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A fundamental study for the crack healing of SiC ceramics and SiC_f/SiC composite ceramics

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After sintering SiC_f/SiC composite ceramics as a material applied to the blanket structure of a fusion reactor, micro-cracks are created by conducting a Vickers test. The presence of a crack was observed using a SEM and the relation between the bending strength and the orientation of the reinforcing fibers were analyzed. There is no relation between the bending strength of SiC_f/SiC composite ceramics and the size of a pre-crack until a limited crack size. The crack healing behavior was investigated on SiC_f/SiC composite ceramics with a coating of SiO_2 nano-colloid on the surface using various methods, such as dipping and a hydrostatic pressure. Although the coating treatment with SiO_2 nano-colloid is conducted according to the hydrostatic pressure method, SiO_2 oxide is only created at the base of a crack. As a result, the recovery of bending strength cannot be anticipated.

Key words: Crack healing, SiC_f/SiC composite ceramics, SiO_2 Nano-colloid.

Introduction

SiC_f/SiC composite ceramics as heat-resistant structural materials have been researched and continues to be studied throughout the world [1]. In particular, the fracture toughness of SiC/SiC composite ceramics is improved by fabricating SiC fibers coated with pyrolytic carbon [2], and research on the strength evaluation and crack healing is also actively in progress by making various SiC composite ceramics [3]. After sintering SiC_f/SiC composite ceramics as a material applied to the blanket structure of a fusion reactor, micro-cracks are created by conducting a Vickers test. The presence of a crack was observed using a scanning electron microscope (SEM) and the relationship analyzed between the bending strength and the crack orientation. The crack healing behavior was investigated on SiC_f/SiC composite ceramics with a coating of SiO_2 nano-colloid on the surface using various methods, such as dipping and a hydrostatic pressure.

Materials and Test method

The preparation of SiC_f/SiC composites having the dimensions 20 mm × 20 mm × 3 mm was performed by the nano-infiltration transient eutectic phase sintering (NITE) process with Tyranno-SA SiC fibers (Φ7.5 μm, thickness of pyrolytic carbon : 500 nm) as a reinforcement material [4-5]. After mixing 90 wt.% β-SiC nano powder(average particle size 100 nm) of a base powder, 6 wt.% Al_2O_3 (average particle size 0.3 μm) and 4 wt.% Y_2O_3 (average particle size 0.4 μm) as sintering additives at the ratio of 90 : 10, the slurry was impregnated in the SiC fiber preform that was made of about 40~50% by volume. The mixtures were subsequently hot-pressed in an Ar gas atmosphere for one hour via hot-pressing conducted under a pressure of 20 MPa at 2123 K. Two types of specimen were prepared, UCS (unidirectional composite specimen) and CCS (cross composite specimen), according to the orientation of the SiC fibers to investigate the relationship with the bending strength. A monolithic specimen (SPS) was prepared for comparison [6]. After the three types of specimens have been cut into 3.0 × 4.0 × 20 mm sizes from the sintered material, all fracture tests were performed on a three-point loading system with a span of 16 mm and a cross head speed of 0.5 mm/minute. To investigate the relationship between the bending strength and crack size, pre-cracks of various lengths were made at the center of the tensile surface of specimens with a Vickers indenter using loads of 24.5 N, 196 N and a drill 1 mm in diameter. The sizes of the pre-cracks were about 210 μm and 400 μm in length, respectively. The size of the hole was about 1 mm in diameter and 260 μm in depth. After a three-point bending test, the cracked and fractured part was observed with a SEM. Also, we conducted heat treatments to investigate the crack-healing effect on cracked SiC_f/SiC composites. In particular, to create the crack-healing substance SiO_2 more effectively, after conducting the dipping method or the hydrostatic pressure method into a SiO_2 nano-colloid, the samples were heat treated at a temperature of 1373 K for one hour in air [6]. Each cracked specimen was observed with a scanning electron microscope (SEM).

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Results and Discussion

Fig. 1 exhibits the bending strength as a function on the crack size. The average bending strengths of these cracked specimens were 377 MPa for UCS, 191 MPa for CCS and 337 MPa for SPS, respectively. The bending strength of the cracked SPS shows about half of that (674 MPa) of the smooth SPS. This is a result of a decrease in the volume ratio of the matrix that consists of a strong covalent bond by reinforcing fibers and an increase of the matrix/fiber interfacial region to be a factor of the fracture. However, the bending strength of SiC$_f$/SiC composites has nothing to do with the crack size. Namely, we found that the cause of fracture until a critical crack size is not the only factor, but also the stress concentration in the interface exposed on the surface acts as a tensile stress, because the matrix/fiber or the fiber/fiber interface leads to crack deflection. Although the Vickers indentation using a load of 24.5 N is on the center of a specimen, the bending strength of the cracked UCS is slightly higher than that of the cracked CCS. This means that although the stress concentration in the cracked part is generally a cause of the occurrence of a crack, we are not able to observe the brittle failure behavior, because the crack growth occurs along the interface that is considered as a defect, and the crack growth energy is used to overcome the interfacial bond strength of the fiber/fiber and fiber/matrix, such as the pull-out phenomenon. The bending strength of the cracked UCS using a load of 196 N is similar to that of the smooth UCS. Also, the cracked UCS using a drill of ϕ1 mm shows that the bending strength is like the other cracked UCS, without a drastic reduction in strength during the crack growth. Therefore, there was no relation between the bending strength of SiC$_f$/SiC composite ceramics and the size of a pre-crack until limited crack size.

Figs. 2 and 3 show the SEM images of the cracked UCS and CCS samples by a Vickers load of 24.5 N. In Fig. 2(b), the side image of a UCS sample, the crack grew along the fiber/matrix interface. In particular, the separation

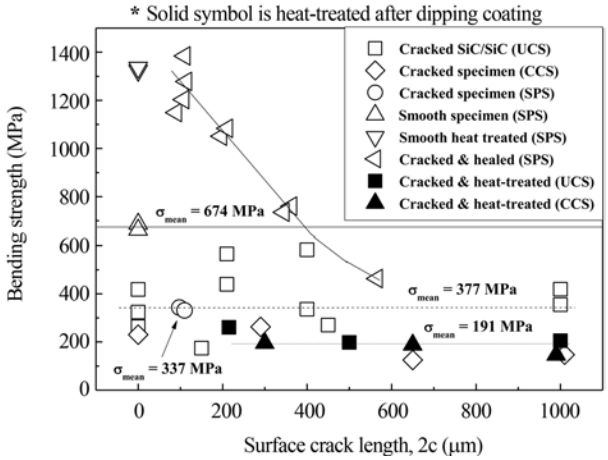

Fig. 1. Relationship between bending strength and surface crack length.

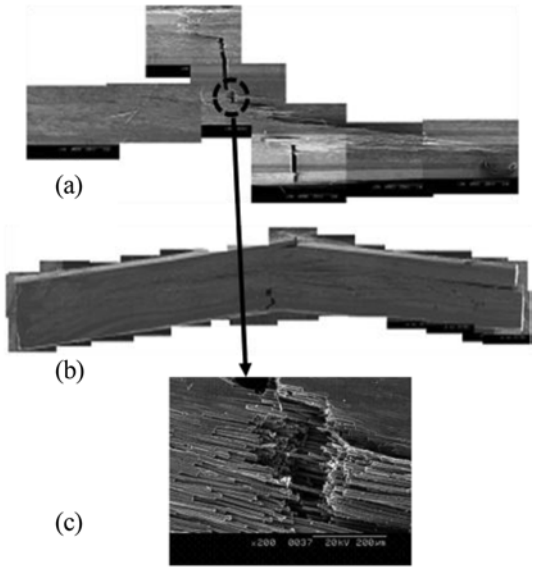

Fig. 2. SEM image of UCS after the bending test; (a) surface, (b) side, (c) enlargement of red circle (a).

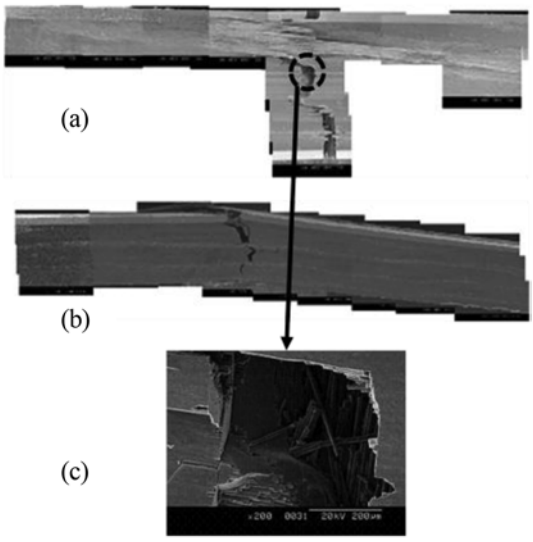

Fig. 3. SEM image of CCS after the bending test; (a) surface, (b) side, (c) enlargement of red circle (a).

of the fiber/matrix interface arises along the longitudinal interface more actively than in the case of the CCS sample, and the crack growth by interfacial separation arises along the longitudinal interface through a wide range of the UCS sample. The delamination of the cracked UCS sample also shows through all longitudinal interfaces. In Fig. 2(a), the surface image of the UCS sample, the crack grew along the fiber/matrix interface with the Vickers pre-crack(c) as its starting point. In Fig. 2(c), locally the SiC fibers broke, because the pre-cracks were made in part of the fibers. Although the UCS sample can be fractured from this broken fiber part because of the stress concentration, the crack grows along the fiber/matrix interface around the cracked part. The crack growth at the matrix-part on the surface occurs in the direction subjected to

the maximum tensile stress as with the SPS sample, then the fiber/matrix interface delaminates, reflecting the crack growth direction. Fig. 2(a) shows that the UCS sample delaminates more easily in a wider range than in the CCS sample. Fig. 3(b), the side image of the CCS sample, shows SiC fibers whose orientation is perpendicularly arranged to the longitudinal direction of the specimen. These perpendicular interfaces are located in the center of the tensile stressed part and are the weakest bonding structure in the three-point bending system, becoming the starting point of the fracture. This phenomenon occurs along the center of the tensile stressed part until reaching the opposite side (the compressive stressed part), and the delamination phenomenon in the longitudinal direction of the CCS sample occurs much less than in that of the UCS sample. In Fig. 3(a), the surface image of the CCS sample, cracks were grown along the fiber/matrix interfaces exposed to the surface when polishing. Fig. 3(c), an enlarged image of Fig. 3(a), shows the pre-crack made at the surface on where fibers were completely broken. Although the pre-crack was made on the surface of the matrix, the cracks grew along the fiber/matrix interfaces. The fracture on the CCS sample began from the fiber/matrix interface whose orientation is perpendicularly arranged to the longitudinal direction of specimens as seen in Fig. 3(b). Therefore, to restrain the occurrence and growth of cracks, we investigated the possibility of crack healing by applying the optimum heat-treatment condition for SiC, a base material. In particular, we conducted a dipping coating treatment, that is to dip the SiC/SiC composite ceramics in SiO_2 nano-colloid for effective crack healing. Fig. 1 shows that the average bending strength of the cracked UCS sample is about 377 MPa which is higher than the 191 MPa of the cracked CCS sample because of the orientation of the reinforcing fibers. In the case of the specimens of the cracked and heat-treated for one hour at 1373 K (■ , ▲) after being dipped into the SiO_2 nano-colloid, the bending strength of the heat-treated CCS sample was had about 176 MPa, which is a lower than about 221 MPa for the heat-treated UCS sample.

Fig. 4 shows the surface morphology of the heat-treated cracked UCS specimen to which the dipping coating method has been applied. In Fig. 4(a) and (b), although the cracks lengthened from 215 μm to 600 μm, the crack healing behavior could not be observed on the cracked part, crack growth part, fiber/fiber interface and fiber/matrix interface as compared with SiC monolithic ceramics. In the specimen cracked by a drill of ϕ1 mm (c), this crack healing behavior also did not occur. From these results, we found that in spite of heat-treatment and dipping the cracked specimen into the SiO_2 nano-colloid, the crack healing effect of a wide crack like at the interface and the synergistic effect of the bending strength of SiC_f/SiC composite could not be anticipated, because a crack healing substance like SiO_2 is difficult to form on the wide crack. Therefore, we conducted a hydrostatic pressure method to infiltrate SiO_2 nano-colloid into a wide crack and interface effectively.

Fig. 5 is the surface image after crack-healing with infiltrated SiO_2 nano-colloid with a hydrostatic pressure on a cracked UCS specimen. The more the Vickers load increases, the more the pre-crack grows and SiO_2 is created on the cracked surface by the hydrostatic pressure as a type of SiO_2 nano-colloid coating. However, the SiO_2 was only created on the surface locally, similar to Fig. 4, and the crack healing behavior at the fiber/fiber interface does not occur either. Therefore, although the coating treatment with a SiO_2 nano-colloid is conducted by applying a hydrostatic pressure, SiO_2 is only created at the base of the crack, because the crack sizes of SiC_f/SiC composite ceramics are very large and wide by the separation of the interface of the fiber/fiber or fiber/matrix, so, the recovery of bending strength by the crack healing effect cannot, as usual, be anticipated.

Conclusions

We investigated the fracture characteristics depending

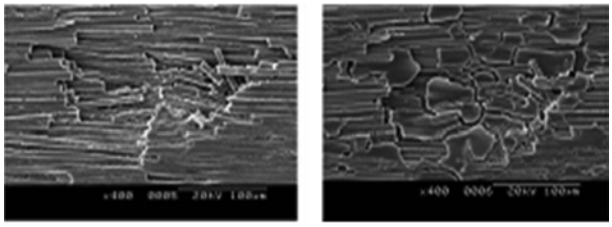

Fig. 5. The surface (a) before and (b) after crack-healing infiltrated SiO_2 nano-colloid with the hydrostatic pressure method on cracked UCS.

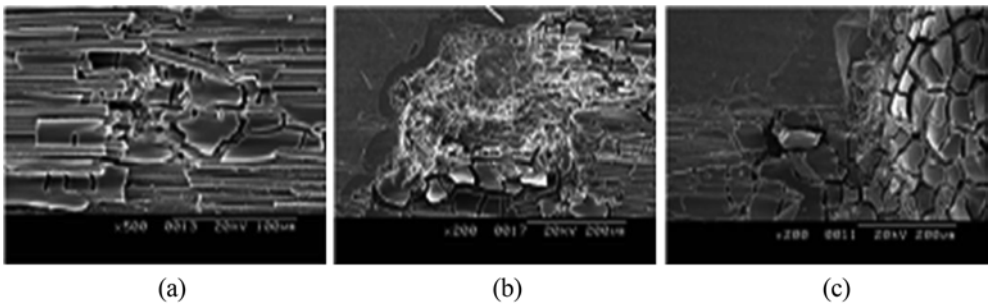

Fig. 4. The surface after crack-healing with a dip coating of SiO_2 nano-colloid on a cracked UCS specimen. (Crack length:(a) 215 μm, (b) 500 μm, (c) 1 mm by ϕ1 mm drill).

on the surface crack size on SiC_f/SiC composite ceramics that were produced by the NITE method, and analyzed the crack-healing possibility by coating SiO_2 nano-colloid on the surface. The bending strength of UCS and CCS samples were about 1/3 of SiC matrix ceramics (SPS), which is the reason that SiC_f/SiC composite ceramics incur surface cracks easily and also have a low volume fraction of 45~50% by reinforcing fibers. Although the stress concentration in cracked parts is generally the cause of the occurrence of cracks, the brittle failure behavior does not occur because the crack growth energy is used to overcome the interfacial bond strength of fiber/fiber and fiber/matrix. There is no relation between the bending strength of SiC_f/SiC composite ceramics and the size of a pre-crack until a limited crack size. Because the crack sizes of SiC_f/SiC composite ceramics are very large and wide by the separation of fiber/fiber or fiber/matrix interfaces, a coating treatment with SiO_2 nano-colloid conducted by applying the dipping method or the hydrostatic pressure method, SiO_2 is only created at the base of a crack, so, the recovery effect of the bending strength could not be expected.

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