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Effect of crack-healing and proof-testing procedures on fatigue strength and reliability of Si_3N_4/SiC composites

K. Ando[a,*], M.C. Chu[a], S. Matsushita[b], S. Sato[c]

[a]*Department of Energy and Safety Engineering, Yokohama National University, 79-5 Tokiwadai, Yokohama, Japan*
[b]*Post-graduate Student, Yokohama National University, 79-5 Tokiwadai Yokohama, Japan*
[c]*NHK Spring, 3-10 Fukuura, Kanazawaku, Yokohama, Japan*

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Abstract

A Si_3N_4/SiC composite was hot-pressed. Using this material, fatigue tests on crack-healed and proof-tested specimens were conducted at 1000–1400 °C. A surface elliptical-crack of about 110 μm in diameter was introduced on the specimens using a Vickers hardness indenter. The crack-healing was performed at 1300 °C for 1 h in air, mainly. The fatigue limit of the crack-healed and proof-tested specimen (C.P specimen) decreased slightly with increasing test temperature. However, the crack-healed specimen is not sensitive to low-cycle fatigue up to 1400 °C, and the fatigue limit is almost equal to the minimum bending strength at each temperature. To investigate the reason, the crack-healing behavior under cyclic stress was carried out systematically at 1200 °C in air. A 110 μm surface crack could be healed perfectly at 1200 °C in air under cyclic stress with a frequency of 0.001–5 Hz. From this, it can be concluded that [crack-healing + proof test] and crack-healing during service are useful techniques for maintaining structural integrity of these ceramic components.

Keywords: Crack-healing, Fatigue; Mechanical properties; Proof testing, Reliability; Si_3N_4/SiC

1. Introduction

Structural ceramics are brittle and sensitive to flaws. As a result, the structural integrity of a ceramic component may be seriously affected. However, some engineering ceramics have the ability to heal a crack.[1–20] If this ability is used on structural components for engineering use, considerable advantages can be anticipated. To use materials with high crack-healing ability, following items should be studied systematically: (a) a method to evaluate a crack healing ability of a material,[5,10,11,16] (b) crack-healing mechanism,[7,10,22,25] (c) effect of chemical compositions on the crack-healing ability of ceramics,[11,13,15] (d) effect of healing condition on the strength of crack-healed zone,[10,11,14–16,21,23] (e) maximum crack size which can be healed completely,[11,13,21] (f) high temperature strength of crack-healed member,[10,11,13,15,18] (g) cyclic and static fatigue strength of crack-healed member at high temperature,[14,16–19,21,22] (h) crack-healing behavior under stress and crack-healing force[16,21,23]

Si_3N_4/SiC[11] and mullite/SiC[13] composite ceramics with high crack-healing ability were developed by the authors, and systematic studies were made on the crack-healing behaviors of both ceramics. If crack of these specimen were healed at the best conditions, the crack-healed zone has higher bending and fatigue strength than those of base material up to 1300 and 1200 °C, respectively.[14,16,17,19,20] Thus most of these specimens failed outside the crack-healed zone. However, embedded flaws and micro-structural flaws such as abnormally large grains cannot be healed. This fact was confirmed many times by examining the crack initiation sites using SEM.[13–20] These facts suggest the importance of proof test for higher reliability.[24,25] There are many useful researches on proof test for ceramic components[26–33] based on linear fracture mechanics, and on probabilistic fatigue S–N curves that can be guaranteed by proof test.[29–31] However, engineering ceramics exhibit non-linear fracture behavior,[34,35] so a new theory related to proof testing and based on non-linear fracture mechanics

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[*] Corresponding author. Fax: +81-45-339-4024.
E-mail address: andokoto@ynu.ac.jp (K. Ando).

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is required. Moreover, ceramic components are not used just at the proof-tested temperature, so a theory to explain the temperature dependence of proof stress based on non-linear fracture mechanics is also necessary. From the above points of view, a new method of [crack-healing + proof test][25–32] was proposed, recently. And the usefulness of this technology for the bending strength at elevated temperature was proved using about 200 samples.[25] Using this technology, reliability of ceramic components can be well guaranteed before service. However, if a crack initiated during service, reliability of ceramic components will decrease considerably depending on the crack size. Therefore, a material being able to heal a crack during service[16,21–23] with crack-healed zone having enough strength at the service temperature will be very desirable for structural integrity as reported by the author[16,21,22] However, crack-healing behavior under cyclic stress is not well understood yet. Thus, the following two objectives were settled in this paper: (a) Crack-healing behavior under cyclic stress at 1200 °C, (b) Fatigue strength of crack-healed and proof-tested sample in temperature ranges where the material can heal a crack by itself.

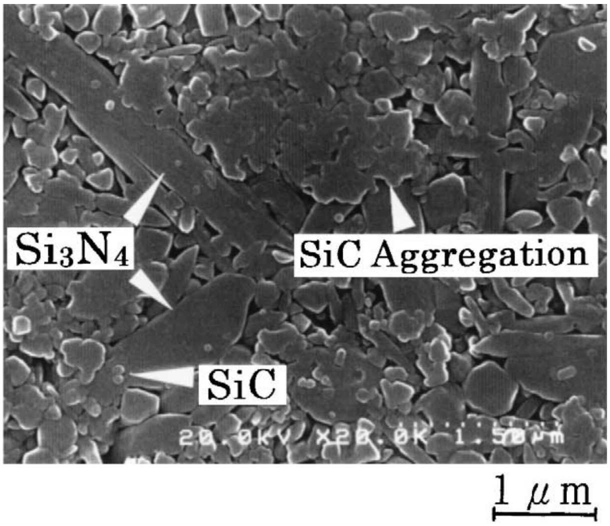

Fig. 1. Microstructure of Si$_3$N$_4$/SiC composite ceramics.

2. Experimental procedure

2.1. Material and specimen

The silicon nitride powder used in this investigation had a mean particle size of 0.2 μm, and a volume ratio of α-Si3N$_4$ of 95% (the rest is β- Si$_3$N$_4$). The SiC powder used had a mean particle size of 0.03 μm, and the sample contained 18.5% SiC powder. Y$_2$O$_3$ 8 wt.% was added to Si$_3$N$_4$ powder with SiC powder as a sintering additive. The powders were then mixed in alcohol for 48h using a nylon ball mill. Subsequently, the mixture was placed in an evaporator to extract the solvent and then in a vacuum desiccator to obtain a dry powder mixture. The mixture was next hot-pressed at 1850 °C and 35 MPa for 2 h in nitrogen gas.

Fig. 1 shows a scanning electron microscopic (SEM) photograph of the microstructure of the sintered material. The sample was heavily etched by immersing it in boiling NaOH liquid for about 1 min. The average Si$_3$N$_4$ grain size was 0.5 μm, and the mean aspect ratio was about 4.4. Most SiC grains were located at grain boundaries. However, SiC particles were not all distributed uniformly, and SiC aggregations could be found, as shown in Fig. 1. These aggregations acted as flaws and reduced bending strength. To prove the usefulness of our concept [crack-healing + proof test], many embedded flaws should be purposely introduced into a sample. In our previous study, the SiC powder having mean particle size of 0.27 μm[16,20] was used. SiC powder of mean particle size 0.27 μm distributed uniformly and did not aggregate. In our previous study,[25] an organic binder was used to form many embedded flaws. This time, however, the very small SiC particles were used to introduce more realistic flaws. The sintered plates (90×90×5 mm) were cut into specimens to the dimensions of 40×4×3 mm, Surfaces of the test specimens were ground and polished before testing in accordance with Japanese Industrial Standards (JIS).[37] A pre-crack was made at the center of the specimen's tensile surface using a Vickers hardness indenter. A load of $P = 19.6$ N was used to introduce a semicircular crack about 110 μm in diameter. A SEM photograph of the crack profile is shown in Fig. 2.

2.2. Crack-healing process

The following two types crack-healing processes were used in this study, mainly. (1) Crack-healing process A: healing temperature of 1300 °C, healing time of 1 h, in air and under no stress. (2) Crack-healing process B:

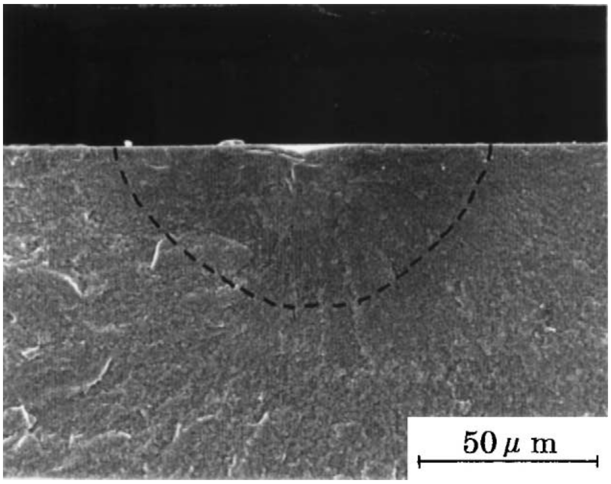

Fig. 2. SEM photograph of crack shape.

healing temperature of 1200 °C, healing time of 70 h, in air and under stress. The healing process B is divided into four stress types; Type B-I has constant stress = 160 MPa; type B-II has cyclic square wave stress with frequency (f) = 0.001 Hz, maximum applied stress ($\sigma_{\max}$) = 160 MPa, stress ratio (R) = 0.5; type B-III has cyclic sine wave stress with f = 1 Hz, $\sigma_{\max}$ = 160 MPa, R = 0.5; and type B-IV has cyclic sine wave stress with f = 5 Hz, $\sigma_{\max}$ = 160 MPa, R = 0.5. The average fracture stress of the indented sample was about 240 MPa. The applied stress of 160 MPa during crack-healing was about 67% of the fracture stress, so the stress was very severe. Crack-healing process A was applied to samples subjected to bending tests and low-cycle fatigue tests at elevated temperature (1000–1400 °C). Crack-healing process B was applied to the sample for investigating the crack-healing behavior under cyclic stress at 1200 °C. The schematic illustration of this crack-healing process B is shown in Fig. 3. In this case, firstly stress was applied to avoid unexpected crack-healing under no stress condition, subsequently power was supplied to increase the furnace temperature at a rate of 10 °C/min and hold it for 70 h. After 70 h, power was turned off. After the furnace was completely cooled down, the stress applied to the sample was removed.

2.3. Fatigue and bending test

All tests were carried out by a three-point loading system with 30 mm bending span. Four kinds of tests were carried out altogether. (a) Bending test on as-received smooth samples to measure the scatter level of bending strength and to decide the proof test stress, also on indented specimens. (b) Bending test at elevated temperature on crack-healed sample, and on crack-healed samples that survived the fatigue test. (c) Low-cycle fatigue test on crack-healed and proof-tested samples at elevated temperature. (d) Bending test related to crack-healing behavior under cyclic stress. The bending strength of the sample was measured at a crosshead speed of 0.5 mm/min at room or elevated temperatures. When the test was carried out at elevated temperature, the furnace temperature was increased to testing temperature at a rate of 10 °C/min and kept for 20 min before testing. The proof test was performed by stressing up to 525 MPa at a crosshead speed of 20 mm/min. The loading pattern of the cyclic fatigue test is illustrated in Fig. 4. All cyclic fatigue testing were carried out by the following conditions; stress ratio (R) of 0.5, stress pattern of square wave and at temperatures ranging from 1000 to 1400 °C in air, and the loading system was also a three-point bending system.

3. Test results and discussion

3.1. Bending strength and proof test at room temperature

The bending strength of as-received smooth specimens at room temperature was plotted in a Weibull diagram as shown in Fig. 5. The mean bending strength is 608 MPa and scale parameter (σ_0) is 672 MPa. The bending strength at room temperature exhibited significant scatter as expected, and its shape parameter (m) is as low as 3.91. It was assumed that a large SiC agglomeration in the sample causes the scattering of bending strength (see Fig. 1). Specimens that showed a low bending strength failed from a large embedded flaw (SiC agglomeration) as shown in Fig. 6. Oxygen from air is necessary^{25} for the crack-healing process. So the embedded flaws can't be healed. And so, proof test to remove defective samples is necessary for higher structural integrity. A proof stress of 525 MPa is adopted in this study, which is equivalent to 3 1.7% of the failure probability.

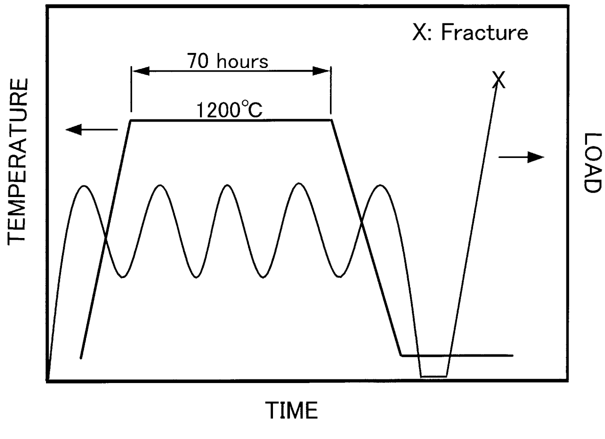

Fig. 3. Schematic illustration of loading and temperature process for the crack-healing test under cyclic stress at 1200 °C.

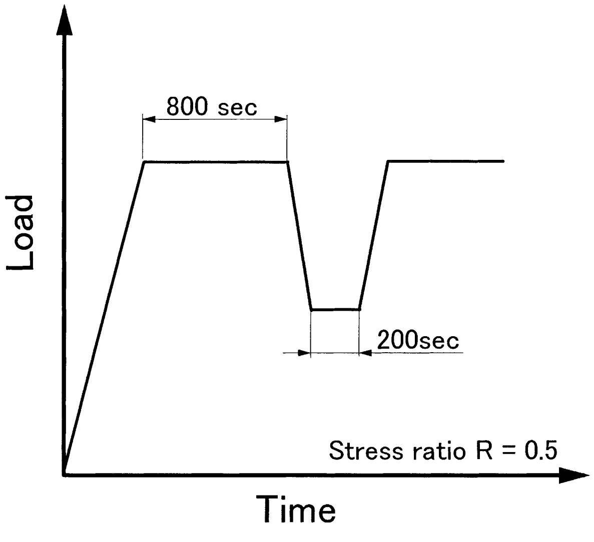

Fig. 4. Schematic illustration of loading pattern for cyclic fatigue test at elevated temperature.

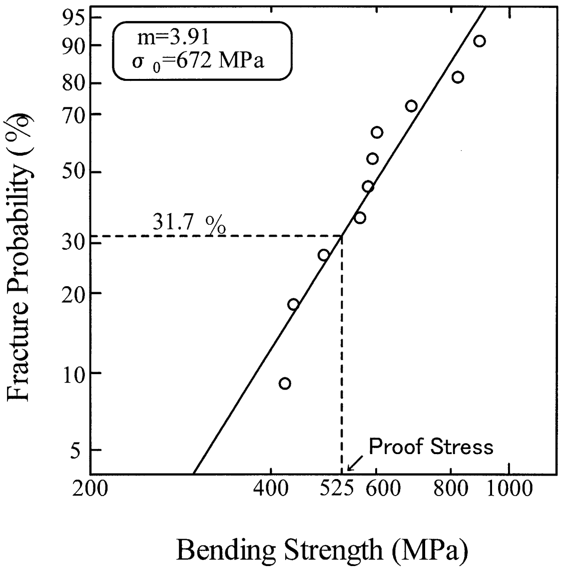

Fig. 5. Weibull plot of bending strength of as-received smooth sample.

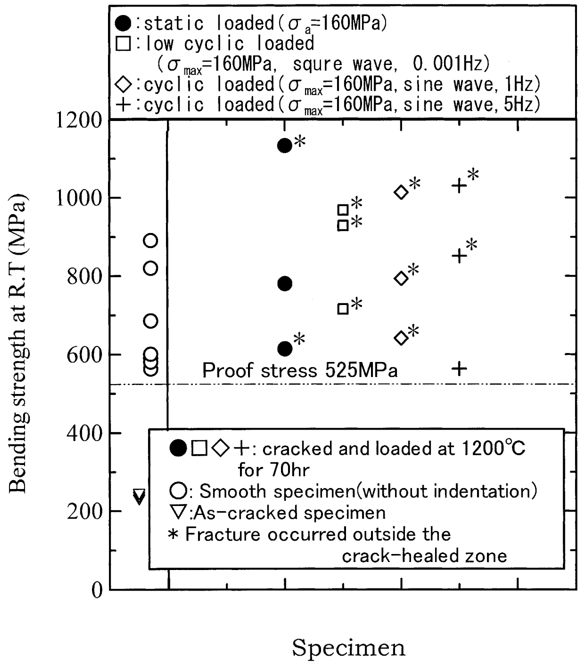

Fig. 7. Bending strength of crack-healed sample under cyclic stress at 1200 °C in air.

3.2. Crack healing behavior under cyclic stress

The crack-healing behaviors under cyclic stress were studied systematically using the crack-healing process B. Three test specimens were used in each experimental condition. No specimens fractured during crack-healing process under stress. Three-point bending test was performed at room temperature on the healed samples. The test results are shown in Fig. 7. The symbol * shows crack-healed zone was not fractured as shown in Fig. 8. The crack-healed zone did not fracture in the ten of twelve samples indicating crack-healing was complete. All samples completely recovered bending strength, and their average bending strength was 836 MPa. The minimum bending strength was about 550 MPa and was a little higher than that of proof stress (525 MPa). The average bending strength was 24% higher than that of the proof-tested smooth sample. In many cases, the sample crack-healed under stress possessed higher strength than that of the sample healed without stress.[21,22] However, the reason is not yet well understood. In conclusion, the bending strength of cracked samples recovered completely during static or cyclic loading at 1200 °C. Of course, this strength recovery was achieved by self

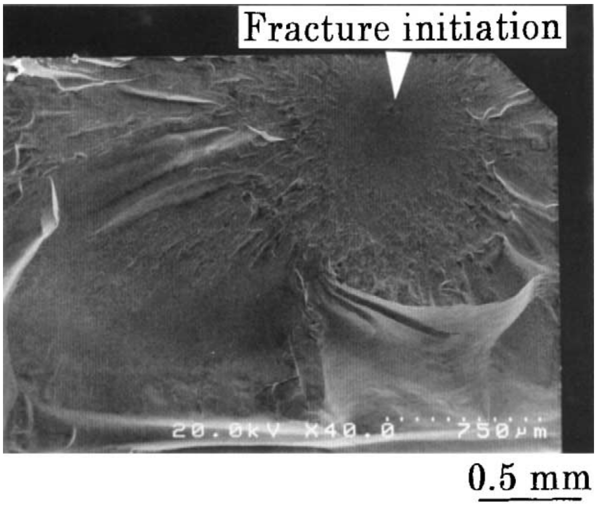

Fig. 6. SEM photograph of fracture surface on as-received specimen (fracture initiated from embedded flaw).

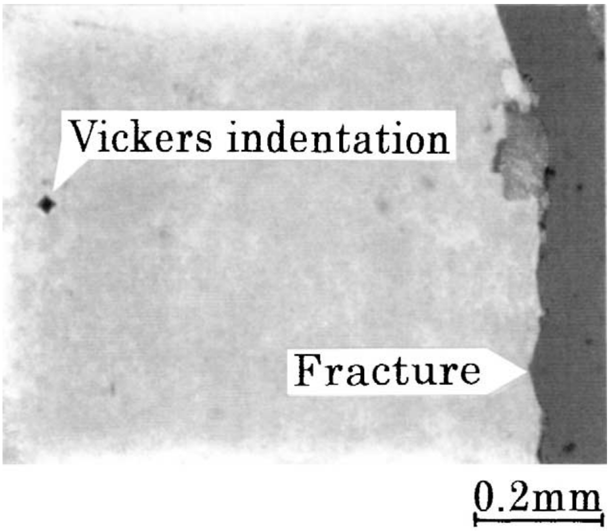

Fig. 8. SEM photograph of the crack path (fracture initiated from outside the crack-healed zone).

crack-healing. The estimated crack-healing reactions of this sample are following.[22,25]

$$Si_3N_4 + 3O_2 \Rightarrow 3SiO_2 + 2N_2 \tag{1}$$

$$SiC + 2O_2 \Rightarrow SiO_2 + CO_2(CO) \tag{2}$$

$$2SiC + Y_2O_3 + 4O2 \Rightarrow Y_2Si_2O_{7+}2CO_2(CO) \tag{3}$$

The SiO_2 has two phases: one is a glassy phase 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 1200–1400 °C. healing reactions (2) and (3) are dominant and both SiO_2 and $Y_2Si_2O_7$ show a crystal phase, if enough SiC particles are added.[22,25,36] However, at 1000 °C, most SiO_2 shows glassy phase, if special healing process is not applied.[22,36]

This result agrees well with the following four previous test results concerning ceramics with very high crack-healing ability. (1) Si_3N_4/SiC composite ceramics exhibited considerable strength recovery under constant stress at 1000 °C.[16] The healing conditions were: 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 air environment. (2) Si_3N_4/SiC composite ceramics demonstrated complete strength recovery under cyclic stress at 1000 °C.[22,36] The healing conditions were: a ratio of applied maximum stress to fracture stress of its cracked sample of about 70% ($R = 0.2$, frequency = 1–10 Hz), a temperature of 1000 °C, and a healing time of 20 h in air. (3) Cracked mullite/SiC showed considerable strength recovery under very slow strain rate testing at 1200 and 1300 °C.[13] (4) Mullite/SiC showed complete strength recovery under static fatigue testing at 1000 °C.[22] The healing conditions of the test were: a ratio of applied constant stress to fracture stress of the cracked sample of about 53%, temperature of 1000 °C, and healing time of 80 h in air. About 50% samples fractured during static fatigue test. However, surviving samples completely recovered bending strength.

From this and above test results (1)–(4), the phenomena occurring during the static or cyclic fatigue test were assumed to be as follows;[16,21,22]; (i) If the crack growth rate (C_{GR}) is larger than the self crack-healing rate (S_{CHR}): a crack will grow during a fatigue test and the sample will finally fracture, (ii) If $S_{CHR} > C_{GR}$: the crack will be healed and the sample will recover strength, (iii) If $S_{CHR} \fallingdotseq C_{GR}$: two rates will compete with each other and the sample's strength will decrease or increase slightly. In this experiment, phenomena (ii) is assumed to have occurred in the sample that recovered bending strength completely. This fact intensely indicates that this sample is able to heal a crack during service.

3.3. Fatigue strength of crack-healed and proof-tested sample at elevated temperature

Samples were cracked, crack-healed by process A and finally proof-tested. About 74% of the samples survived this proof test of 525 MPa. These samples were referred to afterward as C.P samples and were subjected to bending testing or low-cycle fatigue testing at temperatures ranging from 1000–1400 °C. The test results are shown in Fig. 9 with respect to the correlation with maximum applied stress (σ_{max}) and time to failure (t_f). In Fig. 9, symbols ○, △, □, and ◇ indicate the bending strength by monotonic loading, and symbols ●, ▲, ■ and ◆ indicate the very low-cycle fatigue strength which is almost equal to static fatigue strength. The fatigue tests were stopped at $t_f = 1 \times 10^6$ s. The samples that did not, fracture in the test are marked by an arrow (→). The maximum stress at which a specimen did not fail up to $t_f = 1 \times 10^6$ s is defined as the fatigue limit and is denoted as σ_{tf}.

From Fig. 9, it can see the following three interesting characteristics. (1) The minimum bending strength of crack-healed and proof-tested samples (σ_{BCP}) decreases with increasing test temperature, and the minimum σ_{BCP} is lower than the proof stress of 525 MPa except at 1000 °C. Moreover, all samples tested above 1200 °C fracture from the crack-healed zone as shown in Fig. 10. (2) The fatigue limit (σ_{tf}) decreases with increasing test temperature. (3) The fatigue limit is almost equal to the minimum (σ_{BCP}).

As to the characteristics (1) and (2) above; it has been shown theoretically and experimentally that the minimum bending strength of sample proof-stressed at room temperature depends on the test temperature and

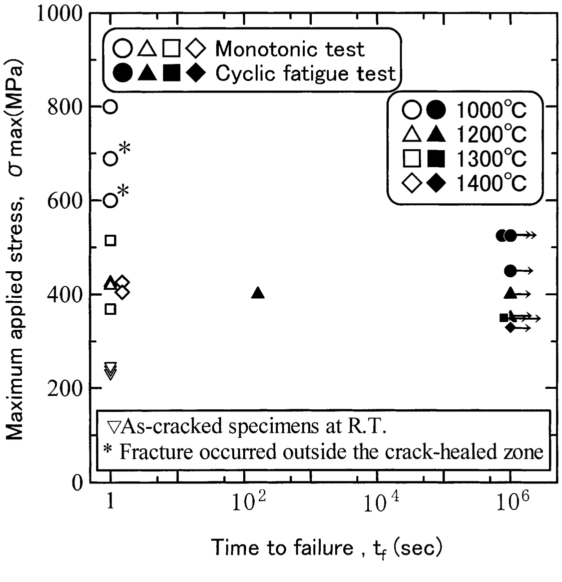

Fig. 9. Low-cycle fatigue strength of CP(crack-healing + proof-test) specimens at elevated temperature.

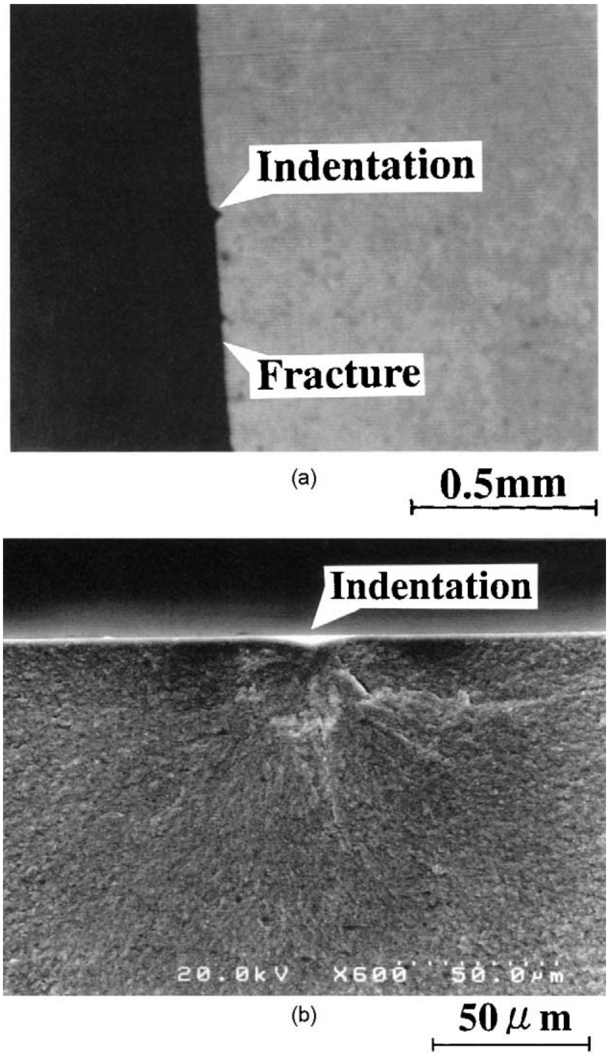

Fig. 10. SEM photograph of the crack path (fracture initiated from the crack-healed zone). (a) Crack path and indentation. (b) Fracture surface and indentation.

decreases with increasing test temperature.[25] The degree of decrease was related to the proof stress level at room temperature and tended to be smaller as the proof stress level decreased. The results obtained in this study are similar to our previous studies.[25] Generally, crack-healed zones in Si_3N_4 with 20% SiC and 8% Y_2O_3 samples exhibited higher bending strength than did the base material, and the fracture initiated from the base material up to 1300 °C in most samples.[11,16,20] However, in this test, fractures initiated from the crack-healed zone in all samples tested from 1200 to 1400 °C. In these samples, 18.5% SiC particles were added. However, many SiC aggregations were purposely formed to introduce embedded flaws. It is thus possible that not enough SiC particles were supplied for good crack-healing. If this happened, it can be easily understood that the bending strength and fatigue limit started to decrease at 1200 °C.

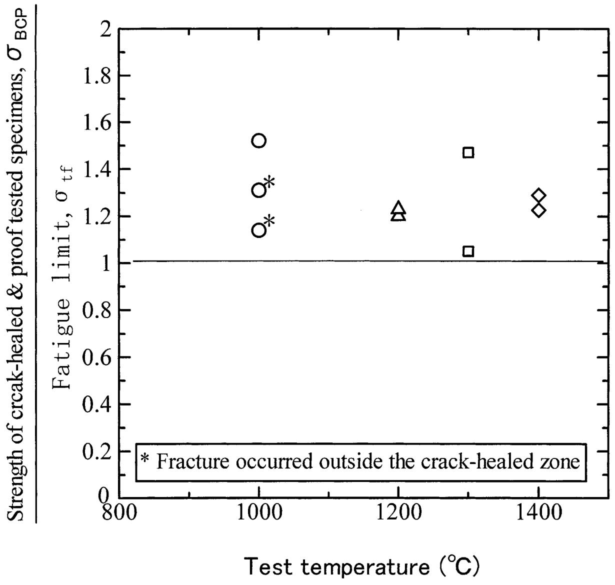

Fig. 11. Relationship between minimum bending strength of crack-healed and proof-tested sample and fatigue limit at elevated temperature.

The bending strength of these samples showed large scatter as shown in Fig. 5. However, crack-healed zone generally shows small scatter in bending strength, if crack was healed for enough time. The reason is that the effective volume of the healed zone is very small. In fact, these samples relatively showed small scatter in bending strength compared to base material at the elevated temperature. The bending strength tested from 1200 to 1400 °C showed lower σ_B than σ_{PR}. However, this behavior is very reasonable as mentioned above. Then minimum fracture stress is well controlled by the [crack-healing + proof-test] technology.

As to the characteristics (3) above: The crack-healing ability depends on healing temperature. The healing ability is higher in the following order: $1300 \geqslant 1200 \fallingdotseq 1400 > 10000$ °C.[11,16] As shown in Fig. 7, this sample can heal a crack even under 5 Hz cyclic stress. Moreover, even at 1000 °C, Si_3N_4/SiC composite ceramics can heal a crack of $2C = 100$ μm even under 5 Hz cyclic stress.[22,36] Assuming that very small fatigue crack initiated during fatigue testing, the crack will be healed during fatigue testing. Because, crack growth rate (C_{GR}) is very small, and crack-healing rate (S_{CHR}) is quite large at 1000–1400 °C as mentioned above, then $S_{CHR} > C_{GR}$ realized easily. It can be conclude that a small crack initiated during fatigue test can easily be healed during fatigue testing, and the fatigue strength becomes almost equal to the minimum bending strength at each temperature. To investigate the above consideration in more detail, the relationship between σ_{BCP} and σ_{tf} was shown in Fig. 11. The ratio (minimum σ_{BCP})/σ_{tf} is 105 to 124%. This test result supports that above consideration is reasonable.

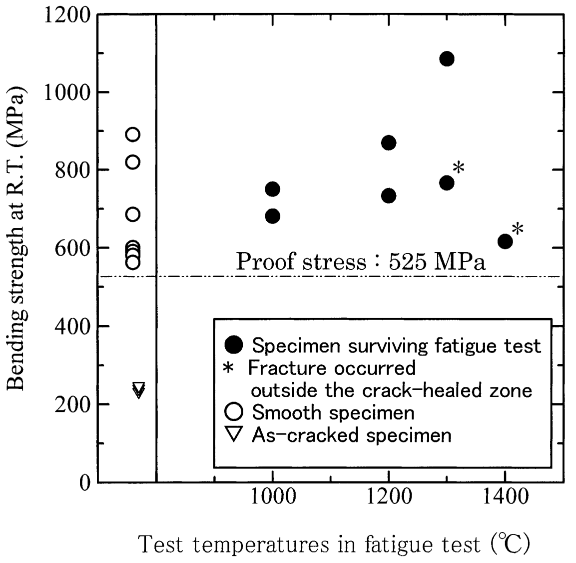

Fig. 12. Bending strength of unfailed samples fatigue-tested at temperatures ranging from 1000 to 1400 °C in air.

3.4. Bending strength of low cycle fatigue-tested samples

Fig. 12 shows the bending strength at room temperature. Symbols ○ and ▽ show the bending strength of proof tested smooth sample and cracked sample, respectively. Average bending strengths of the samples are 676 MPa (smooth) and 240 MPa (cracked). Symbol ● indicates the bending strength of the C.P samples that survived fatigue testing of up to 1×10^6 see at temperatures ranging from 1000 to 1400 °C. Symbol * means crack initiated outside the crack-healed zone. Five of the seven samples failed from the crack-healed zone. Bending strengths of all fatigue-tested samples exceeded 600 MPa and the average bending strength was 786 MPa. The average value is 16% higher than that of the proof-tested smooth sample. The crack-healed samples had Vickers indentations that acted as stress concentrators. This means that the samples were not damaged during the fatigue testing at elevated temperature, and the crack-healed zone had sufficient strength in comparison with the base material at room temperature.

4. Conclusions

Si_3N_4/SiC composite ceramics with large crack-healing ability were sintered. Many flaws were introduced into the sample on purpose. The flaws were formed by SiC aggregations. A surface elliptical-crack of 110 μm in diameter was also introduced in the sample. A crack-healing behavior under static and cyclic stress at 1200 °C was studied, systematically. A low-cycle fatigue testing on crack-healed and proof-tested sample was conducted at 1000–1400 °C. The main results are as follows.

1. This Si_3N_4/SiC material was able to heal a surface elliptical-crack of 110 μm in diameter completely by a standard healing process (1300 °C, 1 h, in air).
2. This Si_3N_4/SiC material was able to heal a surface elliptical-crack of 110 μm in diameter perfectly under both static and cyclic stress at 1200 °C in air. More than 80% of samples fractured outside the crack-healed zone indicating crack-healing was complete. This test result intensely indicates that this material can heal a crack during service.
3. The fatigue limit (σ_{tf}) of crack-healed and proof-tested samples is almost equal to the minimum bending strength of the sample at each temperature. This fact intensely suggests that fatigue cracks initiated during fatigue testing were healed completely during fatigue testing.
4. Before service, [Crack-healing + proof test] is a useful technology to guarantee the reliability of ceramic components in the previous study.[25] However, after service, crack-healing ability under static and cyclic stress is a useful technology to guarantee the reliability of ceramic components, if a ceramics member has a excellent crack-healing ability.

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