A NEW METHODOLOGY TO GUARANTEE THE STRUCTURAL INTEGRITY OF CERAMIC COMPONENTS USING IN-SITU CRACK-HEALING ABILITY

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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: 1573K, 1 h, in air, and the healed zone exhibited the same strength as the base material. Using this good healing ability, a new methodology to guarantee the reliability of ceramic components "crack-healing + proof test" was proposed. However, if a crack initiated during service, reliability would be severely impaired. Therefore, if a material can crack-heal 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 was discussed using the test results in terms of crack-healing behavior and proof test theory by the authors.

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 following methods can overcome these problems ; (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 were used on structural components for engineering use, considerable advantages can be anticipated. With this motivation, the authors developed Si_3N_4, [1] mullite, [2] alumina[3],[4] and SiC with very strong crack-healing abilities.[5] 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, [2],[3],[5] (b) the maximum crack size which can be healed completely, [6] (c) the high temperature strength of the crack-healed member, [4]-[6] (d) the cyclic and static fatigue strength of the crack-healed member at elevated temperature, [6]-[11] (e) a new methodology to guarantee structural integrity of the ceramic component using the crack-healing ability. [6][10][11]

Systematic studies were made on the above subjects by the authors. As a result, in the case of most ceramics above, the crack-healed zone 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. 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. [1],[2] 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. [1]-[4] These facts suggest the importance of a proof test to ensure higher reliability. [12]-[15]

There is much useful research on proof tests for ceramic components [13]-[15] based on linear fracture mechanics, and on probabilistic fatigue S-N curves that can be guaranteed by the proof test.[15] However, engineering ceramics exhibit non-linear fracture behavior, 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.[16],[17] From the above points of view, a new method of "crack-healing + proof test" [12] was proposed, recently.

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 [3]; (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 1073K to 1473K [10],[11] and from 1273K to 1473K, respectively. (2) the healed sample exhibited almost the same mechanical properties as the base material at the temperature of healing. [4],[6],[10] Namely, it can be said that both ceramics have excellent in-situ crack-healing ability.

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-servise) crack-healing. By machining, many surface cracks will be induced and reliability will be decreased considerably. However, by crack-healing under optimized conditions, surface cracks can be healed completely and reliability will be increased. 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 and the usefulness was verified 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}, the total samples used were about 350). [12] 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 be decreased considerably depending on the crack size. However, if a material can crack-heal during 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.

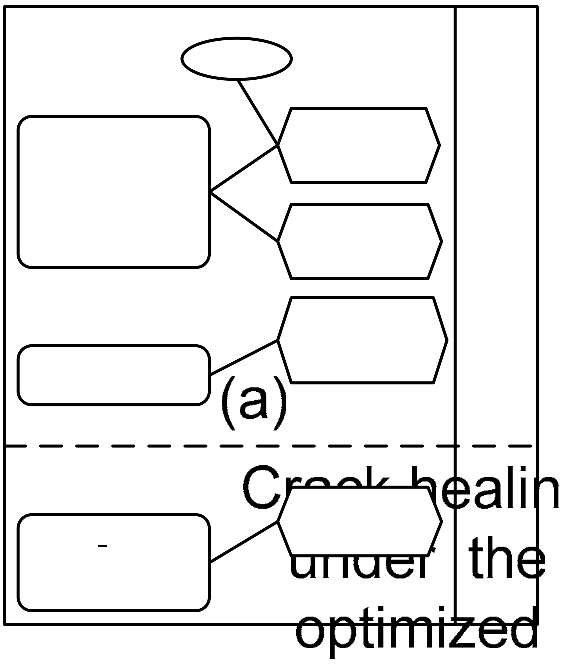

Fig. 1. Flow chart of a new methodology to guarantee the structural integrity of ceramic components using in-situ crack-healing ability.

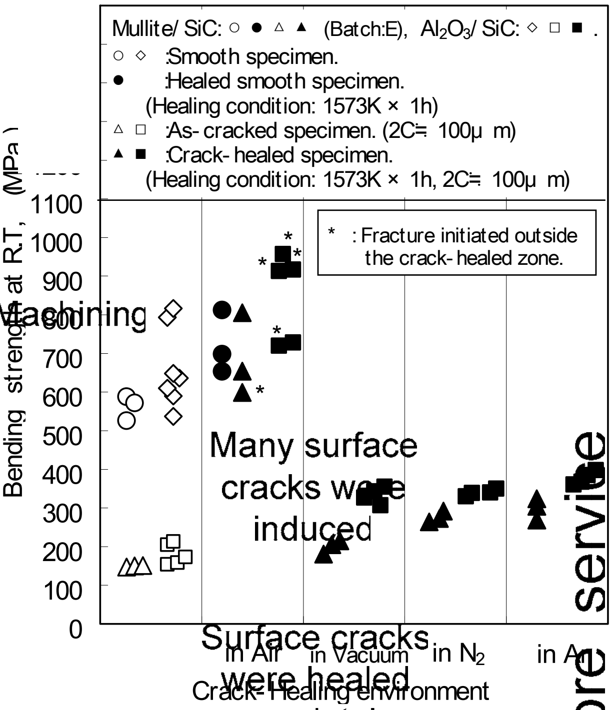

Fig.2 Effect of environment on the crack-healing behaviour of mullite/SiC and alumina/SiC.

3. Basic crack-healing behavior of structural ceramics

3.1. Effect of environment on the crack-healing behavior

The specimens used in this paper were made according to JIS standard. The sizes of bending specimen were 3×4×40mm and 3×4×22mm. The crack was induced by an indentation technique using a Vickers indenter. A semi-elliptical surface crack of 75μm or 100μm in diameter (aspect ratio ≒ 0.9) was introduced on the specimen. The crack of surface crack length 2C≈100μm was defined as the standard crack.

Figure 2 shows the effect of environment on the crack-healing behavior of mullite/SiC and Al_2O_3/SiC. [3] The contrast between bending strength (σ_B) of smooth and cracked samples was shown by the left most column of Fig.2. In these tests, the following three types of fracture were observed; Fracture pattern (a), crack initiation from a pre-crack. This type fracture usually occurred when crack-healing was incomplete. Fracture pattern (b), crack initiation from base material and a crack-healed zone which did not fracture. Fracture pattern (c), the sample fractured into many pieces and 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 sample fractured outside the crack-healed zone. All samples of both ceramics healed in air recovered σ_B completely, and showed that the cracks 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. 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. [1] 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. The test results are shown in Fig.3. The bending strength σ_B of smooth (○) and cracked (△) specimens are compared in the left-most column. The symbol (*) indicates that fracture occurred from outside the crack-healed zone, as mentioned before in fracture pattern (b). The symbol (■) indicates the σ_B obtained by healing time t_H = 1 hour at each healing temperatures. Note that σ_B does not recover below T_H=1223K, but it recovers considerably at T_H=1373K and 1473K. However, when considering that many fractures occurred from a pre-crack, as shown in fracture pattern (a), the strength recovery is not sufficient. On the other hand, at T_H=1573K, the average σ_B of the healed specimen is higher than that of the smooth specimen. In conclusion, the lowest crack-healable temperature for t_{HM} = 1 hour is T_{HL} = 1573K. In the same way, the lowest crack-healable temperature conditions for t_{HM}= 10 hours (□) and t_{HM} = 100 hours (◇) are T_{HL}= 1473K and T_{HL} = 1373K, respectively.

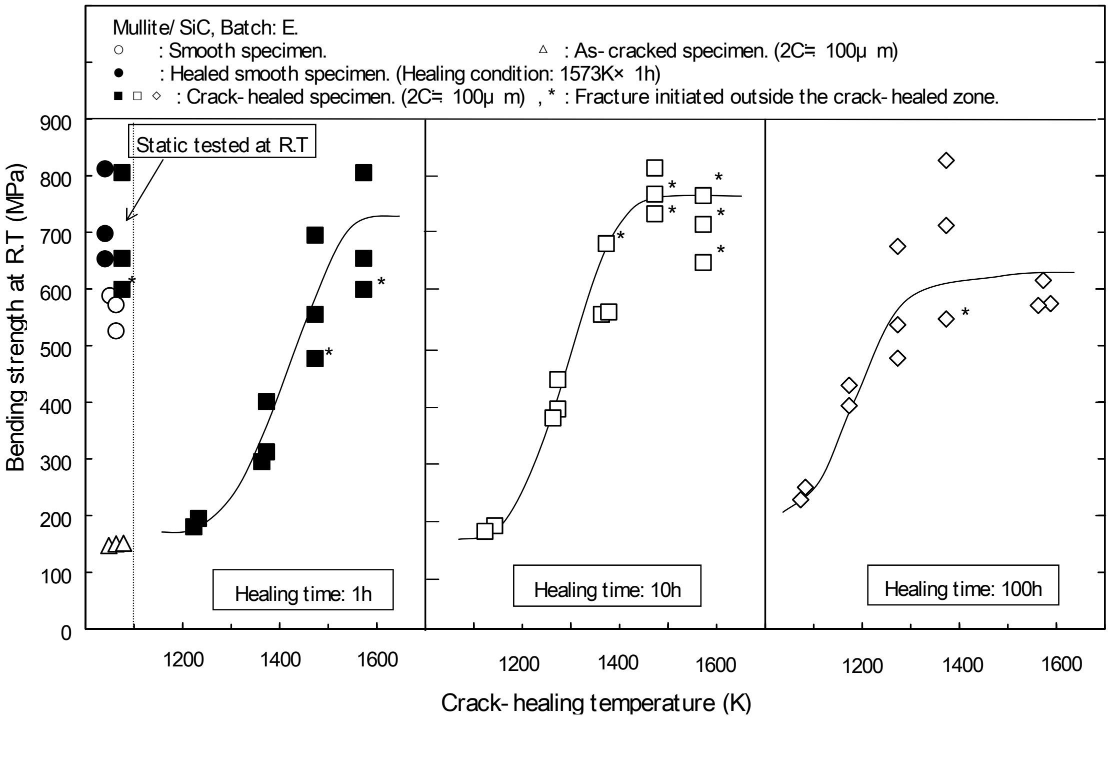

Fig.3 Effect of temperature and time on the crack-healing behavior of mullite/SiC.

3.3 Bending strength of the crack-healed sample at elevated temperature

For the practical use of 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 was shown in Fig.4. Monolithic Al_2O_3 was healed at 1723K, 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 1573K and numerous samples fracture outside the crack-healed zone. Mullite/SiC [18] and Al_2O_3/SiC [3] were healed at 1573K, after 1 h in air. Crack-healed mullite/SiC and Al_2O_3/SiC showed high heat resistance up to 1473K and 1573K, respectively and most samples fracture outside the crack-healed zone up to 1573K. The SiC was healed at 1773K, after 1 h in air. The base material showed a high σ_B up to 1673K, however, heat-proof temperature of the crack-healed sample was about 873K and considerably lower than the base material. Recently, SiC having a heat-proof temperature of 1473K of the crack-healed zone has been developed.[5] The crack-healed zone of SNC-Y5A3 is a glassy phase, so its heat-proof temperature is moderate, being about 1273K, however, the crack-healed zone of SNC-Y8 healed at 1573K, after 1 h in air is crystalline SiO_2, thus the healed zone showed a higher heat-proof temperature of 1673K.

3.4 Crack-healing behavior under constant and cyclic stress

The crack-healing behavior under constant or cyclic stress was investigated systematically. Firstly stress was applied to the sample to prevent unexpected crack-healing under no stress condition. Subsequently power was supplied to increase the furnace temperature at a rate of 10K/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.

Figure 5 shows the crack-healing behavior of mullite/SiC at 1273K as a function of healing time.[3] 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 450MPa. The symbol □ shows the σ_B of crack healed sample under constant stress of 88MPa. For this case, about 50% of the samples were fractured during heating up. The σ_B of the survived samples increased with increasing healing time and exhibited about 600MPa at 80 h healing time. This σ_B is a little higher value than that of sample healed under no stress condition.

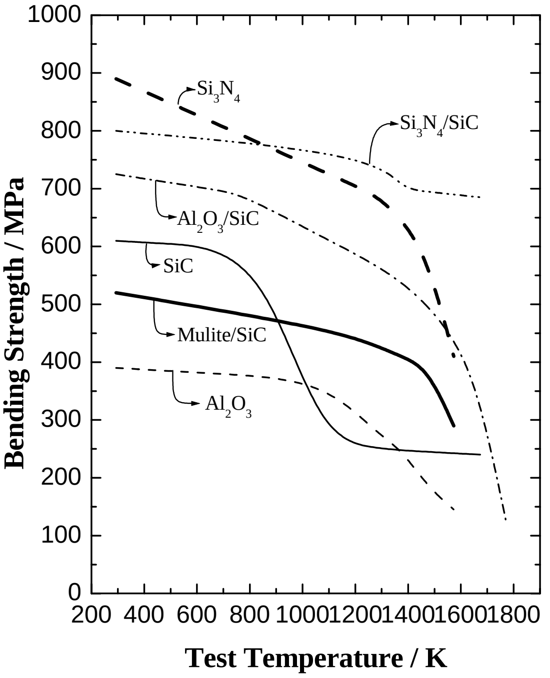

Fig.4 Effect of testing temperature on bending strength of crack-healed sample.

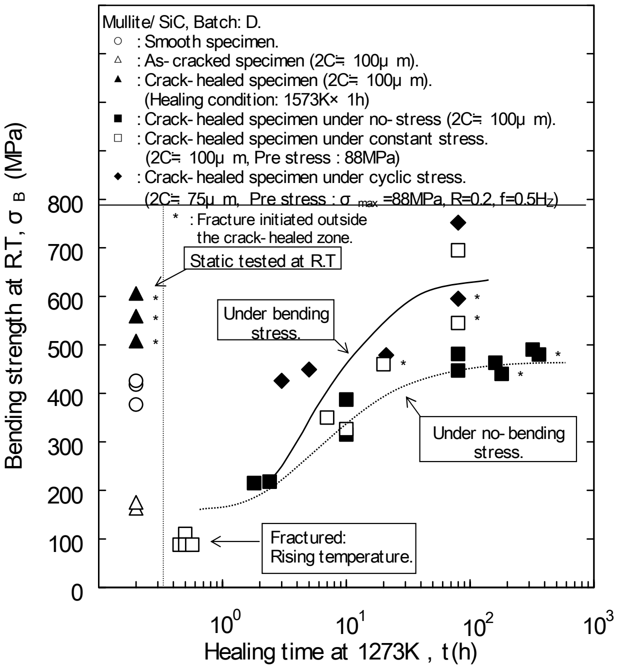

Fig.5 Effect of constant and cyclic stress and crack-healing time on crack-healing behavior and subsequent bending strength.

The symbol ◆ show the σ_B of crack-healed sample under cyclic stress. For this case, pre-crack size was reduced to 75μm, thus no sample fractured during healing and exhibited high level σ_B (≒600MPa) at the 80 h healing time. These test results show that mullite/SiC is able to heal a crack even under stress at 1273K.

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

Figure 6 shows the σ_B of crack-healed sample at the temperature of healing. 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 (1573K for 1 h in air). The symbols ■ and □ shows the σ_B of in-situ crack-healed sample under no-stress and cyclic stress condition, respectively. For example, the crack was healed at 1273K and the σ_B was also measured at 1273K. When looking first, all samples showed almost the same σ_B except a single sample that fractured from the base material and is shown by the symbol (▲*). This test results shows that the mullite/SiC developed by the authors exhibited excellent in-situ crack-healing ability. [3]

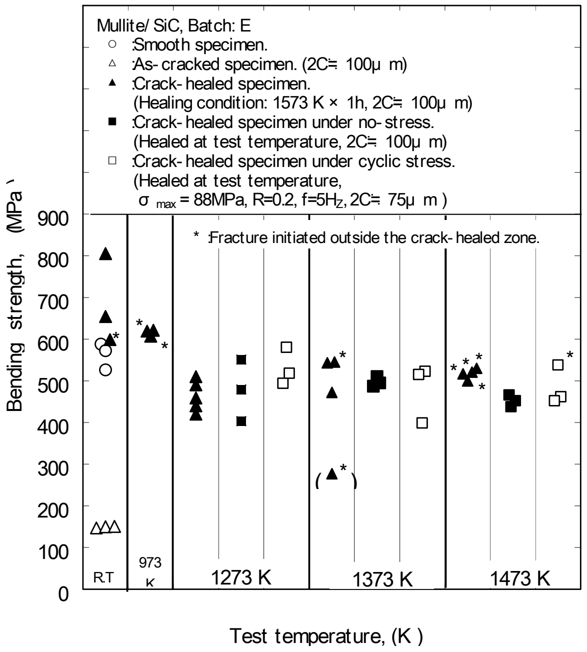

Fig.6 In-situ crack-healing behavior of mullite/SiC at 1273K, 1373K and 1473K.

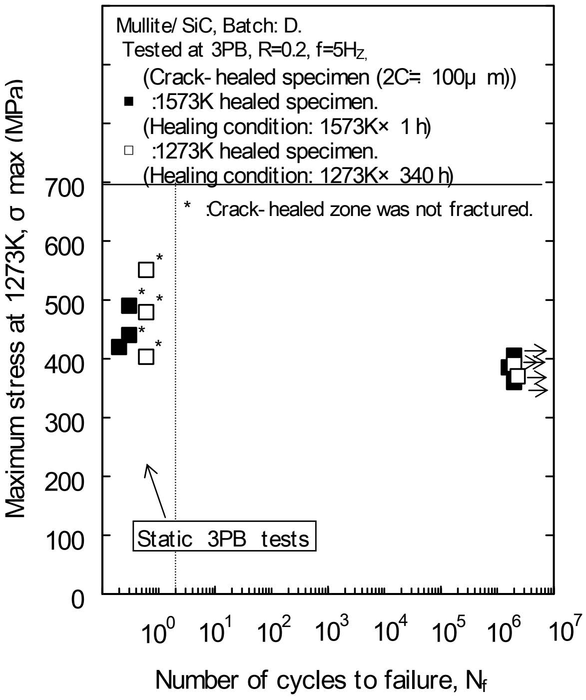

Fig.7 Effect of crack-healing temperature on the fatigue strength of mullite/SiC.

Figure 7 shows the cyclic fatigue strength of mullite/SiC at 1273K. The symbols (■) and (□) indicate that the standard cracks of mullite/SiC were healed at 1573K, 1 h, and 1273K, 340 h, respectively. For this test, Batch D was used, and samples showed a little lower strength. Both samples showed the same fatigue limit (400MPa). From this test results, it can be concluded that the mullite/SiC exhibited excellent in-situ crack-healing ability even for fatigue strength. Studies related to crack-healing behavior under 5Hz cyclic stress and resultant fatigue strength at the temperature of the healing were made systematically.[10][11]

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

There is much useful research on a proof test for ceramic components [13]-[15] based on linear fracture mechanics, and on probabilistic fatigue S-N curves that can be guaranteed by proof test. [15] However, engineering ceramics exhibit non-linear fracture behavior, [16],[17] so a new theory related to proof testing and based on non-linear fracture mechanics was proposed. 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. The theory gives the retained maximum effective crack size a_{em}^R as in equation (1), if proof test was made at room temperature. [12]

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

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 equation (2). [12]

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

Where, K_{1C}^T and σ_0^T are K_{1C} and the fracture stress of the smooth sample at the temperature (T), respectively. The validity of this equation was proved using about 200 samples. [12] Finally, it was shown that “crack-healing + proof test + in-situ crack-healing” is a very useful technology to guarantee the static fatigue limit of Si_3N_4/SiC at 1273K~1673K.[19]

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.

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