Fabrication of SiC_f/SiC Composite by the Whisker Growing Assisted CVI Process

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1. Introduction

In addition to the thermo-mechanical advantages of SiC_f/SiC composites, a low induced activation by neutrons and a good irradiation resistance have also made them quite attractive for fusion reactor applications [1]. The chemical vapor infiltration (CVI) process is an effective method for the fabrication of SiC_f/SiC composite but it is a slow process with an inherent drawback of a substantial residual porosity. As the infiltration process proceeds, the grown matrix obstructs the infiltration of vapor reagents, decreasing the infiltration efficiency [2,3]. Because of this clogging phenomenon, the large closed pores are left in the interior of fiber preform. The whisker growing assisted CVI process was applied to obtain a dense SiC_f/SiC composite by the CVI process [4,5]. During this process, the grown whiskers may serve to divide the large natural pores between the fibers or bundles and then the matrix is effectively filled through the modified pore structure.

In this study, the whisker growing assisted CVI process was applied to investigate the densification behaviors of SiC_f/ SiC composite using 2D plain weave Tyranno-SA^{TM} fabrics. The density and the weight gain rate were measured at the each processing step, respectively. To compare the properties, different SiC_f/SiC composites were also fabricated by the conventional CVI process. Additionally, the flexural strength and the fracture behaviors of these composites were evaluated with the thicknesses of the PyC interlayers.

2. Experimental Procedures

Preparation of SiC whiskers was carried out using a gas mixture of methyltrichlorosilane (CH_3SiCl_3, MTS, Aldrich Co., 99%) and purified H_2 (purity: 5N) where H_2 acted both as a reducing agent and a carrier gas for MTS vapor. The whisker growing was performed at 1100°C and the total pressure of 5 torr with the input gas ratio of H_2 to MTS, α (= $F_{(\text{diluent+carrier gas})}/F_{\text{MTS}}$) of 60 in a hot wall chamber. The flow rate of MTS vapor was controlled by adjusting the bubbler pressure and the flow rate of the carrier gas, maintaining the temperature of the bubbler containing liquid MTS at 0°C. The pressure in the reactor was monitored with a capacitance manometer and controlled at 5 torr with a throttle valve located between the reactor and the mechanical pump. A plain weave fabric of Tyranno-SA^{TM} was used as a reinforced substrate. Ten layers of the fabric with the diameter of 50 mm were stacked as a green preform. Before whisker growth, the preforms were coated with pyrolytic carbon using methane gas. The matrix filling process was performed at 1000°C and the total pressure of 100 torr for up to 24 h.

Crystalline phases were detected by the X-ray diffraction (XRD) method. Microstructures of SiC_f/SiC composites and SiC whiskers were observed using scanning electron microscopy (SEM ; Model JS-5200, Jeol, Japan) and transmission electron microscopy (TEM, Model J, Jeol, Japan). The bulk densities and the weight gain rate were determined by measuring the dimension and weight of the specimens. Three-point bending strength was determined using a fixture with a span of 30 mm. For each material, 5 specimens with dimensions of 45^{l} x 3^{w}x 2^{d} mm were tested. The samples were loaded with a constant cross-head speed of 0.05 mm/s at room temperature.

3. Results and Discussion

Fig. 1 and 2 show the X-ray diffraction patterns detected by the XRD and microstructures observed by SEM and TEM for SiC whiskers grown in the preform by stacking ten layers of Tyranno-SA^{TM} fabrics, respectively. As shown in Fig. 1, β-SiC was observed in the specimens subjected to whisker growing. Fig. 2 (a) shows whiskers grown in the small voids between bundles. These whiskers may divide the voids between fibers and left the open channels for matrix filling. Additionally, these grown whiskers were expected to act as new sites for the deposition of SiC. Therefore, the CVI process using the whisker growing process suggests the possibility of an effective matrix filling.

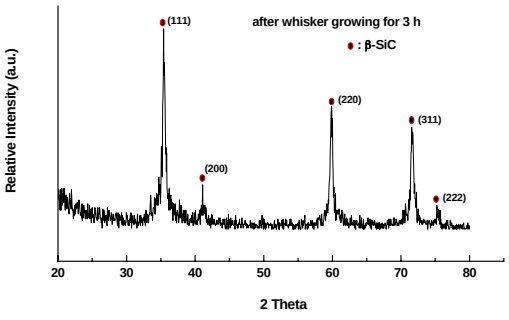

Fig. 1. X-ray diffraction pattern of Tyranno-SA/SiC composite after growing SiC whisker for 3 h.

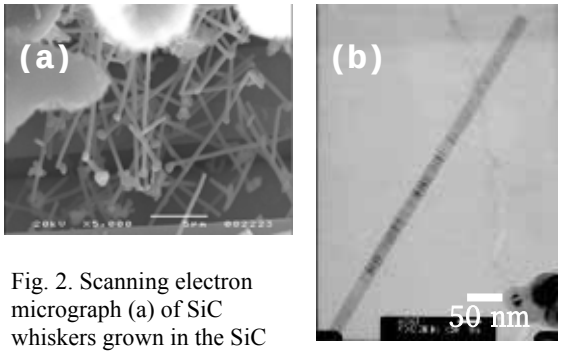

Fig. 2. Scanning electron micrograph (a) of SiC whiskers grown in the SiC fabrics and typical transmission electron micrographs (b) of SiC whiskers containing the stacking faults.

Fig. 3 shows the variations of the weight gain rate of SiC_f/SiC composites with the matrix filling time. By applying the whisker growing assisted CVI process, a higher rate of the weight gain could be obtained during the whole processing time. Additionally, the curve of the weight gain rate by our process shows an easier descent than that by the conventional CVI process. These suggest that the canning effects taking place in the circumferential deposition of SiC in the conventional CVI process can be reduced in our CVI process. Therefore, the densification of the matrix seems to show the different behaviors. In the conventional CVI process, SiC_f/SiC composites generally have the different local densities between the input gas region and the output gas region in a specimen. In the output gas region, the larger voids were apt to remain after finishing of the matrix filling process due to clogging the channels for the reactant gas at the input gas region. But, by using the whisker growing before matrix filling, we could reduce the canning effects and obtain improved microstructure.

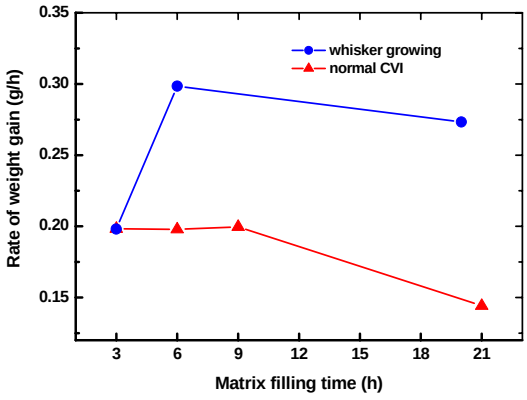

Fig. 3. The rate of the weight gain of SiC_f/SiC composite with the matrix filling time prepared by the different methods.

The nature of the interface between fibers and matrix is one of the key factors to determine the material performance in fiber reinforced composites [2]. The thickness and the constituent of the interlayer significantly affect the properties of fiber reinforced composite [6]. In SiC_f/SiC composite, PyC is the most frequently used as an interlayer. In this study, the PyC interlayer was coated onto the Tyranno-SA fabrics. The thickness of the PyC interlayer was adjusted with the deposition time in the range of 35 nm to 300 nm. The flexural strength of Tyranno-SA/SiC composite was evaluated with the thickness of the PyC layer. All the specimens had a similar density of 2.7 to 2.75 g/cm^3. Fig. 4 shows the flexural strength of SiC_f/SiC composites with the thickness of the PyC interlayer. The composite with the PyC interlayer thickness of 150 nm shows the highest flexural strength of 610 MPa.

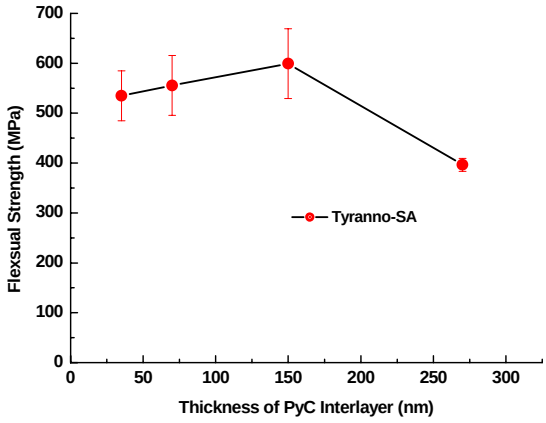

Fig. 4. Flexural strength of SiC_f/SiC composites with the PyC interlayer thickness.

4. Conclusion

β−SiC whiskers were effectively grown between the fibers and the bundles in the Tyranno-SA^{TM}. SiC fabrics using MTS-H_2 gas. A higher weight gain rate could be achieved by the whisker growing assisted CVI process than the conventional CVI process. Tyranno-SA/SiC composite with a PyC interlayer thickness of 150 nm had the flexural strength of about 610 MPa and a density of 2.71 g/cm^3.

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