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Crack-healing behavior and bending strength of Al_2O_3/SiC composite ceramics according to the amount of added Y_2O_3

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Three Al_2O_3/SiC composite ceramics were prepared, which included 1, 3 or 5 wt.% Y_2O_3, and their high-temperature bending strengths and in-situ crack-healing behaviors examined. A surface elliptical-crack of about 100 ß¦ in diameter was introduced in the specimens using a Vickers hardness indenter. From in-situ observations, the Al_2O_3/SiC composite ceramic with 3 wt.% Y_2O_3 showed superior crack-healing ability than the 1 and 5 wt.% Y_2O_3 ceramics. The as-cracked specimen with 3 wt.% Y_2O_3 showed strength recovery on heating for one hour at 1473 K in air, which may have been due to the lower crack-healing temperature on the addition of 3 wt.% Y_2O_3. The heat-resistance limit temperatures of the crack-healed Al_2O_3/SiC composite ceramics were 1073, 1373 and 873 K for 1, 3 and 5 wt.% Y_2O_3, respectively.

Key words: Crack-healing, Al_2O_3/SiC Composites, Strength recovery, Additive powder Y_2O_3, In-situ crack-healing behavior, elevating temperature.

Introduction

Alumina has excellent wear, corrosion and heat resistances; therefore, it is widely used in electrical insulation materials, IC boards, the chemical industry and in cutting tools and bearings, etc. However, it has weaknesses in relation to its bending strength, fracture toughness and heat-resistance limit temperature, which restrict the application of Al_2O_3 for important components. To overcome these weaknesses, three methods exist: (1) toughening with fibers or a particle dispersion and microstructural control, (2) detection and repair of cracks by nondestructive test (NDT) and (3) induction of a self crack healing ability. Many studies have been conducted with regard to (1) and (2), with many useful results reported. [1] Several investigations have found that the strength of ceramics can be increased by heating. [2] Typically, heat treatment of a ceramics specimen containing cracks can result in partial or complete recovery of the strength of the specimen to that of a smooth specimen, which is generally called 'crack healing'. Crack healing has been observed in many ceramics, such as SiC [3, 4], Si_3N_4 [5], Al_2O_3/SiC composites, [6, 7] mullite/SiC [8] and Si_3N_4/SiC composites [9]. However, the crack healing behavior of Al_2O_3/SiC on the addition of Y_2O_3 powder remains to be studied.

In this investigation, three Al_2O_3/SiC composites, with different amounts of Y_2O_3 (as sintering additive powder), were sintered, and small bending specimens and specimens for observation prepared. The objectives of this study were to investigate the crack healing behaviors of Al_2O_3/SiC composite ceramics, and identify the dominant crack healing mechanism operating in the ceramics.

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Material and Test Method

The Al_2O_3 powder used in this study was AA-04 (0.5 μm mean particle size, 99.99% purity) from Sumitomo Chemical Ltd., Japan. The SiC powder used was ultrafine, with a mean particle size of 0.27 μm, from Ibiden Ltd., Japan. The Y_2O_3 was used for sintering (Nippon Yttrium Ltd., Japan) to investigate the effects of various amounts of the additive powder. The quantities of added Y_2O_3 were 1, 3 and 5 wt.%. The mixture of Al_2O_3 powder and 15 wt.% SiC powder were blended well in alcohol for 24 hours using a mill. The solvent was evaporated from the mixture using a dryer, to yield a dry powder mixture. The mixture was subsequently hot pressed in nitrogen gas, at 1873 K under 35 MPa, for one hour. Specimens were cut into 3 × 4 × 22 mm rectangular bars for bending and 3 × 4 × 1 mm thin plates for in-situ observations. The specimens were polished to a mirror finish on a tensile surface. The edges of the specimens were beveled 45° to prevent fracture due to edge-cracks. A surface elliptical-crack, about 100 μm in diameter, was made at the center of the tensile surface of the specimens using a Vickers hardness indenter using a load of 19.6 N.

The crack-healing conditions significantly affect the fracture behavior of a crack-healed specimen. In-situ observations of a cracked-specimen were carried out in an air healing environment at 1573 K for one hour. To investigate the crack healing ability of a cracked specimen, the crack healing was carried out in air at temperatures ranging from 1373 to 1673 K for one hour. The smooth specimens were mainly subjected to heat treatment in air at 1573 K for one hour using the heat treatment conditions previously described. [6] All fracture tests were performed on a three-point bending system, with a 16 mm bending span. The crosshead speed in the monotonic tests was 0.5 mm/minute.

Results and Discussion

Bending strength according of crack healing temperature

Fig. 1 shows the crack-healing behaviors and bending strengths (σ_B) according to the amount of Y_2O_3 powder added. The open, half solid, open with a cross and solid symbols indicate the σ_B of smooth, as-cracked, heat treated smooth, and cracked and healed specimens, respectively. The star symbol indicates the average σ_B without the addition of Y_2O_3. [6]

The open symbols show the average σ_B of smooth specimens at 596 (1 wt.%), 647 (3 wt.%) and 299 MPa (5 wt.%), respectively. The open symbols with a cross show the σ_B of healed smooth specimens at 755 (1 wt.%), 758 (3 wt.%) and 367 MPa (5 wt.%), respectively. Even the smooth specimens contained minute flaws, such as surface cracks and pores, but these flaws were observed to be healed by heat-treating, resulting in an increased strength of approximately 17~27%. The half solid symbols show the σ_B of the as-cracked specimens. The average σ_B of the as-cracked specimens was drastically reduced 287 (1 wt.%), 282 (3 wt.%) and 212 MPa (5 wt.%), respectively. In Fig. 1, the σ_B of the smooth with 1 and 3 wt.% Y_2O_3 was similar to that of heat treated smooth specimen, but both σ_B of the 5 wt.% Y_2O_3 were very low for 1 and 3 wt.% Y_2O_3.

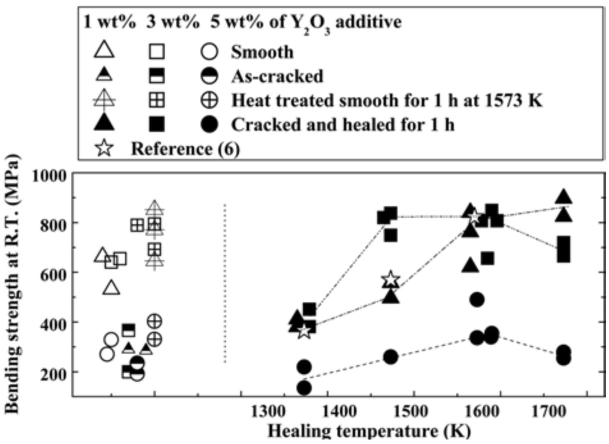

Fig. 1. Relation between bending strength and crack healing temperature according to the amount of added Y_2O_3.

The solid symbols show the σ_B of the crack-healed speci-mens heat treated for one hour between 1,373 and 1673 K. The σ_B of the 1 wt.% Y_2O_3 crack-healed specimen treated for one hour at < 1473 K was low; indicating incomplete crack healing. The 1 wt% Y_2O_3 crack-healed specimen treated for one hour at > 1573 K had a high average σ_B, but this was similar or superior to the average σ_B (755 MPa) of the heat treated smooth specimen for one hour at 1573 K, indicating that the crack had completely healed. The 3 wt.% Y_2O_3 crack-healed specimen treated for one hour at 1473 K had a very high average σ_B (810 MPa), indicating complete crack healing, which was higher than the average σ_B (779 MPa) of the crack healed specimen and the smooth specimen (758 MPa) heat treated at 1573 K. Moreover, this was similar to the average σ_B of Al_2O_3/SiC composite ceramic (☆) without Y_2O_3 obtained by heat treating at 1573 K. This means that the crack-healing temperature could be lowered by the addition 3 wt.% Y_2O_3. Conversely, the σ_B of the crack healed specimen with 5 wt.% Y_2O_3 treated for one hour at 1373 K was lower than that of the as-cracked specimen. The σ_B of healed specimen treated above 1473 K also showed no sufficient strength recovery.

Fig. 2 shows SEM micrographs of smooth specimens both before and after heat treatment for one hour at 1573 K in air. The microstructure of the heat treated specimen with 1 wt.% Y_2O_3 showed the growth of a columnar structure, which contributes to the increased strength. The specimen with 3 wt.% Y_2O_3 showed greater bridging of the columnar structure than that with 1 wt.% Y_2O_3. The specimen with 5 wt.% Y_2O_3 had insufficient strength because of the over-growth of grains. It seems that a suitable addition of Y_2O_3 increased the strength by control of the over-growth of grains and activation of the columnar structure. [10] Furthermore, the strength of the specimen

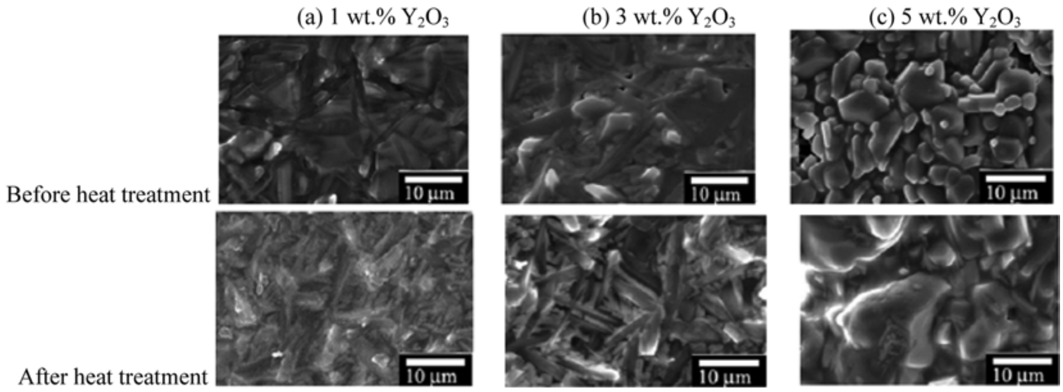

Fig. 2. SEM images. Smoot specimens of before-and-after heat treatment for 1 h at 1573 K.

with 3 wt.% Y_2O_3 was affected by the healing of cracks that occurred during the heat treatment. From Figs. 1 and 2, the optimum conditions for the crack-healing can be defined as being 3 wt.% Y_2O_3.

The σ_B of the crack-healed specimen at elevated temperatures is very important for the practical application at high temperatures. Ceramics are generally used at temperatures exceeding 1273 K. All the cracks in the as-cracked specimens were healed by treatment in air for one hour

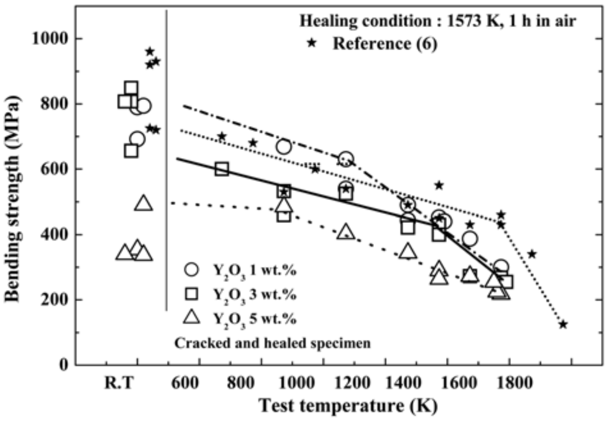

Fig. 3. Effect of test temperature on the bending strength of crack healed specimens.

at 1573 K. The σ_B of crack-healed specimens were measured at elevated temperatures.

Fig. 3 shows the effect of the testing temperature on the σ_B of the crack-healed specimens. The crack healing conditions were standard; 1573 K, one hour and in air. The crack healing was caused by the following chemical reaction [11]; $SiC + 2O_2 = SiO_2 + CO_2$ (CO).

In Fig. 3, all symbols show the σ_B of the crack-healed specimens. The ó$_B$ of the specimens was observed to gradually decrease as the testing temperatures were increased to 1073, 1373 and 873 K with 1, 3 and 5 wt.% Y_2O_3, respectively. However, the σ_B decreased drastically when the testing temperature was increased above 1073, 1373 and 873 K with 1, 3 and 5 wt.% Y_2O_3, respectively. At the stated temperatures, the ó$_B$ were about 590, 415 and 485 MPa, respectively. The symbol(*) was obtained for the specimen without the addition of Y_2O_3. [6] The heat-resistance limit temperature of the crack-healed specimen without Y_2O_3 was 1573 K, with a σ_B of about 420 MPa. The heat-resistance limit temperature of the crack-healed specimen without Y_2O_3 was higher than that of the crack-healed specimen with 3 wt.% Y_2O_3. However, both the σ_B had similar values. From these results, the optimum heat-resistance limit temperature of the specimen with the addition of Y_2O_3 powder can be defined as being 1373 K for the 3 wt.% specimen.

Fig. 4 shows the fractography both before and after

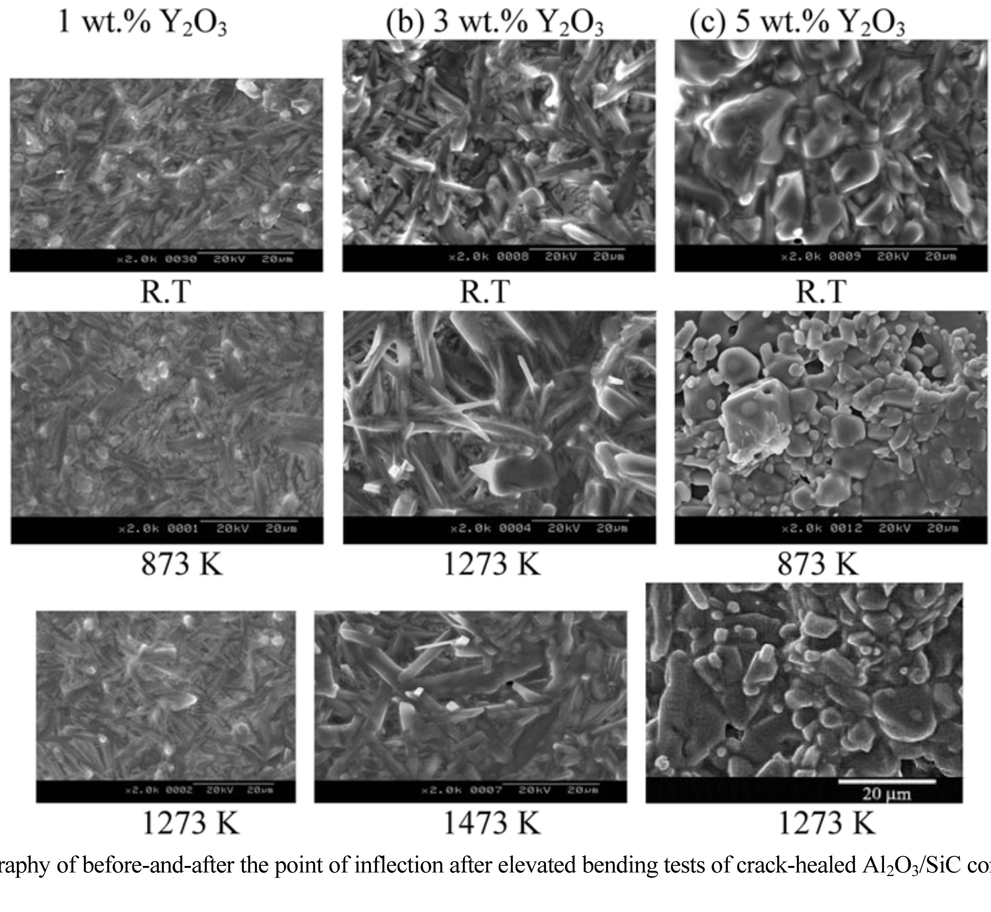

Fig. 4. Fractography of before-and-after the point of inflection after elevated bending tests of crack-healed Al_2O_3/SiC composite ceramics at 1,573 K, 1 h.

the point of inflection for the specimens tested at high temperatures. The microstructure of the specimen with 1 wt.% Y_2O_3 hardly changed, with only a few columnar structures observed. The strength of the specimen with 3 wt.% Y_2O_3 decreased due to the large grains resulting from the growth and agglomeration of the columnar structure. The strength of the specimen with 5 wt.% Y_2O_3 suddenly decreased due to the formation of oversized grains at high temperatures.

Conclusions

In the in-situ observations, the specimen with 3 wt.% Y_2O_3 showed superior crack-healing ability. The bending strength of the smooth and healed smooth specimens with 1 and 3 wt.% Y_2O_3 were almost identical, but the bending strengths of both the 5 wt.% Y_2O_3 specimens were very poor; approximately 48% of the 1 and 3 wt.% specimens. The crack-healed specimen with 3 wt.% Y_2O_3 treated for one hour at 1473 K had a very high average bending strength, indicating complete crack healing, and that the crack-healing temperature could be lowered by the addition 3 wt.% Y_2O_3. The bending strength of the specimens gradually decreased with increasing test temperature, but decreased drastically when the test temperatures were increased above 1073, 1373 and 873 K with the 1, 3 and 5 wt.% Y_2O_3 specimens, respectively. The heat-resistance limit temperatures of the crack-healed specimen with the addition of Y_2O_3 powder were concluded to be 1073, 1373 and 873 K for the specimens with 1, 3 and 5 wt.% Y_2O_3, respectively.

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