

Fabrication of the tube-shaped SiC_f/SiC by hot pressing

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Abstract

A manufacturing technique for fabricating a dense tubular SiC long fiber-reinforced SiC composite (SiC_f/SiC) by hot pressing was developed. After infiltrating a SiC-based matrix phase, containing a 12 wt% of Al₂O₃–Y₂O₃ sintering additive, into the fine voids of a TyrannoTM-SA3 SiC fabric preform by electrophoretic deposition combined with the application of ultrasonic pulses, hot pressing was performed using 2 types of specially designed molds filled with graphite powder to transfer the vertical hot press force efficiently to the sidewalls of the tubular SiC_f/SiC. Compared to the low density (~60%) of SiC_f/SiC hot-pressed using a conventional mold, a density > 95% could be acquired using a special mold filled with graphite powder as a pressure delivering medium. This method is suitable for fabricating a dense tubular SiC_f/SiC, which cannot be obtained using a conventional extrusion method.

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1. Introduction

Owing to its excellent physical and chemical properties, SiC long fiber-reinforced SiC composites (SiC_f/SiC) have attracted considerable interest for high temperature applications, including the structural components of heat exchangers and solar absorbers [1,2]. Moreover, SiC_f/SiC is applicable for future nuclear fusion and advanced fission reactors because of its high stability under neutron irradiation conditions, low tritium permeability and safety features [3,4]. Regarding fusion reactor applications, as an example, SiC_f/SiC has been suggested to be the best material for flow channel inserts in breeding blanket modules with a maximum operating temperature of 1300 °C, where approximately 80% of the fusion power is collected [5,6]. Extensive work on SiC-based composites has been performed by the researchers at Oak Ridge National Laboratory, especially for the fusion reactor applications [4,7–10].

SiC_f/SiC can be fabricated by the infiltration of SiC matrix particles into the fine voids of a SiC fabric preform

followed by a consolidation process. On the other hand, the high covalent nature and low self-diffusivity of SiC make it difficult to acquire a high SiC_f/SiC density without the addition of a sintering additive, even at high temperatures [11]. Therefore, SiC_f/SiC is generally sintered by liquid-phase sintering after adding a sintering additive, such as Al₂O₃–Y₂O₃ [12]. The SiC fibers used for reinforcement are normally coated with a ≤ 1 μm thick pyrolytic carbon (PyC) interphase layer to confer fracture toughness by deflecting the crack at the matrix-fiber interface.

Several techniques for filling a preform with a matrix have been introduced to fabricate high quality SiC_f/SiC, such as chemical vapor infiltration (CVI), polymer impregnation and pyrolysis (PIP), reaction sintering (RS), nano-infiltrated transient eutectoid (NITE), electrophoretic deposition using direct current (DC-EPD), and combinations of these processes [13–18]. On the other hand, most reports using these techniques have focused on the fabrication of planar SiC_f/SiC [13–18], even though a tubular structure is requested more frequently than a plate shape for practical applications [5,6,19,20]. This is due to the lack of a suitable manufacturing technique because typical tube fabrication techniques using ceramic

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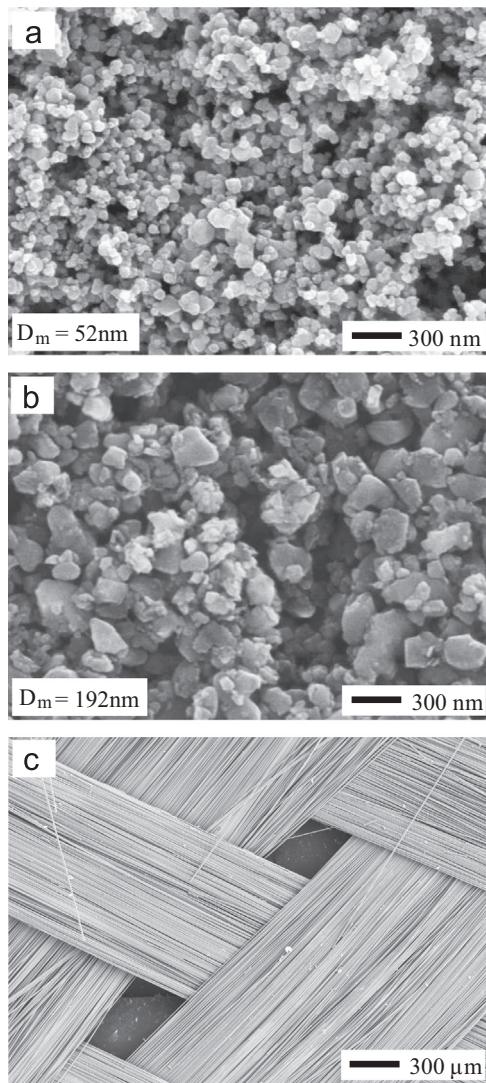


Fig. 1. SEM images of the starting materials; (a) β -SiC particles, (b) high energy-milled Al_2O_3 – Y_2O_3 sintering additive, and (c) 2-dimensionally woven Tyranno™-SA SiC fabric.

powder, such as extrusion and centrifugal molding [21–23], cannot be used for the long fiber-reinforced ceramic matrix tubular composites. According to Krenkel, the fabrication of tubular shape is more difficult than a plate shape because the transverse shrinkage of tubes upon sintering may induce additional matrix stresses in the radial direction, which cannot be observed with plates [24]. Therefore, the development of adequate manufacturing technique for tubular composites is highly needed.

Lorrette et al. recently reported the fabrication of tubular SiC_f/SiC using the CVI method for the preform prepared by the braiding of SiC fibers [25]. Although CVI can minimize the fiber damage because of its low processing temperature ($\sim 1000^\circ\text{C}$), it generally takes many days because of its slow deposition mechanism. On the other hand, CVI with pressure gradients is reported to reduce the infiltration time to several hours, which was developed by the Oak Ridge National Laboratory to address the disadvantage of slow diffusion in CVI process, it is still time-consuming and expensive

process compared to the simple sintering method [26–28]. Moreover, it is difficult to infiltrate the thick preforms by CVI due to preferential deposition of the matrix phase at the surface without filling the deep voids [29], which is known as the ‘surface sealing effect’ or ‘canning effect’. Therefore, composites fabricated by CVI generally contain 10–20% pores, where Lorrette et al. reported a composite density of approximately 2.5 g/cm^3 , which corresponds to $< 80\%$ compared to the theoretical SiC_f/SiC density [25].

In this study, a new fabrication technique for a tubular SiC_f/SiC by hot pressing was developed, which is more convenient and less time-consuming than the CVI method. After preparing the tubular preform using a 2-dimensionally woven Tyranno™-SA fabric, DC-EPD combined with ultrasonic pulses was performed to infiltrate the matrix phase followed by hot pressing. In particular, graphite powder was used as a pressure delivery medium along with a specially designed mold, which can transfer the vertical hot pressing pressure to the sidewalls of the tube effectively to achieve a high SiC_f/SiC density. A PyC coating on the SiC fiber was not considered because this study was a feasibility test for the fabrication of tubular SiC_f/SiC by hot pressing.

2. Experimental

A commercial β -SiC ($> 97.5\%$ purity, 4620KE, NanoAmor Inc., USA) with a mean particle size (D_m) of 52 nm was used as the main matrix phase. A 12 wt% mixture of Al_2O_3 (99.9% purity, $D_m = 1\text{ }\mu\text{m}$, Baikowski, France) and Y_2O_3 (99.99% purity, $D_m = 2\text{ }\mu\text{m}$, Acros Organic, USA) at a weight ratio of 6/4 was used as the sintering additive. Two-dimensionally $0^\circ/90^\circ$ plain-woven Tyranno™-SA grade-3 SiC fabric (Ube Industries, Japan) with a fiber diameter of $7.5\text{ }\mu\text{m}$ and 1600 fibers per yarn was used as reinforcement. The mean particle size of the Al_2O_3 – Y_2O_3 additive was decreased to 192 nm by 2 h of high energy milling (MiniCer,

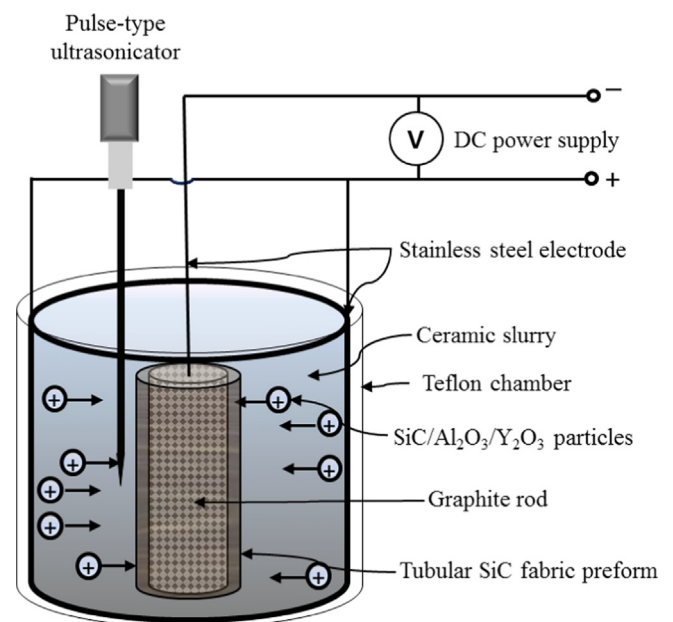


Fig. 2. Schematic diagram of the experimental setup for the matrix phase infiltration for a tubular SiC preform using EPD combined with ultrasonication.

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