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Bending Strength of Crack Healed Si_3N_4/SiC Composite Ceramics by SiO_2 Colloidal

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ABSTRACT: *Si_3N_4/SiC composite ceramics was sintered in order to investigate their bending strength behavior after crack healing. Y_2O_3 and TiO_2 power was added as sintering additives to enhance it's sintering property. A three-point bending specimen was cut out from sintered plates. About 100 ㎛ semi-circular surface cracks were made on the center of the tension surface of the three-point bending specimen using Vickers indenter. After the crack-healing processing from 500 ℃ to 1300 ℃, for 1 h, in air, the bending strength behavior of these crack-healed specimen coated with SiO_2 colloidal were determined systematically at room temperature. Si_3N_4/SiC ceramics using additive powder (Y_2O_3+TiO_2) was superior to that of additive powder Y_2O_3 . The additive powder TiO_2 exerted influence at growth of Si_3N_4 . The optimum crack healing conditions coated SiO_2 colloidal were 1000 ℃ at Si_3N_4/SiC using additive powder (Y_2O_3+TiO_2), and 1300 ℃ at Si_3N_4/SiC using additive powder Y_2O_3.*

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 followings can be an excellent methodology to overcome these problems (Ando et al., 1999) (a) toughen the ceramic by fiber and whisker reinforcement, and microstructure control, (b) activate the crack-healing ability and heal a crack after machining. If a crack-healing ability (Lange, 1970; Lange and Radford, 1970; Greskovich and Rosolowski, 1976; Gupta, 1976) was used on structural components for engineering use, considerable advantages could be anticipated. With this motivation, Ando et al developed Si_3N_4 (Ando et al., 1998), mullite (Lee et al., 2005), alumina [Nakao et al, 2005) and SiC (Lee at al., 2005) with very strong crack-healing abilities. As a result, in the case of most ceramics, the crack-healed zones 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. The temperature where bending strength starts to decrease rapidly with increasing testing temperature is defined as heat-proof temperature. These test results suggest that the crack-healing ability can be used as a method to guarantee the structural integrity of a ceramic component (kim et al., 2005; Nam et al, 2006). However, oxygen is absolutely necessary for the crack-healing process (Ando et al., 1998; Houjou et al., 2004). Thus, in this study, the oxygen effects contributing to toughening and strengthening have initially been studied with SiO_2 colloidal. The crack-healing behaviors of Si_3N_4/SiC composites coated SiO_2 colloidal were investigated from 500 to 1300 °C.

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2. Materials, Specimen and Test 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 over 95 % and the rest is β-Si_3N_4. The SiC powder has a mean particle size 0.27 μm. The TiO_2 powder is the commercial anatase type. The Si_3N_4/SiC composite ceramics were prepared using a mixture of 80 wt% silicone nitride, 20 wt% SiC powder with 8 wt% Y_2O_3 as an additive powder (Sample A). The Si_3N_4/SiC composite ceramics were prepared using 5 wt% Y_2O_3 and 3 wt% TiO_2 as an additive powder (Sample B). Alcohol was added to this mixture and was blended completely for 24 h. The mixture was placed in a desiccator to extract the solvent and to make a dry powder mixture. Circle plates of 60 mm 5 mm were sintered in nitrogen gas for 1 hr via a hot press under 35 MPa at 1850 °C. The sintered plates were cut into the (4 x 3 x 24) mm rectangular bar specimens. The specimens were polished to a mirror finish on one face and the edges of the specimens were beveled 45° to reduce the likelihood of edge initiated failures. SiO_2 colloidal with 12 % SiO_2 particle was used to heal the surface crack.

A semi-elliptical surface crack of about 100 μm in surface length was made at the center of the tensile surface of specimens with a Vickers indenter, using a load of 24.5 N. The ratio of depth (a) to half the surface length (c) of the

crack (aspect ratio a/c) was 0.9. The crack shape is shown in Fig. 1. The specimens cracked and as-received were mainly subjected to crack-healing treatment at 1300 °C for 1 hr in air, where the crack-healing condition is referred [15]. To compare the crack healing ability of a cracked specimen coated using SiO_2 colloidal, the crack healing was carried out in the temperature range from 500 to 1300 °C for 1 hr in air. The coating on the crack surface is 1 time and 3 times. All fracture tests were performed on a three-point loading system with a span of 20 mm at room temperature. The cross-head speed in the monotonic test was 0.5 mm/min.

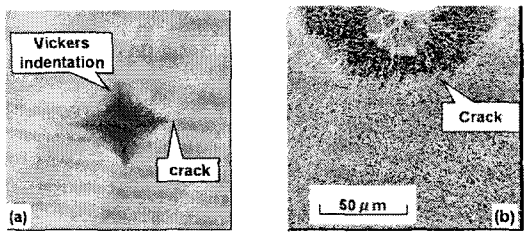


Fig. 1 SEM photographs of (a) Vickers indented crack on the surface and (b) Crack surface.

3. Results and Discussion

Figure 2 shows the bending strength of a smooth specimen and a cracked specimen. The solid symbol and open symbol is Sample A and Sample B, respectively. The solid and open square indicates the bending strength of the smooth specimen, and the solid and open circle indicates that of the cracked specimen. The dot line and solid line is the mean strength of the smooth specimen, respectively. The cracked specimen is about a half the bending strength of the smooth specimen, but Sample B is higher than Sample A. The bending strength of the heat-treated smooth specimen is slightly superior to that of the smooth specimen. The heat-treated smooth specimen of Sample B is far above the smooth specimen. In heat-treated smooth specimen, Sample B is higher than Sample A.

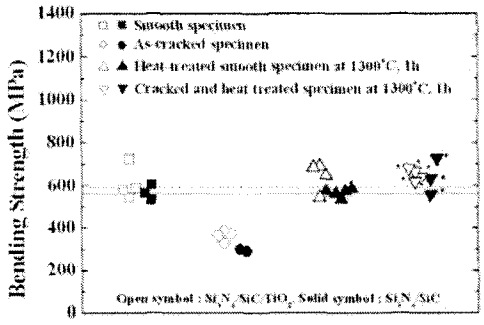
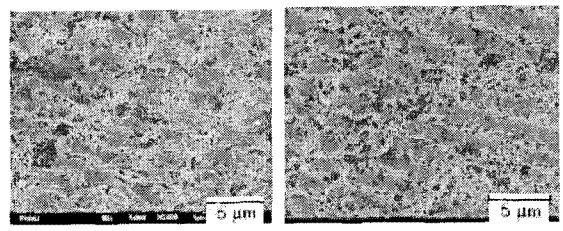


Fig. 2 Bending strength of smooth specimen and cracked specimen.

The SEM image of Sample B shows that the Si3N4has grown and became a column type as shown in Fig. 3(b). This was obtained from 1000 °C. This is estimated that the additive powder TiO_2 affects the growth of Si_3N_4. Fig. 3(a) is SEM image of Sample A from 1300 °C. On the other hand, the triangle and the reversed triangle indicate the bending strength of the heat-treated smooth specimen and the cracked and heat-treated specimen, respectively. The heat treatment was carried out in air at a temperature of 1300 °C for 1 hr. But, the cracked and heat-treated specimen recovered to a higher value than that of smooth specimen. The * marks indicate specimens on which the fracture occurred from the outside of the crack-healed zone. In Fig. 2, all cracked and heat-treated specimens fractured the outside of the crack-healed zone, as shown in Fig. 4(b). Fig. 4(a) shows a fracture at the cracked-healed zone.



(a) 1300 °C (b) 1000 °C

Fig. 3 SEM images of (a) Sample A and (b) Sample B

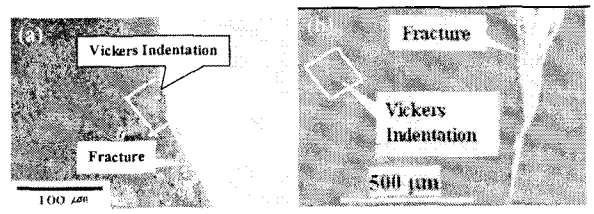


Fig. 4 Fracture patterns fractured from (a) crack-healed zone, (b) outside of crack-healed zone

Figure 5 shows the bending strength for the effect of crack-healing according to coating times of SiO_2 colloidal. The all heat treatment was carried out in air at a temperature of 1300 °C for 1 hr. The bending strength of the as-cracked specimen has nothing to do with coating times. That is, a portion of coating contributes to crack healing and the other portions crystallize to white powder on the surface. Both, the smooth specimen and as-cracked specimen with one time coating had an almost similar bending strength. The bending strength of the heat-treated smooth specimen with one time coating is superior to that of the cracked and heat treated specimen. This phenomenon is far above Sample B in one time coating. The bending strength of three time coating is similar to that of one time coating. Hereafter, all cracked specimens were experimented with one time coating.

Figure 6 shows the effect of crack-healing according to the temperature of a cracked specimen with one time coating of SiO_2 colloidal. All the heat treated crack specimens were fractured from outside the pre-crack. The bending strength of the as-cracked and heat treated specimens coated SiO_2 colloidal are

increased with an increasing heat treatment temperature. In sample A, the bending strength of 900 °C is almost equal to the values of the heat-treated smooth specimens at 1300 °C. The crack-healed specimen coated SiO_2 colloidal at 1300 °C showed dominant crack-healing ability. Sample Bshowed crack healing ability over 700 °C. Moreover, dominant crack-healing ability showed at 1000 °C lower than that of sample A. As above-mentioned, this is estimated that they were piled up the growth of Si_3N_4 by TiO_2 effect on crack-healing by SiO_2 colloidal. SiO_2 colloidal was made SiO_2 crystal over 500 °C.

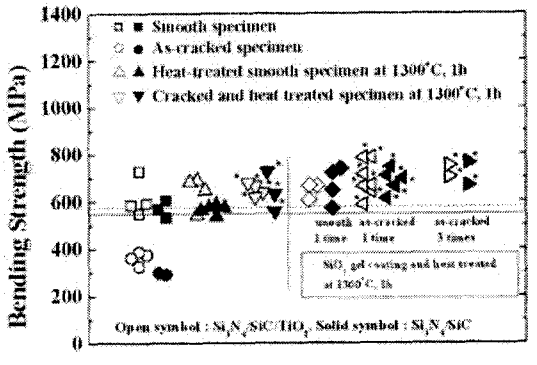


Fig. 5 Effect of crack-healing according to coating time.

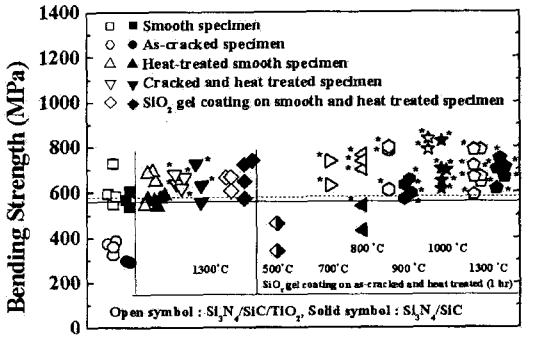


Fig. 6 Effect of crack-healing according to heat treated temperature of as-cracked specimen with SiO_2 colloidal

5. Conclusions

Sample A and Sample B composite ceramics were sintered and subjected to three-point bending tests. A semi-circular crack was made on each specimen surface. Using these specimens, we investigated the bending strength of crack-healed specimens coated with SiO_2 colloidal from 500 to 1300 °C, systematically. The main results are as follows Sample A and Sample B composite ceramics possess remarkable crack-healing ability. The additive powder TiO_2 exerted influence the grain growth of Si_3N_4. The crack-healed specimen coated SiO_2 colloidal showed a dominant crack-healing ability, most fractures occurred outside the pre-cracked zone. The optimum crack healing conditions coated SiO_2 colloidal were 1000 °C at Si_3N_4/SiC using additive powder (Y_2O_3+TiO_2), and 1300 °C at Si_3N_4/SiC using additive powder Y_2O_3. These materials can heal a crack with SiO_2 colloidal while in service.

References

Ando, K., Ikeda. T., Sato. S., Yao. F. and Kobayashi. Y. (1998). "A Preliminary Study on Crack Healing Behavior of Composite Ceramics" *Fatigue Fract. Eng. Mater. Struct.*, Vol. **21**, , pp. 119-122.

Ando, K. Chu. M.C. Kobayashi. Y., Yao. F. and Sato. S. (1999). "Crack Healing Behavior and High Temperature Strength of Silicon Nitride Ceramics", *Jpn. Soc. Mech. Eng.*, Vol. 65, pp. 1132-1139 (in Japanese).

Greskovich. C. and Rosolowski. (1976). "Sintering Covalent Solids", *J. Am. Ceram. Soc.*, Vol. **59**, pp. 336-343.

Gupta, T.K. (1976). "Crack Healing and Strengthening of Thermally Shocked Alumina" *J. Am. Ceram. Soc.*, Vol. **59**, pp. 259-262.

Houjou, K., Ando, K. Liu, S.P. and Sato. S. (2004). "Crack Healing and Oxidation Behavior of Silicon Nitride Ceramics", *J. Eur. Ceram. Soc.*, Vol. 24, pp. 2329-2338.

Kim, J.W., Park, B.W., Ahn, S.H. and Nam, K.W. (2005), "Elastic Wave Signal Characteristics of SiC Ceramics with Crack Healing Ability by Wavelet Analysis", *Key Engng. Mater.*, Vol. **297-300**, pp. 2046-2051.

Lange, F.F. (1970). "Healing of Surface Cracks in SiC by Oxidation", *J. Am. Ceram. Soc.*, Vol. **59**, pp. 290-297.

Lange, F.F. and Radford. (1970). "Healing of Surface Cracks in Polycrystalline Al_2O_3, *J. Am. Ceram. Soc.*, Vol. **53**, pp. 420-421.

Lee, S.K., Ono, Masato., Nakao, Watary., Takahashi, K. and Ando, K. (2005). "Crack-healing behaviour of mullite/SiC/Y_2O_3 composites and its application to the structural integrity of machined components", J. Eur. Ceram. Soc., Vol. 25, pp. 3495-3502.

Lee, S.K., Ishida, W., Lee, S.Y., Nam. K.W. and Ando, K. (2005) "Crack-Healing Behavior and Resultant Strength Properties of Silicon Carbide Ceramic", *J. Eur. Ceram. Soc.*, Vol. **25**, pp. 569-576.

Nam, K.W., Kim, M.K., Kim, H.S., Kim, J.W. and Ahn, S.H. (2006). "Bending Strength and Elastic Wave Characteristics of Si_3N_4 Monolithic and Si_3N_4/SiC Composite Ceramics by Wavelet Analysis", *Int. J.Modern Physics B*, Vol. **20** (in print)

Nakao, W., Ono, M., Lee, S.K., Takahashi, K. and Ando, K. (2005), "Critical crack-healing condition for SiC whisker reinforced alumina under stress", J. Eur. Ceram. Soc., Vol. 25, pp. 3649-3655.

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