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Crack healing of SiC according to SiO_2 colloid coating methods

Kiwoo Nam[a], Heerok Jeong[b], Jongwon Kim[c] and Seokhwan Ahn[d,]*
[a]*Department of Materials Science and Engineering, Pukyong National University, 365, Sinseonro, Nam-ku, Busan 48547, Korea*
[b]*Prepoll Co. Ltd, 1151-1 Daejeo 2-dong, Gangseo-gu, Busan 46719, Korea*
[c]*Busan Technopark, 1277 Jisa-dong, Gangseo-gu, Busan 46742, Korea*
[d]*Department of Mechatronics, Jungwon University, Chungbuk 28024, Korea*

The crack-healing behavior of SiC ceramics with a large crack width has been studied as functions of coating and heat-treatment. The SiO_2 colloid coating was carried out in two types of hydrostatic pressure coating and roll coating. The crack-healing was carried out one hour at 1173 K in air. The crack part formed SiO_2 oxides until the critical time through a hydrostatic pressure method. The crack did not heal anymore if it exceeded the critical times. The crack part and the base part have many O components and Si components regardless of the times of coating and heat treatment. Furthermore, the combined hydrostatic and rolling coating method did not have nearly an effect on crack-healing for a large crack width over 1.4 μm. A study for more effective healing of a large crack width must be carried out in the future.

Key words: Crack Healing, SiC Ceramics, SiO_2 Colloid, Coating Method.

Introduction

Due to a combination of unique properties, silicon carbide (SiC) ceramics are used in extensive applications in several fields of engineering as materials for advanced energy systems, such as high performance combustion systems, fuel-flexible gasification systems, fuel cell/turbine hybrid systems, nuclear fusion reactors and high temperature gas-cooled fission reactors [1-4]. In particular, the SiC/SiC composite material is under study as the first wall material of the blanket due to its excellent heat-resistance and low activation property [5-9]. Further, many studies are being conducted in order to solve the brittle nature of ceramics [10-14], such as (1) toughening by microstructure control and fiber/particles dispersion, (2) detection and repair of crack by nondestructive testing, and (3) crack-healing method. It has also been reported that the cracks formed during machining were healed completely [15, 16]. In particular, some results suggest that the cracks in the silicon carbide, once healed, surprisingly became even stronger than the original silicon carbide. Hence, they concluded that crack length is an important factor of crack-healing by oxidation in silicon carbide [17]. However, there has been no clear explanation regarding the effect of SiO_2 colloid coating for cracks.

In this paper, the SiC ceramic with sintering additives, Y_2O_3 and Al_2O_3, was prepared. We observed the effect of coating method and coating times in relation to crack-healing in SiC ceramic and examined the effect of the crack width for crack-healing.

*Corresponding author:
Tel : +82-43-830-8942
Fax: +82-43-830-8115
E-mail: shahn@jwu.ac.kr

Materials and test Methods

Commercially available SiC (Ultrafine grade, Ibiden Co., Japan), Al_2O_3 (AKP-700, Sumitomo Chemical Co. Ltd., Japan) and Y_2O_3 (CI Chemical Co., Japan) were used as the starting materials. The mean particle sizes of the SiC, Al_2O_3 and Y_2O_3 powders were 0.27 μm, 0.1 μm and 31 nm, respectively.

The SiC ceramic was prepared using a mixture of 90 wt.% SiC powder and sintering additives (Al_2O_3 + Y_2O_3 = 10 wt.%). An individual batch was milled in isopropanol for 24 hrs using the SiC ball (φ5). The mixture was placed in a 363 K furnace in order to extract the solvent as well as to make a dry powder mixture. The dry powder was then passed through a 106 μm sieve. The mixtures were subsequently hot-pressed in N_2 gas for one hour via hot-pressing conducted under 35 MPa at 2053 K. A flowchart of the sintering process is presented in Fig. 1.

For the crack-healing test, the crack was made in the center of the polished face of the specimen by a Vickers indentation in air. The crack-healing by the SiO_2 nano-colloid coating had a large effect on fracture strength. The colloid coating was carried out in two types of methods, hydrostatic pressure coating and roll coating. The crack-healing was carried out one hour at 1173 K in air. Cooling was spontaneous in the furnace. In order to analyze the effect of coating and heat treatment, both were carried out for up to three times. To investigate the crack-healed surface and the

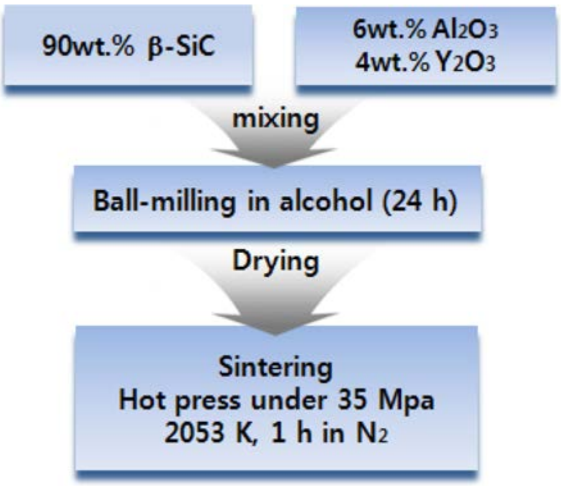


Fig. 1. A flowchart of the sintering process.

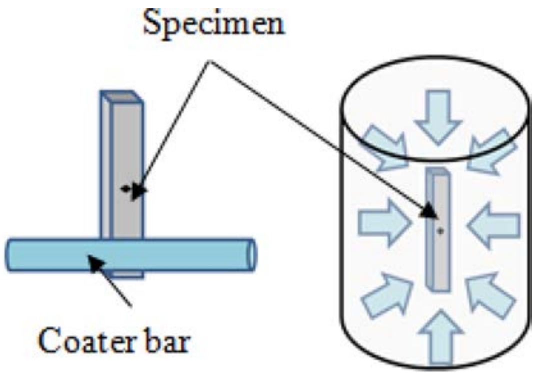


Fig. 2. Two types of coating method. (a) Roll coating, (b) Hydrostatic pressure coating.

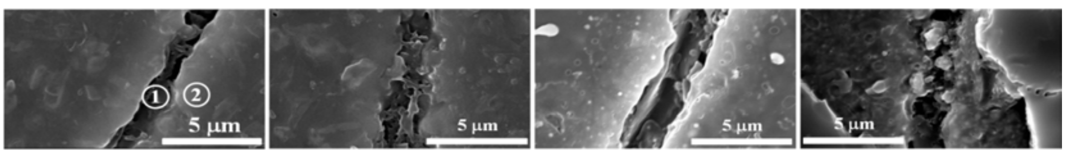
component analysis of crack healing substances, SEM (scanning electron microscope), SPM (scanning probe microscope) and EDX (Energy Dispersive X-ray) analyses were used.

Results and Discussion

All surface cracks of SiC ceramic could heal in the cases in which the cracks are smaller than a crack width of 1.4 μm and a crack length of 450 μm [17]. In order to investigate the healing effect for a larger crack width, this study applied the hydrostatic pressure method as a way to penetrate the SiO_2 nano-colloid. The cracks are healed by being filled with amorphous silica that is produced by the oxidation of silicon carbide [15, 18].

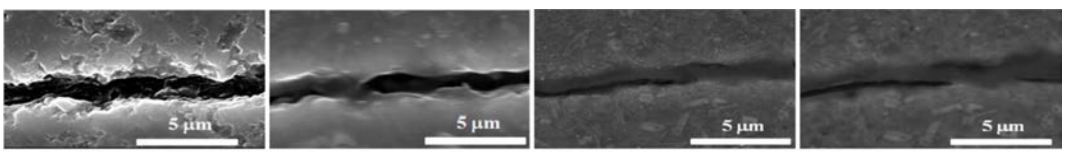
Fig. 3 indicates the surface crack of before-and-after the heat treatment. This infiltrated the SiO_2 nano-colloid at the crack using the hydrostatic pressure method. Fig. 3(a) shows a crack width of about 2 μm. Fig. 3(b) shows the crack shape of crack-healing using the hydrostatic pressure coating one time. In Fig. 3(b), it was difficult to expect crack-healing with a coating of one time. Although the crack part was made of a cross link, the crack did not heal completely. Therefore, in order to infiltrate the cracked part with SiO_2 nano-colloid, the coating and heat treatments were repeated. Fig. 3(c) was carried out the coating twice using the hydrostatic pressure method. Although crack-healing was not perfect, Fig. 3(c) had more healing than Fig. 3(b). In Fig. 3(d), although it underwent the process three times, did the amount of SiO_2 oxide did not increase. We observe that the surface condition is fairly rough. Therefore, if the coating is repeated until the critical times by hydrostatic pressure, SiO_2 oxide can be formed densely. However, if it exceeds the critical times, the crack is judged to no longer heal.

Figs. 4-6 carried out rolling coating after hydrostatic pressure coating. Figs. 4-6(a) show a crack width of about 1.8, 2.0 and 4.05 μm, respectively. Figs. 4-6(b-d) carried out the coating and heat treatments once, twice



(a) (b) (c) (d)

Fig. 3. The surface image of crack-healing on about 2 μm of crack width with SiO_2 nano-colloid coating by the hydrostatic pressure method. (a) Before crack-healing, (b) One time of crack-healing after one time coating, (c) Two times of crack-healing after each time coating, (d) Three times of crack-healing after each time coating.



(a) (b) (c) (d)

Fig. 4. The surface image of crack-healing on about 1.8 μm of crack width with SiO_2 nano-colloid coating by rolling coating method after hydrostatic pressure method and. (a) Before crack-healing, (b) One time crack-healing, (c) Two times crack-healing, (d) Three times crack-healing.

Table 1. Surface elemental analysis of crack-healing part of Fig. 3

Condition \ Element		O (wt.%)	Al (wt.%)	Si (wt.%)	Y (wt.%)
(a) One time	①	15.43	1.80	81.08	1.69
	②	18.30	3.39	78.07	0.25
(b) Two times	①	21.36	2.80	75.59	0.26
	②	31.22	4.80	62.25	1.73
(c) Three times	①	39.14	1.32	58.14	1.40
	②	32.53	4.70	60.36	2.41

Table 2. Surface elemental analysis of crack-healing part of Fig. 6

Condition \ Element		O (wt.%)	Al (wt.%)	Si (wt.%)	Y (wt.%)
(a) One time	①	24.91	2.32	72.54	0.24
	②	13.67	3.31	82.79	0.23
(b) Two times	①	57.02	0.33	41.53	1.11
	②	34.92	2.72	59.66	2.69
(c) Three times	①	56.54	0.40	42.02	1.04
	②	34.60	2.24	60.25	2.91

and three times, respectively. In Figs. 4-6(b), although the reduction of the crack width by SiO_2 oxides was significantly not observed, the inside crack formed a cross link and moreover, the width was reduced. Figs. 4-6(c) still has a crack, but the crack is observed to have healed more than once. However, Figs. 4-6(c-d) did not show much difference.

Tables 1 and 2 show the surface elemental analysis of the crack-healing part of Figs. 3 and 6. In Figs. 3 and 6, amount of Si decreased according to the increase in the number of times and the coating method. This result is

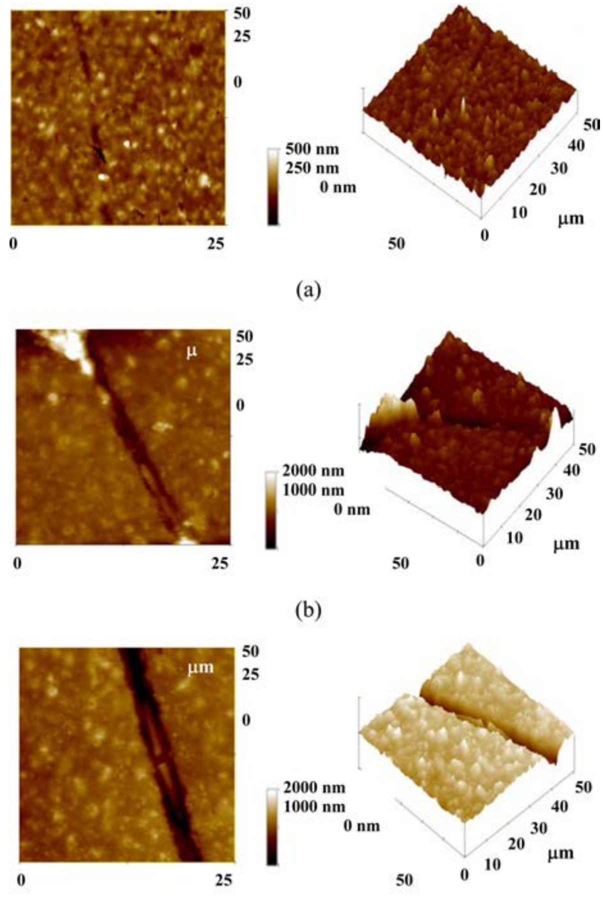
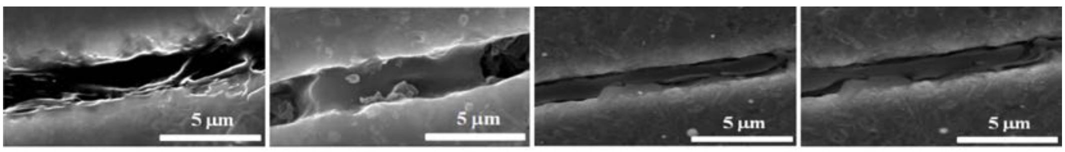
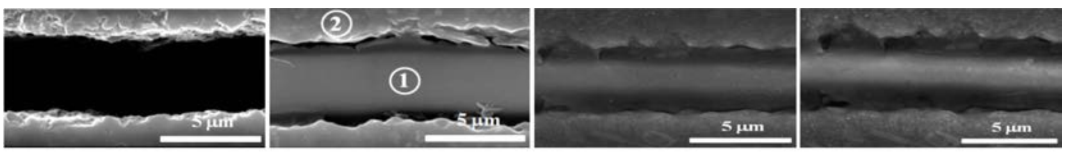


Fig. 7. The SPM image of crack part after coating and heat treatment. (a) Crack width of about 1 µm with crack-healing after roll coating of one time, (b) Crack width of about 2.0 µm with crack-healing for hydrostatic pressure method and roll method of three times, (c) Crack width of about 4.05 µm with crack-healing for hydrostatic pressure method and roll method of three times.



(a) (b) (c) (d)

Fig. 5. The surface image of crack-healing on about 2.0 µm of crack width with SiO_2 nano-colloid coating by rolling coating method after hydrostatic pressure method and. (a) Before crack-healing, (b) One time crack-healing, (c) Two times crack-healing, (d) Three times crack-healing.



(a) (b) (c) (d)

Fig. 6. The surface image of crack-healing on about 4.05 µm of crack width with SiO_2 nano-colloid coating by rolling coating method after hydrostatic pressure method and. (a) Before crack-healing, (b) One time crack-healing, (c) Two times crack-healing, (d) Three times crack-healing.

due to the fact that the crack and base parts are defective by the formation of oxides due to excessive heat treatment. The amount of Si decreased according to the increase in the number of times and the coating method. This result is due to the fact that the crack and base parts are defective by the formation of oxides due to excessive heat treatment. Therefore, if the crack width is wider, crack-healing also hardly has an effect by repeated coating and heat treatment of a variety of ways.

The formation of SiO_2 oxides at the crack part was observed by SPM. Figs. 7(a,b,c) show 1.0, 2.0 and 4.05 μm of crack widths, respectively. Fig. 7(a) has been completely crack healed by roll coating one time. However, in Figs. 7(b) and 7(c), SiO_2 oxides were also locally formed at the crack part for three times. Hence, it can be see that this has not been completely crack-healed compared to Fig. 7(a). Therefore, it was determined that the hydrostatic and rolling coating methods do not have a large effect on crack-healing for SiC ceramics with a large crack width. For a more effective healing of a large crack width, the study on width and depth of crack as well as on the coating method of SiO_2 nano-colloid solution must be carried out in the future.

Conclusions

The crack-healing behaviors of SiC ceramic were investigated effect by the different coating methods, that is, hydrostatic pressure method and roll method. If the coating is repeated until the critical times by the hydrostatic pressure method, the crack part forms SiO_2 oxides. However, if it exceeds the critical times, the crack no longer heals. The crack part and the base part have many O components and Si components regardless of the times of coating and heat treatments, respectively. Further, the amount of O increases and the amount of Si decreases according to the increase in time. The combined hydrostatic and rolling coating method does not have a large effect on crack-healing for SiC ceramics with a large crack width over 1.4 μm. A study regarding more effective healing of a large crack width must be carried out in the future.

References

1. A. Fujishima, K. Hashimoto, and T. Watanabe, "TiO_2 Photocatalysis: Fundamentals and Applications" (BKC, Inc., 1999)
2. A. Fujishima, and K. Honda, Nature 238 (1972) 37-38.
3. R. Kurihara, S. Ueda, S. Nishio, and Y. Seki, Fusion Engineering and Design 54 (2001) 465-471.
4. A. Hasegawa, M. Saito, S. Nogami, K. Abe, R. H. Jones, and H. Takahashi, J. Nucl. Mater. 264 (1999) 355-358.
5. N. N. Ault, "Silicon Carbide Ceramics, Structure and Properties" (Encyclopedia of Materials: Science and Technology, Second Edition, 2001) 8502-8508.
6. R. Morrell, Comprehensive Composite Materials 4 (2000) 1-24.
7. B. Harris, Comprehensive Composite Materials 4 (2000) 489-531.
8. E. Kohn, Comprehensive Microsystems (2008) 131-181.
9. T. Hansson, and R. Warren, Comprehensive Composite Materials 4 (2000) 579-609.
10. K. Ando, K. Houjyou, M. C. Chu, S. Takeshita, K. Takahashi, S. Sakamoto, and S. Sato, J. Eur. Ceram. Soc. 22 (2002) 1339-1346.
11. K. Ando, K. Takahashi, S. Nakayama, and S. Saito, J. Am. Ceram. Soc. 85 (2002) 2268-2272.
12. H. S. Kim, M. K. Kim, S. B. Kang, S. H. Ahn, and K. W. Nam, Materials Science and Engineering A 483 (2008) 672-675.
13. K. Ando, M. C. Chu, S. Matsushita, and S. Sato, J. Eur. Ceram. Soc. 23 (2003) 997-984.
14. K. Ando, K. Furusawa, M. C. Chu, T. Hanagata, K. Tuji, and S. Sato, J. Am. Ceram. Soc. 84 (2001) 2073-2078.
15. S. K. Lee, W. Ishida, S. Y. Lee, K. W. Nam, and K. Ando, Journal of the European Ceramic Society 25 (2005) 569-576.
16. K. W. Nam, M. K. Kim, S. W. Park, S. H. Ahn, and J. S. Kim, Materials Science and Engineering A 471 (2007) 102-105.
17. K. W. Nam, and J. S. Kim, Materials Science and Engineering A 527 (2010) 3236-3239.
18. S. K. Lee, W. Ishida, and K. Ando, J. Soc. Mater. Sci. Jpn. 52 (2003) 674-680.