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Crack-healing behavior of Si_3N_4/SiC ceramics under stress and fatigue strength at the temperature of healing (1000 °C)

K. Ando[a,*], K. Houjyou[a], M.C. Chu[a], S. Takeshita[a], K. Takahashi[a], S. Sakamoto[a], S. Sato[b]

[a]*Department of Energy and Safety Engineering, Yokohama National University, 79-5, Hodogaya, Yokohama, Japan*
[b]*NHK Spring Co., Ltd, 3-10 Fukuura, Kanazawaku, Yokohama, Japan*

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Abstract

Si_3N_4/SiC composite ceramics were sintered and subjected to three-point bending specimens made according to the appropriate JIS standard. A semi-circular surface crack of 100 μm in diameter was made on each specimen. We systematically studied crack-healing behavior, and cyclic and static fatigue strengths at the service temperature (1000 °C) by using three kinds of specimens (smooth, cracked and crack-healed). The main conclusions are as follows: (1) Si_3N_4/SiC composite ceramics have the excellent ability to heal a crack at 1000 °C; (2) this sample could heal a crack even under cyclic stress at 1000 °C; (3) a new crack-healing process was proposed. The sample crack-healed at 1000 °C by the process exhibited a sufficient static and cyclic fatigue strength at 1000 °C.

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

1. Introduction

Some engineering ceramics have the ability to heal a crack.[1–13] If this ability is used on structural components in engineering use, great merits can be anticipated in the following areas:[11,13,14] (1) increases in the reliability of structural ceramic members; (2) decreases in the inspection, machining and polishing costs of ceramic members; and (3) decreases in the maintenance costs and prolongation of the lifetime of ceramic members.

To use this healing ability in structural engineering, many problems must be overcome such as (a) a methodology for evaluating a crack-healing ability,[3,5,8,11,13] (b) the effect of chemical composition on the crack-healing ability,[12,15,16] (c) the effect of healing conditions on the strength of the healed-zone,[5,8,13,16] (d) the maximum crack size that can be healed completely,[11,16] (e) knowledge of the high-temperature strength of crack-healed zones,[8,10,11,13–18] (f) the crack-healing mechanism,[8,11,12] (g) assessment of the cyclic fatigue and static fatigue strengths of crack-healed ceramic member,[13,17–22] and (h) a methodology for guaranteeing the reliability of ceramics member.[10,14,20] Methodology (h) is important, because embedded flaws can not be healed at the present time,[6,11–22] because oxygen from air is necessary. The present authors have already systematically studied several aspects of problems (a) to (h).[8,10–22]

The developed ceramics of high crack-healing ability are Si_3N_4/SiC[8] and mullite/SiC[16] composite ceramics. The best crack-healing condition of both ceramics are: a temperature of 1200 to 1300 °C, healing time of 1 h and an air environment[11,13,16]. The sample healed under the best condition showed the same level bending and fatigue strength as the base material even at elevated temperatures. The limit temperature for fatigue strengths of Si_3N_4/SiC[21] and mullite/SiC[19,22] are 1200 and 1100 °C, respectively.

However, it is very interesting whether a material can heal a crack at service temperature or not and whether the healed zone has enough strength compared to the base material at the healed temperature. A much more important question is a material can heal a crack under service conditions (temperature, stress and environment). In such a case, it is not very important that the healed zone has enough strength at higher temperature

* Corresponding author.
E-mail address: andokoto@ynu.ac.jp (K. Ando).

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than the service one. The present authors have already reported the above possibilities,[13,16,20] but the behavior is not well known.

From the above points of view, we established the followings four purposes in this paper.

1. Study on the crack-healing behaviour of Si_3N_4/SiC at 1000 °C.
2. Study on the effect of the applied stress on the crack-healing behaviour at the service temperature.
3. Develop a new healing process by which the healed zone has enough strength at the healed temperature.
4. Study on the static and cyclic fatigue strength of the crack-healed member at 1000 °C.

In this paper, we selected Si_3N_4/SiC ceramics with very high crack-healing ability[8,11,13,20] as a sample and reported systematic test results on the above four subjects.

2. Material, specimen and test method

The silicon nitride powder used in this investigation has the following properties: mean particle size = 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 a 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 thoroughly 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 at 35 MPa for 1 h in nitrogen gas.

The sintered material has the following properties and microstructure: $K_{IC} \doteqdot 6.5$ MPa$\sqrt{m}$; average grain size of the matrix Si_3N_4 = 0.44 μm; average aspect ratio≑5.0; grain boundary crystals are $Y_{20}N_4Si_{12}O_{18}$, $Y_2Si_3N_4O_3$ and $YNSiO_2$ [8]; most SiC particles are located in grain boundaries; and most SiC particles are distributed uniformly.

The sintered material was then cut into test pieces measuring 3×4×40mm as shown in Fig. 1 according to the standard[23]. This silicon nitride was selected as a test material from our large number of investigations for two reasons. Firstly, this material has excellent crack-healing ability.[8,13,21] Secondly, crack-healed zones of this material have high fatigue strength up to 1200 °C.[18,21]

A semi-circular crack was made at the center of the tension surface of the test pieces with a Vickers indenter using a load of about 20 N. By this method, semi-circular cracks of 100 μm in diameter (aspect ratio≑0.9) were made as shown in Fig. 2. Fig. 2(a) shows the indentation and cracks. Crack shapes were confirmed on the fracture surfaces of cracked specimens as shown in Fig. 2(b).

The crack-healing condition significantly affects fracture behavior of crack-healed samples. The best healing condition for high-temperature strength is a temperature of 1300 °C, and a healing time of 1 h in an air environment.[11,13] Thus, most cracked samples in the past studies were healed under this condition. However, if a material could heal a crack under service conditions (under stress and at service temperature) and if the healed zone were strong enough under those conditions,

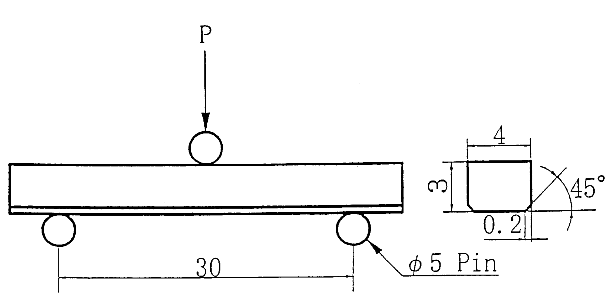

Fig. 1. Three-point loading system and geometry of test specimen: dimensions in mm.

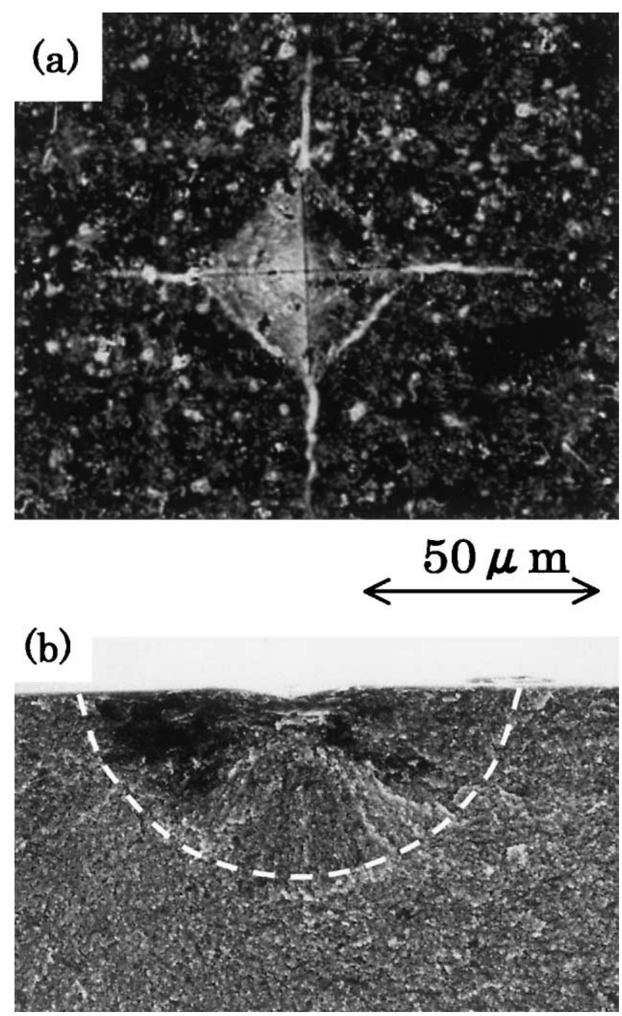

Fig. 2. S.E.M. photographs of (a) indented crack and (b) crack surface.

it would be very beneficial. To investigate the possibility and to develop a new healing process, cracks were healed at 1000 °C and samples were subjected to fatigue tests at 1000 °C, mainly. Moreover, we systematically investigated crack-healing behavior under stress[13,20] at 1000 °C. The crack-healing processes adopted in this paper are shown in schematically Fig. 3. The following six crack-healing processes were selected: (1) healing temperature $T_H = 1000$ °C(constant), applied tensile stress $\sigma_H = 0$, healing time t_H = arbitrary; (2)$T_H = 1000$ °C (constant), $\sigma_H = 210$ MPa (constant), $t_H = 20$ h; (3)$T_H = 1000$ °C (constant), applied cyclic tensile stress $\sigma_H = 210$ MPa (sine wave, $R = 0.2$, frequency = 1,5,10 Hz), $t_H = 20$ h; (4) $T_H = 1000$ °C (cyclic heat:10 h×5 times), applied tensile stress $\sigma_H = 0$, healing time $t_H \fallingdotseq 61$ h; (5) $T_H = 1000$ °C (cyclic heat:10 h×5 times), applied tensile stress $\sigma_H = 210$ MPa, healing time $t_H \fallingdotseq 61$ h, (6) $T_H = 1000$ °C (cyclic heat:10 h×5 times), applied cyclic tensile stress $\sigma_H = 210$ MPa (sine wave, $R = 0.2$, frequency = 1,5,10 Hz), healing time $t_H \fallingdotseq 61$ h, where R is the stress ratio of cyclic stress, and the lower temperature in the cyclic healing process is 200 °C. Fig. 3(a)–(f) shows the healing process (1), (2), (3), (4), (5) and (6), respectively. The reason for cyclic healing process such as type (4) to (6) is that cooling treatment after crack-healing is desirable for improving mechanical properties of the healed zone at elevated temperature.[15] In these healing processes, we first applied stress then increased the furnace temperature

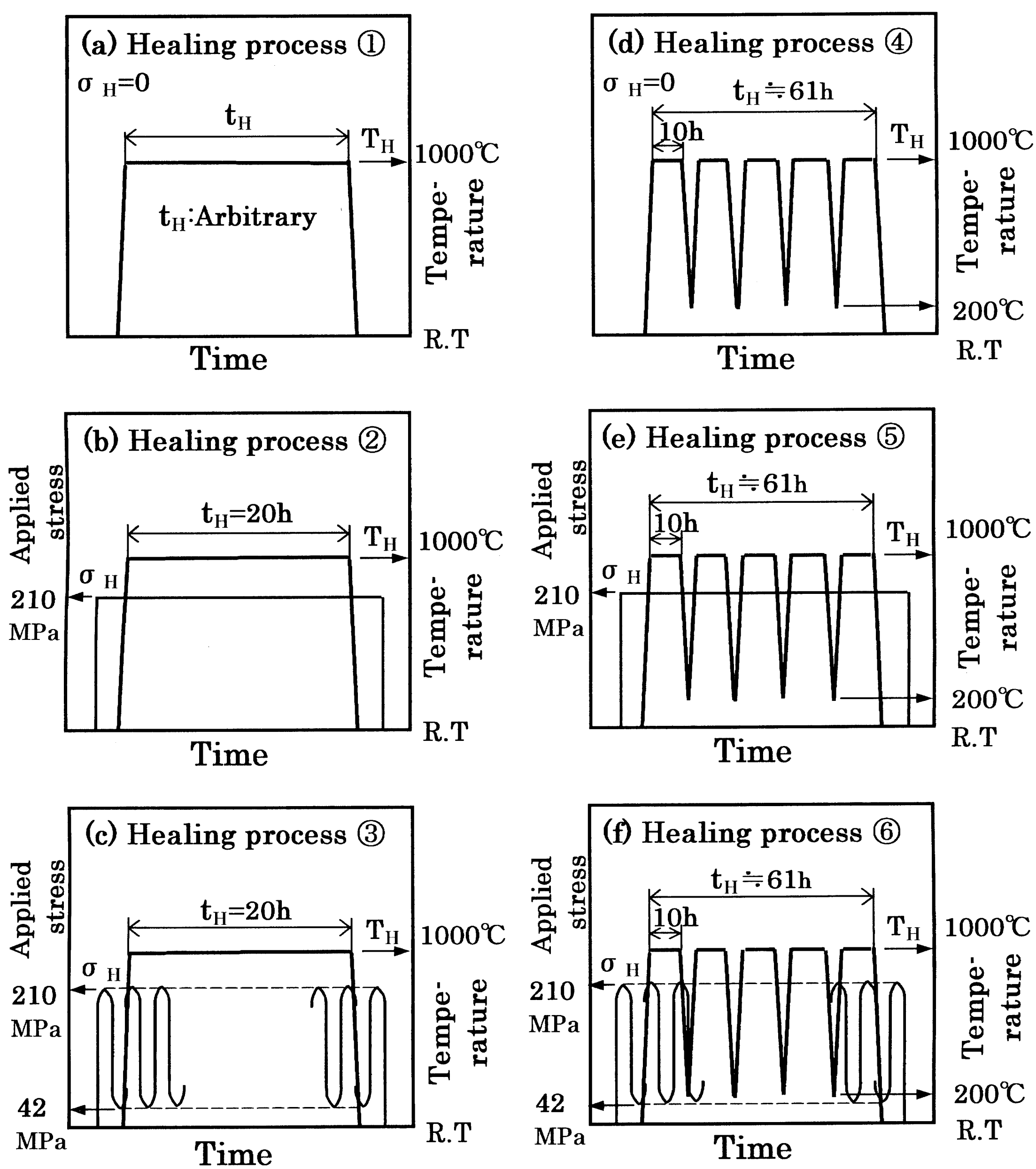

Fig. 3. Schematic illustration of crack-healing process.

at a rate of 10 °C/min. The bending strength of the cracked sample is about 300 MPa, so the applied stress of 210 MPa during healing is 70% of the fracture stress. This is a very severe condition for crack-healing.

All fracture tests were performed on a three-point loading system. The cross-head speed in the monotonic tests was 0.5 mm/min. Cyclic fatigue tests were conducted at 1000 °C at a stress ratio of $R=0.2$ and a frequency of 5 Hz. Static fatigue tests were conducted at 1000 °C using a dead-load type testing machine.

3. Test results and discussion

3.1. *Effect of crack-healing process on bending strength*

In terms of basic knowledge, Fig. 4 shows the effect of crack-healing temperature on the bending strength of crack-healed samples at 1300 °C.[13] For this test, a different batch of Si_3N_4/SiC was used, and the bending strength of the cracked sample was about 350 MPa. When samples were healed from 1200 to 1400 °C, the strength recovery was adequate and most samples fractured outside the crack-healed zone, where symbol (※) indicates a fracture initiated outside the crack-healed zone. When a crack was healed from 900 to 1100 °C, strength recovered considerably, but all sample fractured from crack-healed zone. This fact indicates that the crack-healed zone does not have sufficient strength at 1300 °C. However, if the sample crack-healed at 1000 °C has enough strength at 1000 °C, it would be very desirable. The most important purpose of this paper is to determine whether it is possible or not.

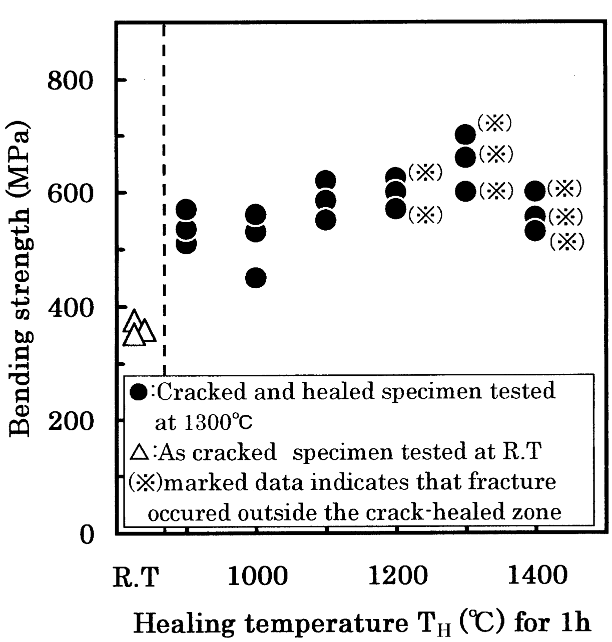

Fig. 4. Relationship between crack-healing temperature and bending strength at 1300 °C.

To investigate the crack-healing behaviour under stress at 1000 °C, we tested the effect of cyclic stress on the crack-healing behaviour, systematically. The test results are shown in Fig. 5 as a function of frequency (H_Z). The bending strength was measured at room temperature. If the sample is heated up to 1000 °C after healing, the crack may be healed at this heating stage. To delete this possibility, we conducted the fracture test at room temperature. In Fig. 5, the bending strength of the crack-healed sample at room temperature recovered completely up to the same strength level as the smooth sample, except for the sample denoted by the symbol ▲. However, the average bending strength of sample ▲ is about 500 MPa, which is a little lower than that of the smooth sample. Surprisingly, these test results showed that this silicon nitride could heal a crack even under 10 Hz cyclic stress at 1000 °C.

These test results showed good agreement with the following three previous test results. (1) Si_3N_4/SiC of a different batch exhibited considerable strength recovery under constant stress at 1000 °C.[13] The healing condition was a ratio of applied constant stress to fracture stress of the cracked sample of about 80%, temperature of 1000 °C, and healing time of about 280 h in an air environment. (2) Si_3N_4/SiC with a different SiC particle size demonstrated complete strength recovery under cyclic stress at 1200 °C.[20] The healing condition was a ratio of applied maximum stress to fracture stress of it's cracked sample of about 65% ($R=0.5$, frequency = 0 to 5 Hz), a temperature of 1200 °C, and a healing time of 20 h in air. (3) Mullite/SiC showed considerable strength recovery under very slow strain rate testing[16] and under static fatigue testing at 1000 °C.[22] The healing condition of the latter test was a ratio of applied constant stress to fracture stress of the cracked sample of

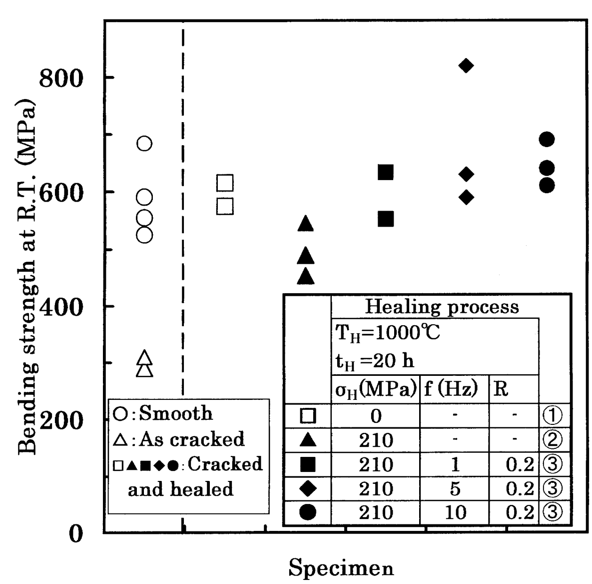

Fig. 5. Effect of crack-healing process at 1000 °C on bending strength at room temperature.

about 53%, temperature of 1000 °C, healing time of 80 h in air.

In Fig. 6, effect of crack-healing process at 1000 °C on bending strength at 1000 °C is shown. The symbol ● indicates the bending strength produced by the healing process (1) for $t_H = 1$ h. Both samples fractured from the crack-healed zone as shown in Fig. 7(a). However, the strength recovered considerably. The symbol ◆ shows the bending strength produced by the healing process (1) for $t_H = 10$ h. In this case, healing is complete and both samples fractured outside the crack-healed zone as shown in Fig. 7(b). Symbols ■ and ▲ represent the bending strengths produced by the healing process (5) and (6), respectively. In these cases, the samples fractured into many pieces and the crack initiation site could not be determined. The bending strength of both samples recovered to almost the same values as that of the as-received smooth sample (○) at 1000 °C. From this, we can conclude that the sample healed at 1000 °C has the same level bending strength as the base material even at 1000 °C.

3.2. Static and cyclic fatigue strength at 1000 °C

Four kinds of samples were used for static fatigue tests. Crack-healing processes selected are (1), (4) and (6). The test results of the static fatigue tests are shown in Fig. 8(a) and (b) as a function of applied stress (σ_{max}) and time to failure (t_f). Monotonic test results are also shown in the left side of Fig. 8. Symbol ○ shows the bending strength of the smooth samples at 1000 °C. Symbols ◆ and ■ show the static fatigue strength of 5 and 100 h crack-healed sample, respectively. Both samples were subjected to crack-healing process (1). The static fatigue tests were stopped at $t = 10^6$ s. The samples that did not fracture in the test are marked by an arrow symbol (→). The applied stress at which a sample did not fracture up to $t = 10^6$ s is denoted as σ_{t0}. The σ_{t0} for the 5 h crack-healed sample is about 350 MPa, and the σ_{t0} for the 100 h crack-healed samples is about 450 MPa. In Fig. 8(b), static fatigue test results related to multi-healing are shown. Symbols ▲ and ● show the static fatigue strength of sample crack-healed by the crack-healing process (4) and (6), respectively. The σ_{t0} for both multi-healed samples are about 500 MPa, regardless of the healing process. The σ_{t0} is considerably higher than that of the 5 h healed sample, and also a little higher than that of the 100 h healed sample. In Fig. 9, cyclic fatigue test results at 1000 °C are shown. Both samples were multi-healed and the crack-healing processes are (4) and (6). The cyclic fatigue tests were

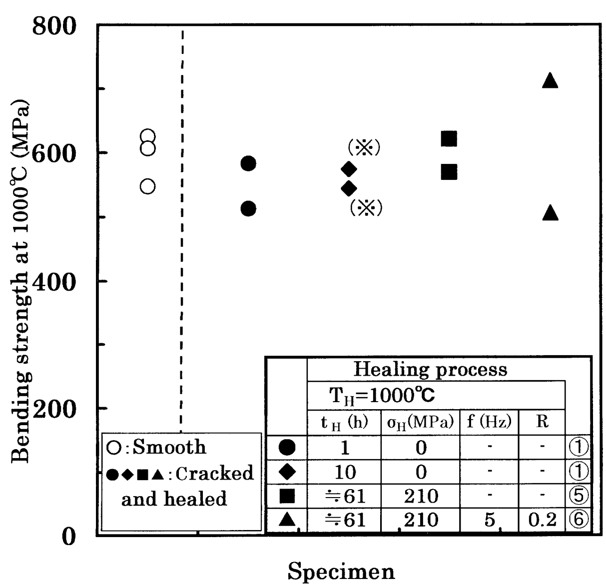

Fig. 6. Effect of crack-healing process at 1000 °C on bending strength at 1000 °C.

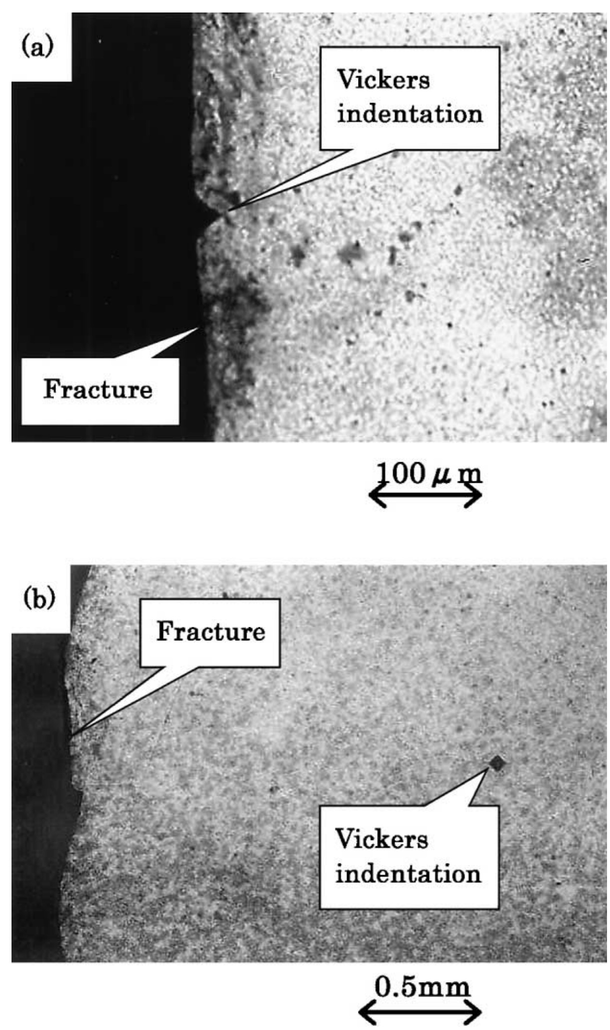

Fig. 7. Fracture surface of crack-healed sample: (a) crack initiated from crack-healed zone (healing process (1), $t_H = 1$ h, bending strength = 513 MPa); (b) crack initiated from the outside of crack-healed zone (healing process (1), $t_H = 10$ h, bending strength = 574 MPa).

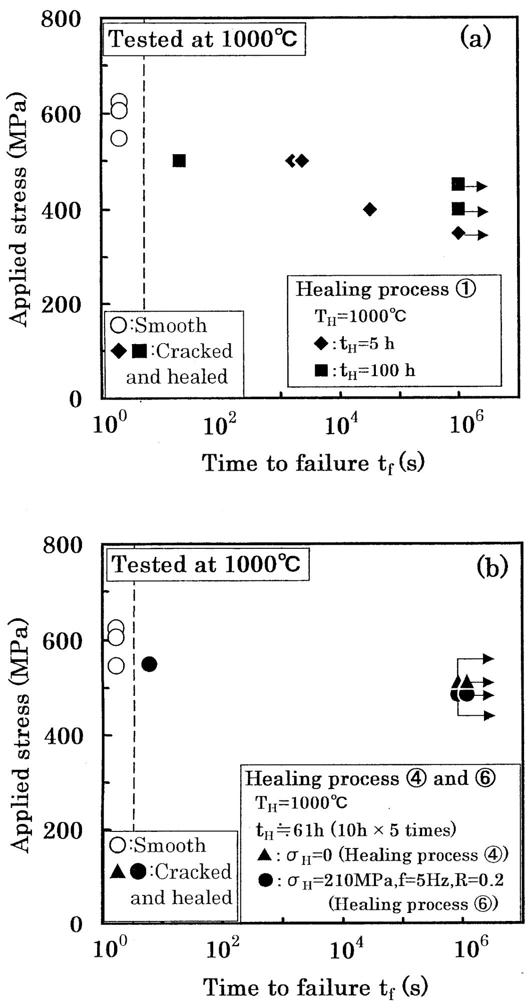

Fig. 8. Relationship between applied stress and time to failure (t_f) at 1000 °C. (a) healing process (1); (b) healing process (4) and (6).

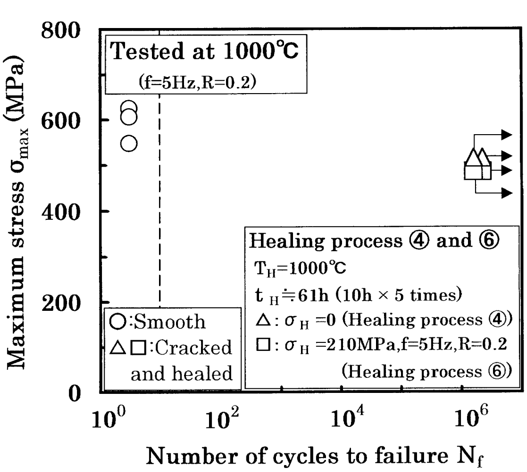

Fig. 9. Relationship between maximum bending stress (σ_{max}) and number of cycles to failure (N_f) at 1000 °C (healing process (4) and (6)).

stopped at $N = 2\times10^6$ cycles. Cyclic fatigue test conditions were: frequency = 5 Hz and stress ratio (R) = 0.2. The samples that did not fracture in the test are marked by an arrow symbol (→). The maximum stress at which a sample did not fracture up to $N = 2\times10^6$ cycles is denoted as σ_{f0}. The σ_{f0} for both multi-healed samples is 500 MPa, which is equal to σ_{t0} for multi-healed samples.

The minimum bending strength of a smooth sample at 1000 °C is 548 MPa. The ratio of the σ_{t0} or σ_{f0} of a multi-healed sample to the minimum bending strength of the smooth sample at 1000 °C is about 91%. The ratio is quite high. We can thus conclude the followings for multi-healing processes: (a) the multi-healed samples demonstrated very high performance and have sufficient strength for static and cyclic fatigue, (b) the newly developed multi-healing processes, such as (4) to (6), are beneficial.

In Fig. 6, all crack-healed samples showed almost the same bending strength at 1000 °C. However, in Fig. 8, static fatigue limit σ_{t0} depends considerably on the crack-healing process from 350 MPa to 500 MPa. To understand this interesting behavior, the relationship between a crack-healing reaction and a healing process was studied. However, the healed zone is so narrow that it is almost impossible to analyze the healing materials. Subsequently, we analyzed surface oxides instead of the healing materials using X-ray analyzer. The estimated crack-healing reactions of this sample are the following. [8,11,14]

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

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

$$2SiC + Y_2O_3 + 4O_2 \Rightarrow Si_2Y_2O_7 + 2CO_2(CO) \quad (3)$$

The SiO_2 has two phases: one is a glassy and another is a crystal phase. In reactions (2) and (3), gases generated during healing are CO_2 or CO depend on healing temperature. At 1200 and 1300 °C, a healing reactions (2) and (3) are dominant and both SiO_2 and $Y_2Si_2O_7$ show a crystal phase.[3,11,21]. However, the healing reactions at 1000 °C are not investigated yet. Typical test results are shown in Fig. 10. A 5 h crack-healed sample showed a very small amount of crystal SiO_2. Then the dominant crack-healing material of the above sample is assumed to be glassy phase SiO_2. However, both 100 h and multi-healed samples showed larger amount of crystal SiO_2. If a healing material is a crystal SiO_2 and $Y_2Si_2O_7$, the healed zone shows high bending strength and fatigue limit even at the healed temperature[13,15,21] such as at 1200 and 1300 °C. On the other hand, if a healing material is a glassy phase SiO_2, the healed zone shows lower bending strength and fatigue limit at elevated temperature.[16,18,19,24,25] As mentioned above, a 5 h crack-healed sample showed a very small amount of crystal SiO_2, and so showed lower fatigue limit σ_{t0} = 350

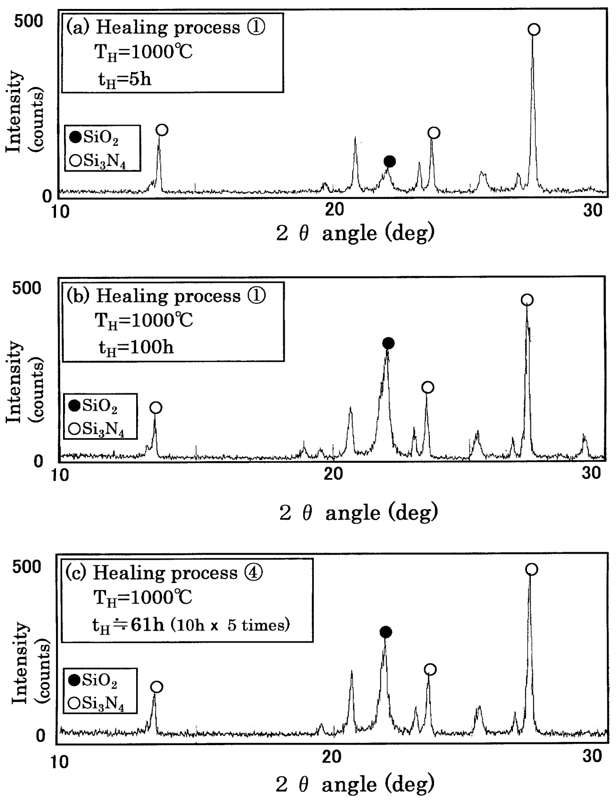

Fig. 10. X-ray diffraction analysis on surface oxide as a function of healing process.

MPa. However, 100 h and multi-healed samples showed larger amount of crystal SiO_2, and so showed higher fatigue limit $\sigma_{t0} = 450$ and 500 MPa, respectively. These test results demonstrated the following two interesting facts: (a) the healing material was crystallized or not had a little effect on bending strength at 1000 °C, however it had a large effect on the static fatigue limit σ_{t0} of crack-healed samples, (b) crystallization behavior of a glassy SiO_2 depended on crack-healing process such as healing time and thermal cycling condition. The (b) above is a very interesting research subjects not only for a materials science aspect but also for a structural integrity; and so more detail studies are expected.

3.3. Bending strength of fatigue-tested samples

Fig. 11 shows the bending strengths of the samples that survived static or cyclic fatigue tests. The bending test was performed at 1000 °C. The symbol ○ indicates the bending strength of the smooth sample at 1000 °C, and the average bending strength is 593 MPa. The symbol ◆ shows the bending strength of the sample healed for 5 h and subjected to 350 MPa static stress for 10^6 s. This sample shows almost the same bending strength. However, six other samples demonstrated bending strengths about 20% higher than that of smooth samples. These test results suggest that the sur-

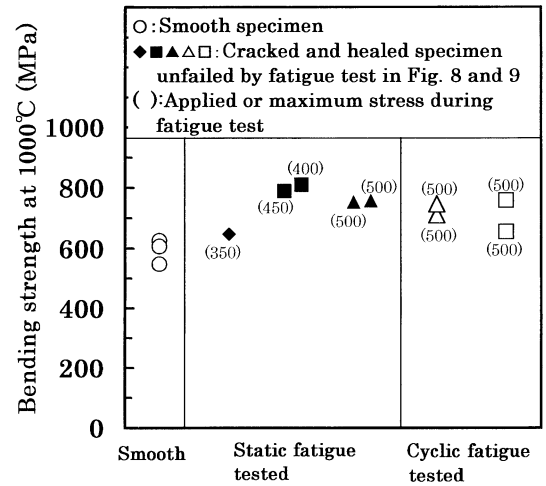

Fig. 11. Bending strength of unfailed samples at 1000 °C.

viving samples had no surface cracks. This interesting behaviour was assumed to be caused by the following three factors: (a) some of the samples showed a very slight plastic deformation during fatigue testing. This deformation introduced compressive residual stress near sample surface. (b) The sample was subjected to 1000 °C in air for a long time, so the sub-surface flaws may have healed. (c) The small surface cracks initiated during fatigue testing were healed during fatigue testing.

4. Conclusions

Si_3N_4/SiC composite ceramics were sintered and subjected to three-point bending tests. A semi-circular crack was made on each sample surface. Using these samples, we investigated crack-healing behaviour under stress at 1000 °C, and static and cyclic fatigue properties of crack-healed members, systematically. The main results are as follows.

1. Sample crack-healed at 1000 °C showed insufficient bending strength at 1300 °C, but sufficient bending strength at 1000 °C.
2. The samples exhibited very interesting crack-healing behaviour. Even under the constant or cyclic stress at 1000 °C, semi-circular crack of 100 μm in diameter was healed completely, and bending strength at RT and 1000 °C recovered completely.
3. The static fatigue limit σ_{t0} of crack-healed sample at 1000 °C depended on the healing process. To increase the static fatigue limit, a new healing process was proposed.
4. The sample healed by the new healing process showed a very high static and cyclic fatigue limit at 1000 °C. Namely, the both fatigue limits are almost the same as the lowest bending strength of smooth sample at 1000 °C.

5. Samples that survived static or cyclic fatigue tests at 1000 °C showed a 20% higher bending strength at 1000 °C than that of smooth specimen.
6. Conclusions 2, 4 and 5 above suggest that this sample can heal a crack while in service.

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