Crack-Healing Behaviour under Cyclic Stress and Resultant Fatigue Strength of Si_3N_4 Composite Ceramics

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ABSTRACT: *Si_3N_4/SiC composite ceramics were sintered and subjected to three-point bending. A semi-elliptical surface crack of 100 μm in surface length was made on each specimen. The crack-healing behavior under cyclic stress of and resultant cyclic fatigue strength at the healing temperature of 1100 ℃ and 1200 ℃ were systematically investigated. The main conclusions are as follows. (1) Si_3N_4/SiC composite ceramics have the excellent ability to heal a crack at 1100℃ and 1200℃. (2) This sample can heal a crack even under cyclic stress at a frequency of 5 Hz. (3) The crack-healed sample exhibited sufficient cyclic fatigue strength at the each crack-healed temperature, 1100 ℃ and 1200 ℃.*

INTRODUCTION

Some engineering ceramics have an ability to heal a crack [1－7]. If this ability is used on structural components in engineering use, great merits can be anticipated, such as increases in the reliability of structural ceramic members, and decreases in the inspection, machining and polishing costs of ceramic components. To use this healing ability in structural engineering, we developed Si_3N_4/SiC [3,6,8,9] and mullite/SiC [10] with very high self crack-healing ability. We also systematically studied important subjects for the practical use, such as the best healing condition for the high temperature strength and cyclic and static fatigue strengths of the crack-healed member [6-10, 12]. To guarantee the reliability of a ceramics member, we proposed a new methodology, so called "crack-healing + proof test" [11]. This methodology is very important for the reliability of ceramics components, because embedded flaws cannot be healed at the present time [3,6-10]. It was shown that the reliability of ceramics component could be guaranteed for monotonic and cyclic loading using this technique. If the crack-healing treatment by the best condition and subsequent proof testing are applied to the ceramics components after machining, the reliability of ceramics component before service can be guaranteed, completely and economically.

However, if a crack initiates during service, the reliability will be reduced considerably, because fracture toughness of structural ceramics is not high. If a material can heal a crack and the crack-healed zone has enough strength at the service conditions, i.e. cyclic stress and high temperature, it would be desirable for the structural integrity. The present authors have already systematically studied the crack-healing behaviour under monotonic and cyclic stress, and resultant fatigue strength at the healed temperature [6,10,12]. However, the crack-healing behaviour under cyclic stress is very complicated and factors affecting crack-healing behaviour are not well understood yet.

In this paper, we selected Si_3N_4/SiC ceramics with very high crack-healing ability as a sample. We investigated the crack-healing behaviour under cyclic stress at 1100℃ and 1200℃, mainly, and resultant cyclic fatigue strength at the healed temperature.

MATERIAL, SPECIMEN AND EXPERIMENTAL METHOD

The silicon nitride powder used in this investigation has the following properties: mean particle size = 0.2 μm, the volume ratio of α-Si_3N_4 is about 95% and the rest is β-Si_3N_4. The SiC powder used has a 0.27 μm mean particle size. The samples were prepared using a mixture of silicon nitride, 20 wt% SiC powder and 8 wt% Y_2O_3 as an additive powder. To this mixture, alcohol was added and blended completely for 48 h. The mixture was placed in an evaporator to extract the solvent and then in a vacuum to produce a dry powder mixture. The mixture was subsequently hot-pressed at 1800 ℃ and 35 MPa for 2 h in nitrogen gas.

The sintered material was then cut into test pieces measuring 3×4×20 mm. A semi-elliptical surface crack of 100 μm in surface length (aspect ratio 0.9) was introduced at the centre of the tension surface of the test pieces with a Vickers indenter using a load of about 20 N.

In this study, a crack was healed at 1100 ℃ and 1200 ℃, mainly under cyclic bending stress, where the maximum bending stress (σ_{max}) was 210 MPa, stress ratio (R) was 0.2 and frequency was 5 Hz. The bending strength of the as-cracked sample is ～400MPa as shown by △ in Fig.1, so that the applied stress of 210 MPa is ～53% of the bending strength of the as-cracked sample. The applied stress of 210 MPa is higher than the cyclic fatigue limit at room temperature (～200 MPa) for a cracked sample [6]. Thus, if a cracked sample is subjected to the cyclic stress of 210 MPa, the sample fails from an initial crack due to the cyclic stress.

In the crack-healing processes, we first applied the cyclic bending stress then increased the furnace temperature at a rate of 10 ℃/min to avoid unexpected crack-healing without applied stress. After the crack-healing treatment, monotonic bending tests were carried out at room temperature and crack-healed temperature of 1100℃ and 1200℃. Cyclic fatigue tests were also carried out at each crack-healed temperature at a stress ratio of 0.2 and frequency of 5 Hz. Both monotonic and cyclic fatigue tests were conducted using a three-point loading system with span of 16 mm. The cross-head speed in the monotonic test was 0.5 mm/min.

Fracture initiation site was identified by optical microscope. Fracture surface was analysed by the scanning electron microscope (SEM).

TEST RESULTS AND DISCUSSION

Effect of healing temperature on the bending strength of Si_3N_4/SiC crack-healed under cyclic stress.

The samples having a surface crack were healed under cyclic stress at a temperature between 800 ℃ to 1200℃ for 5 hours in air. After the crack-healing process, monotonic bending tests were carried out at room temperature. The test results are shown in Fig.1(a). During the heating up of the samples from room temperature to each testing temperature, the initial cracks may be healed. To eliminate this unexpected crack-healing, the bending strength was measured at room temperature.

In Fig.1(a), the bending strengths of as-received smooth samples (○) and cracked samples (△) at room temperature are also indicated. The mean value of the bending strength of smooth samples is ～800 MPa. The Vickers indentation largely reduced bending strength to ～400 MPa. The bending strength of smooth specimen is almost constant up to 1300 ℃ [6,9]. Thus, only the bending strength at room temperature (○) was shown in Fig.1(a). The symbol ＊ shows that fracture occurred outside the crack-healed zone indicating the crack was healed completely.

Above the crack-healing temperature of 1000℃, the samples recovered their bending strength, moreover most of the samples failed outside the crack-healed zone. However, the scatter of the bending strength is large. This is because the bending strength of a smooth sample has a large scatter. On the other hand, at the crack-healing temperature of 800℃, 900℃, most of the samples failed from the crack-healed zone indicating a crack was not healed completely. Thus, in the following sections, the crack-healing behaviours at 1100 ℃ and 1200 ℃ were discussed, where the crack-healing is almost complete.

Effect of healing time on the bending strength of Si_3N_4/SiC crack-healed under cyclic stress.

Fig. 1(b) shows the effect of crack-healing time on the room temperature bending strength of Si_3N_4/SiC crack-healed at 1100℃ and 1200℃ under cyclic stress. The samples crack-healed under cyclic stress at 1100 ℃ (▲) and 1200℃ (□) recovered their bending strength. Most of the samples fractured outside the crack-healed zone. However, a few samples fractured from crack-healed zone.

A difference of healing time between 0.5 h to 5 h had no significant effect on the crack-healing behaviour. It is surprising that the crack can be healed completely in 0.5 h. From these test results, it can be concluded that the

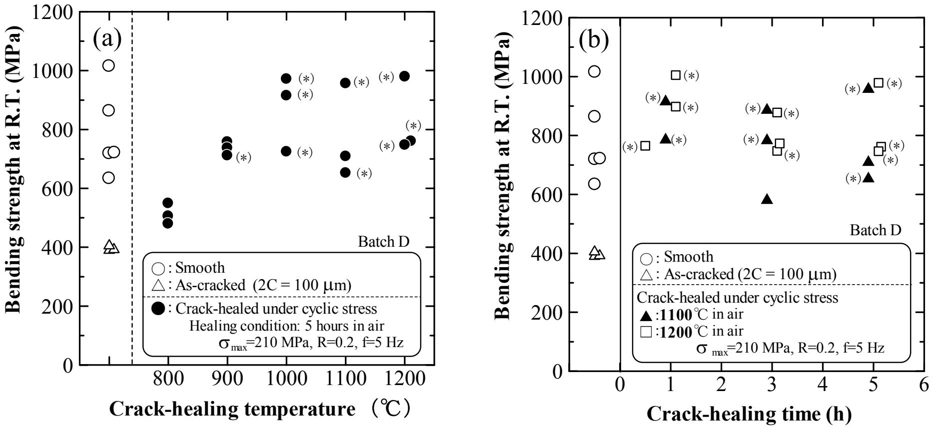

Figure 1: Results of the bending test of Si_3N_4/SiC at room temperature. (a) Effect of crack-healing temperature, (b) Effect of crack-healing time at 1100 ℃ and 1200 ℃. (＊ marked data indicates that fracture occurred outside the crack-healed zone.)

Si_3N_4/SiC ceramics can heal a crack under cyclic stress of 210 MPa which is ～53 % of bending strength of cracked sample.

Cyclic fatigue strength of the samples crack-healed at temperature of 1100℃ and 1200℃

Figs.2(a) and 2(b) show the results of cyclic fatigue tests together with the monotonic bending strengths at each crack-healed temperature, i.e. at 1100℃ and 1200℃, respectively. The monotonic bending test results are shown in the left side of Fig.2 (a) and 2(b). The symbols ● and ▼ in Fig.2(a) show the results of the samples crack-healed at 1100℃ for 5 h and 15 h, respectively. The symbol ◆ in Fig.2(b) shows the results of the samples crack-healed at 1200 ℃ for 5 h. The mean values of monotonic bending strength for 1100℃ (●, ▼) and 1200℃ crack-healed samples (◆) at each crack-healed temperature are 775 MPa and 881 MPa, respectively. These bending strengths are comparable to the room temperature bending strength of smooth samples, ～800 MPa.

The cyclic fatigue tests were stopped at $N=10^6$ cycles. The samples that did not fracture in the tests are marked by an arrow symbol (→). The applied stress at which a sample did not fracture up to $N=10^6$ is defined as cyclic fatigue limit, σ_{f0}. The σ_{f0} for the 1100℃ crack-healed sample is about 600 MPa, and that for the 1200℃ crack-healed sample is about 650 MPa. As

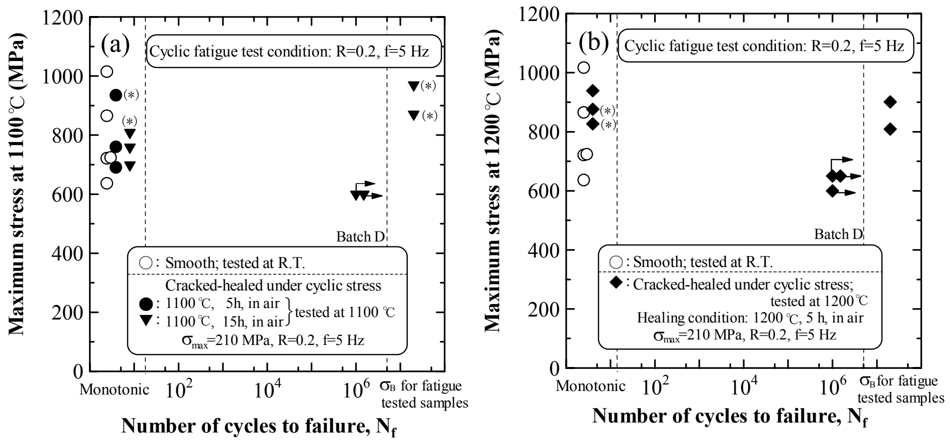

Figure 2: Cyclic fatigue test results of Si_3N_4/SiC at (a)1100 ℃ and (b)1200 ℃. (＊ marked data indicates that fracture occurred outside the crack-healed zone.)

mentioned already, the mean value of room temperature bending strength of the smooth sample (○) is ～800 MPa. The ratio of σ_{f0} to the mean bending strength of the smooth samples is about 75～80 %. Thus, the cyclic fatigue limit is considerably high. The bending strength of the samples which survived cyclic fatigue tests were also investigated and shown in the right side of Figs. 2(a) and 2(b). The bending tests were performed at each crack-healing temperature, i.e. 1100℃ and 1200℃. The fatigue-tested samples exhibited slightly higher bending strengths than that of the monotonically tested samples. This trend is consistent with our previous studies.[6,9,10,12]

Fracture surface and crack-healing mechanism of Si_3N_4/SiC

Fracture surface of the crack-healed samples was analysed by the scanning electron microscope (SEM). Figure 3 show SEM photograph of the fracture surface of Si_3N_4/SiC that was crack-healed at 1100℃ under cyclic stress and tested at room temperature. Most of the samples fractured from an internal flaw rather than crack-healed zone. However, some specimens fractured across the crack-healed zone as shown in Fig. 3. Fig. 3(b) shows the detail within the initial semi-elliptical crack surface, i.e. crack-healed zone. In this region, newly developed reaction product is observed. On the other hand, no clear reaction product is observed outside of semi-elliptical crack surface as shown in Fig. 3(c).

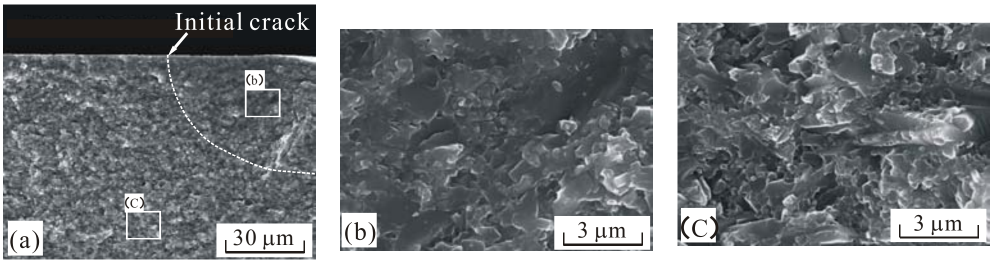

Figure 3:SEM photograph of fracture surface of the crack-healed Si_3N_4/SiC tested at room temperature. Specimen fractured across the crack-healed zone. (Healing condition: 1100 ℃ for 5 h in air, σ_{max} =210 MPa, R=0.2, f=5Hz, bending strength at R.T. = 709 MPa), (a) shows the crack profile, (b) shows the detail of crack-healed zone and (c) shows the detail of outside the crack-healed zone.

The estimated crack-healing reactions for Si_3N_4/SiC are as follows [6,11,12].

$$Si_3N_4 + 3O_2 \rightarrow 3SiO_2 + 2N_2 \quad (1)$$

$$SiC + 2O_2 \rightarrow SiO_2 + CO_2\ (CO) \quad (2)$$

$$2SiC + Y_2O_3 + 4O_2 \rightarrow Y_2Si_2O_7 + 2CO_2\ (CO) \quad (3)$$

The SiO_2 has two phases: one is a glassy phase and another is a crystal phase. At the temperature above 1000℃, such as 1100℃ and 1200℃, reactions (2) and (3) are dominant and both SiO_2 and $Y_2Si_2O_7$ has the crystal phase [12]. The reaction product shown in Fig. 3(b) is considered to be crystallized SiO_2 and $Y_2Si_2O_7$. If a healing material is crystallized, the crack-healed zone exhibits high bending strength and fatigue strength even at the elevated temperature [12]. Thus, the bending strength of the samples crack-healed at 1100℃ and 1200℃ has high bending strength and fatigue limit as shown in Fig. 2.

At a healing temperature of 1000℃, whether SiO_2 is crystallized or not depends on crack-healing time. In the previous study [12], we investigated crack-healing behaviour of Si_3N_4/SiC under cyclic stress at 1000℃. A 100 h crack-healed sample had large amount of crystallized SiO_2. On the other hand, a 5 h crack-healed sample had very small amount of crystallized SiO_2. It was found that whether crack-healing product was crystallized or not had large effect on the static and cyclic fatigue limit at crack-healed temperature, i.e. 1000℃ [12].

Below the crack-healing temperature of 1000℃, such as 800 ℃ and 900 ℃, the reaction of (2) is assumed to be dominant and SiO_2 has the glassy phase [12]. If the healing material is a glassy SiO_2, the crack-healed zone has lower bending strength and fatigue strength, especially at elevated temperature [12]. Thus, most of the samples fractured from the crack-healed zone and the bending strength decreases as the crack-healed temperature decreases as shown in Fig.1(a).

CONCLUSIONS

Si_3N_4/SiC composite ceramics were sintered and subjected to three-point bending. A semi-elliptical surface crack of 100μm in surface length was made on each specimen. Crack-healing behaviour under cyclic stress, and resultant cyclic fatigue strengths at the healing temperature of 1100 ℃ and 1200 ℃ were systematically studied. The main conclusions obtained are as follows.

(1) At the crack-healing temperature of 1100℃ and 1200℃, a surface crack can be healed completely even under the cyclic stress. The crack-healed

samples recovered their bending strength at R.T. and crack-healed temperature. Moreover, most of the samples failed outside the crack-healed zone. Crystallized SiO_2 and $Y_2Si_2O_7$ are considered to contribute the healing of pre-cracked specimen.

(2) Differences of healing time between 0.5 h and 5 h had no significant effect on the crack-healing behaviour. It is surprising that a crack can be healed completely in 0.5 h even under cyclic stress.

(3) Cyclic fatigue limit of the samples crack-healed at 1100℃ and 1200℃ are 600 MPa and 650 MPa at each healing temperature. The ratio of cyclic fatigue limit to the mean bending strength of the smooth samples (～800 MPa) is about 75～80 %. Thus, the cyclic fatigue limit of crack-healed samples is considerably high.

(4) Conclusions (1) to (3) intensely suggest that this sample can heal a crack under service condition, i.e. under cyclic stress and at high temperature above 1000℃.

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