Ceramics International xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Appropriate thickness of pyrolytic carbon coating on SiC fiber reinforcement to secure reasonable quasi-ductility on NITE SiC/SiC composites

Naofumi Nakazato[a,*], Hirotatsu Kishimoto[a,b], Joon-Soo Park[b]

[a] College of Design and Manufacturing Technology, Muroran Institute of Technology, Muroran, Hokkaido, Japan
[b] Organization of Advanced Sustainability Initiative for Energy System/Material (OASIS), Muroran Institute of Technology, Muroran, Hokkaido, Japan

ARTICLE INFO

Keywords:
B. Composites
C. Mechanical property
D. SiC
E. Nuclear application

ABSTRACT

Pyrolytic carbon (PyC) coating of silicon carbide (SiC) fibers is an important technology that creates quasi-ductility to SiC/SiC composites. Nano-infiltration and transient eutectic-phase (NITE) process is appealing for the fabrication of SiC/SiC composites for use in high temperature system structures. However, the appropriate conditions for the PyC coating of the composites have not been sufficiently tested. In this research, SiC fibers, with several thick PyC coatings prepared using a chemical vapor infiltration continuous furnace, were used in the fabrication of NITE SiC/SiC composites. Three point bending tests of the composites revealed that the thickness of the PyC coating affected the quasi-ductility of the composites. The composites reinforced by 300 nm thick coated SiC fibers showed a brittle fracture behavior; the composites reinforced 500 and 1200 nm thick PyC coated SiC fibers exhibited a better quasi-ductility. Transmission electron microscope research revealed that the surface of the as-coated PyC coating on a SiC fiber was almost smooth, but the interface between the PyC coating and SiC matrix in a NITE SiC/SiC composite was very rough. The thickness of the PyC coating was considered to be reduced maximum 400 nm during the composite fabrication procedure. The interface was possibly damaged during the composite fabrication procedure, and therefore, the thickness of the PyC coating on the SiC fibers should be thicker than 500 nm to ensure quasi-ductility of the NITE SiC/SiC composites.

1. Introduction

Silicon carbide (SiC) is an innovative structural material with potential applications in high temperature systems such as turbines for electric power sources, engines, and bodies of aerospace planes. SiC is a light weight material with high strength at elevated temperatures and good chemical stability in various environments. Because SiC is a brittle ceramics material, SiC fiber reinforced SiC matrix composites (SiC/SiC composites) are fabricated to ensure a toughness, creating a quasi-ductility to monolithic SiC [1,2]. Future nuclear systems will require SiC/SiC composites for enhancing the energy efficiency and accident tolerance [2,3]. SiC has an excellent stability in neutron irradiation environments [4]. However, impurities such as oxygen, residual carbon, and silicon in the SiC fibers and matrix tend to reduce the irradiation resistance [5,6]. Therefore, the use of pure and highly crystallized SiC is required for nuclear systems. Third generation SiC fibers, such as Tyranno-SA and Hi-Nicalon Type-S fibers are the promising crystallized SiC fibers [7,8]. The SiC matrix in the SiC/SiC composites also needs to have a high purity and crystallinity, but this strongly depends on the fabrication methods of the composites [9]. A chemical vapor infiltration (CVI) method and nano-infiltration and transient eutectic-phase (NITE) process are appropriate to form the crystallized SiC matrix in the SiC/SiC composites [10].

To create the appropriate quasi-ductility of the SiC/SiC composites, an interface coating process on the SiC fibers, prior to the matrix formation, is important. The interface separates the SiC fibers from the SiC matrix during the composite fabrication processing, preventing integration, which enhances the quasi-ductility of the material [1]. For nuclear systems, pyrolytic carbon (PyC) is an appropriate interface coating material from the view of irradiation induced radio activity [11]. A common method for the formation of the PyC coating is the chemical vapor deposition (CVD) method. In this method, SiC fibers are placed in a CVD furnace and coated. Because the diameters of each SiC fibers is roughly 10 μm or less, 800 or 1600 SiC fibers are bundled for the convenience of handling. In general, the bundled SiC fibers are installed in the shape of fiber ball into the CVD furnace. It makes some issues like unexpected residual deformation of the bundled SiC fibers (twist and/or curvature) and the bonding between adjacent fiber bundles. This results in damage to the fibers, reducing their mechanical properties. A further issue of using a CVD furnace for PyC coating, is

* Correspondence to: Muroran Institute of Technology, 27-1 Mizumoto-cho, Muroran, Hokkaido 050-8585, Japan.
E-mail address: nakazato@mmm.muroran-it.ac.jp (N. Nakazato).

https://doi.org/10.1016/j.ceramint.2018.07.158
Received 19 May 2018; Received in revised form 7 July 2018; Accepted 17 July 2018

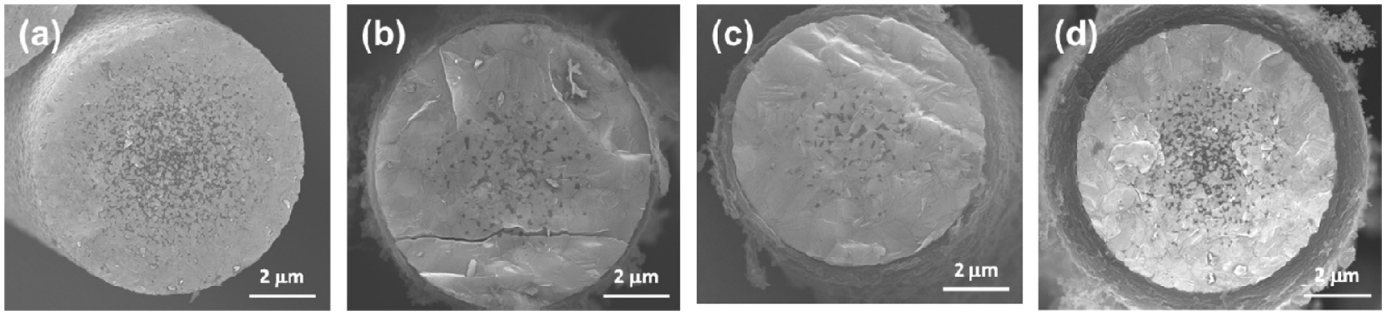

Fig. 1. SEM images of the uncoated SiC and PyC coated SiC fibers fabricated by the CVI continuous furnace; (a) Uncoated SiC fiber. (b)–(d) PyC coated SiC fiber for PyC interface coating thicknesses of (b) 300, (c) 500, and (d) 1200 nm.

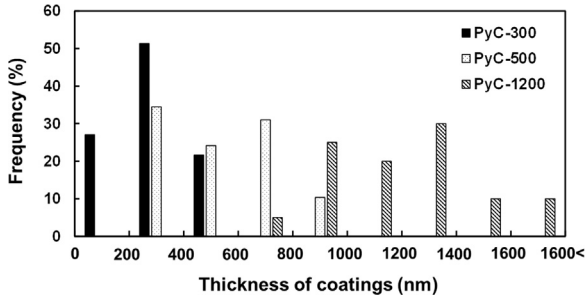

Fig. 2. Thickness distributions of the PyC coatings.

that it is almost impossible to spread the fiber bundles to create space between them, which is needed to enhance the thickness of the PyC coating. It has been reported previously that the appropriate thickness for the PyC coating of CVI SiC/SiC composites is 150 nm [12]. However, NITE SiC/SiC composites tend to be brittle, despite the use of satisfactorily thick PyC coated SiC fibers [13].

Recently, a continuous PyC coating technology for SiC fibers has been developed using an open-end type chemical vapor infiltration (CVI) continuous furnace [14]. The continuous PyC coating technology can be used to spread the fiber bundles, making it possible to increase the thickness of the coating. In this research, we characterized the microstructure of the PyC coating on the SiC fibers before and after the NITE SiC/SiC composite fabrication, and determined an appropriate thickness of the PyC coating for the composites.

2. Experiment

A third generation highly crystalized SiC fiber, the Cef-NITE SiC fiber (GUNZE Limited, Japan), was used as reinforcements in this research. The PyC coatings were deposited on the SiC fibers by a quasi-isothermal type CVD process using the CVI continuous furnace [14]. The precursor for the PyC deposition was methane (CH_4). The intended thickness range of the PyC coating was 300–1200 nm. The microstructure of the PyC coated SiC fibers was evaluated by a scanning electron microscope (SEM). The PyC coated SiC fibers were buried in a resin and cut out using a focused ion beam (FIB) device and observed by a transmission electron microscope (TEM) equipped with an energy dispersive X-ray spectroscopy (EDS) device.

The NITE method for the SiC/SiC composite fabrication is an applied liquid phase sintering method. The SiC mixed slurry for SiC matrix formation was prepared using β-SiC nano-powders (IEST Co. Ltd., Japan, mean grain size of 32 nm), and sintering additives of Al_2O_3 (Kojundo Chemical Laboratory Co. Ltd., Japan, mean grain size of

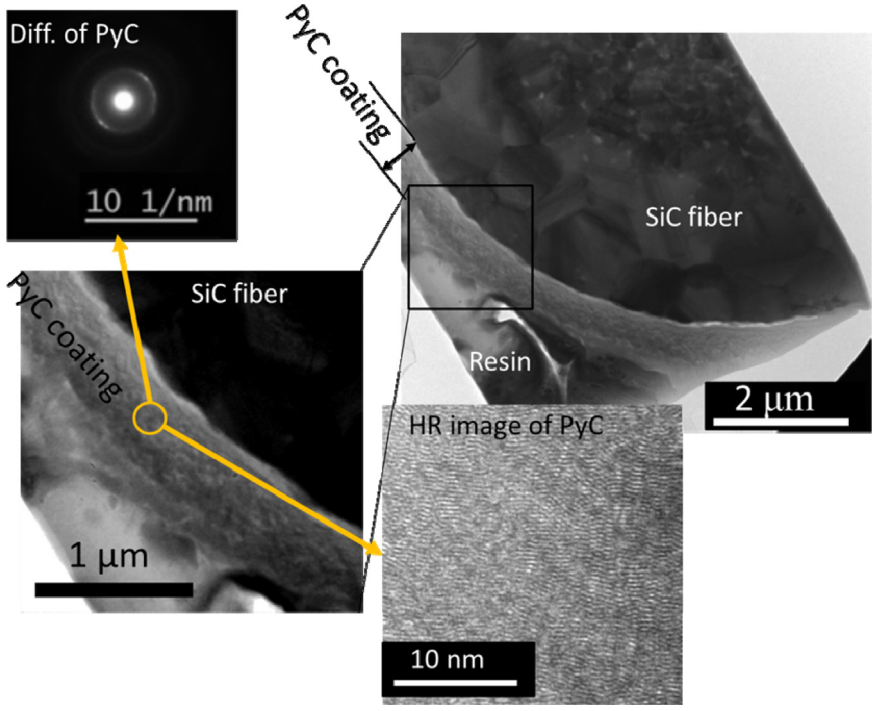

Fig. 3. TEM images of a 500 nm PyC coated SiC fiber.