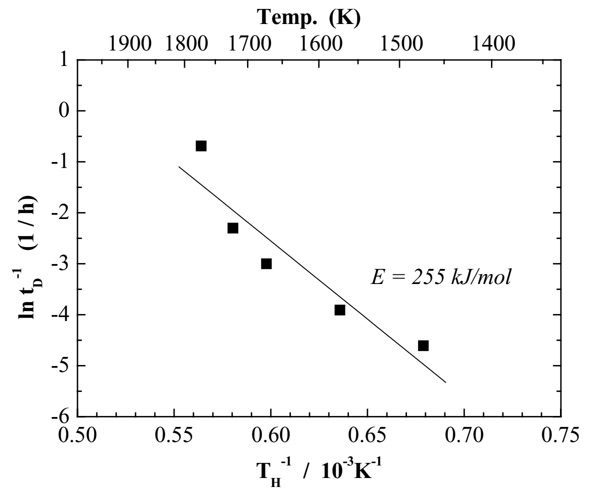


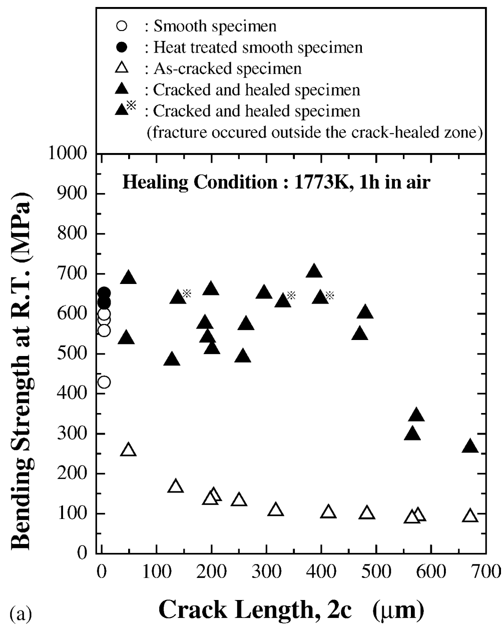
Fig. 6. Arrhenius plots showing the relationship between degradation time (t_D) and healing temperature (T_H) for SiC ceramic.

In the case of $2c \leq 450\ \mu m$, the σ_B of crack-healed specimens exhibited the same level of bending strength to that of smooth sample indicating crack was healed almost completely. However, in the case of $2c > 50\ \mu m$, the σ_B of crack-healed specimens decreased with increasing 2*c*. Thus, it can be concluded that the maximum crack size ($2c_{max}$) that can be healed completely is $2c_{max} \approx 450\ \mu m$ in surface length when the crack is healed at 1773 K, 1 h in air. The value is twice as large compared with the others ceramics having crack-healing ability, such as Si_3N_4/SiC,[23,24] Al_2O_3/SiC[25] and mullite/SiC.[26] As mentioned in Section 3.1, SiC and O_2 is necessary for crack healing. Therefore, it is assumed that the oxidation reaction of this SiC occurred in whole of the crack surface. In contrast, the oxidation reaction of the above ceramics occurred at only SiC on the crack surface.

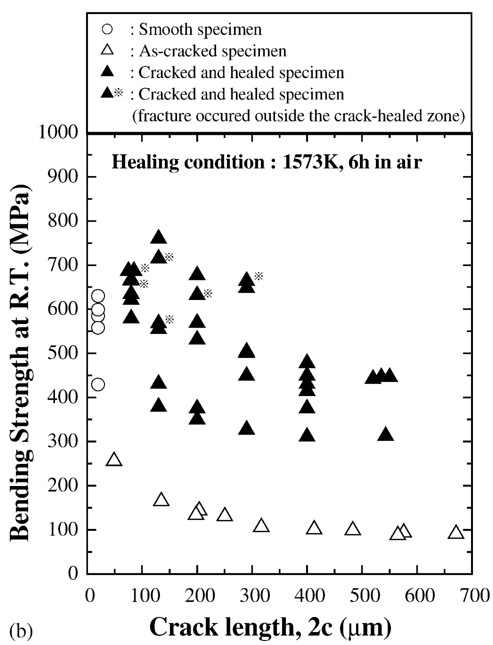
In the case of crack-healing condition at 1573 K, 6 h in air, the bending strength showed large scatter in the $2c > 100\ \mu m$ region, as shown in Fig. 7(b). Therefore, the maximum crack size that can be healed completely is $2c_{max} \approx 50\ \mu m$ in surface length. The previous studies[23–26] showed that sufficient temperature and time is necessary for crack healing. However, the bending strength of specimens crack healed at 1573 K could not recovered despite increasing the holding time, as shown in Fig. 2(b). Therefore, it was assumed that the increasing crack-healing temperature is useful for crack healing of this SiC ceramic rather than increasing crack-healing time.

3.3. Effect of testing temperature on the bending strength of crack-healed specimen

Fig. 8 shows the effect of the testing temperature on the σ_B of crack-healed specimens healed at 1773 K, 1 h in air. The smooth specimen exhibited a high bending strength (≈560 MPa) up to 1573 K. However, the crack-healed specimen showed high σ_B only up to 873 K, and over 873 K,



(a)



(b)

Fig. 7. Effect of pre-crack length on the bending strength of crack healed specimen at room temperature: (a) crack healed at 1773 K, 1 h, (b) crack healed at 1573 K, 6 h.

the σ_B of the crack-healed specimens decreased suddenly and exhibited low values of $\sigma_B \approx 300$ MPa. Therefore, the limiting temperature for bending strength of the crack-healed specimen was defined as 873 K. In a previous study,[8] it was shown that whether the crack-healing material was crystallized or not had a large effect on the high temperature strength and fatigue strength. Therefore, for this crack-healed specimen of SiC ceramic it was assumed that the limiting temperature for bending strength is relatively low, because the crack-healing material of this SiC is glassy SiO_2, as mentioned in Section 3.1. The previous studies[24,26] showed that the limiting temperature for bending strength of Si_3N_4/SiC, mullite/SiC is above 1273 K although the crack-healing material was also glassy SiO_2. This difference is probably due to the bonding force of matrix and crack-healing material, but it is not yet resolved. This matter is the subject of further study.

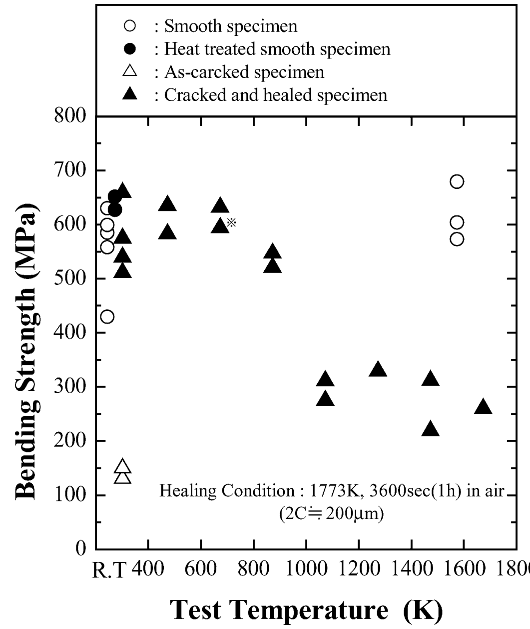


Fig. 8. Effect of test temperature on the bending strength of crack-healed specimen.

Fig. 9 shows the effect of test temperature on the σ_B of crack-healed structural ceramics developed by K. Ando et al.[7,23–26] The phase of grain boundary and crack-healed zone of Si_3N_4 exhibited the glassy phase, thus the bending strength decreased suddenly over 1273 K.[23] On the other

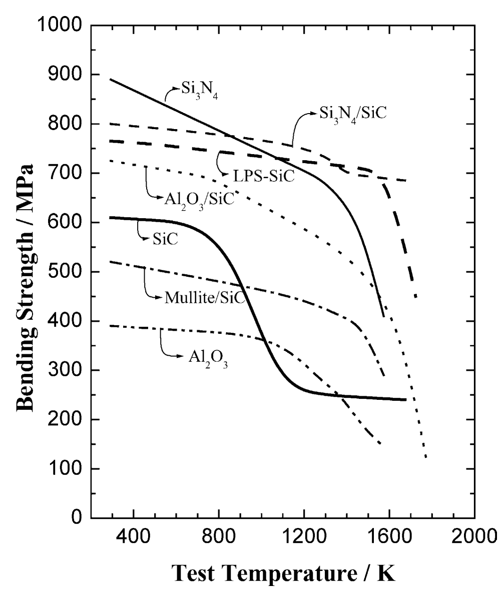


Fig. 9. Effect of testing temperature on the bending strength of crack-healed structural ceramics.

hand, the Si_3N_4/SiC exhibited the crystal phase in both areas, thus it exhibited very high limiting temperature for bending strength (≥1573 K).[24] The Al_2O_3/SiC exhibited a very high limiting temperature for bending strength (≈1573 K) compared to that of monolithic Al_2O_3, because very small SiC particles (0.27 μm) were distributed in Al_2O_3 grain and formed nano-size composite material.[25] The mullite/SiC also exhibited quite a high limiting temperature for bending strength (≈1523 K).[26] Recently, the SiC for which the crack-healed zone exhibited high limiting temperature for bending strength (≈1573 K) was developed.[7]

3.4. *Effect of crack-healing conditions on the bending strength at 1073 K*

This crack-healed specimen of SiC has low limiting temperature for bending strength, as shown in Fig. 8. However, that of the smooth specimen of SiC (base material) is over 1573 K. To increase the limiting temperature for bending strength of the crack-healed specimen, the effect of crack-healing conditions on the bending strength at 1073 K was investigated systematically.

The crack-healed zone of Si_3N_4/SiC healed at 1573 K exhibited high σ_B and fatigue limit at 1273 K,[8,27] the specimen crack healed at 1273 K exhibited the low σ_B and fatigue limit at 1273 K. Then many efforts were made to increase the σ_B and fatigue limit. Finally, it was found that the multi-crack-healing process or long-time crack-healing treatment at 1273 K were able to increase the σ_B and fatigue limit by producing a large amount of crystal SiO_2 in the crack-healed zone.

In this SiC sample, degradation was occurred by long time crack-healing treatment at a relatively high temperature range, thus multi-crack-healing process was applied. Fig. 10 shows the effect of crack-healing conditions on the bending strength at 1073 K. The crack-healing specimen at 1773 K, 1 h exhibited a low value of bending strength at 1073 K (≈300 MPa). And all multi-healed specimen exhibited low bending strength at 1073 K and showed the bending strength similar to crack-healing specimen at 1773 K, 1 h. Therefore, it can be concluded that the crack-healing condition has no effect on the limiting temperature for bending strength of the crack-healed zone of this SiC ceramic.

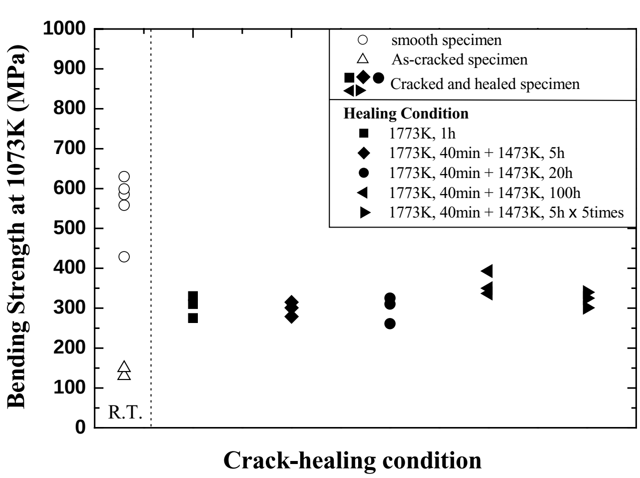


Fig. 10. Effect of crack-healing condition on the bending strength of crack-healed specimens at 1073 K.

4. Conclusions

The basic crack-healing behavior of the commercial SiC which showed the best crack-healing behavior among three tested previously was investigated systematically, as a function of crack-healing temperature, time, crack size and temperature dependence of the resultant bending strength. The main conclusions obtained were as follows:

(1) The optimized crack-healing condition was 1773 K, 1 h in air.
(2) The maximum semi-elliptical crack size ($2c_{max}$) in diameter that could be healed completely was dependent on crack-healing conditions;

At healing conditions of 1773 K, 1 h in air: $2c_{max}$ ≈450 μm in diameter.
At healing conditions of 1573 K, 6 h in air: $2c_{max}$ ≈50 μm in diameter.

(3) The limiting temperature for bending strength for the bending strength of smooth specimen of SiC is over 1573 K. However, that of crack-healed specimen was about 873 K, and crack-healing conditions have no effect on the limiting temperature for bending strength.

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