Spark plasma sintering of SiC/BN-coated SiC fiber

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Highlights

- SPS of SiC fiber (aligned fibers and the BN-coated fibers)
- SPS of SiC/BN-coated SiC fiber
- Mechanical property of SiC/BN-coated SiC fiber

1. Introduction

SiC with a high melting point and a corrosion resistance has been focused as attractive candidates for the applications to turbine blade of aircraft engine [1], fuel cladding tube for a nuclear reactor [2] and blanket for a fusion reactor [3]. Since SiC exhibits extremely low fracture toughness, the ceramics fiber-reinforced ceramics composite (CFCC) has been developed, and the improvement of toughness is devised. For the SiC-CFCC, a preform made of a bundle of SiC fibers (SiC_f) coated with h-BN for the turbine blade and C for a nuclear reactor is fabricated and the voids in the bundles are filled with SiC by repeating the process for several times in which a filling of the polyorganosilane into the voids of bundles made of SiC_f and subsequent pyrolysis to form SiC. A reactive sintering with filling SiC, Si, C particles in the voids of bundles and a liquid sintering of nano SiC with eutectic additive is also processed to fill the voids. Conventionally, a gas phase method such as CVD has been used to coat h-BN and C on the SiC_f surface. We applied a method of coating h-BN on the surface of SiC_f by liquid phase method. The BN-coated SiC_f was also coated by stacking-sequence disordered SiC(SD-SiC). The SD-SiC/BN-coated SiC fiber was sintered under the temperature which the shape of fiber was maintained through BN without the direct sintering between the fibers and the SiC matrix. The sintered SiC-CFCC was also evaluated for mechanical properties.

2. Methods

Nanometric SiC powder with a stacking-sequence disordered structure (heretofore referred as SD-SiC) was synthesized by high energy ball milling of elemental powders of Si and C under Ar atmosphere. The starting materials were powders of Si (-75μm, > 99.99% pure, Kojundo Chemical Co. Ltd., Saitama, Japan) and C (ca.10.0μm, >99.9% pure, Tokai Carbon Co. Ltd., Tokyo, Japan). Stoichiometric 1:1 mixtures of the two elements were milled in a high-energy planetary ball mill (Fritch P-5) for 24 h, using Si_3N_4 vial and balls. The ball-to-powder mass ratio was 40:1 and the spinning rate was 300 rpm. All powders were handled under an argon atmosphere (O_2 < 2 ppm, H_2O < 3 ppm). Then, BN powder was coated with the organozirconium-BN solution made of 10.8% of Zr-181(Nippon Soda Co., Ltd., tri-n-butoxy-mono(acetylacetonate)Zr(IV): 50%, butyl acetate: 30%, 1-butanol: 20%), 82.0% of hexane and 7.2% of BN (UHPS2, ca.1.0 μm, >99.9% pure, Showa Denko K.K.) on polycrystalline SiC fiber (Tyranno® fiber SA grade, 3.10 g/cm^3, 10 μm, Y/F 800, Ube Industries Ltd., Japan). The SiC fibers (aligned fibers and BN-coated fibers) and the SD-SiC/BN-coated SiC fibers were sintered at 1750ºC to 1900ºC under 100MPa by a spark plasma sintering (SPS) (Model 1050, Sumitomo Coal and Mining Co. Tokyo, Japan). The mechanical property was evaluated by 3 points bending test (Electronic universal testing machine, Yonekra MFG Co. Ltd., Osaka, Japan). The fiber pullouts on the fracture surface was also observed by SEM (JSM-6510LV, JEOL Ltd., Tokyo, Japan).

3. Results and discussion

The SiC fibers were sintered in forms of aligned fibers and isotropic compacted fibers. Fig.1(a) shows the fracture surface of the aligned fibers sintered by SPS at 1800ºC for 30min. The cylindrical form deformed to hexagonal column with high density. The grain boundary between the columns may consist of carbon [4].

Fig.1(a) shows the fracture surface of the aligned BN-coated fibers sintered under the same condition. The voids after the pullout of fibers were observed in the fracture surface. The cylindrical form is also maintained in the texture. The coating of BN powder on the surface of the fibers was effective to prevent the sintering of SiC fibers. On the other hand, the fiber form was not observed in the fracture surface of the woven fabric without coating sintered under the same condition.

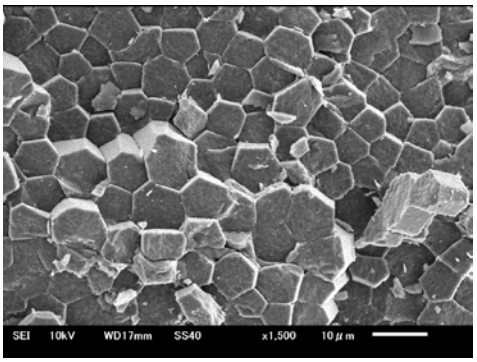

Figure 1. (a) Fracture surface of the aligned fibers sintered by SPS at 1800ºC for 30min.

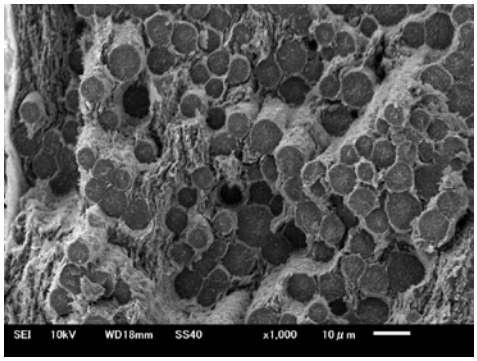

Figure 1. (b) Fracture surface of the aligned BN-coated fibers sintered by SPS at 1800ºC for 30min.

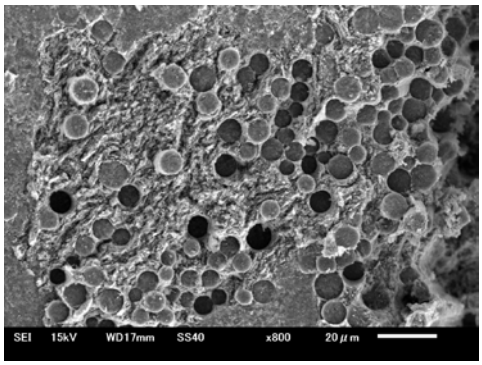

Figure 2. Fracture surface of the aligned SD-SiC/BN-coated fibers sintered by SPS at 1750ºC for 30min.

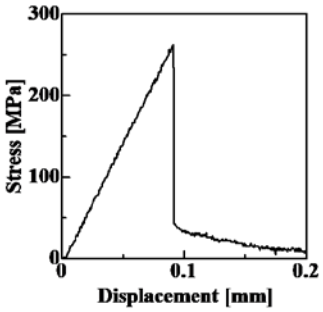

Figure 3. Stress-displacement(deflection) of the aligned SD-SiC/BN-coated fibers sintered by SPS at 1750ºC for 30min.

Figure 2 shows the fracture surface of the SD-SiC/BN-coated SiC fibers sintered at 1750ºC under 100MPa for 30 min by a spark plasma sintering. The residual ZrO_2 decomposed from tri-n-butoxy-mono (acetylacetonate) Zr(IV) in the organozirconium solution accelerate the sintering of SD-SiC between BN-coated SiC fibers. Figure 3 shows stress-deflection curve in 3 points bending test of the sintered SD-SiC/BN-coated SiC fibers. There observed the voids after pullout of the fibers and the slow fracture indicating the higher toughness after the sharp decrease of the stress based on the main fracture.

4. Conclusions

The SD-SiC/BN-coated SiC fibers were sintered by SPS. The pullout of the fibers after the bending test was observed. The slower stress release followed by the main fracture was observed.

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Keywords SiC; SiC$_f$-SiC composite; Fuel cladding tube; Fully ceramics microencapsulated fuel