SELF-CRACK-HELING BEHAVIOR AND FRACTURE STRENGTH OF AL_2O_3/SIC COMPOSITES AT THE HIGH TEMPERATURE

Kotoji ANDO, Shin KODAMA, Masahiro YOKOUCHI, Sang-Kee LEE,
Wataru NAKAO and Koji TAKAHASHI
Department of Energy & Safety Engineering, Yokohama National University
79-5 Tokiwadai, Hodogaya-ku, Yokohama, 240-8501, Japan
e-mail:andokoto@ynu.ac.jp

Abstract

For new approach to improve reliability of structural ceramics, the present authors have developed Si_3N_4 and mullite ceramics having self-crack-healing ability. In this study, mechanical properties at high temperature and crack-healing ability of alumina / SiC particles composite, alumina / SiC whiskers composite, and alumina / SiC particles / Y_2O_3 composites were investigated. Excellent crack-healing ability so that fracture strength reduced by cracking is recovered above that of as-received specimen was endowed for the composites. Fracture strength of alumina / SiC particles composite and alumina / SiC particles / Y_2O_3 composites were 1.8 and 3.0 times larger than the value of monolithic alumina, respectively, because alumina grains after sintering were downsized. Moreover, fracture strength of alumina / SiC whiskers composite was improved up to 2.5 times larger than the value of monolithic alumina by bridging effect of SiC whiskers. The heat-resistance limit temperature for strength, T_{HR}, of the crack-healed alumina / SiC particles composite was improved to 1300 ^oC from 900 ^oC by nano-compositing. Fracture toughness of alumina / SiC whiskers composite was found to be 1.6 times higher than that of monolithic alumina. Bridging and pull-out effects by SiC whiskers improved the fracture toughness.

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. On the other viewpoint to improve reliability of structural ceramics, the present authors [1-12] have proposed endowing the 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 [1-3, 10], the present authors succeeded endowing the excellent crack-healing ability to mullite by admixing with 15 mass% SiC particles. The fracture strength of the mullite composite was reduced almost quarter by cracking, and recovered completely by crack-healing at 1300 ^oC for 1 h. Moreover, it was found that mullite admixed with 15 vol%

SiC whiskers [13] had crack-healing ability as well as two times higher fracture toughness than monolithic mullite.

In this study, alumina / SiC particles composite and alumina / SiC whiskers composite are prepared, and mechanical properties at high temperature and crack-healing ability of the composites are investigated. Moreover, alumina / SiC particles / Y_2O_3 composites are prepared for improvement in fracture strength at high temperature. Also mechanical properties at high temperature and crack-healing ability of alumina / SiC particles / Y_2O_3 composites are investigated.

2. Experimental

Alumina admixed with 15 mass% SiC particles, called as A15SP, alumina admixed with 20 mass% SiC whiskers, called as A20SW, and alumina admixed with 15 mass% SiC particles and 2 mass% Y_2O_3, called as A15SP2Y, were prepared. α-Alumina (α-Al_2O_3) powder (AKP-20, Shumitomo Chemicals Co. Ltd., Japan) used in this study has an average particle size of 0.5 μm. SiC powder (Ultrafine grade, Ibiden Co. Ltd., Japan) used has a mean particle size of 0.27 μm. 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 fixed each composition was mixed well in isopropyl alcohol for 12 h using alumina balls and an alumina mill pot. Rectangular plates of 50 mm ×50 mm × 9 mm were sintered in Ar via hot press under 40 MPa. The sintered conditions of A15SP, A20SW and A15SP2Y were (1600 ^{o}C, 4 h), (1850 ^{o}C, 1 h) and (1600 ^{o}C, 1 h), respectively. The sintered plates were cut into the 3 mm × 4 mm × 22 mm rectangular specimens bar. The specimens were polished to mirror finish on one face and the edges of specimens were beveled 45°, as shown in Fig. 1, 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 a/c) was 0.9. The specimens cracked and as-received were subjected to crack-healing treatment at 1300 ^{o}C for 1 h in air.

All fracture tests of the specimens crack-healed were performed on a three-point loading system with a span of 16 mm, as shown in Fig. 1, at room temperature and temperatures from 600 ^{o}C to 1300 ^{o}C. The cross-head speed in the monotonic test was 0.5 mm/min. Fracture toughness was obtained by indentation fracture method, where Young's modulus used to calculation is 380 GPa [14].

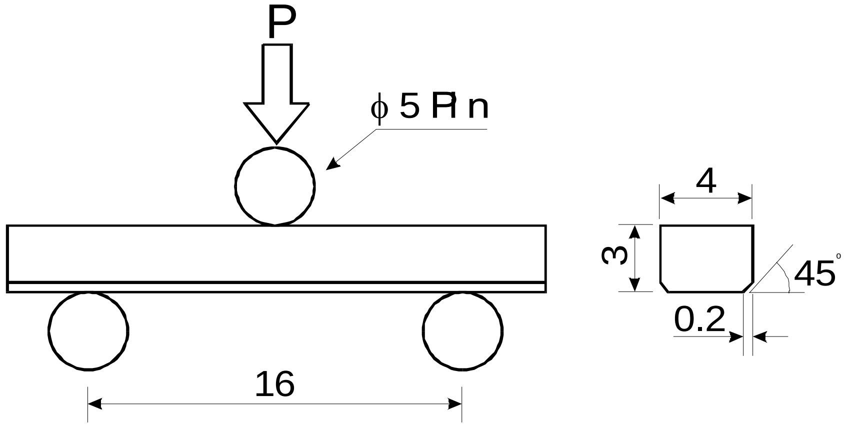


Fig. 1 Three point bending specimen and test system, dimensions in mm

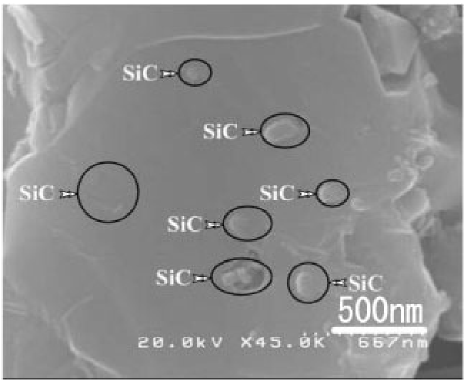
3. Results and discussions

3.1. Microstructure of SiC reinforced alumina.

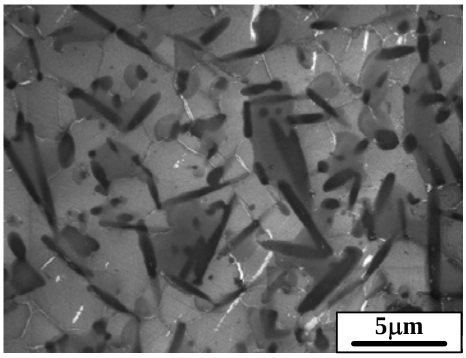
Figure 1 shows the microstructure of (a) A15SP, (b) A20SW and (c) A15SP2Y. From the SEM images, the grain size of A15SP, A20SW and A15SP2Y was found to be 1-2 μm, 5-6 μm and 0.3-0.5 μm, respectively. SiC particles prevented the growth of Al_2O_3 grain. Moreover, admixing Y_2O_3 were reduced the sintered time. Thus, A15SP2Y could be sintered before growing Al_2O_3 grains. Many SiC particles of A15SP located inside the Al_2O_3 grains. In contrast, all SiC particles of A15SP2Y located on the grain boundaries of Al_2O_3. SiC whiskers in A20SW existed along Al_2O_3 grain boundaries.

3.2. Mechanical properties of crack-healed specimens.

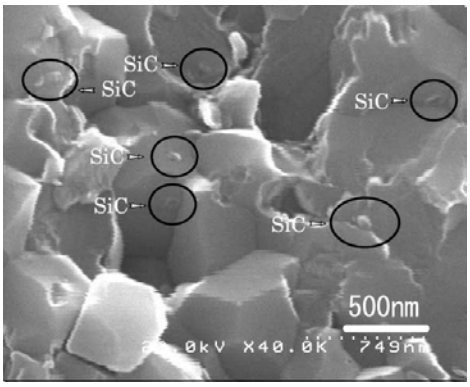
Fracture strengths of all cracked specimens were reduced to half values of each as-received specimen. By crack-healing, fracture strength of A15SP, A20SW and A15SP2Y were improved above the values of each as-received specimen, because small flows containing as-received specimens by machining and polishing were also crack-healed. Thus, it is confirmed that the fracture strengths of the crack-healed specimens were the intrinsic values of the sintered. Fracture strengths of A15SP, A20SW and A15SP2Y were found to be 720 MPa, 1000 MPa and 1200 MPa, respectively. The values were 1.8, 2.5 and 3.0 times larger than the value of monolithic alumina, respectively. It is confirmed that the improvement in the fracture strength for A15SP and A15SP2Y resulted from downsizing alumina grains. Also it is confirmed that bridging effect by SiC whiskers resulted in the improvement in the fracture



(a) A15SP



(b) A20SW



(c) A15SP2Y

Fig. 2 Microstructure of (a) A15SP, (b) A20SW and (c) A15SP2Y strength for A20SW.

Figure 4 shows the temperature dependence of the fracture strengths of the crack-healed specimens. The cross, the open circle and the closed triangle indicate the fracture strengths of A15SP, A20SW and A15SP2Y, respectively. Also the dash line indicates that of monolithic alumina. The fracture strength of A15SP decreases gradually up to 1300 °C, above which it decreases abruptly. A15SP did not exhibited plastic deformation below 1300 °C. However, considerable plastic deformation occurred above 1400 °C. The fracture strength is evaluated using the elastic theory, so that fracture strength has been overestimated if a specimen

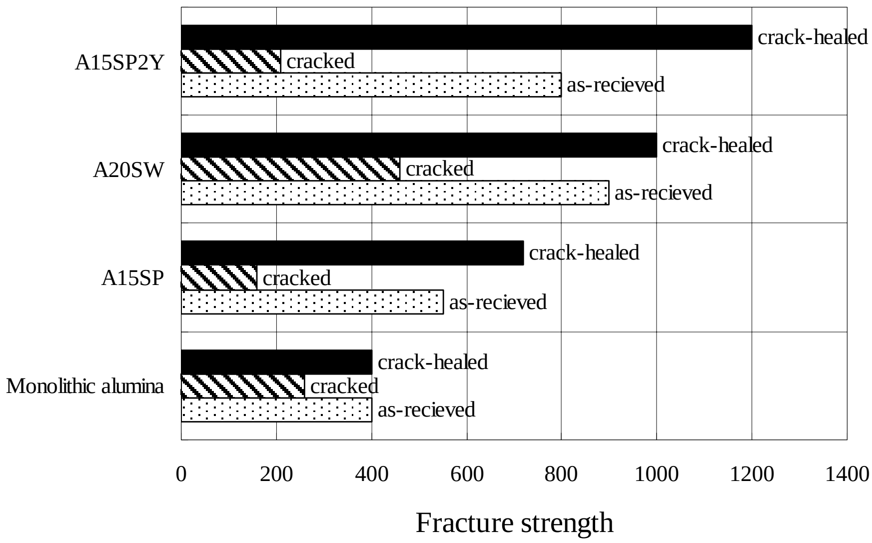


Fig. 3 shows the bending strength at RT of the as-received, cracked and crack-healed specimens, with the values of the monolithic alumina.

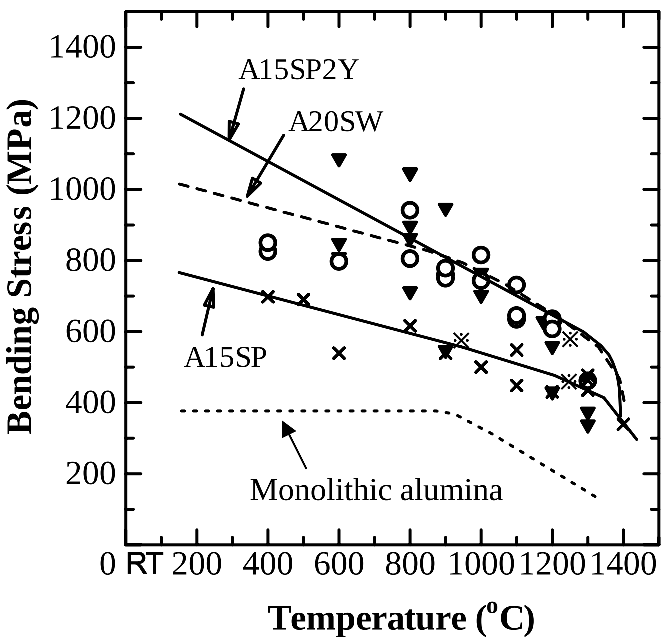


Fig. 4 Temperature dependence of the bending strength of (a) A15SP, (b) A20SW and (c) A15SP2Y crack-healed at 1300 °C for 1 h

Table 1 shows the fracture toughness of A15SP, A20SW and A15SP2Y, with the value of the monolithic alumina.

	Monolithic alumina	A15SP	A20SW	A15SP2Y
Fracture Toughness [$MPam^{1/2}$]	4.0	3.5	6.5	3.3

exhibited plastic deformation. Therefore, the heat-resistance limit temperature for strength, T_{HR}, of the crack-healed A15SP was determined to be 1300 ^{o}C. Using the same estimation, the values of T_{HR} for the crack-healed A20SW and A15SP2Y were determined to be 1200 ^{o}C. Also the value for monolithic alumina was determined to be 900 ^{o}C. The improvements in T_{HR} were attained by nano-compositing.

Table 1 shows the fracture toughness of A15SP, A20SW and A15SP2Y, with the value of the monolithic alumina. Fracture toughness of A20SW was found to be 1.6 times higher than that of monolithic alumina. Bridging and pull-out effects by SiC whiskers improved the fracture toughness of A20SW. Fracture toughness of A15SP and A15SP2Y were slightly reduced to that of monolithic alumina. Small grain size of A15SP and A15SP2Y results in decreasing the fracture toughness.

4. Conclusion

For development of alumina based ceramics having good mechanical properties as well as excellent crack-healing ability, alumina / SiC particles composite, A15SP, alumina / SiC whiskers composite, A20SW, and alumina / SiC particles / Y_2O_3 composites, A15SP2Y, are prepared, and mechanical properties at high temperature and crack-healing ability of the composites are investigated. The following conclusions were derived from the obtained results.

1. Excellent crack-healing ability so that fracture strength reduced by cracking is recovered above that of as-received specimen was endowed for A15SP, A20SW and A15SP2Y.
2. Fracture strengths of A15SP, A20SW and A15SP2Y were found to be 720 MPa, 1000 MPa and 1200 MPa, respectively. The values were 1.8, 2.5 and 3.0 times larger than the value of monolithic alumina, respectively. It is confirmed that the improvement in the fracture strength for A15SP and A15SP2Y resulted from downsizing alumina grains. Also it is confirmed that the improvement in the fracture strength for A20SW resulted from bridging effect by SiC whiskers.
3. The heat-resistance limit temperature for strength, T_{HR}, for the crack-healed A15SP was determined to be 1300 ^{o}C. Also the values of T_{HR} for the crack-healed A20SW and A15SP2Y were determined to be 1200 ^{o}C. The value for monolithic alumina was 900 ^{o}C. The improvements in T_{HR} were attained by nano-compositing.
4. Fracture toughness of A20SW was found to be 1.6 times higher than that of monolithic alumina. Bridging and pull-out effects by SiC whiskers improved the fracture toughness of A20SW.

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