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Fatigue Strength of Crack-Healed Al_2O_3/SiCw at elevated temperature

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Alumina reinforced by SiC whiskers (Al_2O_3/SiCw) was sintered in order to investigate the fatigue strength of crack-healed specimens at high temperature. A semi-elliptical surface crack of 100μm in surface length was introduced on each specimen surface. These specimens were crack-healed at 1300°C for 1 h in air, and static and cyclic fatigue strengths were systematically investigated at room temperature, 900°C and 1100°C by three-point bending. The static and cyclic fatigue limits of the crack-healed specimens were more than 70% of the average bending strength at each testing temperature. Crack-healed specimens of Al_2O_3/SiCw were not sensitive to static and cyclic fatigues at room temperature and high temperature. Therefore, the combination of crack-healing and whiskers reinforcement can play an important role in increasing static and cyclic fatigue strengths at high temperature.
Key words: Alumina, SiC Whisker, Crack-Healing, Fatigue Strength, High Temperature

1. INTRODUCTION

Alumina is a very popular ceramics used in various fields. It retains high strength at elevated temperatures, and it has good corrosion and wear resistance. However, it has the weakness of low fracture toughness (~4MPa $m^{1/2}$), very sensitive to crack and low reliability. To overcome this problem, there are two ways: (a) toughening Al_2O_3 with fibers or whiskers, and (b) inducing a self-crack-healing ability. Many studies have been conducted with the aim of toughening Al_2O_3 by fibers of whiskers reinforcement and many useful results have been reported. [1-4] However, very few studies have been made of method (b). [5-8]

Some engineering ceramics have the ability to heal a crack. If this ability is used in structural components for engineering uses, great benefits can be anticipated, such as increased reliability of the structural ceramic components and reduced inspection, machining, and polishing costs of such components. However, in utility of this healing ability, many problems must be resolved.

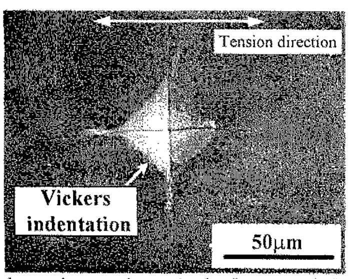
It has been shown that Si_3N_4/SiC [9,10] and mullite/SiC [11-13] have excellent crack-healing abilities. It was also reported that Al_2O_3 reinforced by SiC particles [8] had excellent crack-healing ability similar to Si_3N_4/SiC and mullite/SiC.

However, higher fracture toughness is desirable for the structural integrity of Al_2O_3 ceramics. It is well known that whisker reinforcement is very effective for increasing the fracture toughness of structural ceramics [1-3]. If whisker-reinforced Al_2O_3 could express excellent crack-healing ability, it would be desirable for the structural integrity of Al_2O_3 components. In a previous study [14], we sintered Al_2O_3 reinforced by SiC whiskers (Al_2O_3/SiCw) and studied basic crack-healing behaviors. The addition of SiC whisker is very useful for inducing self-crack-healing ability and increasing monotonic strength and fracture toughness.

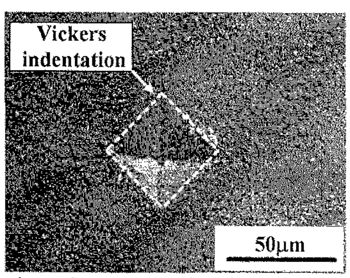
It is essential to determine fatigue behavior under the appropriate loading, such as static, dynamic, or cyclic loadings indispensable in the engineering application of ceramic materials for structural purposes. A considerable number of studies have been conducted with regard to the fatigue behavior of engineering ceramics. [15-20] There have been few critical studies have been conducted regarding static and cyclic fatigue strength of alumina ceramics at high temperatures, although activity in this field has recently been increasing. [15,20] In a previous study [14], we examined the high temperature strength of Al_2O_3/SiCw and found that the limit temperature for the bending strength of crack-healed specimen was 1200°C. However, the fatigue strength of the crack-healed specimen was not studied. This paper focuses on the fatigue behaviors of crack-healed Al_2O_3/SiCw composite ceramics at room and high temperatures.

2. MATERIAL, SPECIMEN AND TEST METHOD

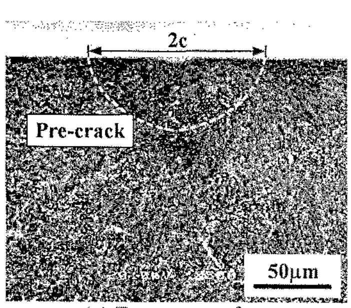
The alumina powder used in this investigation was AKP-20 (mean particle size = 0.4~0.6 μm, purity = 99.99 %) from Sumitomo Chemical. The SiC whiskers used were SCW #1-0.8 (length = 30~100 μm, diameter = 0.8~1.0 μm) from Tateho Chemical Industries. The quantity of SiC whiskers added was 20 vol% relative to Al_2O_3 powder. Isopropyl alcohol was added to the mixture and the mixture was blended completely for 12 h using alumina balls and mill pot. Thereafter, the mixture was placed in an evaporator to extract the solvent, and then in a vacuum desiccator to produce a dry powder mixture. Rectangular plates of 9mm×50 mm×50mm were hot pressed at 1850°C and 40 MPa for 1 h in an argon environment. The density of the test material measured by the Archimedes technique was 3.83 g/cm^3, which was 99.9% of the theoretical density of the material. The average grain size of Al_2O_3 was 1~2μm. The SiC whiskers were located in grain boundaries. The sintered plates were then cut into test specimens measuring 3mm×4mm×23mm. The tensile surface of the test specimen was mirror finished. A semi-elliptical surface crack was made at the center of the tensile surface of the test specimen using a Vickers indenter at a load of 19.6 N. By this method, a semi-elliptical crack of 100 μm in surface length was made. Figures 1(a) and (b) show SEM micrographs of a pre-crack introduced by means of the Vickers indentation method, before and after the crack-healing at 1300°C for 1h in air, respectively. The ratio of depth (a) to half surface length (c) of the crack (aspect ratio) was a/c ≒ 0.9, as shown in Fig. 1(c). Specimens were crack-healed at 1300°C for 1 h in air. The rising rate of the furnace temperature was 10°C/min. This is the optimized healing condition for a surface crack of 100 μm. The bending tests, static and cyclic fatigue tests were conducted using a three-point loading system with a span of 16 mm. The static and cyclic fatigue tests were performed in air at room temperature, and at elevated temperatures of 900°C and 1100°C. The applied stress at which a specimen did not fracture up to 10^6 sec was defined as the static fatigue limit. Cyclic fatigue tests were conducted at a stress ratio of R = 0.2 and using a sine wave with a frequency of 5Hz. The applied stress at which a specimen did not fracture up to $N_f = 2 \times 10^6$ cycles was defined as the cyclic fatigue limit. The surface cracks, before and after crack-healing, and the fracture surface were observed using a scanning electron microscope (SEM).



(a) Indentation and cracks before crack-healing



(b) Indentation and cracks after crack-healing



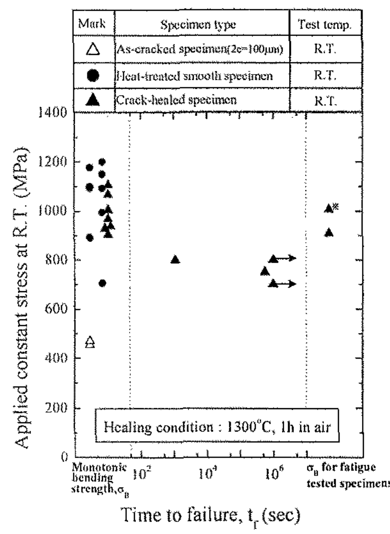
(c) Fracture surface

Fig. 1 SEM micrographs of Al_2O_3/SiCw

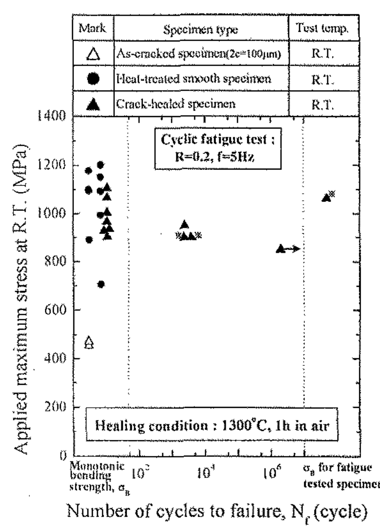
3. TEST RESULTS AND DISCUSSION

3.1 Fatigue strength behavior at room temperature

The results of the static and cyclic fatigue tests at room temperature are shown in Fig. 2. On the left-hand side of Figs. 2(a) and (b), the results of monotonic bending tests are also shown. The symbols ●, △, and ▲ show the bending strength of the smooth specimens heat-treated at 1300°C for 1 h in air, as-cracked, and crack-healed specimens, respectively. The bending strength (σ_B) of the heat-treated smooth specimens scattered to a certain extent, giving a mean fracture stress of about 1000MPa. Due to pre-cracking of 100μm, the bending strength decreased down to ~ 450 MPa. However, the average value of σ_B for the specimen crack-healed at 1300°C, 1h in air is about 1000 MPa.



(a) Static fatigue test results of crack-healed Al_2O_3/SiCw



(b) Cyclic fatigue test results of crack-healed Al_2O_3/SiCw

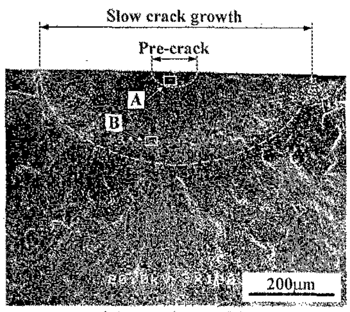
Fig. 2. Fatigue test results of Al_2O_3/SiCw at room Temperature

Thus, σ_B of the crack-healed specimens recovered to a level similar to that of the heat-treated smooth specimens.

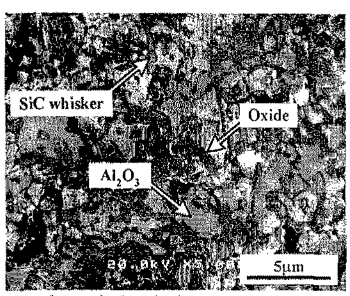
The results of the static fatigue tests are shown in Fig. 2(a) in correlation with applied constant stress and time to failure. The specimens that did not fracture up to 10^6 sec are marked by an arrow symbol (→). Two specimens survived under 700 MPa and 800 MPa up to 10^6 sec; however, one fractured under 750 MPa at 5.6×10^5 sec. Thus, we determined the static fatigue limit (σ_{t0}) for the crack-healed specimen to be about 750 MPa. The cyclic fatigue test results are shown in Fig. 3(b). The specimens that did not fracture up to $N_f = 2 \times 10^6$ cycles are marked by an arrow symbol (→). The cyclic fatigue limit (σ_{f0}) for the crack-healed specimen is about 850 MPa. The cyclic fatigue limit of crack-healed specimens is about 10 % higher than that of the static fatigue limit. These fatigue test results are reasonable considering that the cyclic fatigue limit is defined at a shorter time (4×10^5 sec) than is the static fatigue limit (10^6 sec). The ratio of static fatigue limit (σ_{t0}) and cyclic fatigue limit (σ_{f0}) to the average bending strength of crack-healed specimens (σ_B) is about 75 % and 85 %, respectively. Thus, fatigue limits of crack-healed specimens are quite high.

Bending tests for the specimens which survived the static and cyclic fatigue tests were also conducted at room temperature. The experimental results are shown on the right-hand sides of Fig. 2(a) and (b). The fatigue-tested specimens exhibited similar bending strengths to those of the monotonically tested specimens as shown on the left-hand side of Fig. 2(a) and (b). These experimental results indicate that significant crack growth from the crack-healed zone did not occur.

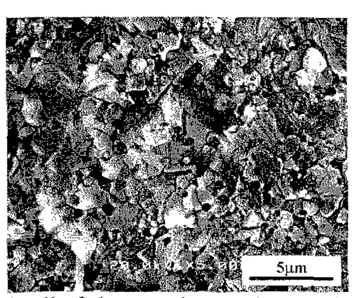
Figure 3 shows SEM micrographs of the fracture surface after static fatigue tests at room temperature. The slow crack growth can be clearly observed, as shown in Fig. 3(a). Figure 3(b) shows the details of the crack-healed zone. The dark regions near SiC whiskers are oxide products, the light regions are unoxidized Al_2O_3. Figure 3(c) shows the details of the slow crack growth zone. SiC whiskers and Al_2O_3 matrix are clearly observed. The fracture surface of the slow crack growth zone is quite rough and predominantly intergranular.



(a) crack profile



(b) detail of crack-healed zone shown by A in (a)



(c) detail of slow crack growth zone shown by B in (a)

Fig. 3. SEM micrographs of fracture surface of the test specimens fractured during static fatigue test at room temperature. (Slow crack growth occurred during the fatigue test, applied stress = 800MPa, t_f=1110 sec)

3.2 Static fatigue strength at high temperature

In a previous study [14], we investigated the high temperature bending strength of Al_2O_3/SiCw which was pre-cracked (2c = 100 μm) and crack-healed at 1300°C for 1 h in air. The heat-resistance limit temperature for bending strength of the crack-healed specimen was 1200°C. In the present study, the cyclic and static fatigue strengths of crack-healed specimens were examined at 900°C and 1100°C.

The results of static fatigue tests at high temperature are shown in Fig. 4. The monotonic bending strength of the crack-healed specimens at 900°C and 1100°C are shown on the left-hand side of Fig. 4. The average bending strength of the crack-healed specimens at 900°C and 1100°C are about 750 MPa and 650 MPa, respectively. The values of the static fatigue limit (σ_{t0}) for the crack-healed specimens at 900°C and 1100°C are about 550 MPa and 450 MPa, respectively. The static fatigue limit of the crack-healed specimen is about 70 % in comparison to the bending strength at each testing temperature. The bending strengths of the specimens that survived static fatigue tests were also investigated at each testing temperature, i.e., 900°C and 1100°C. the bending strengths are shown on the right-hand sides of Fig. 4. The crack-healed specimens that survived the static fatigue test showed bending strengths similar to monotonically tested specimens, indicating that significant crack growth from the crack-healed zone or internal defects did not occur.

3.3 Cyclic fatigue strength at high temperature

The cyclic fatigue strengths of the crack-healed specimen are also investigated at 900°C and 1100°C. The experimental results are shown in Fig. 5. The value of the cycle fatigue limit (σ_{f0}) decreases with increasing test temperature; about 700 MPa at 900°C and 500 MPa at 1100°C. The ratio of cyclic fatigue limit (σ_{f0}) to the bending strength of crack-healed specimens (σ_B) is 93 % at 900°C and 75 % at 1100°C, respectively. The bending strength of the specimens which survived the cyclic fatigue tests were also investigated. The bending strengths are shown on the right-hand sides of Fig. 5.

Bending tests were performed at each crack-healing temperature, i.e., 900°C and 1100°C. Again, the crack-healed specimens that survived the cyclic fatigue test showed bending strengths similar to monotonically tested specimens.

In a previous study[14], the addition of SiC whisker was very useful for increasing the bending strength and the fracture toughness of Al_2O_3. The results of this study show that it is very useful to increase static and cyclic fatigue strengths at high temperatures.

4. CONCLUSIONS

The main conclusions obtained are as follows:

1. The values of static and cyclic fatigue limits for the crack-healed specimens were 750 MPa and 850 MPa, respectively, at room temperature. The static and cyclic fatigue limits were about 75 % and 85 %, respectively, in comparison to the bending strength at room temperature.

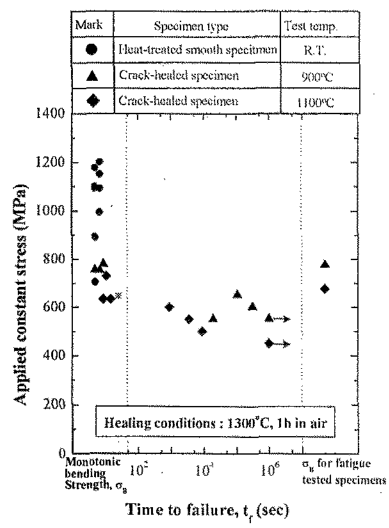


Fig. 4. The results of static fatigue test of crack-healed Al_2O_3/SiCw at 900°C and 1100°C.

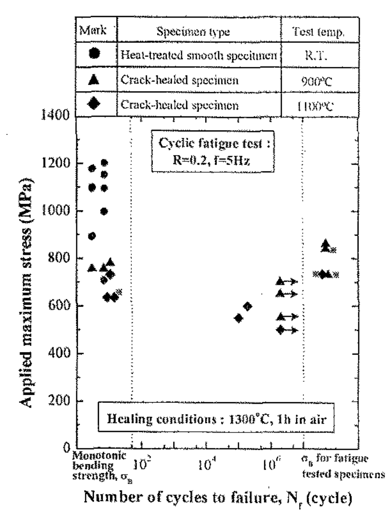


Fig. 5. The results of cyclic fatigue test of crack-healed Al_2O_3/SiCw at 900°C and 1100°C.

2. The static fatigue limits of crack-healed specimens at 900°C and 1100°C were 550 MPa and 450 MPa, respectively. The static fatigue limits of crack-healed specimens were about 70 % at each temperature in comparison to bending strength.
3. The cyclic fatigue limit of the crack-healed specimens at 900°C was 700 MPa. The ratio of cyclic fatigue limit to the bending strength of the crack-healed specimens at 900°C was above 90 %, while the ratio of σ_{f0}/σ_B of the crack-healed specimens at 1100°C was about 75 %.
4. From the above results, crack-healed Al_2O_3/SiCw was not sensitive to static and cyclic fatigues at room temperature and high temperature. Therefore, the addition of SiC whisker is very useful for static and cyclic fatigue at high temperatures.

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