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Corresponding author:

Name: Joon-Soo Park

Mailing address: Institute of Advanced Energy, Kyoto University

Gokasho, Uji, Kyoto 611-0011, Japan

Telephone: +81-774-38-3460

Facsimile: +81-774-38-3467

E-mail address: jspark@iae.kyoto-u.ac.jp

Abstract and keywords

Efforts on Large Scale Production of NITE-SiC/SiC for Test Blanket Module of ITER

Joon-Soo Park[1,2], Akira Kohyama[1], Tatsuya Hinoki[1], Kazuya Shimoda[3], Yi-Hyun Park[3]

[1] *Institute of Advanced Energy, Kyoto University, Uji, Kyoto, Japan*

[2] *Institute of Energy Science and Technology Co., Ltd., Kanda, Tokyo, Japan*

[3] *Graduate School of Energy Science, Kyoto University, Uji, Kyoto, Japan*

Abstract

To indicate the feasibility of utilizing newly developed NITE-SiC/SiC composite materials in a fusion reactor, R & D on NITE process with near net shape forming has been carried out. In order to establish large scale production of NITE-SiC/SiC, the pilot grade (PG) of NITE-SiC/SiC were fabricated and their baseline properties were evaluated. As the key elements, nano-powder fabrication and Tyranno-SA, SAK fabrication are extensively being developed.

The NITE-SiC/SiC composite of cylindrical shape was also fabricated by a near-net shape process called pseudo-HIP, which was a new type HIP using a carbon powder as the pressure transmitter. The microstructure of NITE-SiC/SiC composites, such as fiber volume fraction, porosity and type of pores, can be controlled precisely. It makes it possible to produce Test Blanket Modules (TBMs) with proper thermal conductivity in response to the requirement of fusion reactor design.

Key word codes

C0900, F0100, F0400, F0800, S0400

1. Introduction

A key element of the worldwide fusion program is the development of breeding blankets for commercial fusion power stations. ITER will provide three equatorial ports (size of ~1310×1760 mm) for various types of TBMs(Test Blanket Modules) for the test of tritium breeding module. Each port can accommodate two types of TBM. Reflecting the general direction of breeding blanket developments for fusion, the most developed blanket designs, such as the combinations of He-cooled/ceramic breeder/Be multiplier/RAFs(Reduced Activated Steels), water-cooled/ceramic breeder/Be multiplier/RAFs, He-cooled/Pb-17Li/Rafs and Self-cooled Li blanket/V-4Cr-4Ti alloy are under consideration for the ITER TBMs program. Other advanced concepts are also under discussion by worldwide fusion materials programs. Silicon carbide composites offer the greatest potential for very high temperature operation among the candidate reduced activation fusion structural materials. Recent third-generation SiC/SiC composites have shown no degradation of mechanical properties after irradiation to a dose of ~10 dpa.[10] These encouraging results might enable introduction of SiC composites in low-risk application in ITER TBMs. But, SiC/SiC composites have not been as well-developed commercially and require considerable additional research to investigate engineering feasibility.[1-10]

Based on the improvements in reinforcing SiC fibers and other raw materials, the well know liquid phase sintering (LPS) process was drastically improved to become a new process called the Nano Infiltration and Transient Eutectic Phase (NITE) Process. Laboratory scale NITE-SiC/SiC composites demonstrated excellent mechanical properties, thermal conductivity, hermeticity and microstructural stability which made them attractive for not only nuclear application but many industrial applications.[11-16].

The pilot grade fabrication of the NITE SiC/SiC composite has been conducted with varieties of shapes and sizes to adjust process conditions to meet large scale productions in geometry, size, quality and quantity. To indicate the feasibility of blanket modules for fusion reactors by utilizing newly developed NITE-SiC/SiC composite materials, R & D on NITE process with near net shape forming has been carried out. The pilot grade of NITE-SiC/SiC were fabricated and their baseline properties were evaluated. The microstructure of NITE-SiC/SiC composites, such as fiber volume fraction, porosity and type of pores, can be controlled precisely. This paper provides results and present status of the pilot grade NITE-SiC/SiC composites. Moreover, newly started efforts for the production of porous SiC-materials based on the NITE process are introduced briefly.

2. NITE Process

The NITE process was developed making use of so called liquid phase sintering for silicide ceramics with small amounts of oxide additives, which is one of the common production processes for monolithic SiC. The liquid phase sintering has been applied to SiC many times in the past and has been found inappropriate to high performance SiCf/SiC processing. One of the reasons was the high process temperature required to make a dense matrix with a small amount of sintering additives, which degrades fiber strength. The successful development of NITE owes to fundamental research on surface microchemistry of nano-sized β-SiC powder, the rheological properties of mixed slurry, optimization of sintering conditions, appropriate fiber protection, and emergence of the advanced SiC fibers like Tyranno-SA.

In the laboratory scale production, carbon coated Tyranno-SA preforms were infiltrated with nano-sized β-SiC powder and a small amount of sintering aids (Al_2O_3, Y_2O_3 and SiO_2) and followed by hot pressing at high temperature (1750°C -1800°C) under pressures ranging from 15 to 20 MPa. The mastrix of NITE SiC/SiC is composed of polycrystalline SiC and small amount of isolated oxides. The details of the NITE process were introduced elsewhere.[12-14]

3. Efforts on Large Scale Production

3.1 Continuous UD prepreg sheet forming line

For the stable supply of high performance SiC fiber, the large scale production line for SiC fiber is being built under the strong correlation between Kyoto University and other Japanese companies, such as Institute of Energy Science and Technology, Co., Ltd. Ube Industries, Ltd. and Japan Ultra-high Temperature Materials Research Institute (JUTEM).

In spite of growing demand for prepreg, which is a kind of fabric consisted of the carbon coated reinforcing fiber including raw materials for the formation of SiC matrix, only limited amount of prepregs can be supplied due to its manual production system and the short supply of carbon coated SiC fiber. To raise the production efficiency the establishment of the continuous unidirectional (UD) prepreg forming facilities including CVD furnace for carbon coating as fiber/matrix interphase was strongly required as well as the stable supply of highly qualified raw materials. Fig. 1 shows the rough concept of continuous UD prepreg sheet forming line for the large scale production. This forming line consists of bobbin mount, fiber desizing furnaces, CVD furnace for carbon coating, slurry infiltration, dryer, and correcting spool.

[Fig. 1]

3.2 Chracterization of SiC nano powder

For the reduction of porosity and good sinterability in the NITE process, the handling of SiC nano-powder and process aids are emphasized as key techniques. The stable supply of SiC nano powders with precise characterization and severe quality control is also an important issue. It was well known that the surface of SiC powder is covered by silicon oxide, silicon oxycarbide or free carbon, and strongly affected by fabrication/preservation condition.

X-ray photoelectron spectroscopy is one of the most powerful methods for the investigation of powder surface chemistry. Fig. 2 shows XPS spectra of SiC nano powders which were examined for the production of laboratory scale NITE-SiC/SiC composites. As an example, N3 SiC powder has highest O_{1s} peak compared to that of other SiC powders. It means that a large amount of oxygen exists on the surface of N3 SiC powder as the status of SiO_2 or SiO_xC_y. Although, SiO_2 assists the formation of liquid phase at lower temperature, it is well known as the main vitrifying component.[17] Based on the screening test including XPS, FT-IR and XRD, the SiC nano powder with proper properties for NITE process was selected with special care and used in large scale production.

[Fig. 2]

3.3 Pilot grade products, characteristics and performance

The brief history of the large scale (here after called pilot grade) fabrication, which aims at industrialization and commercialization, of NITE-SiC/SiC is summarized in Table 1.

[Table 1]

The first trial to make pilot grade NITE was done in 2002 based on the optimized condition for laboratory scale fabrication. For the case of UD reinforcement with PyC interface, it was difficult to make the inter-bundle matrix and the result was not satisfactory from a density and mechanical properties point of view. For the case of plain woven fabrics of Tyranno-SA without fiber coating, a fully dense composite without any detectable cracks under optical and SEM observation was successfully produced.

Figure 3 shows the appearance of a 1000 cc block of NITE-SiC/SiC. The center portion was saw cut and a plate with 3mm thickness was obtained (shown in the upper right picture in Fig. 3) and specimens for mechanical test was obtained. The result of tensile test was shown bottom left of the figure.

[Fig. 3]

To improve the workability for workers in charge of fabrication at the Ube process line, the amount of process additive was increased for pilot grade #2. Furthermore, in the case of the pilot grade #3 fabrication a new Tyranno fibers was applied. The tentative name of the fiber is Tyranno-SAK (the new version of SA for Kyoto University) where 800 fibers make one fiber bundle for improving inter bundle matrix formation and improving weavability for textile fabrication. Although a fiber/matrix interphase, mainly CVD-carbon, is necessary to protect the reinforcing fiber during sintering and increase the toughness of composites, fiber filaments stick together and a tangle of fiber filaments can be an obstacle to intrabundle matrix formation. Through the pilot grade productions, process improvement was conducted. As a result, density and the deformation of fibers during processing and mechanical properties have been improved. Fig. 4 summarizes specific features of products comparing laboratory grade, pilot grade #1 to #3.

[Fig. 4]

As shown in Fig. 4, the pilot grade #1 was insufficient in density, tensile strength and elastic modulus. The only exception was for the case of the no fiber coating product where density was near full-dense and elastic modulus was quite high. From the pilot grade #2 to #3, all characteristics were improved and the results for UD NITE-SiC/SiC utilizing Tyranno-SAK with PyC interface became very similar to the laboratory grade material. But, the stress-strain curves in the left side graph in Fig. 4

indicate a big difference in those two materials. The laboratory scale product shows large elongation (fiber pull outs) after reaching the proportional limit stress, whereas the pilot grade #3 sample (denoted as PG#3PCUD) showed relatively small fiber pull outs. These results indicate the insufficient interface formation for the case of pilot grade #3.

3.4. Near net shaping

The fabrication process for cylindrical shaped NITE-SiC/SiC composites is schematically shown in Fig. 5. The cut UD prepreg sheets were stacked on the graphite mold. Then the preform was densified by the pseudo-HIP process, which was a new type HIP using a carbon powder as the pressure transmitter and a carbon mold with near-net shape cavity. In this process, pressure was applied to the upper graphite die the same as hot pressing, however, the pressure was transmitted through the carbon powder filled in the mold. For the small diameter cylindrical shape, pressure was designed to be transmitted from outside to inside as shown in Fig. 5 (a). For the large diameter cylindrical shape, pressure was designed to be transmitted from inside to outside as shown in Fig. 5 (b).

[Fig. 5]

3.5. Porous SiC materials

Fusion reactors and advanced generation fission reactors like the Gas-Cooled Fast Reactor (GFR) are considered to be operated near or over 1000°C. Therefore, the reactors need high temperature materials for their components. Porous SiC materials and SiC/SiC composites are considered as the partition and perforated containment wall for a blanket module, and structural material for the coated particle fuel compartment for a horizontal flow cooling concept with direct cooling system for the GFR. SiC and

SiC/SiC with through thickness channels should maintain high thermal conductivity and high helium leakage rate.

Based on the NITE process, new trial efforts for the production of SiC ceramic materials with open pores and through thickness channels have been started. Due to the high degree of freedom in the NITE process, various types and sizes of pore can be formed in a robust SiC matrix as shown in Fig. 6.

These NITE-SiC matrices with the various sizes of pores were achieved in different ways, like simple hot pressing with different contents of precursors, and the decarburization of precursors in a SiC matrix. It means that the sizes and shapes of pores or channels in SiC matrix can be controlled by the selection of precursors with appropriate sizes and shapes. The slight problem of heterogeneous microstructure around through-thickness channels remains. Further R&D is on-going to form more robust and sound NITE-matrix.

[Fig. 6]

IV. CONCLUSIONS

Pilot grade production of NITE SiC/SiC composites has been extensively conducted in recent years and much progress towards industrialization and commercialization has been accomplished. Although many crucial needs remain for the application to advanced energy systems, the results up to now are quite encouraging and the efforts to initiate mass production of SiC nano-powders and Tyranno SAK fibers for NITE are rapidly maturing. Development of porous SiC materials based on the NITE process is also on going with high potential and attractiveness.

Acknowledgments

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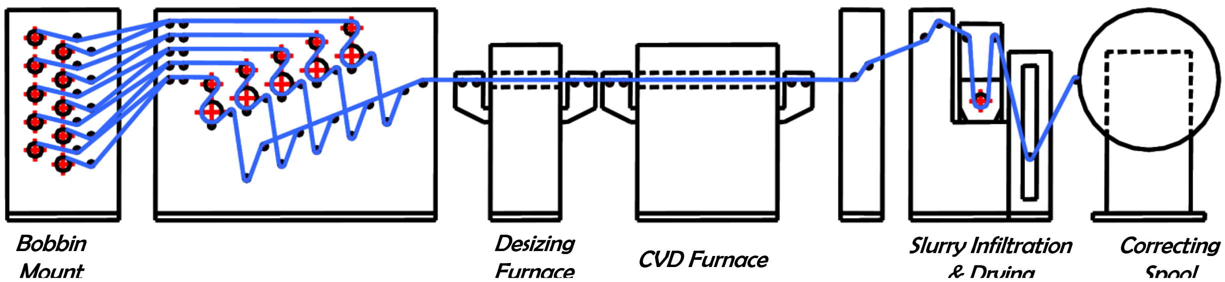

Fig. 1 Continuous UD prepreg sheet forming line for the large scale production of NITE-SiC/SiC

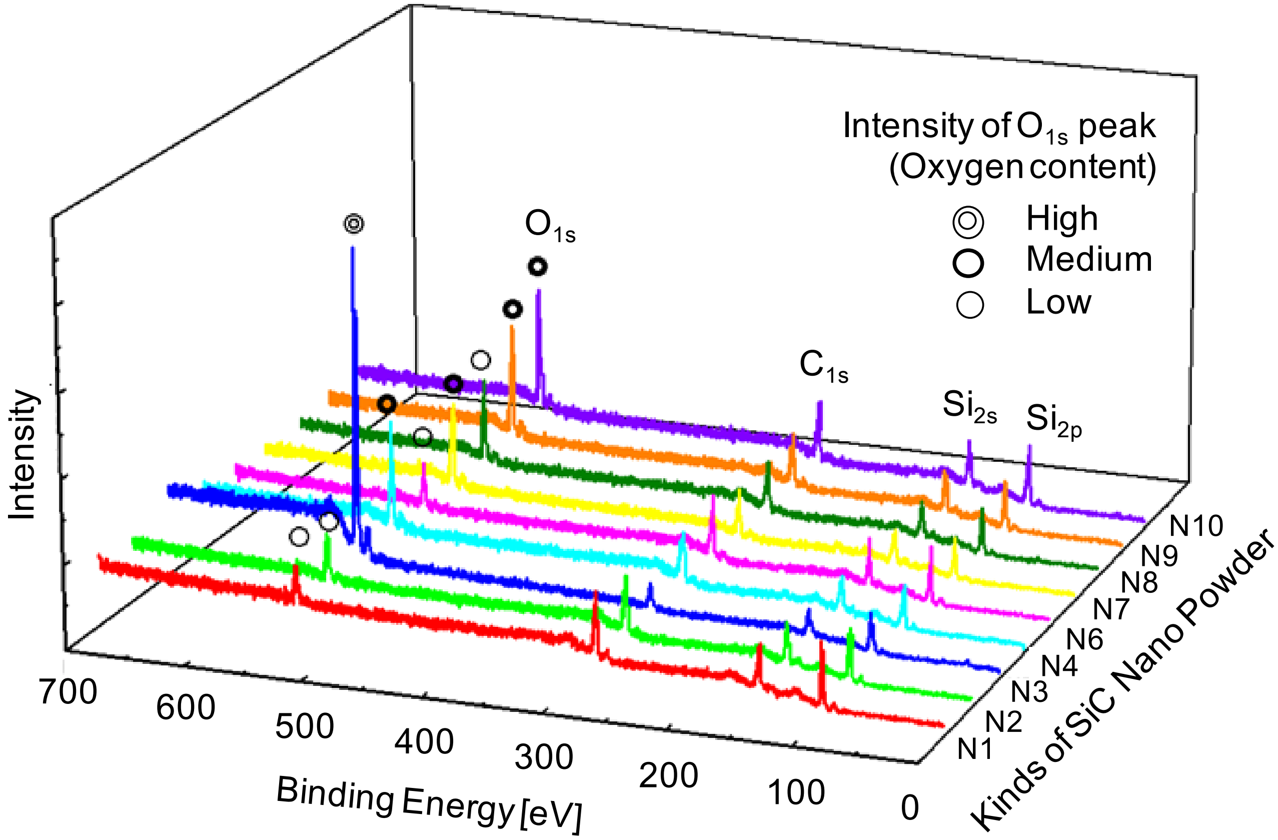

Fig. 2 XPS spectra of SiC nano powders

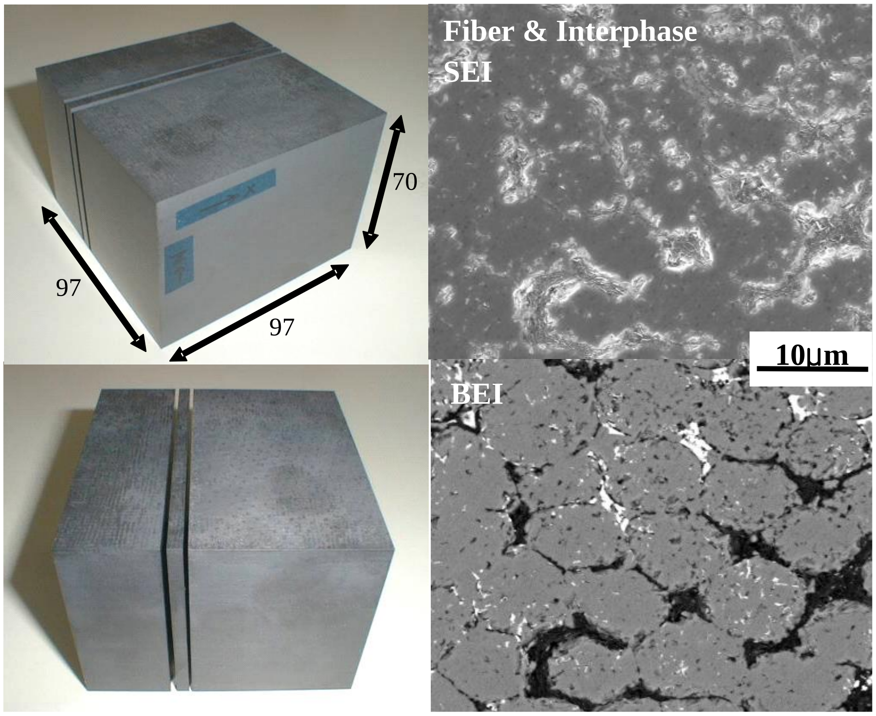

Fig. 3 Pilot grade #1 NITE-SiC/SiC 1000CC cubic block

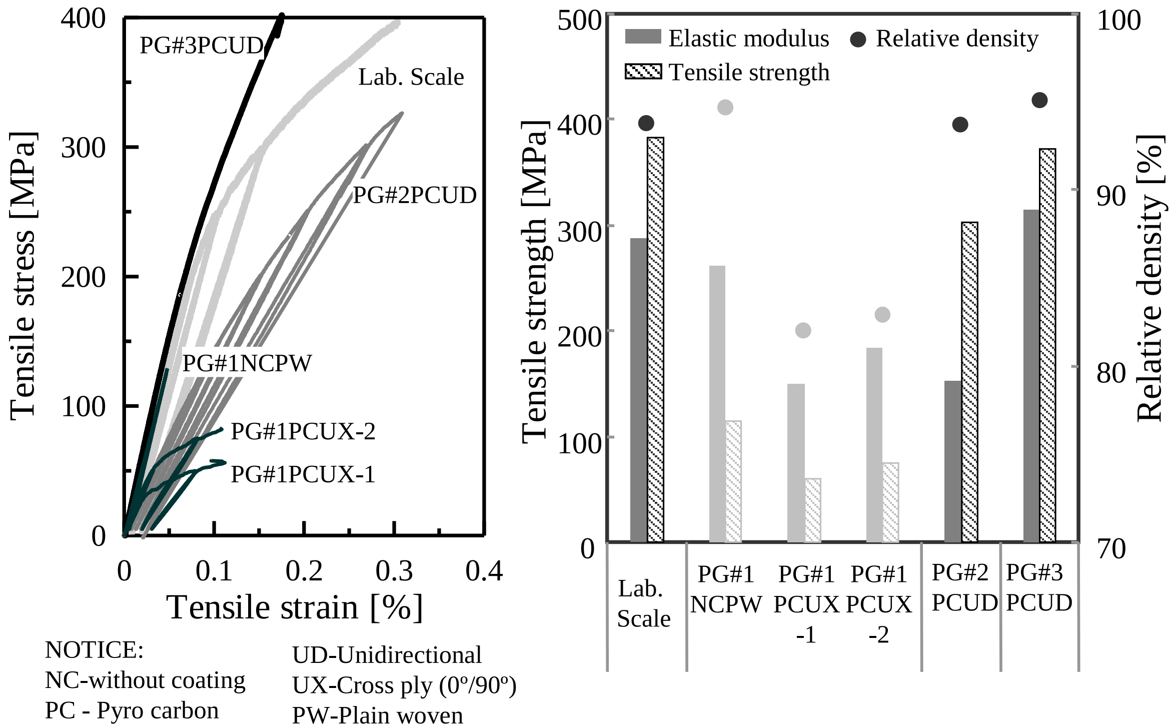

Fig. 4 Characteristics of NITE-SiC/SiC composites

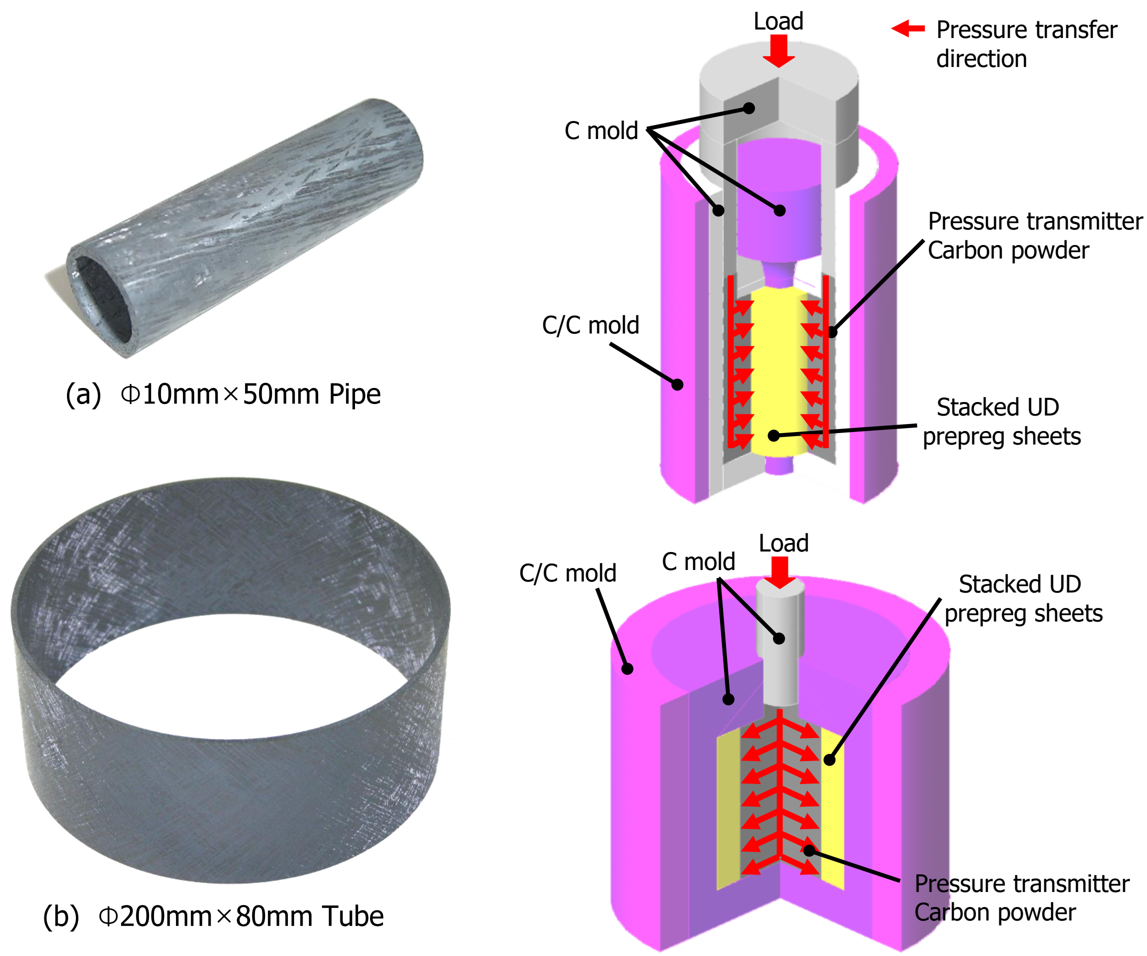

Fig. 5 Fabrication process of cylindrical shaped NITE-SiC/SiC composites

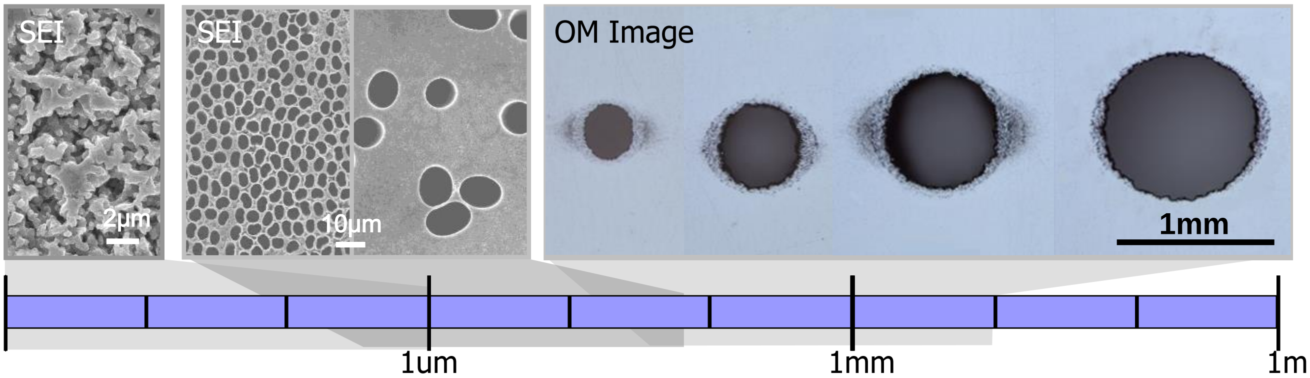

Fig. 6 Porous SiC materials with various types and sizes of pore manufactured by NITE Process

Table 1 NITE-SiC/SiC R&D history toward industrialization/commercialization.

	Lab. Scale	Pilot Grade #1	Pilot Grade #2	Pilot Grade #3
Reinforcements	Tyranno-SA (Φ7.5 μm, 1600Fil./Bundle)	Tyranno-SA (Φ7.5 μm, 1600Fil./Bundle)	Tyranno-SA (Φ7.5 μm, 1600Fil./Bundle)	Tyranno-SAK (Φ7.5 μm, 800Fil./Bundle)
Reinforcement Architecture	UD	UD X-ply, P/W	UD	UD
F/M Interface	PyC	None, PyC, BN	PyC	PyC
Amount of Process Additives	Standard	X1	X2	X2
Shape	Plate	Plate, Block, Tube	Plate, Tube	Plate, Tube