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Crack-healing behavior and static fatigue strength of Si_3N_4/SiC ceramics held under stress at temperature (800, 900, 1000 °C)

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Abstract

Si_3N_4/SiC composite ceramics were sintered and subjected to three-point bending. A semi-elliptical surface crack of 100 μm in surface length was introduced on each specimen. The pre-crack was healed under constant bending stress of 210 MPa at 800, 900 and 1000 °C. Applied stress of 210 MPa is ~70% of the bending strength of pre-cracked specimen. Bending strength and static fatigue strength of crack-healed specimens were systematically investigated at each crack-healing temperature. The bending strength of crack healed specimen showed almost the same value as smooth specimen. Thus, Si_3N_4/SiC composite ceramics could heal a crack even under constant bending stress of 210 MPa at 800, 900 and 1000 °C. Moreover, crack-healed zone had quite high static fatigue limit at each crack-healing temperature. These conclusions indicate that Si_3N_4/SiC composite ceramics has an ability to heal a crack under service condition, i.e. high temperature and applied stress.

Keywords: Crack-healing; Fatigue; Healing under stress; Mechanical properties; Si_3N_4/SiC

1. Introduction

Some engineering ceramics have the ability to heal cracks.[1–9] If this ability is used on structural components in engineering use, great merits can be anticipated, such as increases in the reliability of structural ceramic components, and decreases in the inspection, machining and polishing costs of ceramic components. Si_3N_4/SiC[10–13] and mullite/SiC [6,16,19] with very high self crack-healing ability were sintered by the present authors. Following important subjects were investigated using these ceramics, such as the best healing conditions[11,12,14] the maximum crack size that can be healed completely,[11] and cyclic or static fatigue strengths of a crack-healed specimen.[12,15–21] To guarantee the reliability of ceramic components, we proposed a new methodology, so called ‘‘crack-healing + proof test’’.[18] This methodology is very important since embedded flaws can not be healed at the present time.[10–22] It was shown that the reliability of ceramic components could be increased for monotonic and cyclic loading using this technique.[22] Applying optimized crack-healing treatment and subsequent proof testing to the ceramic components after machining, the reliability of ceramic components can be increased, simply and economically.[18]

If a surface crack could be healed under service conditions, the reliability and lifetime of ceramic components could be increased. Here, we define the crack-healing under service conditions as ‘‘in-situ crack-healing’’. The crack-healing behavior of Si_3N_4/SiC under stress at 1000 °C was investigated by the present authors.[20] It was found that a surface crack of 100 μm could be healed completely even under constant or cyclic stress at 1000 °C.[20] Recently, the crack-healing behavior of Si_3N_4/SiC under cyclic stress at 1100 and 1200 °C was investigated.[21] It was shown that a surface crack of 100 μm could be healed completely just in 0.5–1 h even under cyclic stress at 1100 and 1200 °C.[21]

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These experimental results indicate that Si_3N_4/SiC have an excellent in-situ crack-healing ability.

However, the crack-healing behavior under stress below 1000 °C is not studied. Thus, in this study, the crack-healing behavior of Si_3N_4/SiC under constant stress was investigated at 800, 900 and 1000 °C. Static fatigue strength of crack-healed specimens was also investigated at each healing temperature.

2. Material, specimen and experimental method

The silicon nitride powder used in this investigation has the following properties: mean particle size is 0.2 μm, the volume ratio of α-Si_3N_4 is about 95% and the rest is β-Si_3N_4. The SiC powder used has 0.27 μm mean particle size. The samples were prepared using a mixture of silicon nitride, 20 wt.% SiC powder and 8 wt.% Y_2O_3 as an additive powder. To this mixture, alcohol was added and blended completely for 48 h. The mixture was placed in an evaporator to extract the solvent and then in a vacuum to produce a dry powder mixture. The mixture was subsequently hot-pressed at 1850 °C and 35 MPa for 1 h in nitrogen gas.

The sintered material has the following properties and microstructure: $K_{IC} \doteqdot 6.5$ MPavm, average grain size of the matrix Si_3N_4 is 0.44 μm, average aspect ratio of Si_3N_4 is ~5.0. Most of SiC particles located in grain boundary distributed uniformly. This silicon nitride was selected as a test material because it has an excellent crack-healing ability and high temperature strength of crack-healed zone up to 1300 °C.[11,12]

The sintered material was then cut into test specimens measuring 3×4×40 mm according to the standards.[23] Then, a semi-elliptical surface crack of 100 μm in surface length was introduced at the center of the tension surface of the test specimens with a Vickers indenter using a load of ~20 N as shown in Fig. 1. The ratio of depth (a) to half surface length (c) of crack (aspect ratio) was $a/c \doteqdot 0.9$.

Table 1 shows the crack-healing process adopted in this study. Fig. 2 shows the crack-healing process of I–IV, schematically. The healing processes of I–V were

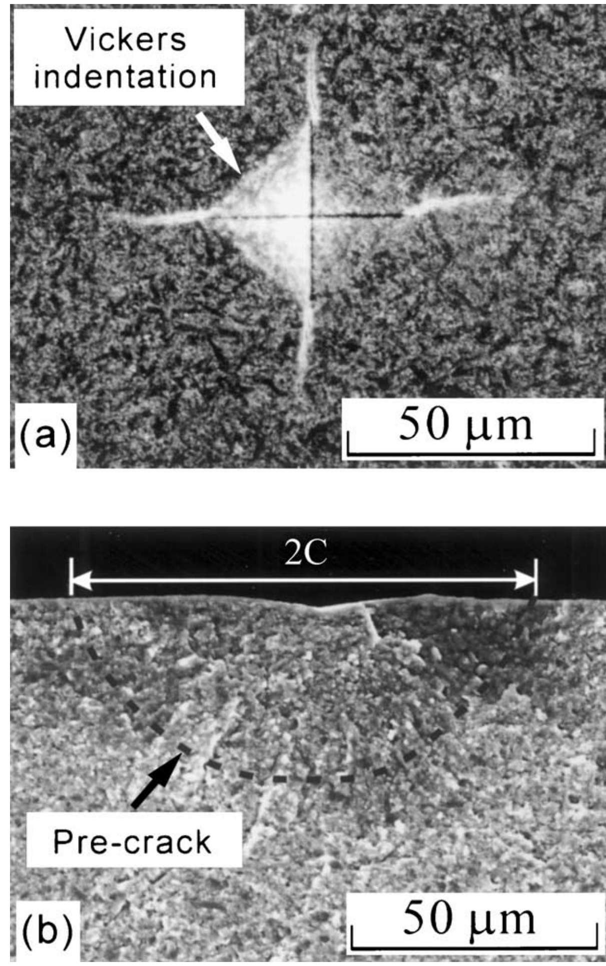

Fig. 1. SEM micrographs of (a) indentation crack and (b) fracture surface.

selected to discuss the effect of healing conditions at 800 °C. In the healing process I, pre-cracked specimens were crack-healed at 800 °C for 70 h without applied stress. In the healing process II, pre-cracked specimens were healed in the process I followed by the crack-healing at 800 °C for 10 h five times without applied stress. We call the crack-healing under cyclic heating as "multi healing" in this paper. In the healing process III, pre-cracked specimens were crack-healed at 800 °C for 70 h

Table 1
Processes of crack-healing

Healing process	First healing (constant temperature)			Second healing (multi-healing)	
	Temperature T_H (°C)	Time t_H (h)	Constant stress σ_H (MPa)	Temperature T_H (°C)	Time t_H (h)
I	800	70	–	–	–
II	800	70	–	200–800	10h×5
III	800	70	210	–	–
IV	800	20	210	200–800	10h×5
V	900	70	210	–	–
VI	1000	70	210	–	–

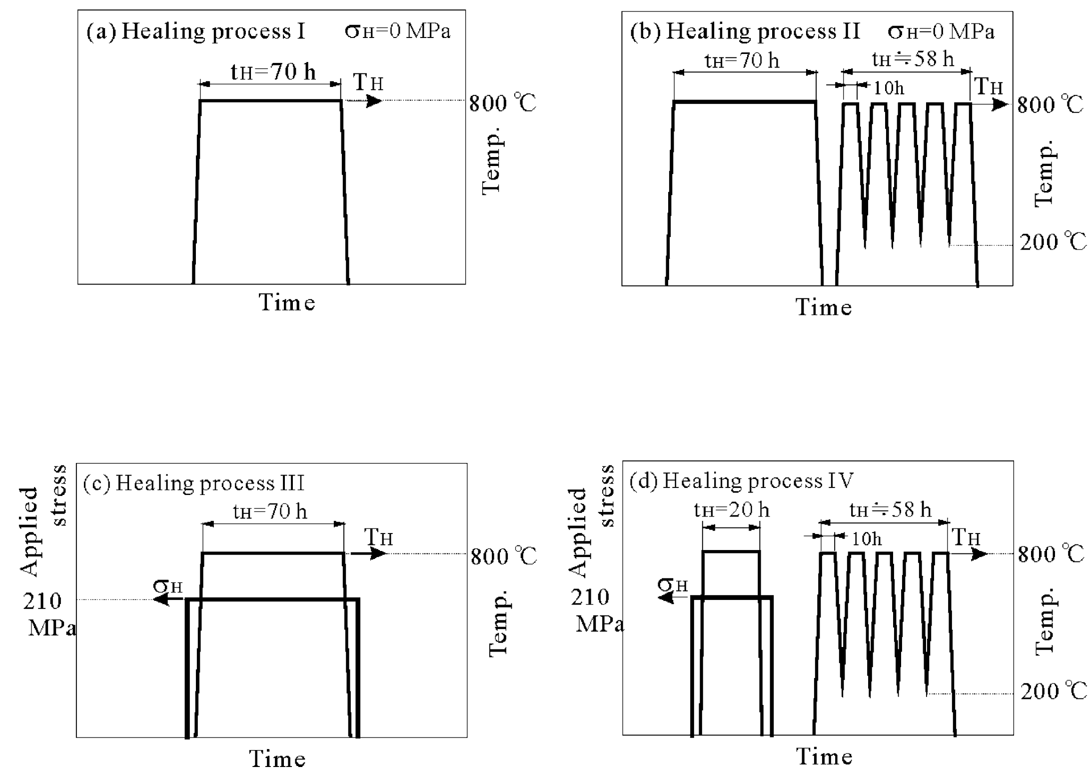

Fig. 2. Schematics of crack-healing process. (a) healing process I, (b) healing process II, (c) healing process III, (d) healing process IV.

under constant stress of $\sigma_H = 210$ MPa. In the healing process IV, pre-cracked specimens were crack-healed at 800 °C for 20 h under constant stress of $\sigma_H = 210$ MPa followed by crack-healing at 800 °C for 10 h five times without applied stress. In the healing processes V and VI, pre-cracked specimens were crack-healed for 70 h under constant stress of $\sigma_H = 210$ MPa at 900 and 1000 °C, respectively. All crack-healing was carried out in air.

The bending strength of a pre-cracked specimen is ~300 MPa as shown by △ in Fig. 4 (or Fig. 5), so that the applied stress of 210 MPa is ~70% of the bending strength of a pre-cracked specimen. The applied stress

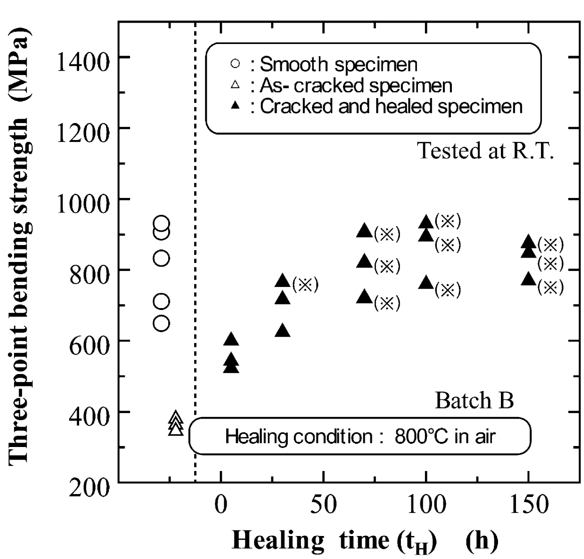

Fig. 3. Relationship between healing time and bending strength of Si_3N_4/SiC ceramics at room temperature. Data with an asterisk indicates that fracture occurred outside of the crack-healed zone.

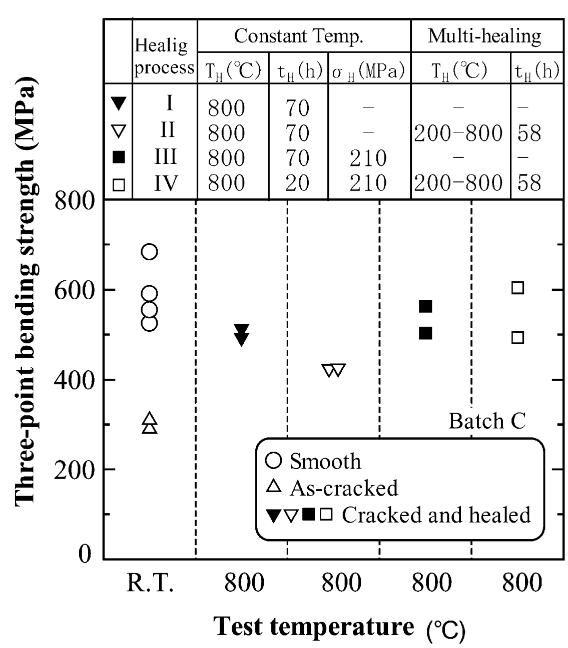

Fig. 4. Effect of crack-healing process at 800 °C on bending strength at 800 °C.

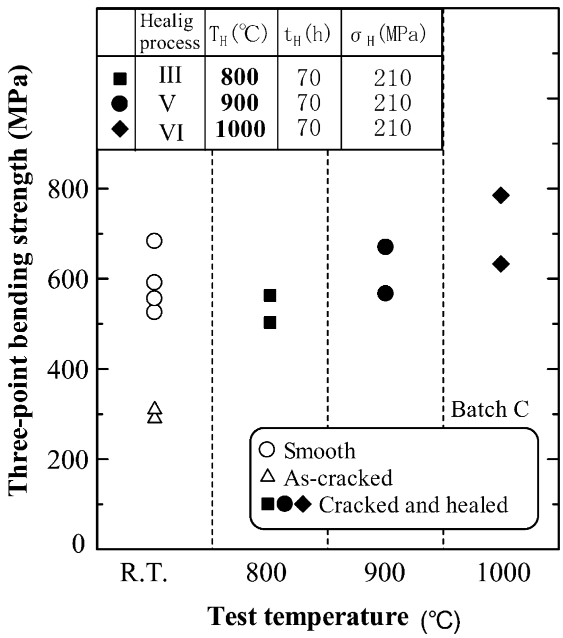

Fig. 5. Effect of crack-healing process at 800, 900 and 1000 °C on bending strength at each crack-healed temperature.

of 210 MPa is higher than the room temperature cyclic fatigue limit (~200 MPa) of the pre-cracked specimen.[12] Thus, the stress condition during crack-healing is quite severe.

In the crack-healing processes, we first applied the bending stress then increased furnace temperature at a rate of 10 °C/min to avoid unexpected crack-healing without applied stress. After the crack-healing, the monotonic bending tests were conducted at each crack-healing temperature. Static fatigue tests were also conducted at each crack-healing temperature. Both monotonic bending tests and static fatigue tests were conducted using a three-point loading system with a span of 30 mm. The cross-head speed in the monotonic bending test was 0.5 mm/min. Crack-healing under stress and static fatigue tests were conducted using dead-load type testing machine equipped with an electric furnace. The fracture initiation site was identified by optical microscope. The fracture surface was analyzed by scanning electron microscopy (SEM).

3. Test results and discussion

3.1. *Effects of healing temperature and time on the crack-healing behavior of Si_3N_4/SiC*

Some experimental results in the previous study[13] will be briefly explained. In the previous study,[13] Si_3N_4/SiC specimens with a semi-elliptical surface crack of 100 μm were crack-healed at the temperatures between 900 and 1400 °C for 1 h in air. Bending strength of the crack-healed specimens was measured at 1300 °C. It was shown that the bending strengths of the specimen crack-healed at between 1200 and 1400 °C were as high as the smooth specimen, moreover, most specimens fractured outside of the crack-healed zone. On the other hand, all specimen crack-healed at 900 and 1000 °C fractured from the crack-healed zone. The bending strength of the crack-healed zone that was healed at 900 and 1000 °C is not enough at 1300 °C. However, when the service temperature of the material is between 900 and 1000 °C, it would be desirable if specimens crack-healed at 900 °C and 1000 °C have high strength at 900 and 1000 °C. Fig. 3 shows the relationship between bending strength at room temperature and crack-healing time for Si_3N_4/SiC.[13] The specimens with a surface crack ($2C = 100$ μm) were crack-healed at 800 °C in air. Bending strength of the crack-healed specimen recovered if the crack-healing time was longer than 70h.[13] However, whether the crack-healed specimens have enough strength at 800 °C is not studied yet.

3.2. *Bending strength of the crack healed specimen at the healing temperature of 800 °C*

Pre-cracked specimens were crack-healed by the healing processes of I–IV shown in Table 1 or Fig. 2. After the crack-healing, monotonic bending tests were conducted at 800 °C. Fig. 4 shows the results of the bending tests. In Fig. 4, the bending strengths of smooth specimens (○) and pre-cracked specimens (△) at room temperature are also indicated. The mean value of the bending strength of smooth specimens is ~590 MPa. The Vickers indentation largely reduced the bending strength to ~300 MPa. The bending strength of smooth specimen is almost constant up to 1300 °C.[11,12] Thus, only the bending strength at room temperature (○) was shown in Fig. 4. The crack-healed specimens fractured from the crack-healed zone since the Vickers indentation in the crack-healed zone acted as stress concentrator. However, the bending strength of these specimens recovered. The pre-cracked specimens crack-healed by the process of III and IV were subjected to a constant bending stress of 210 MPa. The pre-crack of 100 μm can be healed by the crack-healing at 800 °C for 70 h even under constant stress of 210 MPa and the bending strength of the crack-healed zone is quite high at 800 °C. The bending strengths of the specimens crack-healed under stress (■, □) are slightly higher than that of the specimens crack-healed without stress (▼, ▽). However, the difference of bending strength is not detrimental considering the scatter of the bending strength of smooth and crack-healed specimens. The bending strength of the specimens applied to multi-healing (▽, □) was similar to that of the specimens crack-healed at constant temperature (▼, ■). Thus, the effect of multi-healing on the crack-healing behavior was not large when the healing temperature was 800 °C.

3.3. Bending strength of the crack healed specimen at the healing temperature of 900 °C and 1000 °C

The pre-cracked specimens were crack-healed by the healing processes of V and VI shown in Table 1. After the crack-healing, monotonic bending tests were conducted at the healing temperatures of 900 and 1000 °C. Fig. 5 shows the results of the bending tests. The experimental results of the specimens crack-healed at 800 °C were also indicated in Fig. 5. The symbols of ■, ● and ◆ show the bending strength of the crack-healed specimens at the healing temperature of 800, 900 and 1000 °C, respectively. Although the crack-healed specimens fractured from the crack-healed zone, the crack-healed specimens recovered their bending strength at the healing temperature. Thus, it can be said that the pre-crack of 100 μm can be healed even under stress of 210 MPa by the crack-healing at 900 and 1000 °C for 70 h and the bending strength of the crack-healed zone is quite high even at the healing temperature.

Ogasawara et al.[5] conducted crack-healing under constant bending stress using pre-cracked specimens of $Si_3N_4/Al_2O_3/Y_2O_3$. After the crack-healing at 1100 °C for 3 or 20 h, four point bending tests were conducted at room temperature. It was pointed out that if the applied stress during crack-healing was less than ~50% of the bending strength of the pre-cracked specimens, the bending strength of crack-healed specimens recovered. However, bending strength at the healing temperature was not investigated by Ogasawara et al.[5]

It can be concluded that Si_3N_4/SiC ceramics can heal a surface crack during service. Off course, whether the

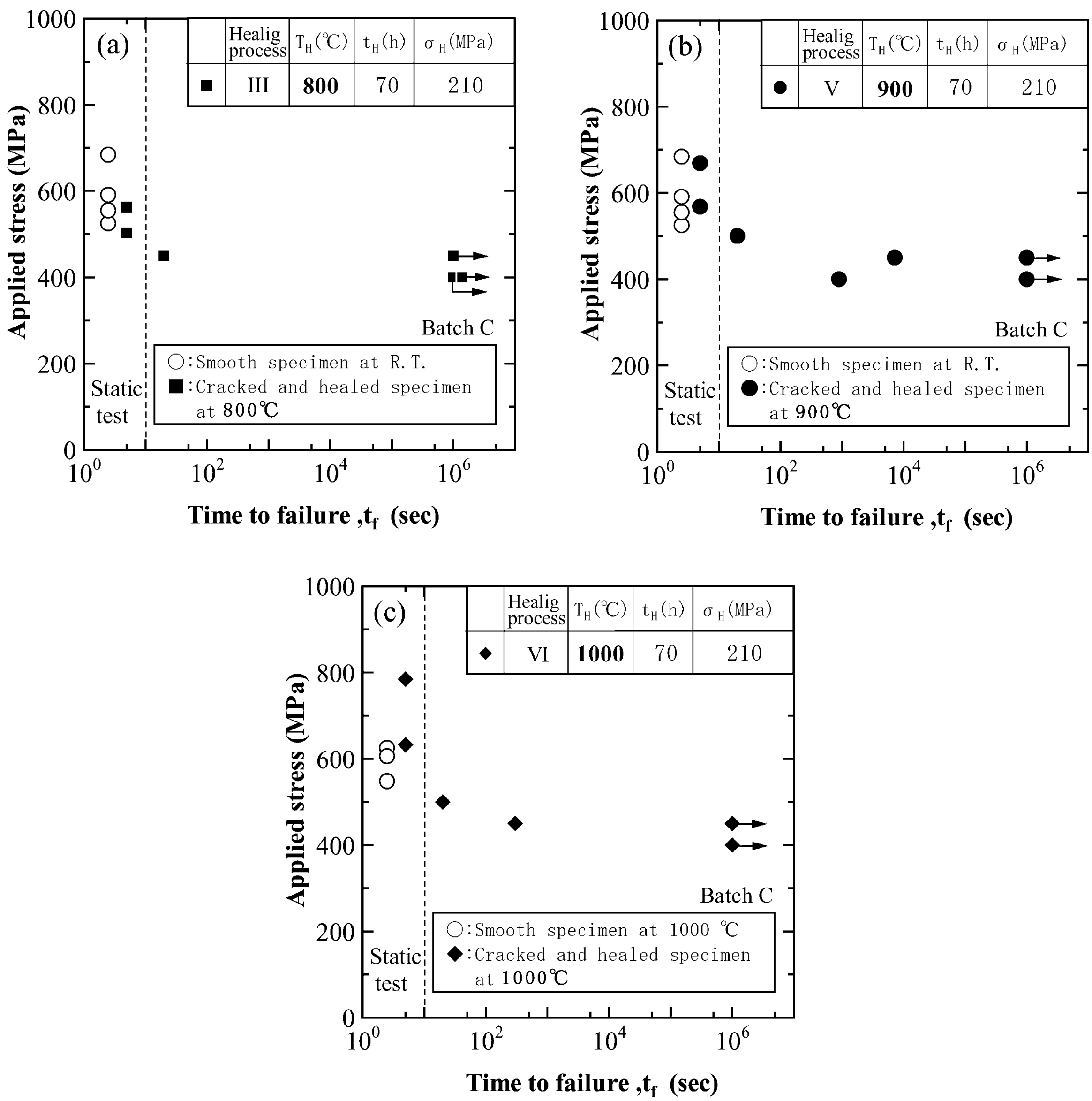

Fig. 6. Relationship between applied stress and time to failure (t_f), (a) 800 °C crack-healed samples (Healing process III), (b) 900 °C crack-healed samples (healing process V), (c) 1000 °C crack-healed samples (healing process VI).

healing proceeds or not depends on applied stress, temperature and environment.

3.4. Static fatigue strength of the crack-healed specimen

Fig. 6 shows the results of static fatigue tests conducted at the healing temperatures of 800, 900 and 1000 °C, respectively. The pre-cracked specimens were crack-healed by the processes of III, V and VI in Table 1. Monotonic bending strengths of smooth specimens (○) and crack-healed specimens are shown on the left side of Fig. 6. The static fatigue tests were stopped at $t=10^6$. The specimens that did not fracture in the tests are marked by arrow symbols (→). The applied stress at which a specimen did not fracture up to $t=10^6$. is defined as static fatigue limit, σ_{t0}. The value of static fatigue limit (σ_{t0}) for the specimens that were crack-healed at 800, 900 and 1000 °C is ~400 MPa at each healing temperature. As mentioned already, the mean value of room temperature bending strength of the smooth specimen (○) is ~590 MPa. The ratio of σ_{t0} to the mean bending strength of the smooth specimens is about 70%. Thus, static fatigue limit of the crack-healed specimens is quite high.

Fig. 7 shows the effect of crack-healing process at 1000 °C on the static fatigue strength at 1000 °C. In Fig. 7, experimental results obtained in the previous study[20] (□,▽,△) are also indicated. Symbols □ and ▽ show static fatigue strength of the specimens crack-healed at 1000 °C without applied stress for 5 and 100 h, respectively. The static fatigue limit of 5 h crack-healed specimen is ~350 MPa and that of 100 h crack-healed specimen is ~450 MPa.[20] Static fatigue limit increase with the crack-healing time. On the other hand, static fatigue limit of the specimen crack-healed for 70 h under constant stress (healing process VI) is ~400 MPa. Static fatigue limit of the multi-healed specimen shows the highest static fatigue limit (~500 MPa) in Fig. 7.[20] Surface oxide layer was analyzed by X-ray diffraction in order to investigate the crack-healing material.[20] A 100 h crack-healed specimen had large amount of crystallized SiO_2. On the other hand, a 5 h crack-healed specimen had a very small amount of crystallized SiO_2. It was found that the crystallinity of SiO_2 had large effect on the fatigue strength at elevated temperature.[20]

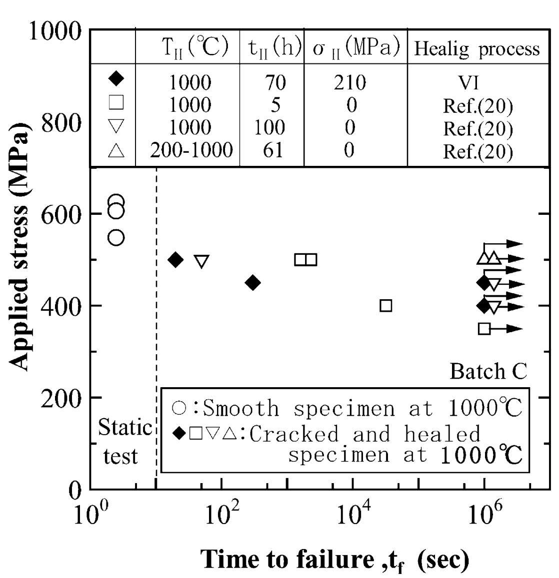

Fig. 7. Effect of crack healing process at 1000 °C on static fatigue strength at 1000 °C.

3.5. Bending strength of the fatigue tested specimen

The monotonic bending strength of fatigue-tested specimens at each crack-healing temperature was indicated on the right side of Fig. 8. The monotonic bending strengths of crack-healed specimens at each crack-healing temperature were also indicated on the left side of Fig. 8. The values in brackets show the applied stress during static fatigue tests. The fatigue tested specimens exhibited slightly higher bending strength than that of the monotonically tested specimens except a few results shown by symbol ◆. This increase of bending strength was probably caused by the healing of small sub-surface crack since the specimens were subjected to high temperature for a long time (280 h). This tendency is consistent with our previous studies.[20]

3.6. Fracture surface of crack-healed specimen

The fracture surface of the crack-healed specimens was analyzed by the scanning electron microscopy (SEM). Fig. 9 shows SEM micrograph of the fracture

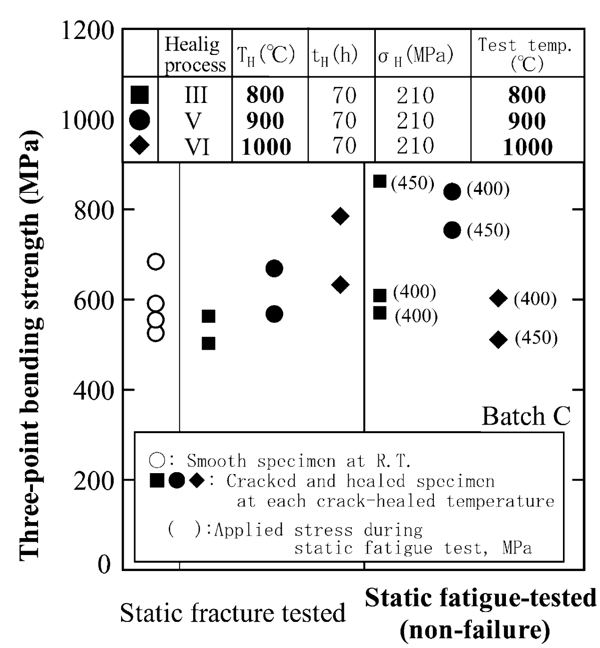

Fig. 8. Bending strength of fatigue-tested samples at each crack-healed temperature.

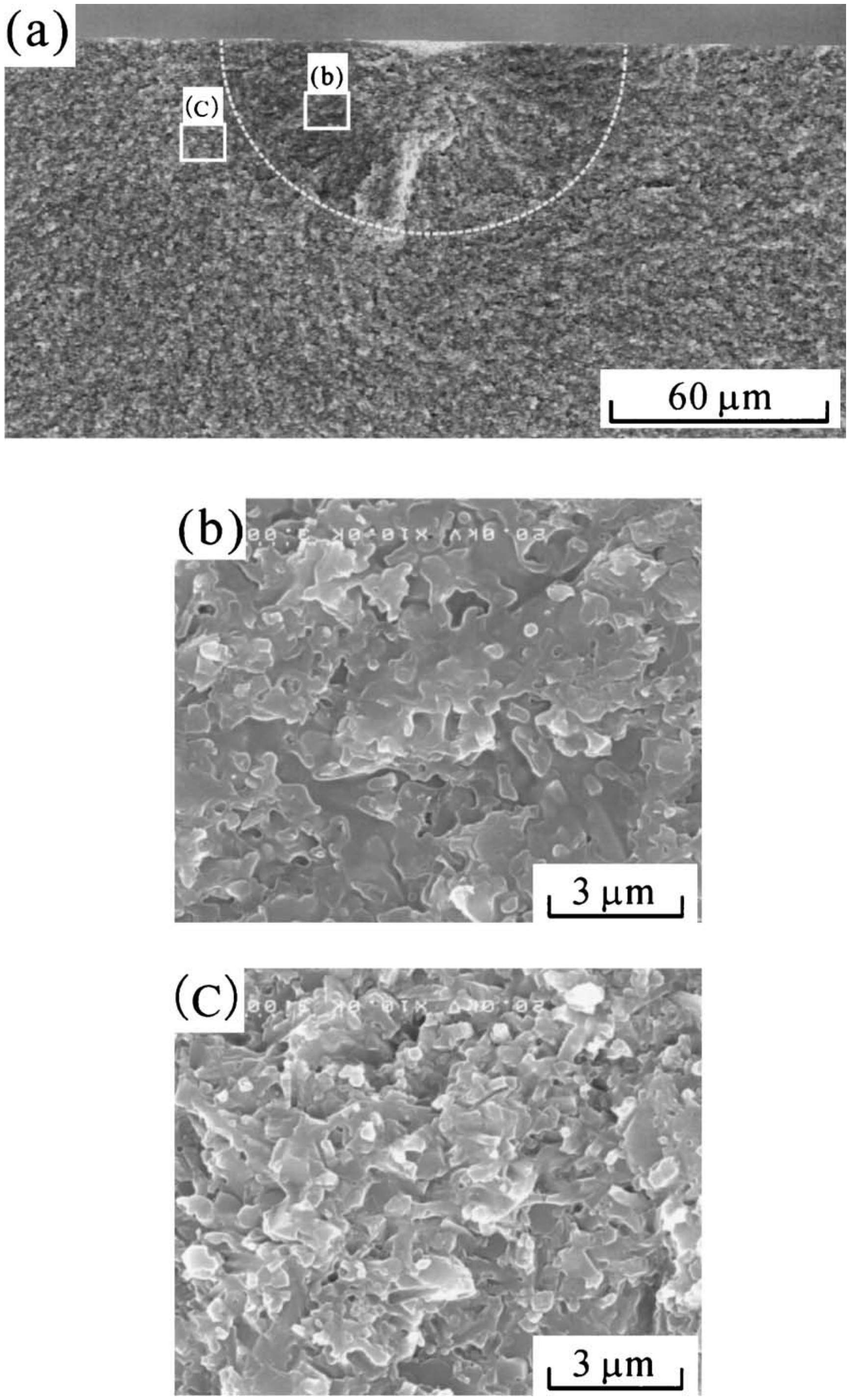

Fig. 9. SEM micrographs of fracture surface of a crack-healed Si_3N_4/SiC ceramics tested at 800 °C. (Healing process III, $T_H = 800$ °C, $t_H = 70$ h, $\sigma_H = 210$ MPa) (b) shows the detail of crack-healed zone, (c) shows the detail of outside of crack-healed zone.

surface of Si_3N_4/SiC that was crack-healed at 800 °C under bending stress followed by the bending test at 800 °C. The morphology of the fracture surface within crack-healed zone shown in Fig. 9(b) is clearly different from that of outside of the crack-healed zone shown in Fig. 9(c). Reaction product is observed at the fracture surface within the crack-healed zone. Similar reaction product is also observed in the specimens crack-healed at 900 and 1000 °C. On the other hand, no clear reaction product is observed at the fracture surface outside of the crack-healed zone. The estimated crack-healing reactions for Si_3N_4/SiC are as follows.[10,12,20]

$$Si_3N_4 + 3O_2 \rightarrow 3SiO_2 + 2N_2 \quad (1)$$

$$SiC + 2O_2 \rightarrow SiO_2 + CO_2(CO) \quad (2)$$

$$2SiC + Y_2O_3 + 4O_2 \rightarrow Y_2Si_2O_7 + 2CO_2(CO) \quad (3)$$

The SiO_2 has two phases: one is a glassy phase and another is a crystal phase. The reaction product shown in Fig. 9(b) is probably glassy phase of SiO_2 since clear X-ray peek of SiO_2 was not detected.

4. Conclusions

A semi-elliptical surface crack of 100 μm in surface length was made on Si_3N_4/SiC ceramics. The pre-crack was healed under constant bending stress of 210 MPa at 800, 900 and 1000 °C. Bending strength and static fatigue strengths of crack-healed specimens were systematically investigated at each crack-healing temperature. The main conclusions obtained are as follows.

1. The bending strength of crack-healed zone is almost as same as that of smooth specimen at room temperature. Thus, a surface crack of 100 μm, which reduces the bending strength by half, can be healed completely even under constant stress of 210 MPa. Applied stress of 210 MPa is ~70% of the bending strength of pre-cracked specimen.
2. The value of static fatigue limit (σ_{t0}) of the specimens that were crack-healed at 800, 900 and 1000 °C is ~400 MPa at each healing temperature. The ratio of σ_{t0} to the mean bending strength of the smooth specimens is about 70%. Thus, the static fatigue limit is quite high.
3. The results suggest that Si_3N_4/SiC ceramics can heal a crack under service condition, i.e. under stress and at temperatures between 800 and 1000 °C.

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