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Critical Crack Healing Condition under Stress and Subsequent Strength at the Healing Temperature

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Alumina and mullite reinforced by SiC whisker, Alumina(W) and Mullite(W), were developed for aim to improve fracture toughness and crack-healing ability. The composites were crack-healed at 1473 K for 8 h in air under elevated static and cyclic stresses and the bending strength at 1473 K of the composites crack-healed were also investigated. Alumina(W) crack-healed under static stress below 250 MPa were never fractured during crack-healing, and have the same bending strength as the specimens crack-healed under no-stress. Therefore, the threshold static stress during crack-healing of Alumina(W) has been determined to be 250 MPa. The threshold cyclic stress has been also determined to be 300 MPa. Considering that the crack growth is time-dependence, the threshold stress of every condition during crack-healing of Alumina(W) has been concluded to be 250 MPa. For the same assessment of Alumina(W), the value of Mullite(W) have been also determined to be 100 MPa.

Key words: Crack-healing, SiC whisker, Alumina, Mullite

1. INTRODUCTION

Structural ceramics such as alumina, mullite and silicon nitride have excellent heat, corrosion and wear resistance. However, fracture toughness is low. This low reliability has, thus, limited their applications. Many investigators tried to improve the fracture toughness of structural ceramics by admixing with whisker and fiber. Guo *et al.* [1] improved fracture toughness of silicon nitride from 6.0 MPam$^{1/2}$ to 15.6 MPam$^{1/2}$ by admixing with carbon fiber.

On the other viewpoint to improve reliability of structural ceramics, the present authors [2-14] have progressed in development of the structural ceramics attached crack-healing ability by using oxidation of SiC. When ceramics admixed with SiC are kept in air at high temperature, SiC located on the crack surface reacts with O_2 in air and then crack is completely restored by the products and the heat of the reaction. Moreover, the restored part is mechanically stronger than the other parts. If the above mechanism so called as crack-healing is used on structural components in engineering use, great benefits can be anticipated improvement in reliability as well as a decrease in machining and polishing costs of ceramics elements. In the previous studies [15, 16], alumina admixed with 20 mass% SiC whisker, Alumina(W) [15], and mullite admixed with 15 mass% SiC whisker, Mullite(W) [16], were sintered and investigated mechanical properties for improvement in fracture toughness and attaching crack-healing ability. Both composites have indicated excellent crack-healing ability and the fracture toughness of the composites have been determined to be 6.5 MPam$^{1/2}$ and 4.0 MPam$^{1/2}$, respectively. The value of Alumina(W) is 2 times lager than that of monolithic Alumina, and the value of Mullite(W) is 1.6 times larger than that of monolithic Mullite.

Structural ceramics attached crack-healing ability have an interesting mechanical property [6, 10] that crack-healing occurs though the crack is applied tensile stress. As a result, crack-healing prevents fatigue crack growth and the fatigue strength at high temperature was reduced slightly compared to fracture strength at the same temperature. A prolongation of the lifetime of ceramics can be anticipated by using this behavior. It is necessary for applying fully crack-healing under stress, that upper limit stresses to be safely able to apply during crack-healing are determined.

Therefore, Alumina(W) and Mullite(W) are subjected to crack-healing under elevated static and cyclic stresses at 1473 K, corresponding to the limit temperature for bending strength of Alumina(W), and the bending strength of Alumina(W) and Mullite(W) crack-healed under stress are measured at the crack-healing temperature. From the result, upper limit stresses to be safely able to apply during crack-healing are determined for Alumina(W) and Mullite(W).

2. EXPERIMENTAL

α-Al_2O_3 powder (AKP-20, Sumitomo Chemicals Co. Ltd., Japan) used in this study has purity of 99.999 % and a mean particle size of 0.4 μm to 0.6 μm. Mullite powder (KM 101, Kioritzz Co. Ltd., Japan) used is an average particle size of 0.2 μm and Al_2O_3 content of 71.8 %. SiC whisker (SCW, Tateho Chemical Industry Co. Ltd., Japan) used has a diameter of 0.8 μm to 1.0 μm and a length of 30 μm to 100 μm. The mixture of α-Al_2O_3 powder and 20 mass% SiC whiskers was blended well in isopropyl alcohol for 12 h using alumina balls and an alumina mill pot. The mixture of mullite powder and 15 mass% SiC whiskers was also blended in

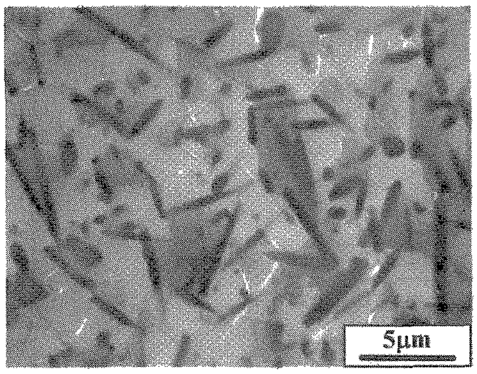

Fig. 1 Typical microstructure of Alumina(W)

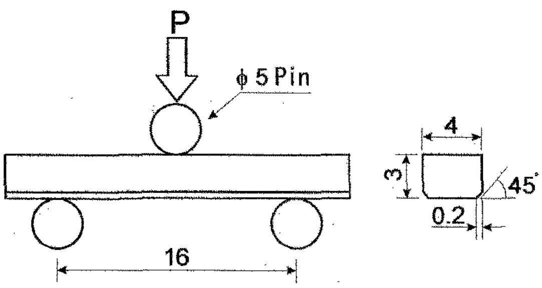

Fig. 2 Three point bending specimen and test system, dimensions in mm

the alcohol. Then, the slurries were dried. Rectangular plates of 50 mm ×50 mm × 9 mm of Alumina(W) and Mullite(W) were sintered in argon for 1 h via hot press under 40 MPa at 2123 K and 1973 K, respectively. Figure 1 shows microstructure of Alumina(w). The grain size of α-Al_2O_3 was 1 to 2 μm. The SiC whiskers locate at grain boundaries between α-Al_2O_3 grains and are preferentially oriented within the plane perpendicular to the pressing axis. The density of the sintered plate was 3.83 g/cm^3.The sintered plates were cut into the 3 mm × 4 mm × 23 mm rectangular specimens bar that were polished to mirror finished on one face and the edges of specimens were beveled 45°, as shown in Fig. 2, to reduce the likelihood of edge initiated failures.

A semi-elliptical surface crack of 100 μm in surface length was made at the center of the tensile surface of specimens with a Vickers indenter, using a load of 19.6 N. The ratio of depth (a) to half the surface length (c) of the crack (aspect ratio) was a/c = 0.9. The specimens were heat-treated to heal the pre-crack in air. In this investigation, the pre-crack was healed with loading tensile stress by a three-point loading system shown in Fig. 2. To avoid the crack-healing under no-stress, the pre-crack was loaded tensile stress before the crack-healing was started by heating the furnace to 1473 K. The specimens were kept the above condition for 8 h to finish completely crack-healing. The applying stresses were static or cyclic (R = 0.2, 5 Hz).

All fracture tests of the specimens crack-healed were performed on a three-point loading system with a span of 16 mm at 1473 K, corresponding to the crack-healing temperature. The cross-head speed in the monotonic test was 0.5 mm/min.

3. RESULTS AND DISCUSSION

In terms of basic knowledge, figure 3 shows temperature dependence of the bending strength of Alumina(W) crack-healed at 1573 K for 1 h. The crack-healing condition was selected from the previous reported [15], and pre-crack size crack-healed was 100 μm. The bending strength at room temperature was recovered from 480 MPa to 1000 MPa by crack-healing. The bending strength was not affected by an increase in temperatures form 673 K to 1273 K but decreased with increasing temperature above 1273 K. The specimens were fractured brittlely below 1473 K, and fractured with a creep deformation at 1573 K. Thus, the limit temperature for bending strength of Alumina(w) was determined to be 1473 K. Figure 4 also shows temperature dependence of the bending strength of Mullite(W) crack-healed at 1573 K for 1 h. The limit temperature for bending strength was also determined to be 1573 K for the same assessment as Alumina(W).

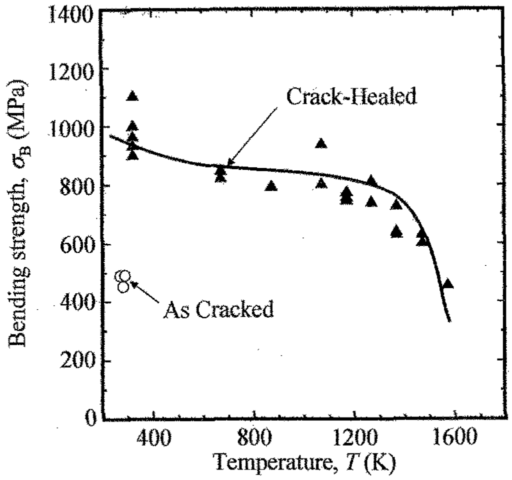

Fig. 3 Temperature dependence of bending strength of Alumina(W) crack-healed at 1573 K for 1 h.

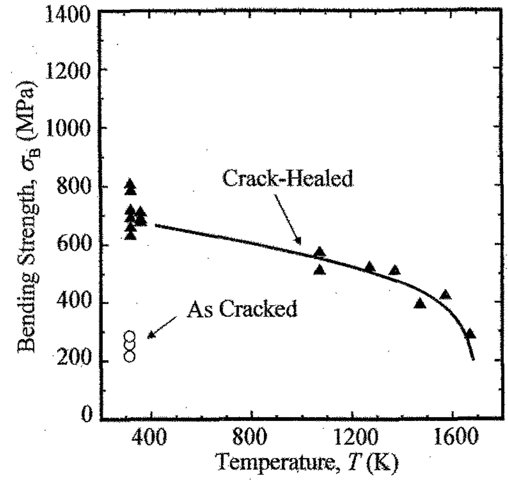

Fig. 4 Temperature dependence of bending strength of Mullite(W) crack-healed at 1573 K for 1 h.

Figure 5 shows the bending strength at the crack-healing temperature of Alumina(W) crack-healed at 1473 K for 8 h under stress. The open circle and the closed triangle indicate the bending strength of the specimen crack-healed under static stress, $\sigma_{app,S}$, and cyclic stress, $\sigma_{app,C}$, respectively. The bending stress of 0

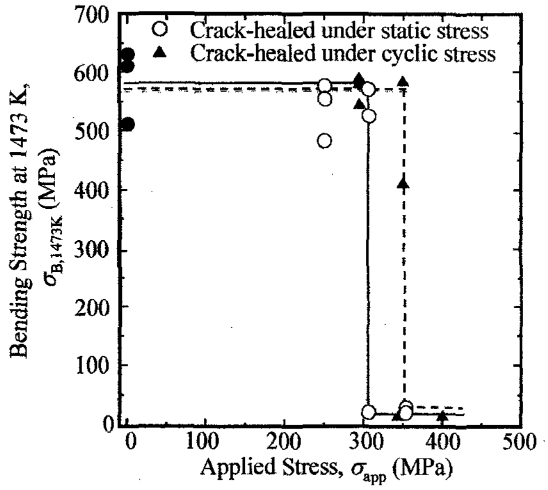

Fig. 5 Bending strength at the crack-healing temperature of Alumina(W) crack-healed at 1473 K for 8 h under stress

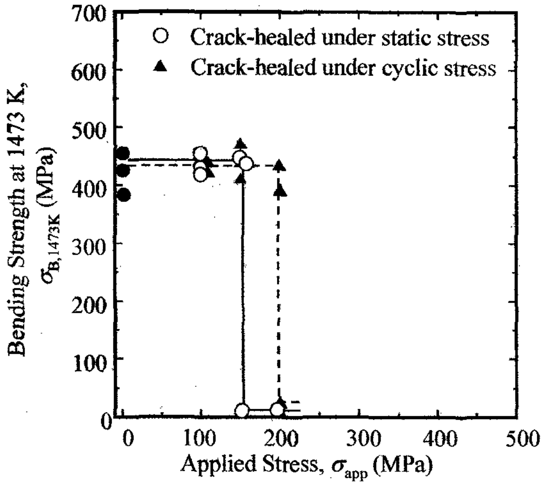

Fig. 6 Bending strength at the crack-healing temperature of Mullite(W) crack-healed at 1473 K for 8 h under stress

MPa indicates the specimen fractured during crack-healing. The specimens crack-healed under static stresses below 250 MPa were never fractured during crack-healing, and have the same bending strength as the specimens crack-healed under no-stress. A few specimens crack-healed under static stress of 300 MPa were fractured, and all specimens crack-healed under static stress of 350 MPa were fractured during crack-healing. Therefore, the threshold static stress during crack-healing of Alumina(W) having pre-crack of 100 μm, $\sigma^C_{app,S}$, has been determined to be 250 MPa where the threshold stress during crack-healing is defined the stress which is upper limit stress not to fracture during crack-healing, The threshold cyclic stress of it, $\sigma^C_{app,C}$, has been also determined to be 300 MPa. These values correspond to 63 % and 75 % of bending strength of Alumina(W) as cracked, respectively.

Figure 6 shows the bending strength at the crack-healing temperature of Mullite(W) crack-healed at 1473 K for 8 h under stress. For the same assessment as Alumina(W), the threshold static and cyclic stresses during crack-healing of Mullite(W) having pre-crack of 100 μm have been determined to be 100 MPa and 150 MPa, corresponding to 40 % and 60 % of bending strength of Mullite(W) as cracked, respectively.

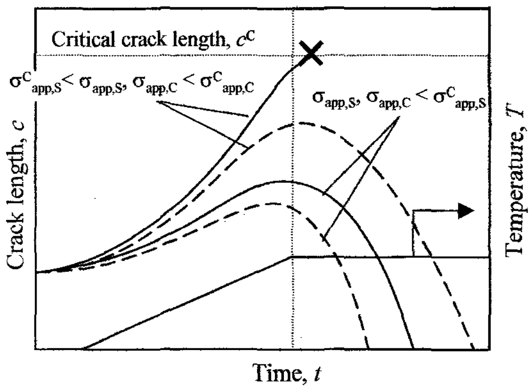

Fig. 7 Schematic diagram of the crack-growth and the crack-healing behavior during the crack-healing treatment under stress

Figure 7 shows the schematic diagram of the crack-growth and crack-healing behavior during the crack-healing treatment under stress. The driving force of crack growth, which is called as crack driving force by Irwin, increases with an increase in applied stress and crack length. On the other hands, the driving force of crack-healing, which is called as crack healing force in short, is not affected applied stress but increases remarkably with increasing temperature. Crack-healing starts preventing crack growth above the temperature that crack healing force becomes enough large not to neglect compared to crack driving force. Moreover, crack starts being healed when crack healing force becomes larger than crack driving force. The threshold stress gives upper limit crack growth rate that crack length does not reach to critical crack length before starting crack-healing. From a comparison with the values of $\sigma^C_{app,S}$ and $\sigma^C_{app,C}$, it may be confirmed that the crack growth behaviors of Alumina(W) and Mullite(W) is time dependence rather than cyclic dependence. It is, therefore, summarized that applying static stress is most fracturable condition and threshold stresses of every condition during crack-healing of Alumina(W) and Mullite(W) have been determined to be 250 MPa and 100 MPa, respectively.

4. CONCLUSION

For the determining threshold stress during crack-healing, Alumina(W) and Mullite(W) having pre-crack of 100 μm were subjected to crack-healing under elevated static and cyclic stresses at 1473 K. Alumina(W) crack-healed under static stresses below 250 MPa were never fractured during crack-healing In the other words, Alumina(W) can be crack-healed not to fracture at applying static stress of 250 MPa. Alumina(W) crack-healed under 250 MPa has the same bending strength as the specimens crack-healed under no-stress. Therefore, the threshold static stress during crack-healing of Alumina(W) has been determined to be 250 MPa. The threshold cyclic stress has been also determined to be 300 MPa. Moreover, the threshold static and cyclic stresses during crack-healing of Mullite(W) have been determined to be 100 MPa and

150 MPa, respectively. Considering crack growth and healing mechanism, the threshold stress of every condition during crack-healing of Alumina(W) has been determined to be 250 MPa. The threshold stress of every condition during crack-healing of Mullite(W) has been also determined to be 100 MPa.

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