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Microstructure of CVI SiC Matrix on Thickness of SiC Green Body Manufactured by Binder Jet Printing

Young-Seok Jeong[a*], Duhyun Kim[a], YoungSuk Jung[a], JiWon Oh[a] and ShinHu Cho[a]
[a]*Madde Inc., Process Design Dept., 89, Yeonmujang-gil, Seongdong-gu, Seoul, Republic of Korea 04783*
[*]*Corresponding author: zhcm123@madde.co.kr*
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1. Introduction

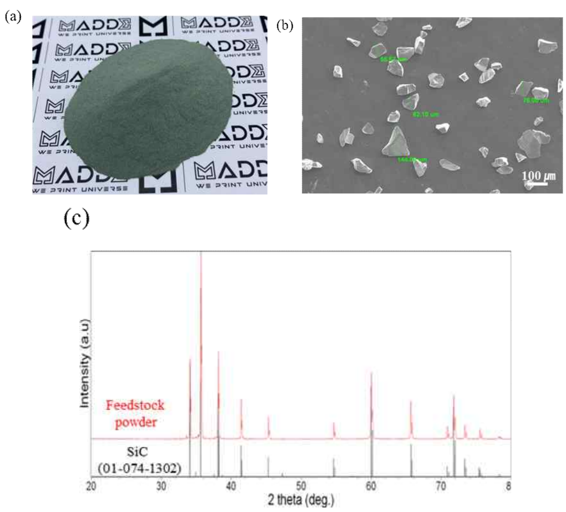
With the advancement of AI technologies, the importance of nuclear power for supplying electricity to data centers is increasing. Many countries are developing technologies to commercialize the fourth-generation nuclear reactor, the Very High Temperature Reactor (VHTR), as it is both economical and safe [1]. The VHTR uses TRi-structural ISOtropic (TRISO) fuel. TRISO particles are dispersed inside the SiC cladding to fabricate Fully Ceramic Micro-encapsulated (FCM) fuel to minimize radioactive materials. FCM fuel can significantly improve both the safety and performance of reactors. However, it is challenging to evenly disperse TRISO particles without damage, and designing internal channels to allow coolant to flow efficiently is also difficult.

Recently, the U.S. TCR program proposed a technology to fabricate FCM fuel using additive manufacturing (AM) [2]. The proposed FCM fuel disperses TRISO particles inside AM SiC cladding and forms the SiC matrix through chemical vapor infiltration (CVI). CVI is a method in which process gases containing Si and C are passed through a porous preform, where thermal decomposition and chemical reactions form a high purity SiC matrix. In this method, the SiC matrix first forms on the preform surface. If the process gas cannot flow further into the structure, voids are created inside. The SiC density gradient greatly influences the mechanical properties. Terrani et al. [3] analyzed the failure behavior of FCM fuel fabricated by AM and the CVI process under irradiation. It was confirmed that the structural stability of FCM fuel is related to the distribution of TRISO particles and the densification of the SiC matrix. Schappel and Terrani [4] conducted lifetime testing of FCM under high-radiation environments. This study showed that the stress in the matrix significantly changed over the irradiation period. According to the report, the microstructure of the SiC matrix greatly affects the stability of FCM fuel. When FCM is fabricated by AM and CVI, the microstructure of the SiC matrix can vary depending on the thickness of the shell. Therefore, we investigated the effect of the thickness of binder-jet-printed green bodies on the microstructure of the CVI SiC matrix.

2.Experimental

We fabricated green bodies using 3D Binder Jetting Printing (BJT). The size of the BJT build box was 500 mm (length) × 400 mm (width) × 300 mm (height). The particle size distribution, phase, and morphology of the SiC powder used in the 3D BJT process are shown in Fig. 1. The green SiC powder corresponds to the α-SiC phase (ICDD card number: 01-074-1302). The D50 of the SiC powder was approximately 55 μm. SiC green bodies with various thicknesses were printed using this powder. First, the desired thickness of the green body structures was designed using 3D CAD software. The designed structures were then sliced layer by layer, and the sliced data were transferred to the binder jetting printer. Based on the transferred data, green bodies were fabricated according to the desired structures.

During the binder jetting process, SiC powder was spread on a flat plate. Polymer binder was dispersed in the desired layer pattern. The polymer binder was cured by infrared radiation (IR). This sequential process was repeated multiple times to fabricate the green body of the desired structure. The printed green bodies were obtained by eliminating loose powders. The fabricated green body thicknesses were 2, 5, 8, and 30 mm. Finally, the SiC matrix was densified using CVI. In the CVI process, methyltri-chlorosilane (MTS, CH_3SiCl_3), containing silicon and carbon, was used as the precursor, and hydrogen (H_2) was used as the carrier/dilution gas. The temperature, pressure, and MTS concentration were fixed at 1050 °C, 5 Torr, and 20%, respectively.



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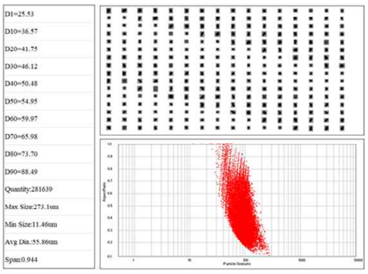

Fig. 1. (a) Green SiC powder used as feedstock for green body, (b) microstructure of SiC powder, (c) X-ray Diffraction pattern of SiC powder, and (d) particle size distribution of SiC powder.

3. Results

The green body printed by binder jetting consists of entangled SiC powders bonded together by a polymer binder (Fig. 2). In the binder jetting process, the polymer binder wets the surface of the SiC powders or infiltrates between the particles through capillary force. Subsequently, volatile substances are removed by IR irradiation. The remaining polymer binder connects the particles. The powders bonded by the polymer maintain sufficient binding strength to prevent damage to the green body. The strength of the fabricated green body was measured to be 2.58 ± 0.02 MPa.

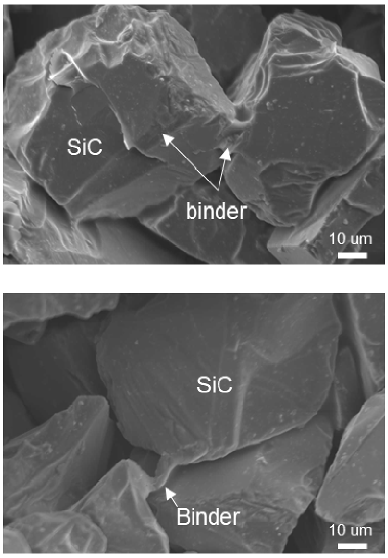

Fig. 2. The microstructure of SiC particles covered and bridged by binder.

The SiC powders in the printed green body are randomly distributed. Because of this random arrangement, pores are uniformly distributed throughout the green body (Fig. 3). It indicates that the pore size and distribution are isotropic, regardless of direction. Therefore, in the CVI process, process gases can uniformly infiltrate from all directions. In contrast, anisotropic materials with aligned structures may hinder the gas flow during CVI. The uniformity of the pore structure enables easier control of the infiltration rate and densification during CVI, while ensuring consistent thermal and physical properties. Fig. 4 shows the density of the binder-jet-printed green body, measured from 30 specimens. The average density was 1.32 ± 0.02 g/cc. It is confirmed that binder jetting can fabricate green bodies with similar density across a large area.

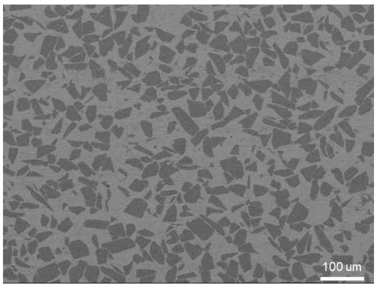

Fig. 3. SEM micrographs of randomly arranged SiC powders.

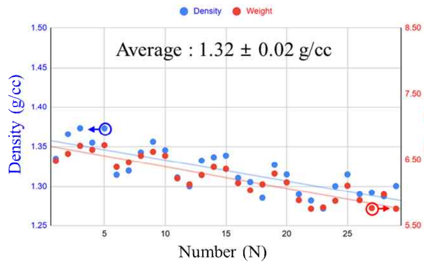

Fig. 4. Density distribution of binder jet printed green body.

Fig. 5 shows the cross-section of a specimen subjected to the CVI process with a green body thickness of 0.5 mm. The CVI matrix filled the voids between the SiC powders. However, closed micro-pores were formed inside the specimen. As the CVI SiC matrix sequentially deposited on the surfaces of adjacent powders, necking formed between neighboring particles [5]. Subsequently, as the necks were filled with SiC matrix, the remaining voids were completely sealed, preventing the process gases from infiltrating further inside, thereby creating closed pores. The density of this specimen after CVI was 2.93 g/cc. The infiltration capacity of the CVI SiC matrix is inherently limited. In general, the residual porosity of CMCs fabricated by CVI is about 10–15% [6]. Thus, it is difficult to achieve the theoretical density of the SiC matrix through CVI.

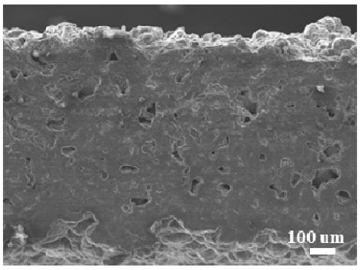

Fig. 5. Microstructure of a 0.5 mm thick sample after the CVI process.

Fig. 6 shows the cross-sections of specimens with green body thicknesses of 2, 5, 8, and 30 mm after CVI SiC process. Regardless of the green body thickness, the surface regions were mostly filled with SiC matrix. However, the center of the structures unfilled the SiC matrix as thickness increased. The CVI process forms the SiC matrix by infiltrating into the porous structure. However, since the SiC matrix forms first on the surface, the surface pores become blocked [7]. This prevents the processing gas from diffusing into the inner regions of the structure, leaving unfilled voids inside. Such voids degrade the mechanical and thermal properties of the material.

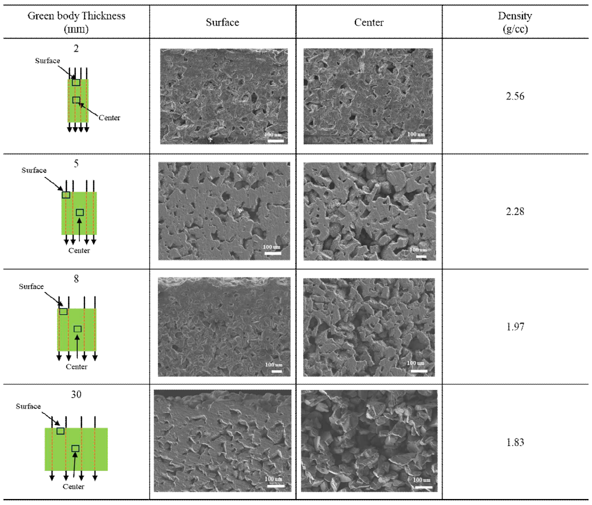

Fig. 6. Surface and central microstructure of specimens with thicknesses of 2, 5, 8, and 30 mm after the CVI process.

4. Conclusions

In this study, SiC green bodies were fabricated using 3D binder jet printing and SiC matrix were subsequently formed by CVI. The green bodies manufactured by binder jet printing exhibited adequate binding strength, as the SiC powders were interconnected by polymer binders. The pores within the green body are randomly arranged. Therefore, we confirmed that isotropic structures can be manufactured using binder jet printing. A density difference in the CVI SiC matrix was observed between the surface and the central regions of green bodies with different thicknesses. For the green body with a thickness of 5 mm, a similar CVI SiC matrix was formed in both the surface and central regions. However, when the thickness exceeded 5 mm, the central region showed insufficient SiC matrix formation. Therefore, the process condition control is essential to achieve a uniform SiC matrix within thicker green bodies. In future work, we aim to optimize the CVI process for thick green bodies by controlling parameters such as temperature, gas flow rate, and pressure to achieve a more homogeneous SiC matrix distribution.

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