Microstructure Characterization of SiC_f/SiC Composites by X-ray Computed Microtomography

Weon-Ju Kim[a*], Daejong Kim[a], Choong Hwan Jung[a], Ji Yeon Park[a], Lance L. Snead[b]
[a]*Nuclear Materials Division, Korea Atomic Energy Research Institute, 989-111 Daedeok-daero, Yuseong-gu, Daejeon 305-353, Korea*
[b]*Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA*
[*]*Corresponding author: weonjkim@kaeri.re.kr*

1. Introduction

Silicon carbide fiber-reinforced silicon carbide matrix (SiC_f/SiC) composites have been studied extensively for fusion and advanced fission energy systems due to the excellent high temperature properties, irradiation tolerance, inherent low activation and other superior physical/chemical properties [1]. Recently, there have also been efforts on applying the SiC_f/SiC composites to fuel claddings of various types of reactors such as liquid salt-cooled advanced high temperature reactor (LS-AHTR), gas-cooled fast reactor (GFR), sodium-cooled fast reactor (SFR), and light water reactor (LWR) [2-4].

In general, the chemical vapor infiltration (CVI) technique has been used most widely to produce SiC_f/SiC composites [4]. Although the technique produces a highly pure SiC matrix and thus an irradiation tolerant composite, it results in a complex distribution of porosity. The porosity consists of the interfiber micro porosity and interbundle/interply macro porosities. The complex geometry of porosity and fiber architecture also induces a highly anisotropic behavior of composite properties. The inhomogeneity of fiber-reinforced composites renders the conventional, destructive metallographic techniques difficult to obtain representative microstructural features throughout the specimen volume.

In this study, we applied X-ray computed microtomography (X-ray μCT) to nondestructively analyze the microstructure of SiC_f/SiC composites. The pore structure and porosity distribution were analyzed for disk shape and triplex tubular SiC_f/SiC composite specimens.

2. Methods and Results

Two kinds of SiC_f/SiC composite specimens were used for the X-ray μCT analyses, disk shape and triple-layered (triplex) tubular composites. The disk specimen was 2 dimensional (2D) plain-woven composite reinforced with Hi-Nicalon™ (Nippon Carbon, Japan) fibers. The matrix was infiltrated by the CVI method and the dimension was Φ6 x 2.8t mm. The tubular specimen consists of triplex layers. The innermost layer is high-density monolithic CVD SiC and the second layer consists of SiC fiber-reinforced SiC matrix (SiC_f/SiC) composite to increase a mechanical property and prevent a brittle fracture of the composite tube. We used Tyranno SA3 (Ube Ind., Japan) fibers for the reinforcement of the composite layer. Another CVD SiC is finally coated to improve the corrosion resistance of the composite layer.

In the X-ray μCT technique, several hundreds or more than a thousand 2D X-ray radiographic images are taken at different angles by rotating the specimen. Then, using 3D reconstruction software, the spatial distribution of the absorption coefficient within the sample is reconstructed to produce a 3D volume image of the specimen. In this study, we used laboratory-based polychromatic X-ray source and Al/Cu filters were used to reduce a beam hardening effect. For the disk sample, the spatial resolution of X-ray μCT equipment (microXCT, Xradia) was 8 μm at the field of view of 4.5 mm. The X-ray power was 100 kV, 80 μA with an exposure time of 15 seconds per image and the number of acquired radiographic images were 361. For the tubular sample, the spatial resolution was 3.85 μm for the equipment (Skyscan1172, Bruker) used.

Fig. 1 shows the reconstructed 3D volume image of the disk specimen and the 3D image of porosity extracted from the volume image. Lighter and darker regions of the 3D rendered volume correspond to SiC matrix/fiber and porosity, respectively. It can be seen

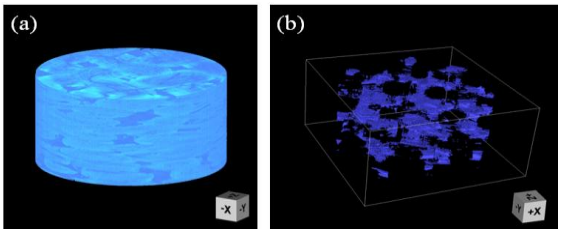

Fig. 1. Reconstructed 3D volume image (a) of SiC_f/SiC composite and porosity image (b) extracted from the volume image.

from the 3D porosity image that some macro pores are interconnected by channel-shaped pores.

From the volume image, we can obtain several hundreds of cross-sectional sliced images along various directions, e.g., transaxial, coronal, sagittal. In order to evaluate the distribution of porosity in the sliced images, each sliced image was binarized into two phases (pore and solid phases) through a threshold operation using

ImageJ software [5]. Fig. 2 shows a typical sliced image in the transaxial direction and the binarized image through the threshold operation. It can be seen that there are two types of pores, intrabundle micro pores and interbundle macro pores.

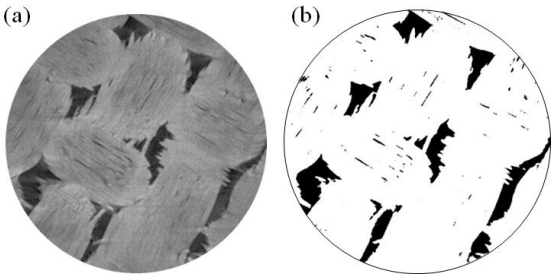

Fig. 2. Typical transaxial sliced image (a) and the binarized image for the calculation of porosity.

Fig. 3 shows the porosity distribution of the SiC_f/SiC composite in the three directions, XY (transaxial), XZ, and YZ slices. In the transaxial direction, it shows a significant variation of the porosity due to the stacking nature of the 2D composite, a lower porosity within the plies and a higher porosity between the plies. On the other hand, the XZ and YZ slices show a less variation of porosity.

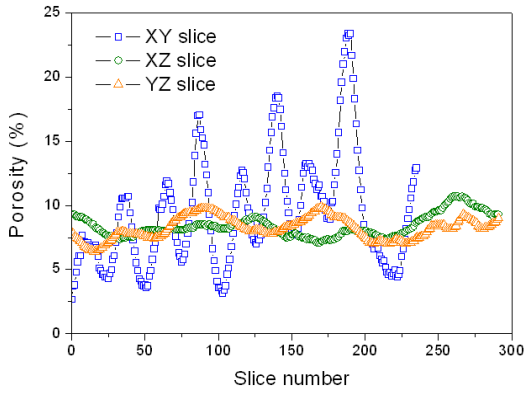

Fig. 3. Porosity distribution of SiC_f/SiC composite calculated from sliced images in three directions.

Microstructure of triplex SiC composite tube was also analyzed using the X-ray μCT. Fig. 4 shows the 3D volume of SiC composite tube and sliced images in three directions. The inner CVD SiC tube shows a very uniform thickness throughout the tube length. However, the outer surface of the composite tube depicts a rather rough feature. This is attributed to the fracture of individual fibers during the winding step of fiber yarn. The brittle nature of SiC ceramic fiber in spite of the fine diameter (7.5 μm for Tyranno SA3) makes the fiber susceptible to fracture during the winding process even at a small tension. Currently, the winding process has been improved significantly to minimize the fiber fracture.

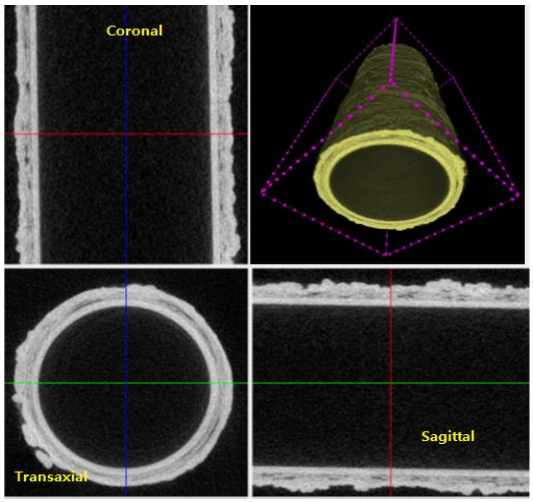

Fig. 4. Reconstructed 3D volume image and sliced cross-sectional images of triplex SiC composite tube.

3. Summary

X-ray computed microtomography is a powerful tool to analyze materials with an inhomogeneous structure such as continuous fiber-reinforced ceramic matrix composites. The technique was able to characterize the distribution of interfiber and interbundle/interply porosities throughout the specimen volume. It is also expected that the technique can apply in-situ mechanical tests or performance tests under severe accident conditions to explore crack initiation and propagation behavior.

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