

# Effect of Self-Healing on Fatigue Behaviour of Structural Ceramics and Influence Factors on Fatigue Strength of Healed Ceramics

Wataru Nakao  
*Yokohama National University*  
*Japan*

## 1. Introduction

Silicon carbide particles embedded in ceramic matrices give rise to self-healing function in the structural ceramics operated at high temperature in air. This feature is taken advantage of to enhance life time of the ceramic components with high mechanical reliability.

Ceramics are well-known to tend to have brittle fracture that usually occurs in a rapid and catastrophic manner. Brittle fracture is usually caused by the stress concentration at the tip of the flaws. For brittle fracture under pure mode I loading, under which crack is subjected to opening, the fracture criterion is that the stress intensity factor,  $K_I$ , is equal to the fracture toughness,  $K_{IC}$ . Since the value of  $K_I$  is determined from the flaw size and the geometry between flaw and loading, one can understand that the fracture strength of ceramic components is not an intrinsic strength but is determined from fracture toughness and flaw size. Especially surface cracks are the most severe flaws because surface cracks lead to the highest stress concentration. If surface cracks are introduced during service, e.g., crash or thermal shock, the strength of ceramic components decreases significantly. The behaviour leads to low mechanical reliability of ceramic components.

The ceramic composites containing silicon carbide particles can heal surface cracks by themselves, as shown in Fig. 1 (Nakao et al., 2010, Ando et al., 2004). Surface cracking allows the silicon carbide particles on the crack walls to contact the oxygen in the surrounding atmosphere. If the components operated at high temperature, the contact would cause the oxidation of silicon carbide. The oxidation includes almost two times volume expansion of the condensed phases and the huge exothermic heat. Due to the volume expansion, the space between the crack walls can be completely filled with the formed oxide. Furthermore, the reaction heat leads to strong bonding at the interface between the matrix and the formed oxide. As a result, the self-healing induced by the oxidation of the embedded silicon carbide particles can recover the degraded strength completely and can enhance the life time of the ceramic components with high mechanical reliability.

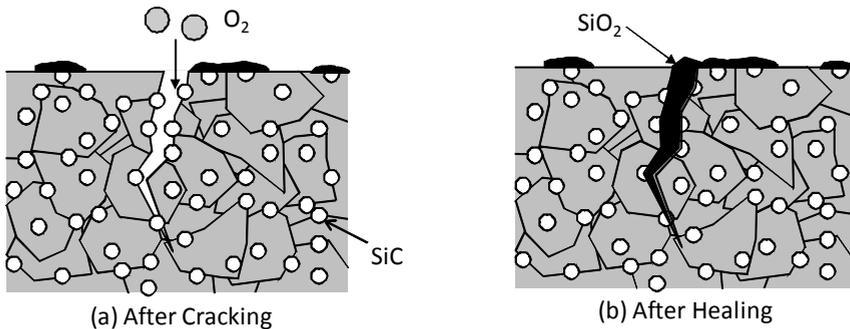


Fig. 1. Schematic diagram of the self crack-healing mechanism

To discuss the life time of the ceramic components, it is also important to know the fatigue behaviour that caused the crack growth when the stress intensity factor is lower value of  $K_{IC}$ . The mechanism has been analyzed to describe the slow crack growth behaviour including chemical reaction kinetics, and it is called as stress corrosion cracking (SCC). Figure 2 shows the typical example of SCC, in which Si-O bonds in silica glass are de-bonded by the SCC with the moisture in the surrounding atmosphere. At the stressed crack tip, the accumulated elastic energy activates the Si-O bond, thereby enhancing the hydrolysis of the bond. As a result, surface cracks propagate with the progression of the hydrolysis. This suggests that the fatigue degradation of the structural ceramics has also been generated by the presence of surface cracks, which not only leads to the highest stress concentration but it is also able to react with the reactant in the surrounding atmosphere. Thus, one can understand fatigue strength and life time are also determined by the size of surface cracks.

This chapter will introduce the effect of self crack-healing on fatigue strength in structural ceramics. As mentioned above, fatigue strength is also significantly influenced by the presence of surface cracks. Therefore, self-healing of surface cracks gives large advantage to the fatigue strength in structural ceramics, and the knowledge of the effects contributes to the realization of a long life time with high strength integrity of ceramic components.

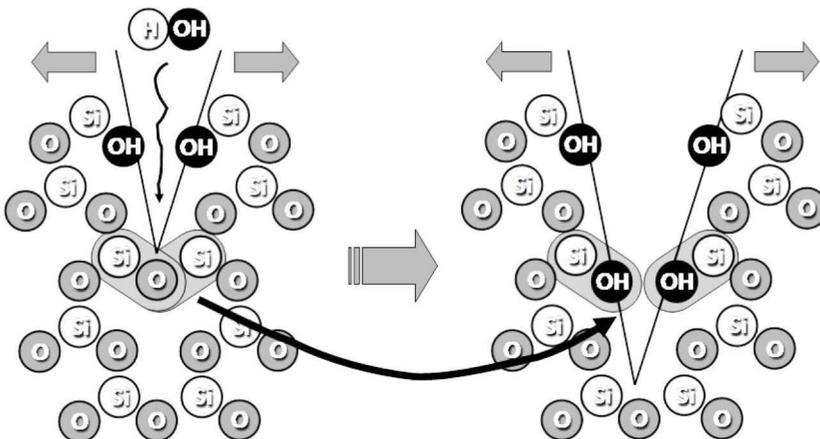


Fig. 2. Schematic illustration of the mechanism of stress corrosion cracking

## 2. Fatigue Behaviour of Crack-Healed Surface

### 2.1 Effect of Self Crack-Healing on Dynamic Fatigue Behaviour

The fatigue strength enhancement by the self crack-healing has been clearly found in dynamic fatigue behaviour, as shown in Fig. 3 (Nakao et al., 2006).

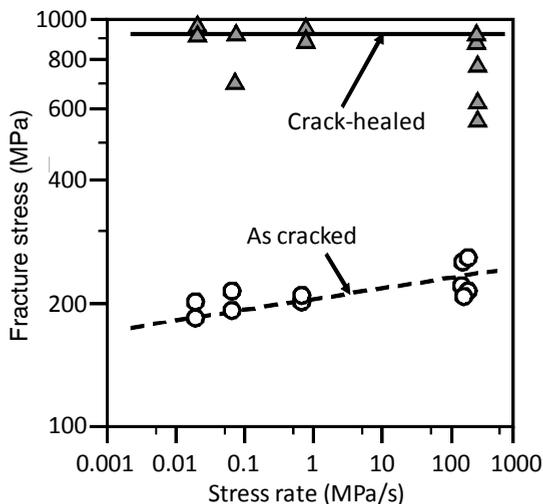


Fig. 3. Dynamic fatigue results of the crack-healed mullite containing 15 vol% SiC whiskers and 10 vol% SiC particles composite with that of the composite having a semi-elliptical crack of 100  $\mu$ m in surface length

Dynamic fatigue behaviour can be obtained from the fracture strength as a function of the applied stress rate. If the specimen exhibits the slow crack growth due to SCC, lower stress rate allows the surface cracks to progress larger by the applied stress until the fracture, thereby giving lower fracture strength. Thus, demonstrating the logarithmic plot of the fracture strength versus the stress rate, one can find the positive slope in the materials exhibiting the SCC crack propagation. The gradient of the slope implies the indicator of the fatigue sensitivity.

Figure 3 shows the dynamic fatigue results of the crack-healed mullite containing 15 vol% SiC whiskers and 10 vol% SiC particles composite (MS15W10P), which possesses the excellent self crack-healing ability (Nakao et al., 2006) and high crack growth resistance by SiC whiskers reinforcement. In order to test the self crack-healing effect, the specimen contained a semi-elliptical surface crack having surface length of 0.1 mm, which comes from the prolongation of the diagonal line of the indentation introduced by the Vickers indentation, and the indenter test, pre-crack was completely healed by the high temperature heat treatment (Nakao et al., 2006) at 1300 °C for 2 h in air. In comparison, Figure 3 also shows the dynamic fatigue behaviour of the as-cracked mullite based composite, i.e., the specimens were subjected to no healing treatment, thereby exhibiting the SCC crack growth.

The crack-healed MS15W10P sample shows a constant fracture strength over whole the stress rate, while the as-cracked MS15W10P exhibits the positive slope in the dynamic fatigue curve.

Furthermore, the fracture initiation of the crack-healed MS15W10P is not the healed pre-crack but the embedded flaws, e.g., the aggregation of SiC particles. The embedded flaws cannot be reacted with the moisture in the surrounding atmosphere. Then the flaws cannot propagate by the SCC crack growth. A similar behaviour was reported in the fatigue behaviour of the sintered alumina in toluene (Evans, 1972). Therefore, the result demonstrates clearly that the self crack-healing makes the fatigue sensitivity decrease significantly.

## 2.2 Effect of Surface Morphology on the Fatigue Strength of Self Crack-Healed Specimens

Surface morphology of the healed specimen was found to affect the fatigue strength in the situation when the continuous stress is applied for a long period. Here, the effect demonstrates the fatigue behaviour of three alumina- 30 vol% SiC composite having different SiC whiskers content (20% in this case). These composites possess excellent self crack-healing ability (Nakao et al., 2005).

Static fatigue testing, in which the constant stress is continuously applied, is well-known to be the most severe fatigue situation for alumina based ceramics, because SCC crack growth is mechanically enhanced by only the stress intensity factor at the crack tip, and not enhanced by the fluctuation of the applied stress. According to Japan Industrial Standard (JIS) R1632, the optimal test finish time is 100 h, and the maximum stress under which the specimen survived until the test finish time is determined as the static fatigue limit.

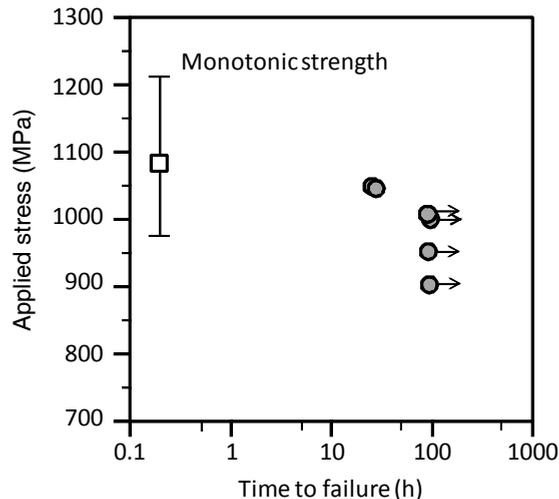


Fig. 4. Stress- time to failure diagram of the crack-healed alumina- 20 vol% SiC whiskers- 10 vol% SiC particles composite, with its monotonic strength

The stress-time to failure diagram of the crack-healed alumina- 20 vol% SiC whiskers and 10 vol% SiC particles composite (AS20W10P) is shown in Fig. 4 (Sugiyama et al., 2008). In order to test for the self crack-healing effect, the specimen contained the indentation pre-crack having surface length of 0.1 mm. The indentation pre-crack was completely healed by the high temperature heat treatment (Nakao et al., 2005) at 1300 °C for 5 h in air. For comparison,

the monotonic strength is also shown in Fig. 4 using open squares. Under the applied stress below 1000 MPa, all the specimens survived up to test finish time, while two specimens fractured after 30 h under 1050 MPa. Therefore, the static fatigue limit of the crack-healed AS20W10P has been determined to be 1000 MPa. The fatigue limit present in the distribution range of the monotonic strength. Furthermore, the fracture initiation was found not to be the healed pre-crack.

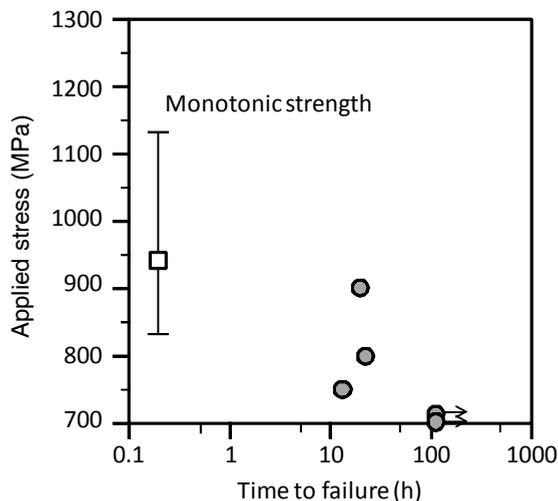


Fig. 5. Stress- time to failure diagram of the crack-healed alumina- 30 vol% SiC whiskers composite, with its monotonic strength

Alternatively, the static fatigue limit of the healed alumina-30 vol% SiC whiskers composite (AS30W) containing the healed pre-crack is less than the monotonic strength as shown in Fig.5. Also the healed pre-crack was found not to act as the fracture (fatigue) initiation in the healed AS30W, but large strength degradation due to static fatigue occurs from the tensile surface. The strength degradation results from that the surface morphology of the healed surface, which affects significantly the static fatigue behaviour.

The fatigue strength degradation has a relation to the surface morphology, such as surface roughness, as shown in Fig. 6. The progression of the SiC oxidation, inducing the self crack-healing, generated the island like formed oxide on the healed surface, as shown in Fig. 1. If coarse SiC particle, for example SiC whisker, exist on the surface, the surface roughness significantly increases with the progression of the oxidation. Thus, Sugiyama et al. (Sugiyama et al. 2009) concluded that the fatigue crack of the healed ceramics which has large surface roughness is initiated from the “valley” in the rough surface, which can generate too high a stress concentration to induce SCC reaction. Although the healed surface of alumina-30 vol% SiC particles composite (AS30P) has low surface roughness, the composite exhibits large fatigue strength degradation. From the SEM investigation, AS30P had the grain-pullout traces, which were about 3 μm in diameter, on the healed surface as

shown in Fig. 7. The pullouts were introduced during polishing and cannot be eliminated by crack healing. Therefore, the fatigue crack initiated from these traces.

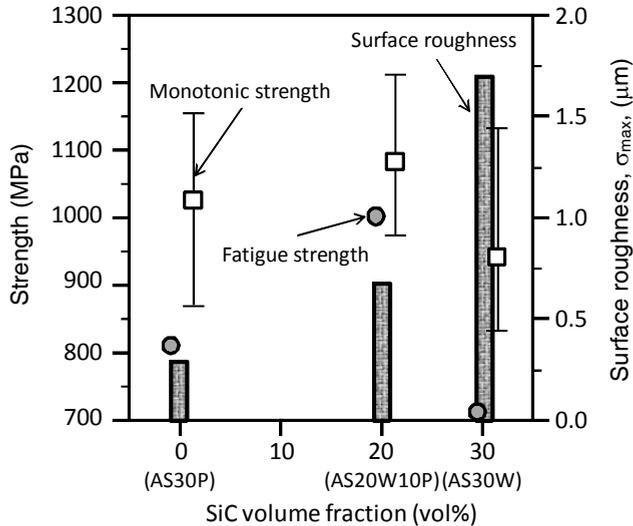


Fig. 6. The influence of SiC whiskers content on the surface roughness of the healed surface, monotonic strength and static fatigue limit

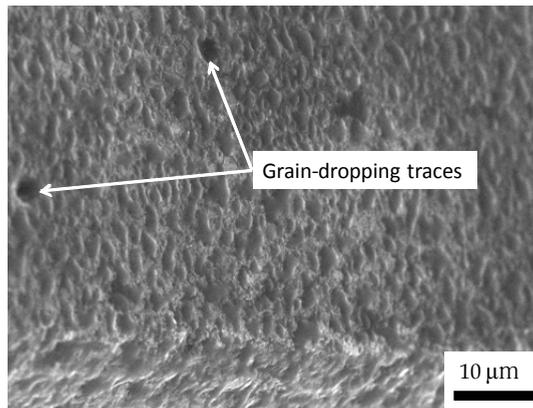


Fig. 7. The grain-pullout traces on the surface of the crack-healed alumina- 30 vol% SiC particles composite

From the above results, it is noted that if the design requires extreme low fatigue sensitivity, it is necessary not only to heal the whole surface cracks but also to manage the surface roughness of the healed surface.

### 3. Fatigue Behaviour with Self Crack-Healing

In high temperature fatigue, there is another interesting phenomenon, in order that self crack-healing occurs at the same time as fatigue damage. For example, figure 8 (Nakao et al., 2006) shows a logarithmic plot of life time in terms of the applied stress for the crack-healed mullite containing 15 vol.% SiC whiskers and 10 vol.% SiC particles composite at 1000 °C. In general, i.e. the slow crack growth is included, the life time increases as the applied stress decreases.

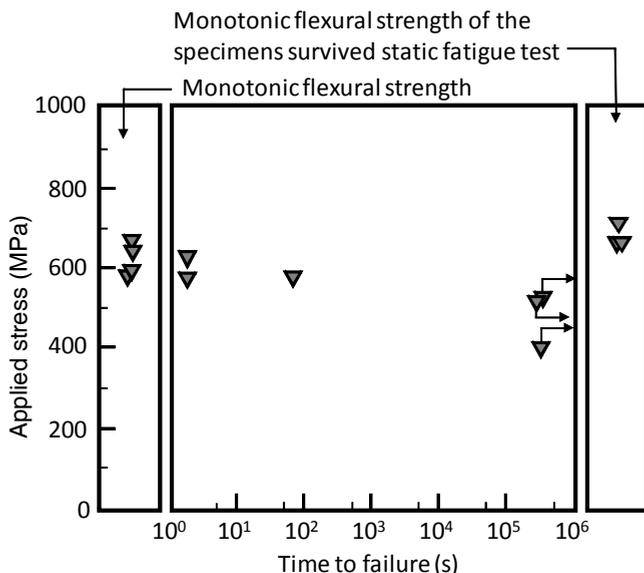


Fig. 8 . Logarithmic plot of life time in terms of the applied stress for the the crack-healed mullite containing 15 vol% SiC whiskers and 10 vol% SiC particles composite at 1000 °C

However, all crack-healed test specimens survived up to finish time of 100 h under static stresses of 50 MPa less than the lower bound of the monotonic strength at the same temperature. Alternatively, the specimens fractured at less than 100 s under stresses corresponding to the lower bound of the flexural strength. This failure is not fatigue but rather rapid fracture. Therefore, it is confirmed that the crack-healed composite is not degraded by the static fatigue at 1000 °C. The behaviour would result from the self crack-healing that occurs rapidly compared with the fatigue damage.

### 4. Summary

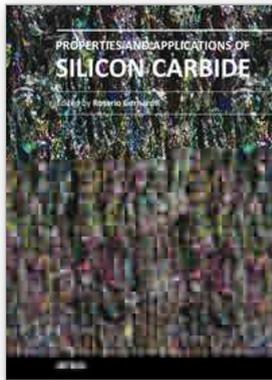
Self-healing induced by the high temperature oxidation of the dispersed silicon carbide particle can eliminate the surface cracks, leading to a great benefit to ensure the high monotonic strength of ceramics. Since most mechanical and structural components usually

work under applied continuous stresses, it is important to know the self-healing effect on fatigue behaviour.

The fatigue sensitivity, which corresponds to the strength degradation is caused by the continuous loading, has been decreased significantly in the healed ceramics, because the self-healing of surface cracks can prevent stress corrosion cracking from occurring in the original surface cracks. Furthermore, the improvement on the fatigue sensitivity has been affected by the surface roughness of the healed ceramics. Another attractive phenomenon has occurred in high temperature fatigue behaviour. This results from the fact that self-healing occurs at the same time as fatigue damage. As a result, the fatigue limit has been equal to the minimum monotonic strength at the same temperatures.

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