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Crack-healing behaviour of mullite/SiC/Y_2O_3 composites and its application to the structural integrity of machined components

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Abstract

High-strength mullite/SiC/Y_2O_3 composite ceramics with a great crack-healing ability were developed and subjected to three-point bending. A semicircular surface crack 100 μm in diameter was made in each specimen. Crack-healing behaviour was systematically studied as a function of crack-healing temperature and healing time, and the bending strength of the specimens crack-healed up to 1573 K was investigated. The effects of the crack-healing treatment on the structural integrity and reliability of machined components were studied systematically. Four main conclusions were drawn from the present study: (1) mullite/SiC/Y_2O_3 composite ceramics have the ability to heal after cracking from 1423 K to 1673 K in air, (2) the heat-resistance limit temperature for strength of the crack-healed specimen is ≅1473 K, while at 68% of the specimens fractured from outside the crack-healed zone in the tested-temperature range of 300–1573 K, (3) due to the crack-healing treatment, the strength of the machined specimen increased by 40–200%. Local fracture strength was assumed to recover completely, and the cracks formed by machining were healed completely, and (4) a large self-crack-healing ability is desirable for obtaining a higher structural integrity in ceramic components.

Keywords: Mullite/SiC/Y_2O_3 composite; Crack-healing; Machined components; Strength recovery; Structural integrity

1. Introduction

Mullite is a chemical compound composed of Al_2O_3 and SiO_2, and has superior oxidation resistance and heat-resistance limit temperature ($T_{HR} \cong 1423\,K$) for strength. However, it has two weak points: low bending strength (σ_B) (≅350 MPa) and low fracture toughness (≅2.5 MPa $m^{1/2}$). These weaknesses restrict the application of mullite for important components. To overcome these weaknesses, the authors adopted the following three objectives: (1) to increase strength up to at least 500 MPa, (2) induce a large self-crack-healing ability and overcome the low fracture toughness, and (3) make clarify the crack-healing behaviour of machined mullite/SiC components.

Objective (1) was achieved successfully by adding 15–20 mass% nano-size SiC particles to prevent the grain growth of mullite during sintering.[1–4] However, the fracture toughness is low yet; thus, the mullite/SiC is very sensitive to flaws such as cracks and pores.

As to objective (2), monolithic mullite has no crack-healing ability. However, a large self-crack-healing ability was successfully induced similar to that of Al_2O_3/SiC[5–10] and Si_3N_4/SiC[11–16] by adding 15–20 mass% nano-size SiC particles.[1–4] Especially, the mullite/SiC composite exhibits very interesting in situ crack-healing ability at 1273–1473 K.[17–19] In other words, the mullite/SiC can heal a crack even under 5 Hz cyclic stress and exhibit the same fatigue limit as the base material at the healing temperature. As to objective (3), the basic crack-healing behaviour of an indented crack was studied systematically in terms of the functions of crack-healing temperature and time, the crack-healing mechanism, and the resultant

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strength behaviour of the crack-healed components at elevated temperatures.[1–4,17–19] However, the strength recovery behaviour of machined component have not yet been studied.[20]

Applying the high crack-healing ability of ceramics to structural components for engineering use could result in great benefits, such as the increased reliability of the structural ceramic components and reduced inspection, machining, and polishing costs for the ceramic components.

On the other hand, a Y_2O_3 is very useful sintering additive.[21–25] Especially, $Si_3N_4/SiC/Y_2O_3$ also exhibits very interesting in situ crack-healing ability at 1073–1473 K.[26–28] $Al_2O_3/SiC/Y_2O_3$ exhibits very high bending strength (≈1300 MPa) at room temperature.[29] It has been reported that the optimal Y_2O_3 content of mullite is 0.5 mass%.[22,23] It has also been reported that the optimal Y_2O_3 content of mullite/SiC is 1–2 mass%.[24] However, the basic crack-healing behaviour of mullite/SiC/Y_2O_3 has not yet been studied.

From this perspective, the following three research objectives related to mullite/SiC/Y_2O_3 were chosen for the present study: (1) to clarify basic crack-healing behaviour as functions of crack-healing temperature and time, (2) to determine the effect of testing temperature on the bending strength of crack-healed specimen, and (3) to determine the effect of crack-healing treatment on the structural integrity and reliability of machined mullite/SiC and mullite/SiC/Y_2O_3 components.

2. Material, specimen and test method

The mullite powder used in this investigation was KM101 (average particle size = 0.2 μm, Al_2O_3 content = 71.8 wt.%, Kyoritz Co., Ltd., Nagoya, Japan) and SiC powder was ultrafine (average particle size = 0.27 μm, Ibiden Co., Ltd., Oogaki, Japan). Y_2O_3 power (Fine grade, Nippon Yttrium Co., Ltd., Oomuta, Japan) has 0.4 μm mean particle size. The specimens were prepared using a mixture of mullite, 15 vol.% SiC powders and 1.5 vol.% Y_2O_3 powders as additive powders. The mixture was added to alcohol and then blended for 48 h. The mixture was then placed in an evaporator to extract the solvent, and in a vacuum desiccator to produce a dry powder mixture. Following this, the mixture was hot-pressed at 1923 K and 35 MPa in nitrogen for 4 h.

The sintered plate was then cut into specimens measuring 3 mm × 4 mm × 23 mm. The tensile surface of the test specimen was mirror finished. A semi-elliptical surface crack of 100 μm in surface length was made at the centre of the tensile surface of the test specimen using a Vickers Indenter at a load of 19.6 N, as shown in Fig. 1(a). Fig. 1(b) showed the indentation and cracks after crack-healing at 1573 K for 1 h in air. From a micrograph of the crack profile as observed by a SEM in Fig. 1(c), the ratio of depth (a) to half surface length (c) of the crack (aspect ratio) was $a/c \approx 0.9$.

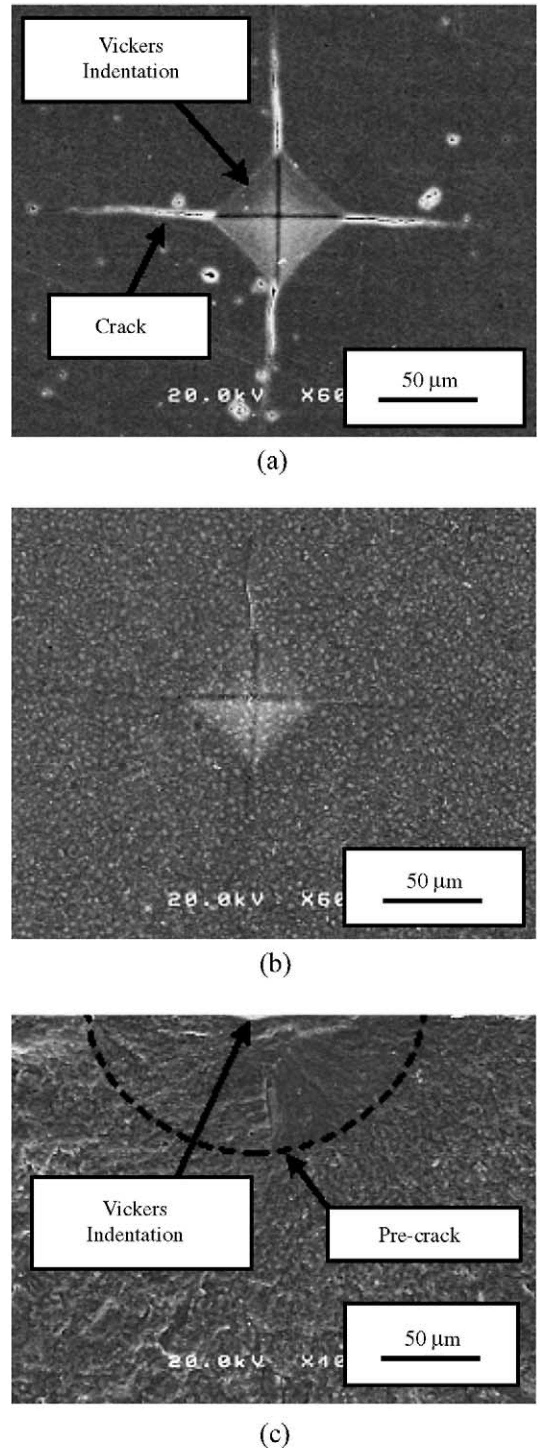

Fig. 1. SEM photomicrographs of (a) indentation and cracks before crack-healing, (b) indentation and cracks after crack-healing and (c) pre-crack shape.

Specimens were crack-healed at 1273–1573 K for 1–100 h in air. The furnace temperature's rate of increase was 10 K/min. Bending tests of the crack-healed specimens were conducted at RT ∼1573 K using a three-point loading system with a span of 16 mm, as shown in Fig. 2. The cross-head speed in the bending tests was 0.5 mm/min.

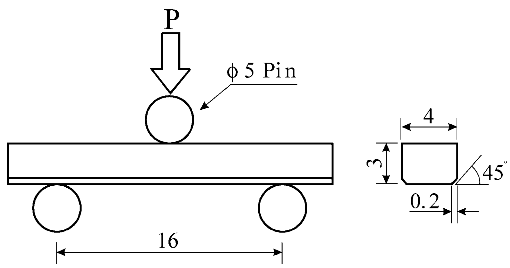

Fig. 2. Three-point loading system and geometry of test specimen, dimensions in millimeters.

Five kinds of machined specimens were made, as shown in Fig. 3(a)–(e). Fig. 3 shows top and front views of the five kinds of machined specimen, respectively. Fig. 4 shows optical micrographs of two specimens. Machining operations were performed by expert workman using a diamond drill or grinder with # 400. These machined specimens were healed in air at 1473 K for 10 h, 1573 K for 1 h and 1623 K for 1 h, respectively, in order to investigate the strength recovery of the machined components by crack-healing. The bending tests of the machined specimens and healed specimens were also performed at RT.

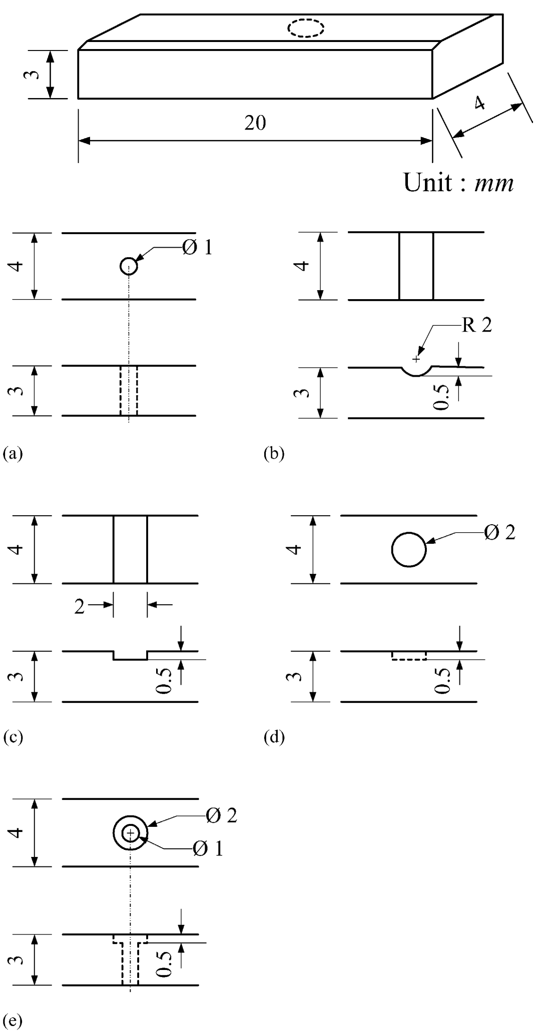

Fig. 3. Schematic drawing of machined specimen: (a) type A: through-hole, (b) type B: semicircular groove, (c) type C: square groove, (d) type D: spot facing with bottom and (e) type E: spot facing.

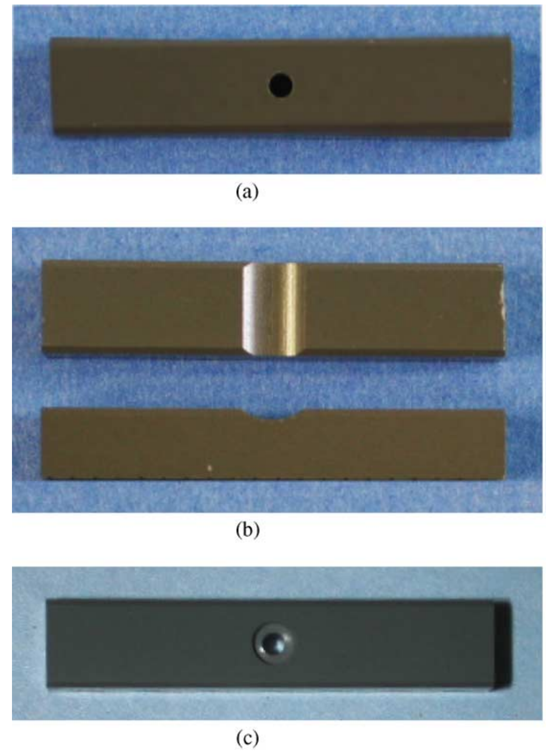

Fig. 4. Optical micrographs of machined specimens of mullite/SiC/Y_2O_3 composite ceramics: (a) type A: through-hole, (b) type B: semicircular groove and (c) type E: spot facing.

3. Test results and discussion

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

Fig. 5 shows the relationship between bending strength at room temperature and crack-healing temperature. The closed and open symbols indicate the test results of mullite/SiC/Y_2O_3 and mullite/SiC[3], respectively, where the SiC content of the mullite/SiC composite is 15 vol.%. The symbols with an asterisk show specimens in which the fracture occurred outside of the crack-healed zone. Fig. 5(a) shows the test results on specimens crack-healed for 1 h. The average σ_B of smooth specimen (●) is ≈450 MPa. By cracking, the bending strength decreased to ≈130 MPa (▲). The closed diamonds (♦) show the σ_B of the specimens crack-healed for 1 h from 1273 K to 1673 K. The σ_B of the specimens crack-healed for 1 h at 1573 K was 600 MPa, and this value was higher than that of the smooth specimen. However, the σ_B of the specimens crack-healed for 1 h below 1473 K was lower, indicating that crack healing was incomplete. In

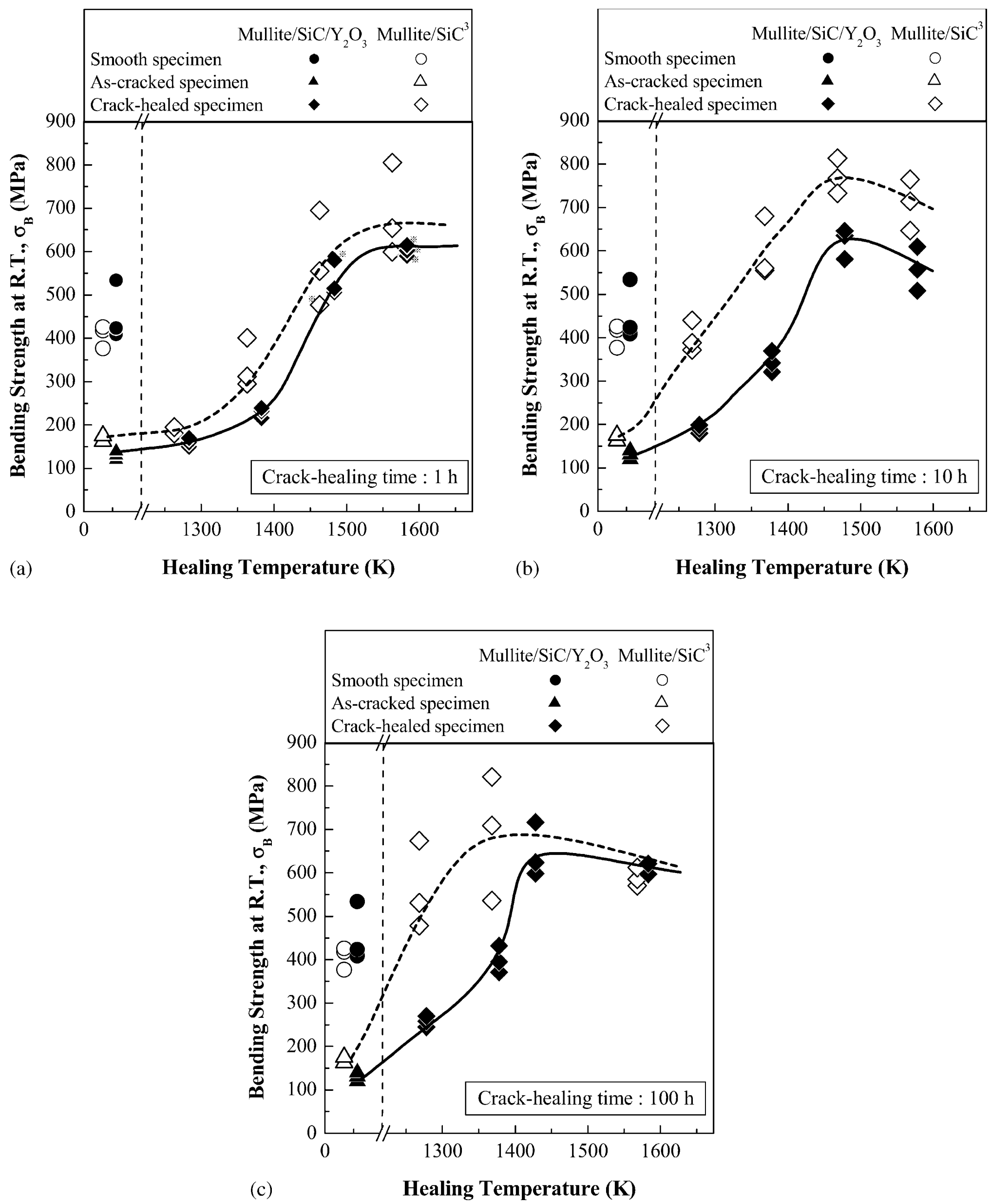

Fig. 5. Relationship between bending strength at room temperature and crack-healing temperature. Data marked with an asterisk indicate that fracture occurred outside of the crack-healed zone: (a) crack-healing time: 1 h, (b) crack-healing time: 10 h and (c) crack-healing time: 100 h.

a crack-healing time of 1 h, the minimum healing temperature (T_H) for the sufficient crack-healing of the as-cracked specimen with $2c = 100\ \mu m$ was determined to be 1573 K. If an as-cracked specimen is healed above T_H, the crack can be healed completely. The open diamonds (⧫) show the σ_B of crack-healed mullite/SiC. The T_H of mullite/SiC was found to be equal to that of mullite/SiC/Y_2O_3. Fig. 5(b) shows the test results of specimens crack-healed for 10 h. The σ_B of the specimens crack-healed for 10 h above 1473 K recovered almost completely. However, the specimens crack-healed below 1373 K had lower σ_B values. Therefore, the T_H at a crack-healing time of 10 h was determined to be 1473 K. Thus, this T_H was also equal to that of the mullite/SiC. Fig. 5(c) shows the test results on specimens crack-healed for 100 h. In this case, the T_H of the mullite/SiC/Y_2O_3 was determined to be 1423 K, and this value was 50 K higher than that of mullite/SiC.

The results confirm that the crack-healing ability of mullite/SiC/Y_2O_3 is similar to that of mullite/SiC. However, the average σ_B of mullite/SiC/Y_2O_3 is lower than that of mullite/SiC. Nevertheless, most specimens of mullite/SiC/Y_2O_3 crack-healed above T_H fractured from a point outside of the crack-healed zone. Therefore, the σ_B of crack-healed mullite/SiC/Y_2O_3 recovered completely, thus the crack-healing condition at 1573 K for 1 h in air was determined to be optimal.

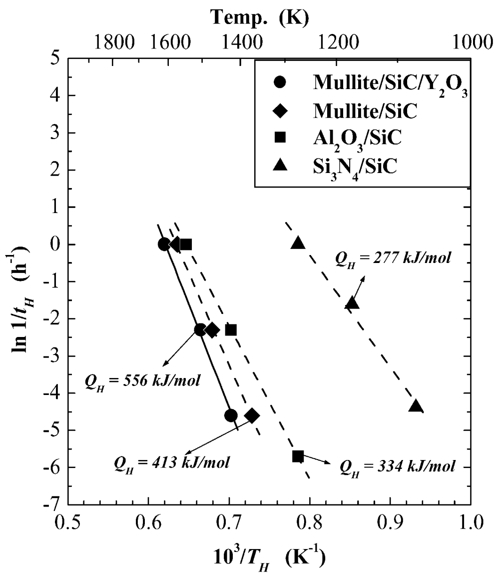

Fig. 6. Arrhenius plots of crack-healing behaviour for mullite/SiC/Y_2O_3, mullite/SiC, Al_2O_3/SiC and Si_3N_4/SiC/Y_2O_3.

It has been reported that crack-healing is caused by the oxidation reaction as follows:[14]

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

$$2SiC + Y_2O_3 + 4O_2 \rightarrow Si_2Y_2O_7 + 2CO_2(CO) \tag{2}$$

Therefore, the crack-healing rate obeys the Arrhenius law. Fig. 5 shows the minimum temperatures (T_H) at which the average σ_B of the crack-healed specimen exceeded the average σ_B of the smooth specimens. For each healing time (t_H = 1, 10 and 100 h), the T_H was determined and the relationship t_H and T_H was expressed as Arrhenius plots, as shown in Fig. 6. The crack-healing behaviour follows Eq. (3),[14,16–17]

$$\frac{1}{t_H} = A_H\, e^{-Q_H/RT_H} \tag{3}$$

where t_H is the minimum healing time for complete strength recovery (h), A_H is proportionality constant (h^{-1}), Q_H is the activation energy (kJ/mol), and R is a gas constant (kJ/mol K). The activation energy (Q_H) for the crack-healing of mullite/SiC/Y_2O_3 is determined to be ≈566 kJ/mol. In earlier studies,[14,16–17] the activation energies for the crack-healing of several ceramics were examined, and are also shown in Fig. 6. The evaluated Q_H of mullite/SiC, Al_2O_3/SiC and Si_3N_4/SiC are 413 kJ/mol, 334 kJ/mol and 227 kJ/mol, respectively. The activation energy for the crack-healing of mullite/SiC/Y_2O_3 is similar to that of mullite/SiC.

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

Fig. 7 shows the effect of testing temperature on the bending strength (σ_B) of a crack-healed specimen. The closed diamonds (♦) indicate the σ_B of mullite/SiC/Y_2O_3 crack-healed at 1573K for 1 h in air. The average σ_B of the crack-healed specimen is about 600 MPa at RT. The σ_B of the crack-healed specimen decreased gradually as the testing temperature increased up to 1473 K. However, the σ_B decreased drastically when the testing temperature increased above 1573 K. Below 1473 K, the specimens did not exhibit plastic deformation before fracture. However, considerable plastic deformation occurred at above 1573 K. The elastic theory was used to the evaluation of σ_B, so that σ_B has been overestimated if a spec-

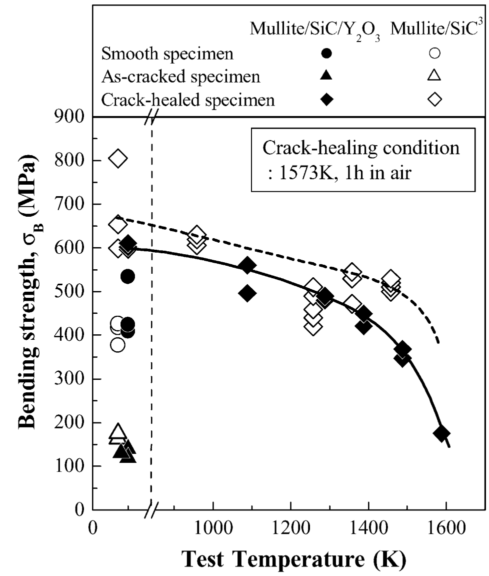

Fig. 7. Effect of test temperature on the bending strength of a crack-healed specimen for mullite/SiC/Y_2O_3 and mullite/SiC. Data marked with an asterisk indicate that fracture occurred outside of the crack-healed zone.

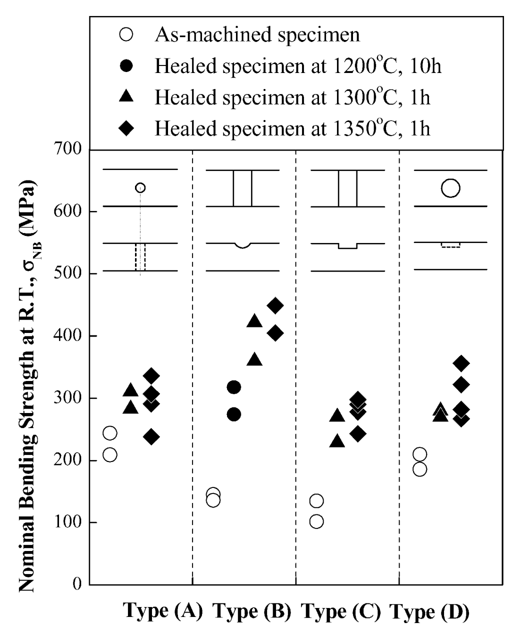

Fig. 8. Effect of crack-healing on the nominal bending strength of the machined specimens of mullite/SiC/Y_2O_3.

imen exhibited plastic deformation. Thus, the heat-resisting limit temperature for bending strength (T_{HR}) was found to be 1473 K for mullite/SiC/Y_2O_3. However, the σ_B of crack-healed mullite/SiC (◊) is almost constant up to 1473 K.

Previous studies have reported that the crack-healing ability and strength properties of Si_3N_4/SiC[11–14] and Al_2O_3/SiC[26] were increased by the addition of Y_2O_3. However, the bending strengths (σ_B) of mullite/SiC/Y_2O_3 at elevated temperatures were lower than those of mullite/SiC ceramics. Therefore, it can be concluded that the 1.5 vol.%. Y_2O_3 additions do not improve the crack-healing behaviour and strength property of the mullite/SiC/Y_2O_3. This was probably due to the additive amount of Y_2O_3 and the sintering condition. This matter is the subject of further study.

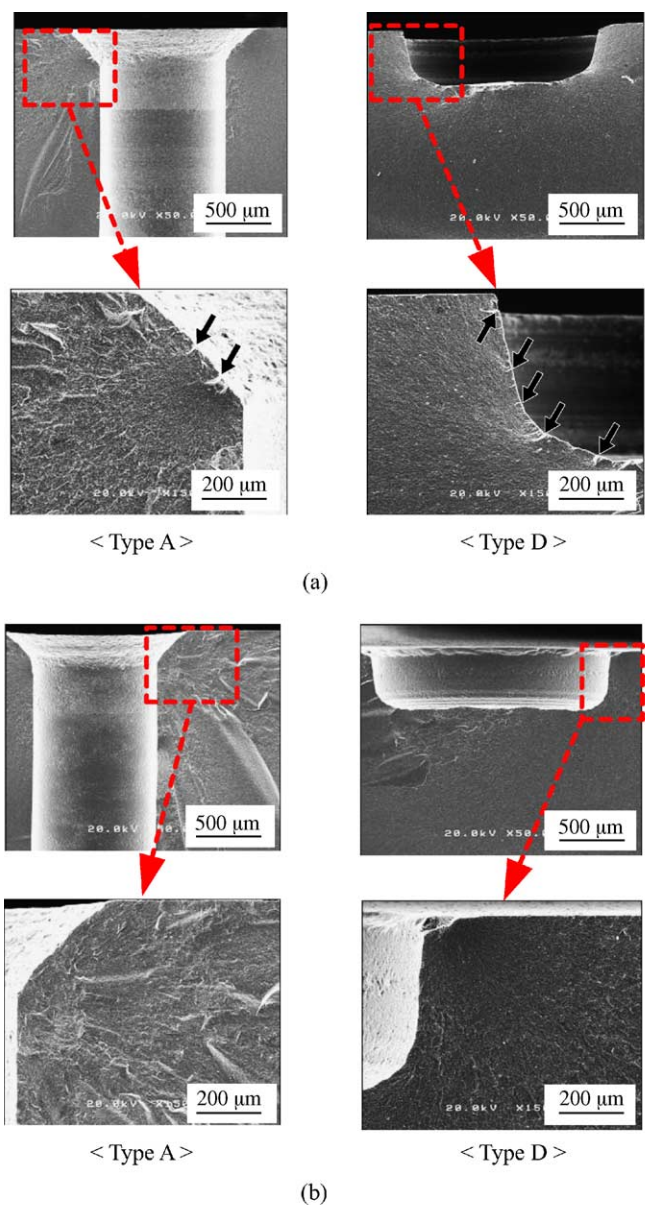

Fig. 9. SEM photomicrographs of fracture surface. The arrow symbols indicate that the surface micro-crack formed during machining: (a) as-machined specimen (before crack-healing) and (b) crack-healed specimen (after crack-healing).

3.3. *Strength recovery behaviour of machined components by crack-healing*

Fig. 8 shows the effect of crack-healing on the strength recovery for machined specimens of mullite/SiC/Y_2O_3. The symbols (○), (●), (▲) and (⧫) show the nominal bending strength (σ_{NB}) of an as-machined specimen, a crack-healed specimen at 1473 K for 10 h, a crack-healed specimen at 1573 K for 1 h and a crack-healed specimen at 1623 K for 1 h, respectively. The σ_{NB} of as-machined specimens of types A and D are about 200–250 MPa. These values were higher than those of types B and C, due to the differences of the stress concentration factor and the size of the surface micro-cracks formed by machining.

The σ_{NB} of the specimen crack-healed at 1473 K for 10 h could not be completely recovered. However, in the case of specimen crack-healed at 1573 K and 1623 K for 1 h, the σ_{NB} were increased by 40–200% compared to those of the as-machined specimen. Fig. 9 shows the fracture surface of as-machined specimens and crack-healed specimens; the arrow symbols indicate that the surface micro-cracks formed during machining. Four kinds of the as-machined specimens were found to fracture due to surface micro-cracks formed during machining, which were observed in the SEM observation. In contrast, there were no surface micro-cracks on the fracture surface of the crack-healed specimens, as shown in Fig. 9(b). Therefore, the σ_{NB} of the crack-healed specimen was increased by the crack-healing of the surface micro-crack formed during machining.

The stress concentration factors (α) of types A and B were reported as 2.2 and 1.4, respectively.[30] Considering the stress concentration factor of types A and B, it can be said that the

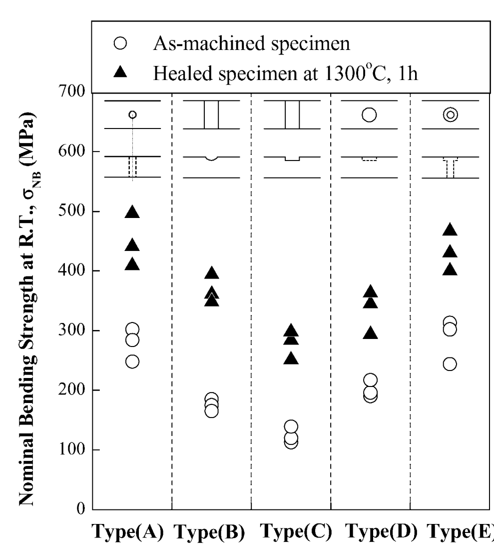

Fig. 10. Effect of crack-healing on the nominal bending strength of the machined specimens of mullite/SiC.

σ_{NB} of the specimens crack-healed at 1573 K and 1623 K for 1 h have sufficient strength. Therefore, these results suggested the surface micro-crack of the as-machined specimens formed during machining can be healed completely.

Fig. 10 shows the effect of crack-healing on the five kinds of machined specimens of mullite/SiC. The symbols (◯) and (▲) show the nominal bending strength (σ_{NB}) of the as-machined and specimens crack-healed at 1573 K for 1 h, respectively. The σ_{NB} of the specimens crack-healed at 1573 K for 1 h were increased by 50–130% compared to those of the as-machined specimens. The surface micro-cracks of the as-machined specimens of mullite/SiC were also completely healed.

Therefore, it can be concluded that the crack-healing is very useful for improvement of strength and structural integrity of machined components.

4. Conclusions

(1) Mullite/SiC/Y_2O_3 composite ceramics have the ability to heal after cracking from 1423 K to 1673 K in air.
(2) The heat-resistance limit temperature for strength of the crack-healed specimen is ≅1473 K, and (68% of the specimens fractured from outside the crack-healed zone at the tested-temperature range 300–1573 K.
(3) Due to the crack-healing treatment, the strength of the machined specimen increased by 40–200%. Local fracture strength was assumed to recover completely, and the cracks formed by machining were healed completely.
(4) A large self-crack-healing ability is desirable for achieving a higher structural integrity in ceramic components.

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