Available online at www.sciencedirect.com

SCIENCE DIRECT®

Journal of the European Ceramic Society 25 (2005) 549–558

www.elsevier.com/locate/jeurceramsoc

Crack-healing ability of structural ceramics and a new methodology to guarantee the structural integrity using the ability and proof-test

Kotoji Ando [a,*], Kotokaze Furusawa [b], Koji Takahashi [a], Shigemi Sato [c]

[a] *Department of Energy & Safety Engineering, Yokohama National University, 79-5 Hodogaya, Yokohama 240-8501, Japan*
[b] *Yokohama National University, 79-5 Hodogaya, Yokohama 240-8501, Japan*
[c] *R&D center, NHK Spring Ltd., Kanazawaku, Yokohama 236-0004, Japan*

Received 19 June 2003; received in revised form 22 December 2003; accepted 10 January 2004

Available online 15 June 2004

Abstract

Recently, the authors developed Si_3N_4, Al_2O_3 and mullite ceramics with good self-crack-healing abilities. It was shown that the optimized crack-healing condition to get high temperature strength was: 1573 K, 1 h, in air, and the healed zone exhibited the same strength as the base material up to about 1573 K (Si_3N_4 and Al_2O_3) and 1473 K (mullite), respectively. Using this good crack-healing ability, a new methodology to guarantee the reliability of ceramic components [crack-healing + proof test] was proposed. It was shown that reliability could be guaranteed before service by this technique, using about 200 samples. However, if a crack initiated during service, reliability would be severely impaired. Therefore, if a material can heal a crack during service, and if the healed zone has enough strength at the temperature of healing, it would be very desirable for structural integrity. From the above points of view, a new methodology to guarantee the structural integrity of ceramic components using in situ crack-healing ability was proposed and the usefulness is discussed using the test results in terms of crack-healing behavior and proof test theory by the authors.

Keywords: Mullite/SiC; Si_3N_4/SiC; Crack-healing; Proof test; Structural integrity

1. Introduction

Structural ceramics are brittle and sensitive to flaws. As a result, the structural integrity of a ceramic component may be seriously affected. The followings can be the excellent methodology to overcome these problems;[1] (a) toughen the ceramic by fiber reinforcement, etc., (b) activate the crack-healing ability and heal a crack after machining. If a crack-healing ability[2–6] was used on structural components for engineering use, considerable advantages can be anticipated. With this motivation, the authors developed Si_3N_4,[7] mullite,[8,9] alumina[10,11] and SiC[12] with very strong crack-healing abilities. To use these materials with a high degree of efficiency, the following topics should be studied systematically: (a) the effect of the healing condition on the strength of the crack-healed zone,[1,9,10,12,13] (b) the maximum crack size which can be healed completely,[14] (c) the high temperature strength of the crack-healed member,[1,11–14] (d) the cyclic and static fatigue strength of the crack-healed member at elevated temperature,[14–20] (e) a new methodology to guarantee structural integrity of the ceramic component using the crack-healing ability.[21]

Systematic studies were made on the above subjects by the authors. As a result, in the case of most ceramics above, the crack-healed zones exhibited excellent mechanical properties almost up to the heat-proof temperature for the strength of the base material, if the ceramics were healed at the optimized conditions. The temperature where bending strength starts to decrease rapidly with increasing testing temperature is defined as heat-proof temperature. These test results suggest that the crack-healing ability can be used as a method to guarantee the structural integrity of a ceramic component. However, oxygen is necessary for the crack-healing process.[7–9] Thus, 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.[7–12] These facts suggest the importance of a proof test to ensure higher reliability.[21–27]

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

0955-2219/$ – see front matter
doi:10.1016/j.jeurceramsoc.2004.01.027

There is much useful research on proof tests for ceramic components[22–26] based on linear fracture mechanics, and on probabilistic fatigue *S–N* curves that can be guaranteed by the proof test.[25–27] However, engineering ceramics exhibit non-linear fracture behavior,[28–30] so a new theory related to proof testing and based on non-linear fracture mechanics 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.[29,30] From the above points of view, a new method of [crack-healing + proof test][21] was proposed, recently. And the usefulness of this technology for the bending strength at elevated temperature was proved using about 200 samples (if one counts the total samples that were fractured by the proof test and used to evaluate fracture strength of the smooth sample and K_{1C}, a total of 350 samples was used).[21] Using this technology, the reliability of ceramic components can be well guaranteed before service. However, if a crack initiates during service, the reliability of ceramic components will decrease considerably depending on the crack size. There are two ways of overcoming this problem:[10] (a) a periodic proof test to remove the components with non-acceptable flaws; (b) activating the in situ crack-healing ability and heal the crack which initiated during service. Recently, the following interesting test results were obtained by the authors: (1) Si_3N_4 and mullite showed excellent crack-healing ability even under constant and cyclic stress at temperatures from 800 to 1200 °C[19,20] and from 1000 °C to 1200 °C,[14] respectively; (2) the healed sample exhibited almost the same mechanical properties as the base material at the temperature of healing.[11,14,19] Namely, it can be said that both ceramics have excellent in situ crack-healing ability. However, the systematic study has not been made on in situ crack-healing behavior. In this paper, in situ crack-healing behavior of mullite/SiC composite has been made systematically, and a new concept; [crack-healing + proof test + in situ crack-healing] is proposed. The usefulness of the new concept is discussed using these and previous test results in terms of crack-healing and proof test theory by the authors.

2. A new concept of [crack-healing + proof test + in situ crack-healing]

Flow chart of a new methodology to guarantee the structural integrity of a ceramic component is shown in Fig. 1. This new concept consisted of the following three stages; (a) crack-healing under optimized conditions, (b) proof testing, and (c) in situ (in-service) crack-healing.

By machining, many surface cracks will be induced and reliability will decrease considerably. However, by crack-healing under optimized conditions, surface cracks can be healed completely and reliability will be increased. The basic crack-healing behavior of the structural ceramics at optimized temperature is discussed in detail in Section 3.

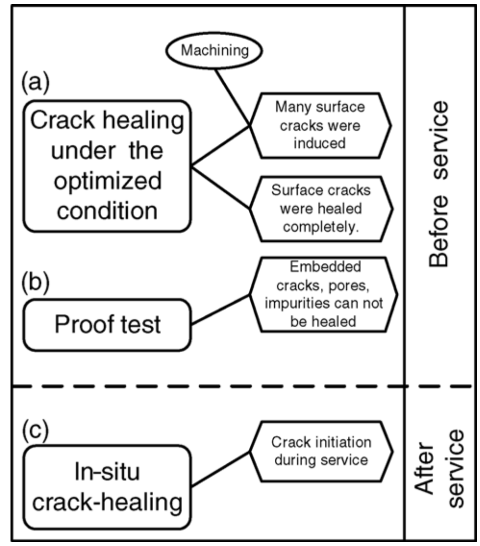

Fig. 1. Flow chart of a new methodology to guarantee the structural integrity of ceramic components using in situ crack-healing ability and proof-testing.

However, for the crack-healing of the above ceramics, oxygen is necessary. Consequently an embedded crack cannot be healed at all. This fact means that structural integrity before service cannot be guaranteed only by crack-healing technology. Thus a proof test is necessary. Recently, a new theory to explain the temperature dependence of proof stress based on non-linear fracture mechanics was proposed.[21] The outline of this theory is explained in Section 5. Thus, before service, the structural integrity of ceramic components can be confidently guaranteed using the concept; crack-healing + proof test. After service, if a crack initiated, structural integrity will decrease considerably depending on the crack size. However, if a material can heal a crack in service (that is to say, if a material has an in situ crack-healing ability), it would be very desirable for structural integrity. Thus, for the whole lifetime, a new concept which may be called [crack-healing + proof test + in situ crack-healing] will be very desirable. In situ crack-healing ability of the structural ceramics is explained in Section 4.

3. Basic crack-healing behavior of structural ceramics

3.1. Effect of environment on the crack-healing behavior

Fig. 2 shows the standard surface crack used for evaluating the basic crack-healing behavior. The specimens used in this paper were made according to JIS standard.[31] The summary of chemical compositions, sintering conditions and optimized crack-healing conditions of the materials used is listed in Table 1. The details of the above information of mullite/SiC[14,36] used mainly in this paper were as follows: The mullite powder used has a mean particle size of 0.2 μm and an alumina content of 71.8%. The SiC powder has a

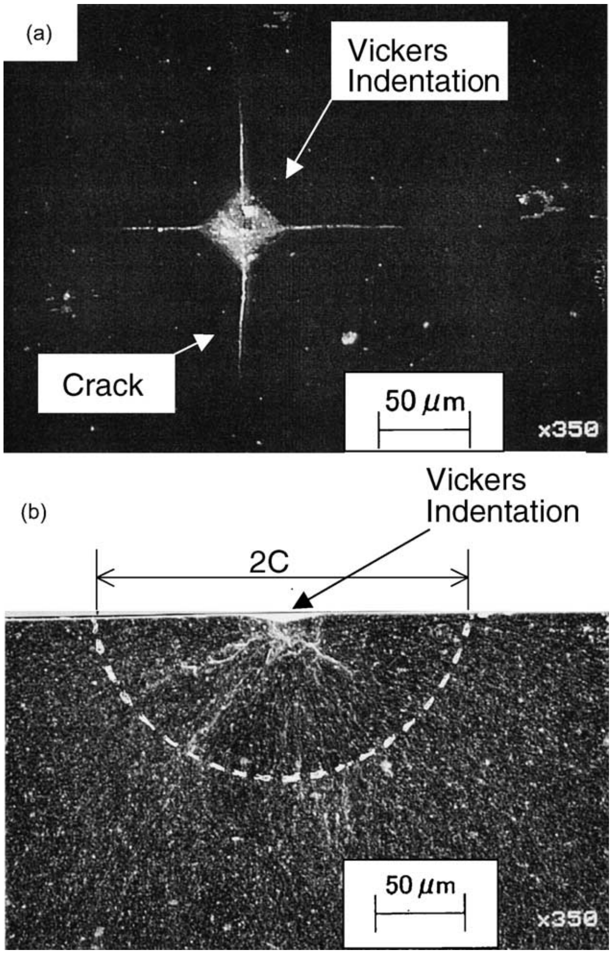

Fig. 2. (a) Pre-crack and indentation, and (b) standard crack shape.

mean particle size of 0.27 μm. The quantity of SiC powder added is 15 mass%, in contrast to mullite powder. The mixture is hot-pressed at 923 K, 4 h and 35 MPa in nitrogen. The sintered material has an average grain size of 0.46 μm, and SiC particles are located in grain boundary and distributed uniformly.

The semi-elliptical surface crack was induced by an indentation technique using a Vickers indenter. Fig. 2(a) shows cracks and indentation, and Fig. 2(b) shows the surface crack shape obtained for mullite/SiC. A surface crack length $2C \approx 100$ μm was induced by controlling the Vickers indentation load, and the crack depth was about 45 μm. This crack was defined as the standard crack. Fig. 3 shows the effect of environment on the crack-healing behavior of mullite/SiC[32] and Al_2O_3/SiC.[10] The contrast between bending strength (σ_B) of smooth and cracked samples was shown by the left-most column of Fig. 3. The sintering process and the basic properties of these samples were described in detail in the Table 1. In these tests, the following three types of fracture were observed:(1) crack initiation from a pre-crack as shown in Fig. 4(a). This type of fracture usually occurred when crack-healing was incomplete. (2) Crack initiation from the base material, and the crack-healed zone did not fracture, as

Table 1
Chemical composition of ceramics, sintering condition, maximum crack size that can be healed completely, reduction ratio of bending strength by the maximum crack and optimized crack-healing condition

Sample	Composition	Sintering conditions				$2C_{max}$ (μm)[a]	Reduction ratio of bending strength by the crack with $2C_{max}$ (%)	Optimized crack-healing conditions for $2C = 100$ μm surface crack		
		Temperature (K)	Time (h)	Environment	Hot-pressing (MPa)			Temperature (K)	Time (h)	Environment
Al_2O_3	–	–	–	–	–	200	50	1773	1	Air, N_2, Ar, vacuum
SiC	–	–	–	–	–	450	84	1773	1	Air
Mullite/SiC	Mullite + 15 vol.% SiC(p)	1923	4	N_2	35	200	70	1573	1	Air
Al_2O_3/SiC	Al_2O_3 + 15 vol.% SiC(p)	1873	4	N_2	35	200	83	1573	1	Air
SNC-Y8	(Si_3N_4 + 20 wt.% SiC) + 5 wt.% Y_2O_3 + 3 wt.% Al_2O_3	2123	2	N_2	35	200	60	1573	1	Air
SNC-Y5A3	(Si_3N_4 + 20 wt.% SiC) + 8 wt.% Y_2O_3	2123	2	N_2	35	200	60	1573	1	Air

[a] $2C_{max}$: maximum crack size that can be healed completely.

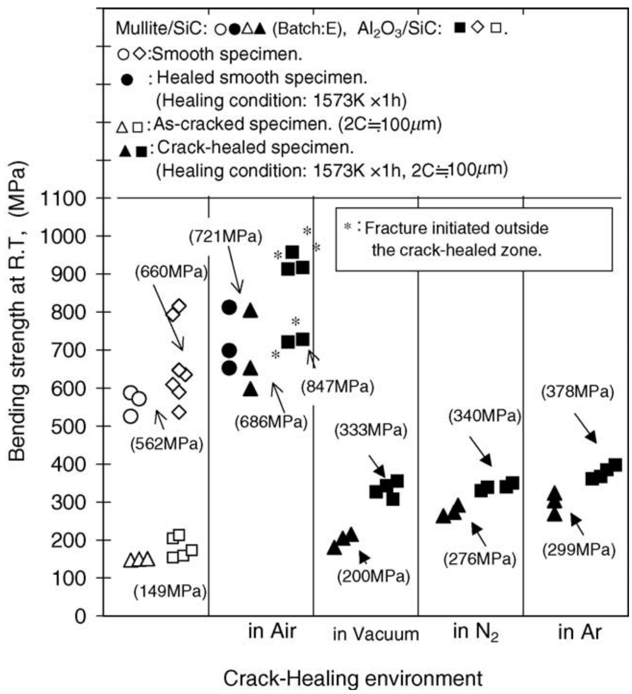

Fig. 3. Effect of environment on the crack-healing behavior of mullite/SiC and alumina/SiC.

shown in Fig. 4(b). Fig. 4(c) shows the crack initiation site of Fig. 4(b) and no special flaw can be seen on the site. (3) The sample fractured into many pieces and the crack initiation site could not be found. In the case of high bending strength (σ_B), most samples showed this type of fracture. The symbol (*) indicates that samples fractured outside the crack-healed zone as shown in Fig. 4(b). All samples of both ceramics healed in air recovered σ_B completely, and showed that the cracks were healed completely. Especially, healed smooth specimen and crack-healed sample exhibited higher bending strength than that of smooth sample, because either small cracks on smooth sample and standard crack ($2C = 100\,\mu m$) were healed completely. Samples of both ceramics healed in vacuum, Ar gas and N_2 gas indicated that the strength recovery was insufficient, and all samples fractured from the crack-healed zone as shown in Fig. 4(a). These test results showed that a crack in mullite/SiC and Al_2O_3/SiC can be healed completely only in an air environment similar to experience with silicon nitride.[7] This test result clearly shows that crack-healing needs oxygen in the air, thus an embedded crack cannot be healed.

3.2. Effect of temperature and time on the crack-healing behavior

Crack-healing behavior depends on both healing temperature (T_H) and time (t_H). To find this relationship, 14 kinds of healing conditions were tested, using mullite/SiC.[32] The test results are shown in Fig. 5. The bending strength σ_B of smooth (○) and cracked (△) specimens are compared in the

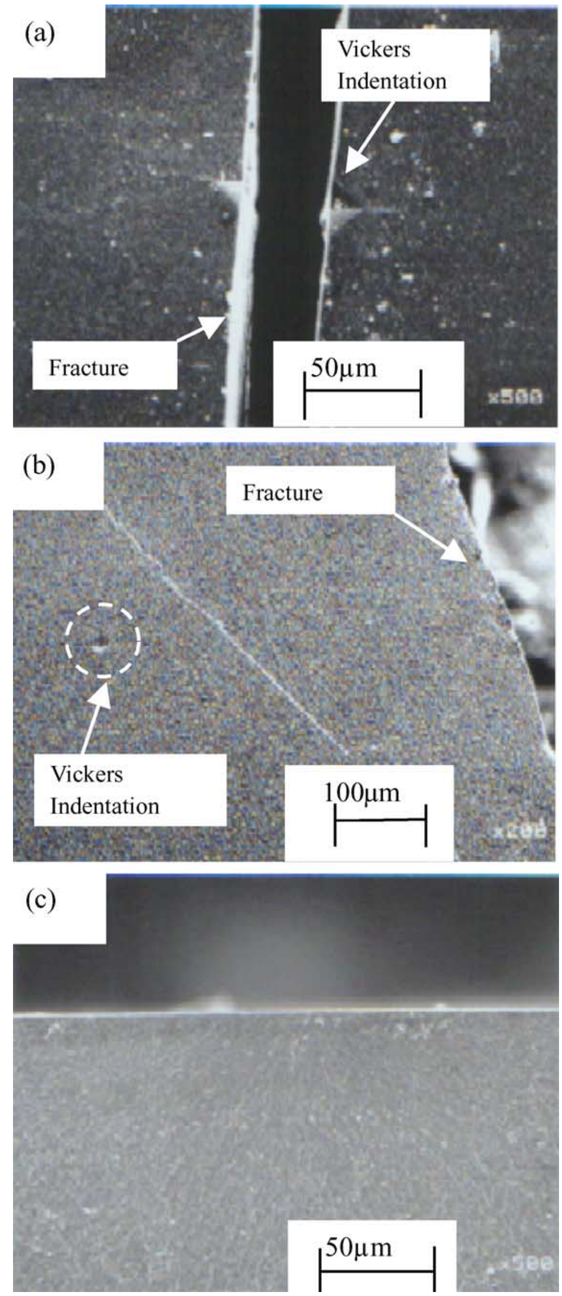

Fig. 4. SEM images of the crack path of mullite/SiC. (a) Crack initiation from the crack-healed zone. (b) Crack initiation from outside the crack-healed zone. (c) Crack initiation site with no flaw.

left-most column. The symbol (*) indicates that fracture occurred from outside the crack-healed zone, as mentioned before in Fig. 4(b). The symbol (■) indicates the σ_B obtained by healing time $t_H = 1$ h at each healing temperatures. Note that σ_B does not recover below $T_H = 1223$ K, but it recovers considerably at $T_H = 1373$ and 1473 K. However, when considering that many fractures occurred from a pre-crack, as shown in Fig. 4(a), the strength recovery is not sufficient. On the other hand, at $T_H = 1573$ K, the average σ_B of the healed specimen is higher than that of the smooth specimen.

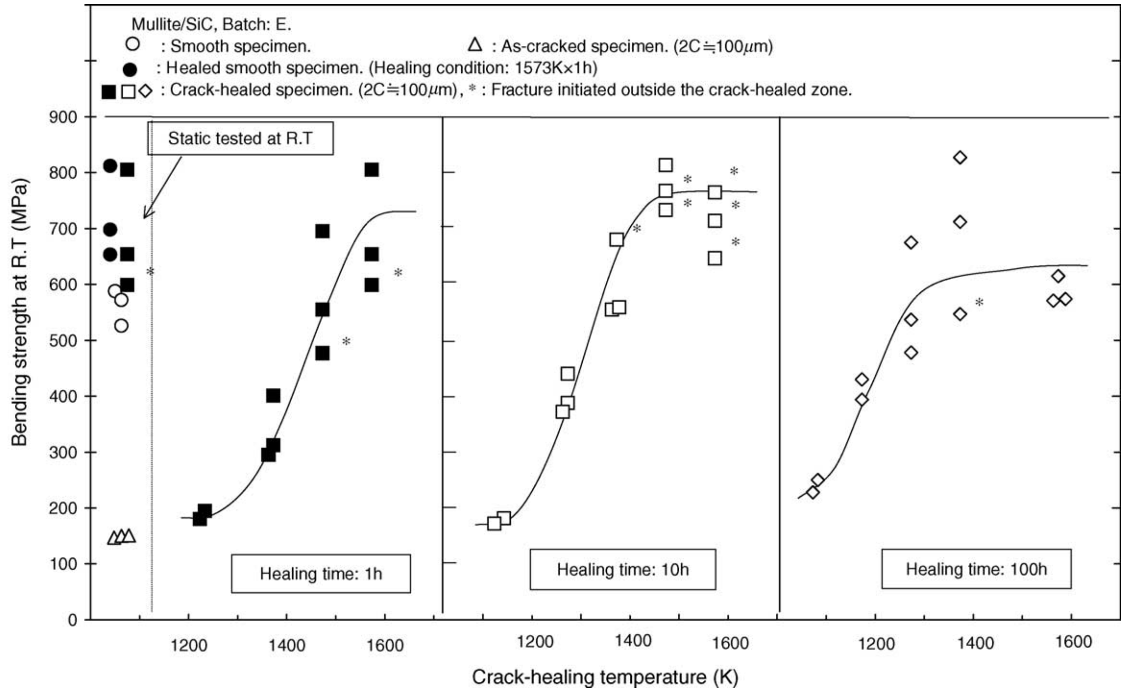

Fig. 5. Effect of temperature and time on the crack-healing behavior of mullite/SiC.

In conclusion, the lowest crack-healable temperature for $t_{HM} = 1$ h is $T_{HL} = 1573$ K. In the same way, the lowest crack-healable temperature conditions for $t_{HM} = 10$ h (□) and $t_{HM} = 100$ h (◇) are $T_{HL} = 1473$ K and $T_{HL} = 1373$ K, respectively.

From the σ_B versus healing temperature curve in Fig. 5, the lowest temperatures (T_{HL}) were selected, where the average σ_B of the crack-healed sample exceeded the average σ_B of smooth specimens, for each healing time ($t_{HM} = 1$, 10, 100 h), and the T_{HL} were plotted in the Arrhenius graph as shown in Fig. 6.[10,33] In short, $T_{HL} = 1573$ K is for $t_{HM} =$ 1 h, $T_{HL} = 1473$ K is for $t_{HM} = 10$ h, and $T_H = 1373$ K is for $t_{HM} = 100$ h. Where, the minimum healing time for complete strength recovery is denoted by (t_{HM}), since this expression is convenient for the Arrhenius graph. The Arrhenius plots of four kinds of ceramics are also shown in Fig. 6. Symbol (□) indicates the result on mullite/SiC[23] used mainly for this study, symbols (○), (●) and (■) show the results for monolithic alumina, Al_2O_3/SiC[22] and SNC-Y8,[25] respectively. The crack sizes of these three specimens are $2C \fallingdotseq 100$ μm, and they are healed in an air environment. Symbol (↑) shows that the crack can be healed within this time period. With respect to these four kinds of ceramics, both ($T_{HL}{}^{-1}$) and ($1/t_{HM}$) are in closely proportional relation. Therefore, the crack-healing behavior of these ceramics follows the Eq. (1).[10,33]

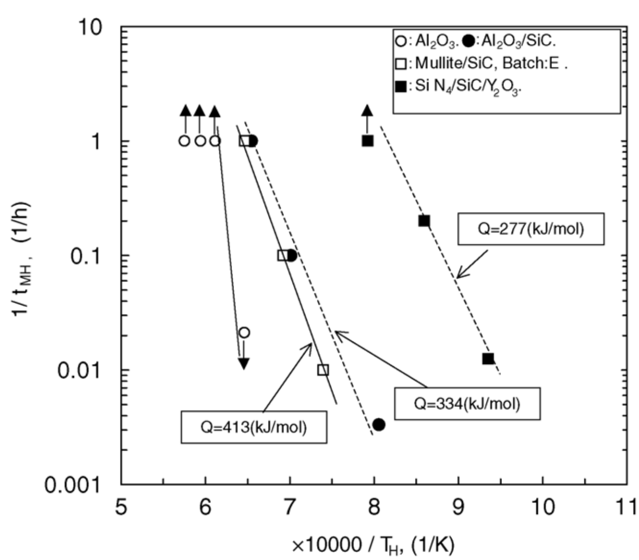

Fig. 6. Arrhenius diagram and activation energy of crack-healing behavior.

$$\left(\frac{1}{t_{HM}}\right) = A_H \times \exp\left(-\frac{Q_{aH}}{R \times T_{HL}}\right) \tag{1}$$

where A_H is a proportionality constant (h^{-1}), Q_{aH} is the activation energy of crack-healing (kJ/mol), R is the gas constant (kJ/mol K) and T_{HL} is the lowest absolute temperature of the healing (K). The Q_{aH} and A_H of each of the ceramics are shown in Table 2. A crack-healable condition of the standard crack can be evaluated easily as a function of temperature and time, using Eq. (1) and Table 2.

3.3. *Effect of pre-crack size on the crack-healing behavior*

A surface crack ($2C = 100-250$ μm, aspect ratio $= 0.9$) was introduced on the mullite/SiC sample,[14] and healed

Table 2
Activation energy (Q_{aH}) and proportionality constant (A_H) of crack-healing behavior

	Sample name	Q_{aH} (kJ/mol)	A_H (h^{-1})
1	SNC-Y5A3	150	5.3×10^4
2	SNC-Y8	277	2.7×10^9
3	Al_2O_3/SiC	334	7.3×10^{10}
4	Mullite/SiC	413	4.7×10^{13}

under the standard condition of mullite/SiC ($T_H = 1573$ K, $t_H = 1$ h, air environment). Subsequently the bending test was carried out at room temperature. The relationship between the crack length ($2C$) and bending strength (σ_B) is shown in Fig. 7. The symbols (●) and (▲) shows the σ_B of the heat-treated smooth and crack-healed sample, respectively. The σ_B of crack-healed sample regains the same level σ_B as the heat-treated smooth specimens, when $2C$ is smaller than 200 μm. But the σ_B decreases suddenly, when $2C$ is over 200 μm. Therefore, the maximum crack size to be healed completely is $2C = 200$ μm. The critical parameter for the complete crack-healing is not crack-length but crack-depth. If a crack is deep enough, oxygen cannot supplied well, thus the crack cannot be healed completely. SNC-Y8, SNC-Y5A3, Al_2O_3/SiC and SiC were also tested in the same way. Table 1 lists the maximum crack size to be healed completely for six kinds of structural ceramics.[14,34,35] Only SiC could heal a large crack of $2C = 450$ μm by itself.[34] The other materials can heal a crack up to $2C = 200$ μm, completely.

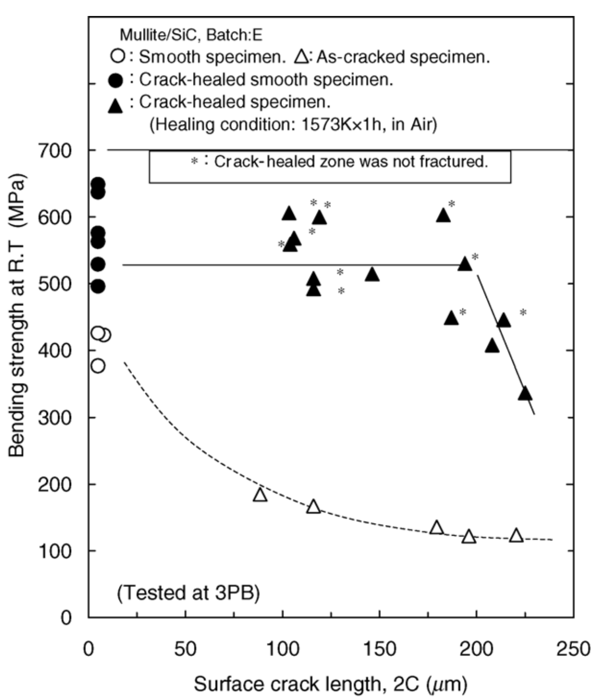

Fig. 7. Effect of crack size on the crack-healing behavior of mullite/SiC.

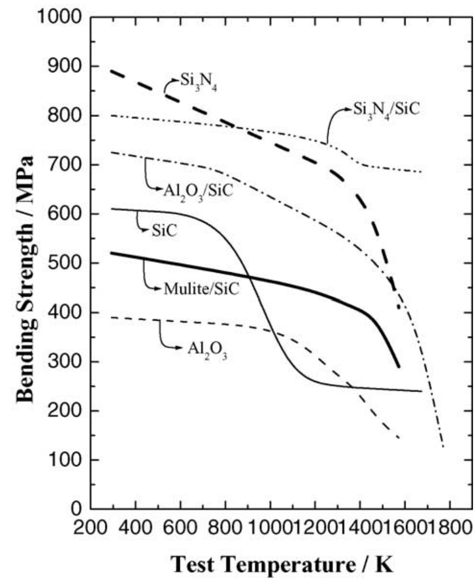

Fig. 8. Effect of testing temperature on the bending strength of crack-healed sample.

3.4. Bending strength of the crack-healed sample at elevated temperature

For the practical use of the crack-healing technology, the bending strength (σ_B) of the crack-healed sample at elevated temperature is very important. The temperature dependence of the σ_B in six crack-healed ceramics is shown in Fig. 8. Monolithic Al_2O_3 was healed at 1723 K, 1 h in air. For this case, crack-healing is a re-sintering mechanism, and the heated sample showed the same value of σ_B as that of the base material up to 1573 K and numerous samples fracture outside the crack-healed zone.[35] Mullite/SiC[36] and Al_2O_3/SiC[10] were healed at 1573 K, after 1 h in air. Crack-healed mullite/SiC and Al_2O_3/SiC showed high heat resistance up to 1473 and 1573 K, respectively and most samples fractured outside the crack-healed zone up to 1573 K. The SiC was healed at 1773 K, after 1 h in air. The base material showed a high σ_B up to 1673 K, however, the heat-proof temperature of the crack-healed sample was about 873 K and considerably lower than that of base material.[34] Recently, SiC having a heat-proof temperature of 1473 K of the crack-healed zone has been developed.[12] The crack-healed zone of SNC-Y5A3 is a glassy phase, so its heat-proof temperature is moderate, being about 1273 K, however, the crack-healed zone of SNC-Y8 healed at 1573 K, after 1 h in air is crystalline SiO_2, thus the healed zone showed a higher heat-proof temperature of 1673 K.[1]

3.5. Crack-healing behavior under constant and cyclic stress

The crack-healing behavior under constant or cyclic stress was investigated systematically. First stress was applied to the sample to prevent unexpected crack-healing under no

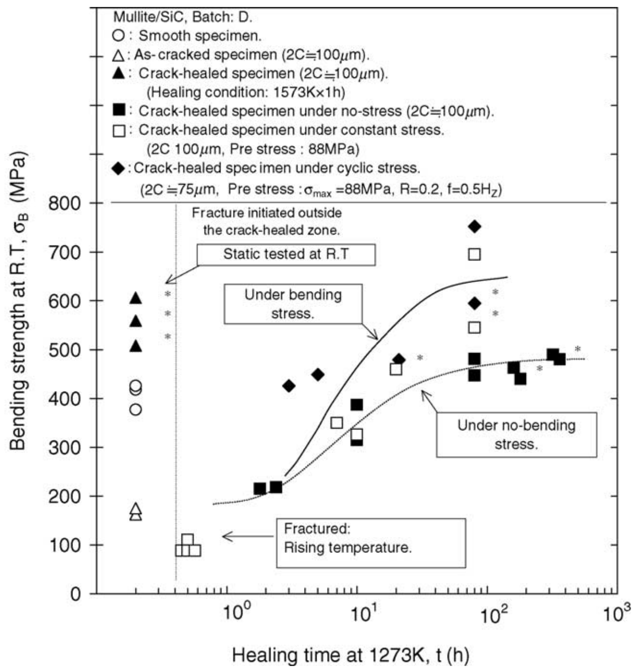

Fig. 9. Effect of applied stress (constant and cyclic) and crack-healing time on the bending strength of mullite/SiC at room temperature.

stress condition. Subsequently power was supplied to increase the furnace temperature at a rate of 10 K/min and hold it for an arbitrary time. After the time, power was turned off. After the furnace had completely cooled, the stress applied to the sample was removed, and bending strength was measured at room temperature.

Fig. 9 shows the crack-healing behavior of mullite/SiC at 1273 K as a function of healing time.[32] The symbol (△) shows the σ_B of cracked sample. The symbol (■) shows the σ_B of crack-healed sample under no stress condition. The σ_B increased with increasing healing time and above 80 h the σ_B was saturated to about 450 MPa. The symbol (□) shows the σ_B of crack healed samples under constant stress of 88 MPa. For this case, about 50% of the samples fractured during heating up, because applied stress was so high compared with bending strength of the cracked sample. The σ_B of the surviving samples increased with increasing healing time and reached about 600 MPa at 80 h healing time. This σ_B is a little higher value than that of samples healed under no stress condition. However, the reason was not well understood yet. The symbol (◆) shows the σ_B of crack-healed sample under cyclic stress. For this case, the pre-crack size was reduced to 75 μm, thus no sample fractured during healing and exhibited a high level σ_B (≒600 MPa) at 80 h healing time. These test results show that mullite/SiC is able to heal a crack even under stress at 1273 K.

Fig. 10 shows the fracture surface of the sample that was healed at 1273 K for 10 h and fractured at room temperature. The bending strength of the sample was 326 MPa and fractured from the crack-healed zone. The dark area of Fig. 10 shows the cracked zone that was healed slightly after 10 h.

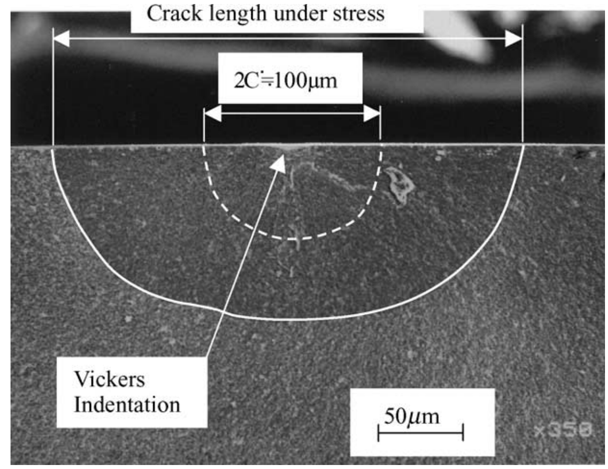

Fig. 10. Fracture surface of crack-healed mullite/SiC under constant stress. Batch: D. Applied constant stress: 88 MPa. Healing condition: 1273 K × 10 h. σ_B: 326 MPa.

The crack size was $2C \approx 250\,\mu m$, thus the pre-crack enlarged considerably during heating up and the enlarged crack was healed at 1273 K for 10 h.

Fig. 11 shows the effect of frequency of applied stress on the crack-healing behavior of the mullite/SiC.[10] For this tests, a smaller crack ($2C = 75\,\mu m$) than standard crack was used, so as to avoid the unexpected fracture during crack-healing as mentioned above. The crack was healed at 1273 K for 80 h in air. The σ_B of crack-healed sample under stress (88 MPa) showed considerably large scatter. However,

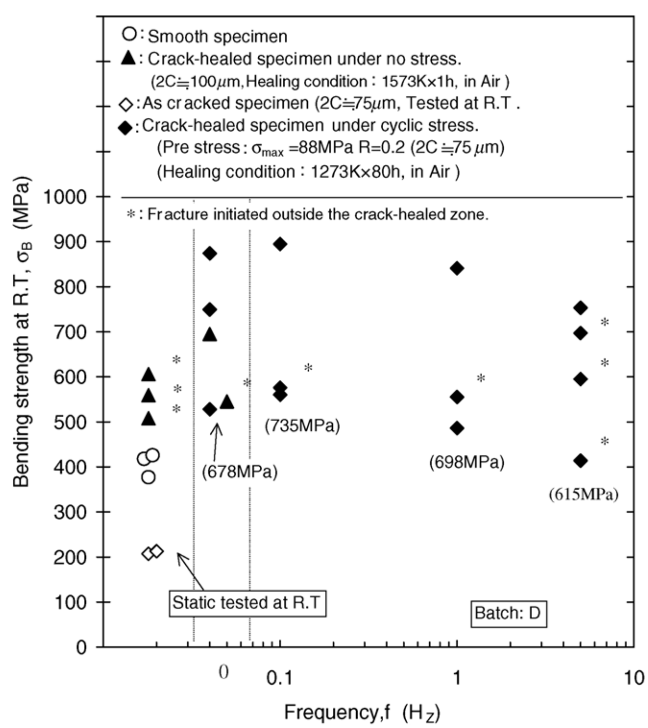

Fig. 11. Effect of frequency on the crack-healing behavior of mullite/SiC.

the minimum σ_B was about 400 MPa and the σ_B was almost equal to the σ_B of a smooth sample. Moreover, the average σ_B of a crack-healed sample under stress was almost independent of the stress frequency (f), and the value was considerably larger than that of a crack-healed sample under no stress condition. This fact showed that this mullite/SiC can heal a crack completely even under cyclic stress of 5 Hz.[32]

4. In situ crack-healing behavior and resultant strength at the temperature of healing

4.1. In situ crack-healing behavior of mullite/SiC

Fig. 12 shows the σ_B of crack-healed sample at the temperature of healing.[32] This behavior was defined as in situ crack-healing behavior. The symbol (▲) shows the σ_B of the sample crack-healed under the optimized conditions (1573 K for 1 h in air). The symbols (■) and (□) show the σ_B of in situ crack-healed samples under no-stress and cyclic stress condition, respectively. For example, the crack was healed at 1273 K and the σ_B was also measured at 1273 K. When looking first, all samples show almost the same σ_B except a single sample that fractured from the base material and is shown by the symbol (▲*). This test results show that the mullite/SiC developed by the authors exhibits excellent in situ crack-healing ability.[10]

Fig. 13 shows the cyclic fatigue strength of mullite/SiC at 1273 K.[32] The symbols (■) and (□) indicate that the standard cracks of mullite/SiC were healed at 1573 K, 1 h, and 1273 K, 340 h, respectively. For this test, Batch D was used, and the samples showed a little lower strength. Both samples

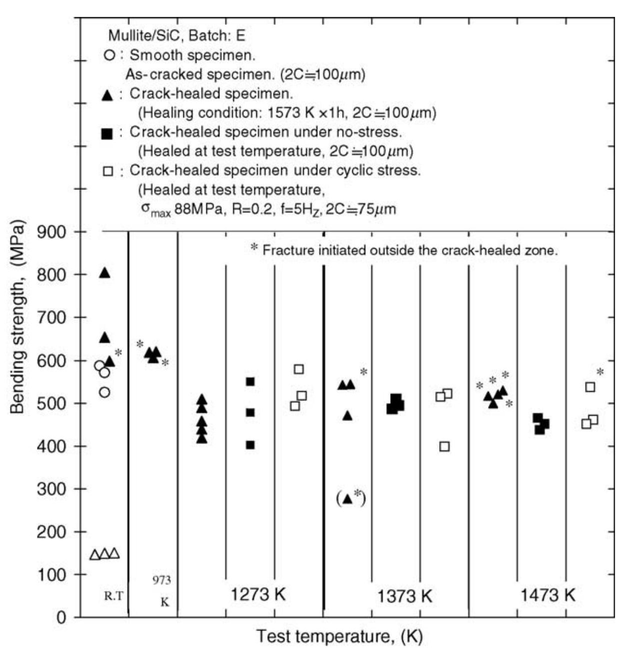

Fig. 12. In situ crack-healing behavior of mullite/SiC at 1273, 1373 and 1473 K.

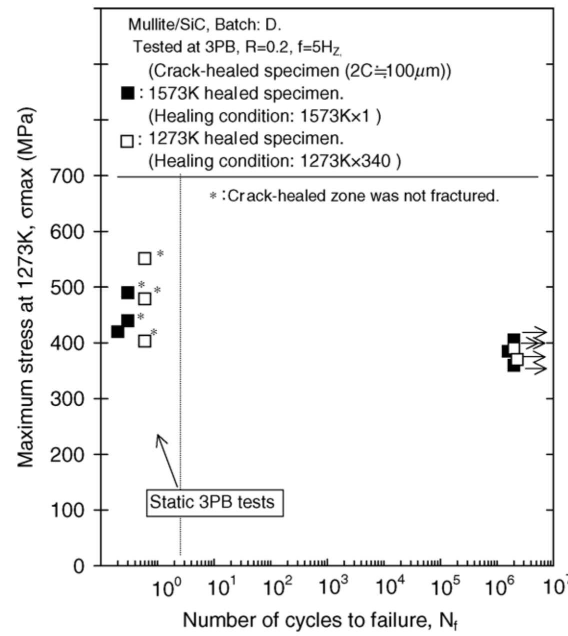

Fig. 13. Effect of crack-healing temperature on the fatigue strength of mullite/SiC.

showed the same fatigue limit (400 MPa). From this test results, it can be concluded that the mullite/SiC exhibits excellent in situ crack-healing ability even for fatigue strength.

4.2. In situ crack-healing behavior of Si_3N_4/SiC

Fig. 14 shows the cyclic fatigue strength at 1273 and 1473 K of sample, that were crack-healed under the cyclic stress at 1273 and 1473 K, respectively.[20] The crack healed under the very severe condition (5 Hz cyclic stress of 210 MPa). The stress 210 MPa is the fatigue limit of this sample with $2C = 100\,\mu$m surface crack at room temperature.[16] At 1273 K, the 15 h-healed sample exhibited a rather low fatigue limit of 550 MPa, however, the 50 h-healed sample exhibited the very high fatigue limit of 650 MPa. At both temperatures, samples crack-healed under cyclic stress exhibited a high fatigue limit of 650 MPa. Thus, it can be concluded that this Si_3N_4 developed by the authors also exhibits excellent in situ crack-healing ability even under cyclic stress. Finally, from Figs. 13 and 14, the following two conclusions can be drawn; (a) these mullite/SiC and Si_3N_4 materials have an ability to heal a crack even in service conditions, and (b) these samples exhibit excellent fatigue strength at the temperature of healing.

5. Proof test theory and temperature dependence of minimum fracture stress

As mentioned before, oxygen is necessary for the crack-healing, thus embedded crack cannot be healed. Consequently, proof test is very important for the higher reliability. This proof test should be done before service as shown in Fig. 1.

Unexpectedly, engineering ceramics exhibit non-linear fracture behavior,[28–30] so a new theory related to proof testing and based on non-linear fracture mechanics was proposed.[21] Moreover, ceramic components are not used just at the temperature proof-tested, so a theory to explain the temperature dependence of proof stress based on non-linear fracture mechanics was also proposed.[21] The theory gives the retained maximum effective crack size a_{em}^{R} as in Eq. (2), if proof test was made at room temperature.[21]

$$a_{\mathrm{em}}^{R} = \frac{\pi}{8}\left(\frac{K_{1C}^{R}}{\sigma_0^{R}}\right)^2 \left\{\sec\left(\frac{\pi\sigma_P^{R}}{2\sigma_0^{R}}\right) - 1\right\}^{-1} \tag{2}$$

where K_{1C}^{R}, σ_0^{R} and σ_P^{R} are K_{1C}, the fracture stress of the smooth sample and the proof test stress at room temperature, respectively. Thus, the minimum guaranteed fracture stress (σ_{mf}^{T}) at temperature T was given by the following Eq. (3).[21]

$$\sigma_{\mathrm{mf}}^{T} = \frac{2\sigma_0^{T}}{\pi}\arccos\left[\left\{\left(\frac{K_{1C}^{T}}{K_{1C}^{R}}\right)^2 \left(\frac{\sigma_0^{R}}{\sigma_0^{T}}\right)^2 \left(\sec\frac{\pi\sigma_P^{R}}{2\sigma_0^{R}} - 1\right) + 1\right\}^{-1}\right] \tag{3}$$

where K_{1C}^{T} and σ_0^{T} are $K_{1\mathrm{C}}$ and the fracture stress of the smooth sample at the temperature (T), respectively. The validity of this equation was proved using about 200 samples.[21]

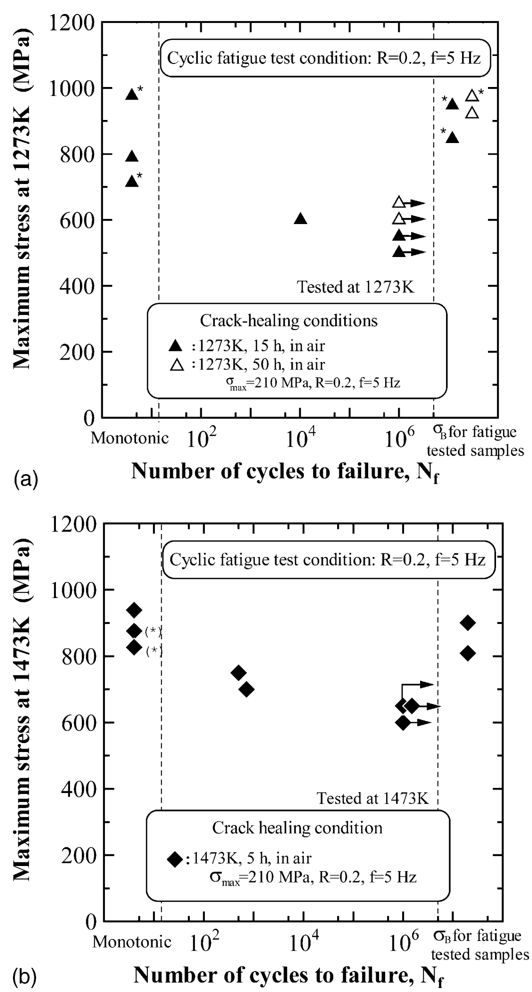

Fig. 14. Cyclic fatigue strength of crack-healed Si_3N_4 at the crack-healing temperature of 1273 and 1473 K. Data marked with an asterisk indicate that fracture occurred outside of the crack-healed zone.

6. Conclusion

A new methodology to guarantee the structural integrity of ceramic components which may be called "[crack-healing + proof test + in situ crack-healing]" was proposed and the flow chart was shown. During machining, many surface cracks may be induced in ceramic components. By the crack-healing under the optimized condition, the surface cracks can be healed completely and strength recovered completely. However, oxygen is necessary for the crack-healing, thus embedded cracks cannot be healed at all. Proof test is very useful to reject the member that has unacceptable flaws. Thus, the structural integrity of a ceramics component before service can be guaranteed by [crack-healing + proof test]. However, if a crack initiates during service, the reliability of the component will decrease considerably depending on the crack size. If the materials used have excellent crack-healing ability during service (namely; in situ crack-healing ability), this problem will be overcome easily. Then a new concept [crack-healing + proof test + in situ crack-healing] is a very useful technology to guarantee the structural integrity of a ceramic component over all its lifetime, if the material used has large crack-healing ability.

Acknowledgements

This study was supported by a Grant-in-Aid for scientific research from the Japan Ministry of Education, Basic Research Category (B) (2), No. 15360047 for the term from 2003 to 2004.

References

1. Ando, K., Chu, M. C., Sato, S., Yao, F. and Kobayashi, Y., The study on crack healing behavior of silicon nitride ceramics. *Jpn. Soc. Mech. Eng.* 1998, **64-623A**, 1936–1942 (in Japanese).
2. Lange, J. J. and Gupta, T. K., Crack-healing by heat treatment. *J. Am. Ceram. Soc.* 1970, **53**, 54–55.
3. Lange, J. J. and Radford, K. C., Healing of surface cracks in polycrystalline Al_2O_3. *J. Am. Ceram. Soc.* 1970, **53**, 420–421.

4. Petrovic, J. J. and Jacobson, L. A., Controlled surface flaws in hot-pressed SiC. *J. Am. Ceram. Soc.* 1976, **59**, 34–37.
5. Gupta, T. K., Crack-healing and strengthening of thermally shocked alumina. *J. Am. Ceram. Soc.* 1976, **59**, 259–262.
6. Choi, S. R. and Tikara, V., Crack-healing behavior of hot-pressed silicon nitride due to oxidation. *Scr. Metall. Mater.* 1992, **26**, 1263–1268.
7. Ando, K., Ikeda, T., Sato, S., Yao, F. and Kobayashi, Y., A preliminary study on crack healing behavior of Si_3N_4/SiC composite ceramics. *Fatigue Fract. Eng. Mater. Struct.* 1998, **21**, 119–122.
8. Chu, M. C., Sato, S., Kobayashi, Y. and Ando, K., Study on strengthening of mullite by dispersion of carbide ceramics particles. *Jpn. Soc. Mech. Eng.* 1994, **60-580A**, 2829–2834 (in Japanese).
9. Chu, M. C., Sato, S., Kobayashi, Y. and Ando, K., Damage healing and strengthening behavior in intelligent mullite/SiC ceramics. *Fatigue Fract. Eng. Mater. Struct.* 1995, **18-9**, 1019–1029.
10. Ando, K., Furusawa, K., Takahashi, K., Chu, M. C. and Sato, S., Crack-healing behavior of structural ceramics under constant and cyclic stress at elevated temperature. *J. Ceram. Soc. Jpn.* 2002, **110**, 741–747 (in Japanese).
11. Takahashi, K., Yokouchi, M., Lee, S. K. and Ando, K., *Crack-healing behavior of* Al_3O_3 *toughened by SiC whiskers J. Am. Ceram. Soc.* 2003, **86**(12), 2143–2147.
12. Kim, Y. W., Ando, K. and Chu, M. C., Crack-healing behavior of liquid-phase sintered silicon carbide ceramics. *J. Am. Ceram. Soc.* 2003, **86**, 465–470.
13. Korous, Y., Chu, M. C., Nakatani, M. and Ando, K., Crack healing behavior of SiC ceramics. *J. Am. Ceram. Soc.* 2000, **83**, 2788–2792.
14. Ando, K., Furusawa, K., Chu, M. C., Hanagata, T., Tuji, K. and Sato, S., Crack-healing behavior under stress of mullite/SiC ceramics and the resultant fatigue strength. *J. Am. Ceram. Soc.* 2001, **84**(9), 2073–2078.
15. Ando, K., Tsuji, K., Ariga, M. and Sato, S., Fatigue strength properties of crack healed mullite/SiC composite ceramics. *J. Soc. Mater. Sci. Jpn.* 1999, **48-10**, 1173–1178 (in Japanese).
16. Ando, K., Chu, M. C. and Sato, S., Fatigue strength of crack-healed Si3N4/SiC composite ceramics. *Fatigue Fract. Eng. Mater. Struct.* 1999, **22**, 897–903.
17. Yao, F., Ando, K., Chu, M. C. and Sato, S., Crack-healing behavior, high-temperature and fatigue strength of SiC-reinforced silicon nitride composite. *J. Mater. Sci. Lett.* 2000, **12**(19), 1081–1084.
18. Yao, F., Ando, K., Chu, M. C. and Sato, S., Static and cyclic behaviour of crack-healed Si_3N_4/SiC composite ceramics. *J. Eur. Ceram. Soc.* 2001, **21**, 991–997.
19. Ando, K., Houjyou, K., Chu, M. C., Takeshita, S., Takahashi, K., Sakamoto, S. *et al.*, Crack-healing behavior of Si_3N_4/SiC ceramics under stress and fatigue strength at the healed temperature(1000 °C). *J. Eur. Ceram. Soc.* 2002, **22**, 1339–1346.
20. Ando, K., Takahashi, K., Nakayama, S. and Saito, S., Crack-healing behavior of Si_3N_4/SiC ceramics under cyclic stress and resultant fatigue strength at the healing temperature. *J. Am. Ceram. Soc.* 2002, **85**, 2268–2272.
21. Ando, K., Shirai, Y., Nakatani, M., Kobayashi, Y. and Sato, S., [Crack-healing + Proof test]: a new methodology to guarantee the structural integrity of a ceramics component. *J. Eur. Ceram. Soc.* 2002, **22**, 121–128.
22. Wiederhorn, S. M. and Tighe, N. J., Proof-testing of hot-pressed silicon nitride. *J. Mater. Sci.* 1978, **13**, 1781–1793.
23. Ritter, Jr. J.E., Oates, P. B., Fuller, Jr. E.R. and Wiederhorn, S. M., Proof testing of ceramics (Part 1. Experiment). *J. Mater. Sci.* 1980, **15**, 2275–2281.
24. Ritter, Jr. J.E., Oates, P. B., Fuller, Jr. E.R. and Wiederhorn, S. M., Proof testing of ceramics (Part 2. Theory). *J. Mater. Sci.* 1980, **15**, 2282–2295.
25. Hoshide, T., Sato, T. and Inoue, T., Fatigue properties of ceramics after proof testing (1st report theoretical analysis). *Jpn. Soc. Mech. Eng.* 1990, **56A**, 212–218 (in Japanese).
26. Hoshide, T., Sato, T., Ohara, T. and Inoue, T., Fatigue properties of ceramics after proof testing (2nd report experimental study). *Jpn. Soc. Mech. Eng.* 1990, **56A**, 220–223 (in Japanese).
27. Ando, K., Sato, S., Sone, S. and Kobayashi, Y., Probabilistic study on fatigue life of proof tested ceramics spring. In *Fracture from Defects (ECF-12)*, ed. M. W. Brown, E. R. de los Rios and K. J. Miller. EMAS Publishing, 1998, pp. 569–574.
28. Kimoto, H., Usami, S. and Miyata, H., Relationship between strength and flaw size in glass and polycrystalline ceramics. *Jpn. Soc. Mech. Eng.* 1985, **51-471**, 2482–2488 (in Japanese).
29. Ando, K., Kim, B. A., Iwasa, M. and Ogura, N., Process zone size failure criterion and probabilistic fracture assessment curves for ceramics. *Fatigue Fract. Eng. Mater. Struct.* 1992, **15**(2), 139–149.
30. Ando, K., Iwasa, M., Kim, B. A., Chu, M. C. and Sato, S., Effect of crack length, notch root radius and grain size on fracture toughness of fine ceramics. *Fatigue Fract. Eng. Mater. Struct.* 1993, **16**, 995–1006.
31. Japan Standard Association, Testing method for flexural strength (modulus of rupture) of fine ceramics. *JIS* 1993, R1601.
32. Furusawa, K., *In Situ Crack-Healing Behavior of Mullite/SiC and Resultant Strength at the Temperature of Healing*. Doctor thesis presented to Yokohama National University, December 2002.
33. Houjyou, K., Ando, K., Liu, S. P. and Sato, S., Crack-healing and oxidation behavior of silicon nitride ceramics. *J. Eur. Ceram. Soc.*, Accepted for publication.
34. Lee, S. K., Ishida, W. and Ando, K., Strength properties of crack-healed silicon carbide. *J. Soc. Mater. Sci. Jpn.*. 2003, **52**(6), 674–680 (in Japanese).
35. Kim, B. S., Ando, K., Chu, M. C. and Saito, S., Crack-healing behavior of monolithic alumina and strength of crack-healed member. *J. Soc. Mater. Sci. Jpn.* 2003, **52**(6), 667–673 (in Japanese).
36. Ando, K., Chu, M. C., Tsuji, K., Hirasawa, T., Kobayashi, Y. and Sato, S., Crack healing behavior and high-temperature strength of mullite/SiC composite ceramics. *J. Eur. Ceram. Soc.* 2002, **22**, 1313–1319.