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Crack-Healing Behavior of SiC with Additive SiO_2 Colloid

Seok-Hwan Ahn*, Chang-Kwon Moon** and Ki-Woo Nam**†

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Abstract: This study focuses on the crack-healing behavior and bending strength of SiC ceramics with sintering additives of SiO_2colloid. Optimized crack-healing condition was found to be 1hr at an atmosphere of 1373 K. The maximum crack size that can be healed at the optimized condition was a semi-elliptical surface crack of 450 μm in diameter. After heat treatment at the optimum temperature in air, the crack morphology almost entirely disappeared and the strength recovered to the value of the smooth specimens at room temperature for the investigated crack sizes up to 450 ㎛. The crack with width 1.4 ㎛ can be completely heal the surface crack.

Key Words : Crack-Healing, SiC, Additive, SiO_2 Colloid, Crack Length, Crack Width

1. Introduction

Silicon carbide (SiC) has good high temperature strength and is resistant to radioactivity. It is a leading candidate material for future generation gas turbines and the inner containment of nuclear fusion reactors.[1-3] The demand is for the highest structural integrity.

A critical semi-circular crack diameter of SiC is about 20. 50 ㎛, the strength is reduced to about 50% by this crack, thus it is almost impossible to detect the crack with high reliability. Then the application of the above (a) technology to SiC structure is almost impossible. There are much useful researches related to the above (b) technology. However, a crack-healing ability is assumed to be the pre-eminent technology similar to (b) above. It is well known that SiC exhibits a very interesting crack-healing ability.[4-8] However, very few studies have been made on the crack-healing behavior systematically, as a function of crack-healing temperature and time, crack-healing environment and chemical composition of SiC, and also very few studies have been made on the fracture behavior of crack-healed components, such as bending strength and fatigue strength at elevated temperature.

To meet this demand, the technology is very useful to introduce self-crack-healing ability. To overcome this, many studies[9-13] have attempted to achieve the crack healing such as SiC, Si_3N_4, Al_2O_3-SiC composites, mullite-SiC composites, and Si_3N_4-SiC composites. In this regard, while SiC is a ceramic that has a high potential for crack-healing, very few studies have been conducted on its crack-healing behavior.

**† Ki-Woo Nam(corresponding author) : Department of Materials Science and Engineering, Pukyong National University.
E-mail : namkw@pknu.ac.kr, Tel : 051-629-6358
*Seok-Hwan Ahn : Department of Mechatronics, Jungwon University.
**Chang-Kwon Moon : Department of Materials Science and Engineering, Pukyong National University

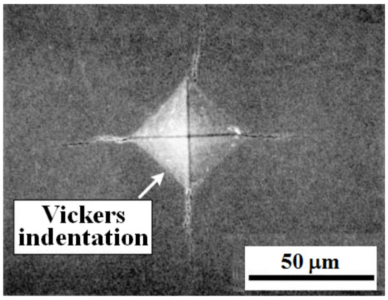
This study investigates the crack-healing behavior of SiC ceramics with additive SiO_2 colloid, and identified the critical width for crack-healing.

2. Materials and Experimental method

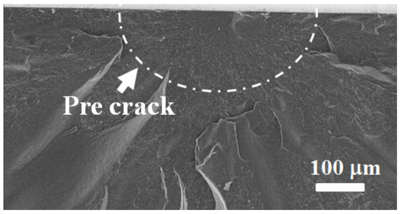
An ultra-fine SiC powder (mean size = 0.27 ㎛) was employed for this experiment. The Al_2O_3 powder and Y_2O_3 powder were 0.1 ㎛ and 33 nm of mean size, respectively. The SiO_2 employed consisted of a colloid (SiO_2 12 wt.% solid solution). The SiC ceramics were prepared using a mixture of 85 wt.% SiC powder and sintering additives (Al_2O_3 + Y_2O_3 = 12 wt.% (Al_2O_3 : Y_2O_3 = 60 : 40) and SiO_2 = 3 wt.%). Powders were milled in isopropanol using planetary mill (premium line) for 24 hours using Si_3N_4 balls(ϕ5). The mixture was placed in a 363 K furnace to extract solvent and to make a dry powder mixture. The mixtures were hot-pressed in N_2 for 60 minute via hot-pressing under 35 MPa at 2053 K.

The sintered plate was cut into test specimens measuring 3 x 4 x 18 mm. The specimens were polished to a mirror finish on one face, and the edges of specimens were beveled 45^o to reduce the likelihood of edge-initiated failures. A semi-elliptical surface crack was made at the center of the tension surface of the test specimen with a Vickers hardness (HV-112) using a load of 9.8 ~ 196 N. In this manner, semicircular cracks of 100 to 550 ㎛ in diameter were, as shown in Fig. 1(a), created. A micrograph of crack profile observed by scanning electron microscopy (SEM) is shown in Fig. 1(b).

All fracture tests were performed on a three point loading system with a span of 16 mm at room temperature. The cross head speed in the monotonic test was 0.5 mm/min. Crack-healing appearance was observed by FESEM.



(a)



(b)

Fig. 1 SEM micrographs of (a) indentation crack, (b) crack shape

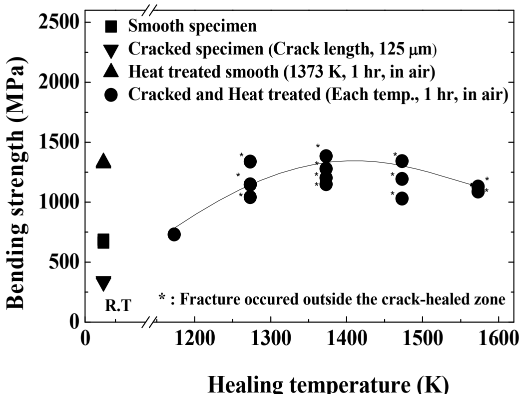


Fig. 2 Effect of healing temperature on the strength of the healed specimen

The condition of crack-healing has a large effect on fracture strength. The crack-healing time in air was one hour at various temperatures ranging from 1173 K to 1483 K. Cooling was spontaneous in the furnace. The crack-healed specimens were subsequently tested in three point bending at a cross head speed of 0.5 mm/min, using a fixture with a span of 16 mm. After testing, all specimens were inspected to ensure that failures had initiated from the indentation sites by optical microscope. FESEM was used to investigate crack-healed surface.

3. Experimental results and discussion

Fig. 2 shows the effect of the crack-healing temperature on bending strength at room temperature. The strengths of smooth specimen were found to be 720 MPa. The strengths of the cracked specimens with 2c≈125 ㎛ were 353 MPa. As such, the strengths of the cracked specimens were 49 % of that of the smooth specimens.

Each material generally has an optimum crack healing temperature at which the maximum strength has been obtained. After healing from 1173 to 1473 K, not only had the cracked specimens recovered to the strength similar to that of the smooth specimens, but in most cases had actually surpassed it. Based on the room temperature strength, the optimum healing temperature of cracked specimens was found to be 1373 K. The bending strengths of the crack healed specimens at 1373 K were found to be 1337 MPa. This constituted a 179 % increase over the value achieved by the smooth specimens. The bending strength of the smooth healed specimens at 1373 K was revealed to 1427 MPa. This marked a respective increase of 195 % and 198 % over the value achieved by the smooth specimens.

The dependence of strength on the surface crack size (2c) for the crack-healing behavior is shown in Fig.3. The strengths of the cracked specimens were half of that of the smooth specimens. The strengths of the cracked specimens where healing did take place decreased linearly as the surface crack length increased. The crack healed specimens exhibited far higher strength than the smooth specimens when the crack length was ≤350 μm. Although both materials were fractured at the crack healed part at a crack length 2c≒ 350 μm, they nevertheless displayed a good strength property. These result indicate that the SiO_2 colloid of additive material investigated may be able to heal cracks of up to nearly 2c≒ 450 μm.

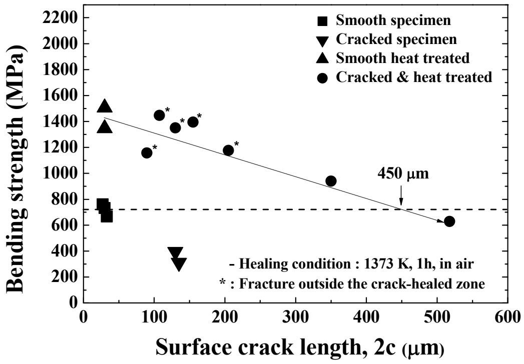


Fig. 3 Dependence of strength on surface crack length for crack-healed specimens

To investigate this result in detail, Fig. 4 shows FESEM images of crack-part before and after crack-healing. The crack width also gradually

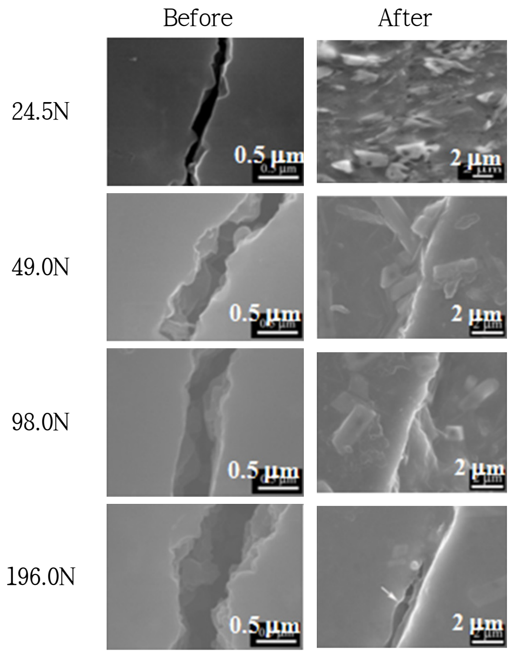


Fig. 4 FESEM micrographs of crack width. (healing condition : 1373 K, 1 h in air)

Fig. 5 shows the relationship between crack length and crack width. As the Vickers indentation load increased, crack length and crack width also increased linearly. The surface crack length of the crack-healing possibility was about 2c ≒ 450 μm from figure 3. The crack-width at that time was about 1.4 μm from figure 5. These results show that this is the critical size of crack-healing possibility, which indicates that crack-width is very important to crack-healing. That is, if the crack-width is below 1.4 μm, the material is likely to heal even if crack-length is above 450 μm.

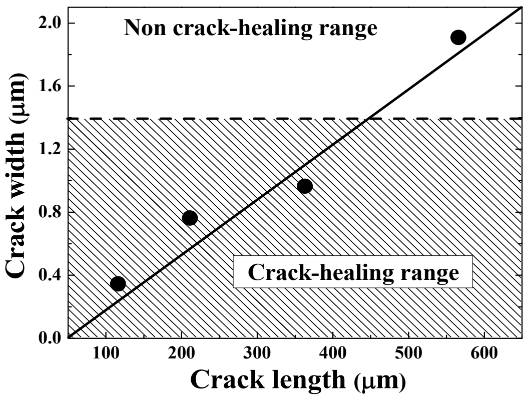


Fig. 5 Relationship between surface crack length and crack width of healing possibility

4. Conclusions

The crack-healing behaviors of SiC ceramics featuring different sintering additives were investigated. The optimum crack-healing condition at 2c≒ 125 μm was found to be for one hour at an atmospheric level of 1373 K, and crack-healed specimens at this temperature had a high crack-healing ability. The crack-healing is mainly dependent on crack width, not crack length. If the crack width of SiC ceramics is below 1.4 μm, surface cracks can heal regardless of the surface crack length.

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