SiC/SiC Composite for Fusion by NITE Process and Its Performance

A. Kohyama [1)], Y. Katoh [1)], S.M. Dong [1)], T. Hino [2)] and Y. Hirohata [2)]

1) Institute of Advanced Energy, Kyoto Univ. and CREST-ACE, Uji, Kyoto, Japan
2) Faculty of Engineering, Hokkaido University, Kita-ku, Sapporo 060-8628, Japan

e-mail contact of main author: kohyama@iae.kyoto-u.ac.jp

Abstract: A new process, named Nano-powder Infiltration and Transient Eutectoid (NITE) process, has been developed and unidirectional SiC/SiC composites were prepared, and the effects of densification conditions on the microstructural evolution and mechanical properties were investigated. Carbon coated fibers were used as reinforcement and SiC nano-powders were used for matrix formation with certain amount of sintering additives. Density of the composites was improved with the increment of either temperature or pressure. Highest tensile strength was obtained at 1780C under 20MPa from the laboratory scale production. Many advantages of the NITE process were suggested as, promising mechanical properties, moderate to high thermal conductivity, very low gas permeability and low production cost. Based on the results, a large scale production was performed as the first trial at Ube Industries Ltd., Japan. Where 10cm cubic two-dimensionally reinforced SiC/SiC composites, 10cm diameter and 1cm diameter tubes and even a model component of real sized motor generator combustion liner head were produced by the NITE process. The excellent performance of these 2D SiC/SiC composites is provided.

1. Introduction

As the key technological challenge to make an attractive and competitive fusion reactor, silicon carbide (SiC) fiber reinforced SiC matrix (SiC/SiC) composites process developments have been extensively studied in these years [1-3]. One of the important accomplishments is based on the liquid phase sintering (LPS) process modification [4], where the new process called Nano-powder Infiltration and Transient Eutectoid (NITE) Process has been developed. This paper provides a remarkable breakthrough in SiC/SiC process, NITE process, development done by the CREST-ACE (Core Research for Evolutional Science and Technology, Advanced Composite Systems for Energy Conversion) program .

2. UD-SiC/SiC Composites by NITE Process

Pyrolytic Carbon (PyC) coated Tyranno SA fiber tows (Ube Industries Ltd., Japan) were used as reinforcement. Beta-SiC nano-phase powder (Marketek International, Port Townsent, US) with an average particle size of 20 nm was used for matrix formation, and Al_2O_3 (Sumitomo Chemical Industries Ltd., Japan) and Y_2O_3 (Johnson Matthey, UK) were used as sintering additives. Carbon coated fiber tows were first wound and fixed on the frames to form the aligned UD fiber sheets which were used to make SiC/SiC by NITE process [5]. By NITE process, even at 1750C with 15MPa pressure, 0.92 of theoretical density was achieved by a single process. At 1800C with 20MPa, it reached almost theoretical density. Representing microstructure of the SiC/SiC by NITE process is shown in Fig. 1, together with

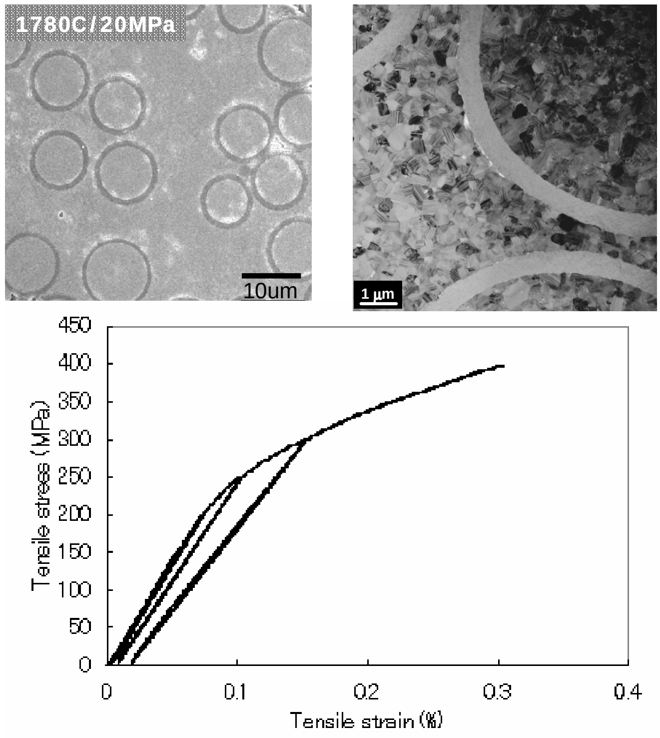

Fig.1 Representing microstructures and mechanical behavior of SiC/SiC composites fabricated through 'NITE' process.

the tensile test result. The SEM image (upper left), indicates highly densified matrix in intra-fiber bundle and the TEM image (upper right), indicates well crystallized matrix and fiber with uniform formation of carbon interface. The tensile test results (lower) provide high proportional limit strength, as high as 200MPa, and large strain at fracture. This is the result from load-unload tensile test, where after each unloading nominal Young's modulus became slightly smaller but proportional limit stress became larger. This is indicating the micro-cracks produced at higher than the proportional limit stress were very stable up to the stress level applied. This is an important mechanism that this material showed pseudo-ductile behavior under tensile test and flexural tests.

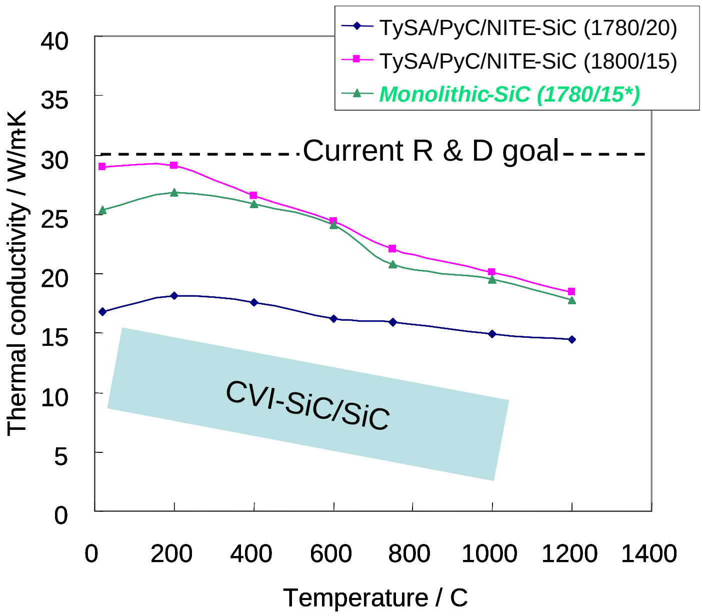

Fig. 2: Thermal conductivity of UD-SiC/SiC by NITE process

3. Characteristic Features of UD-SiC/SiC Composites by NITE Process

Figure 2 shows the through-thickness thermal conductivity measured for the UD composites made by NITE process. Comparing with the data band seen in the recent literature for the case of the composites made by CVI process, the thermal conductivity obtained from the composites made by NITE process exhibits significantly higher than those of F-CVI composites. Since the current process R & D efforts are emphasized on densification and mechanical property improvement, with the efforts to improve thermal property may bring further attractiveness to SiC/SiC. The recent efforts are indicating that by the purification of matrix, reduction of sintering aids, enhancement in crystallinity and selection of fibers and fiber-architecture optimization, further thermal conductivity improvement would be accomplished. Another significant feature is the resistance to thermal stresses. This is evaluated by using the thermal stress figure of merit shown in Fig.3. Ceramics materials, like SiC, are known to be advantageous with metallic materials only at temperatures higher than

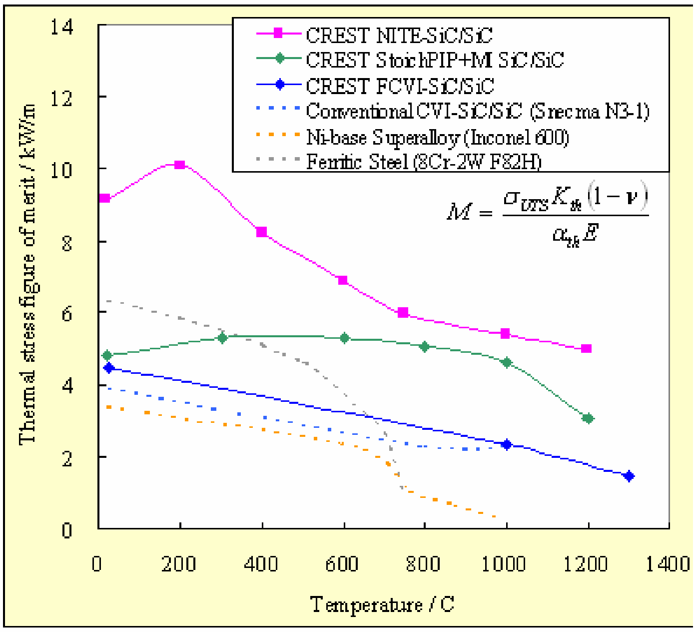

Fig. 3 Thermal stress tolerance of UD-SiC/SiC by NITE process comparing with other high temperature materials

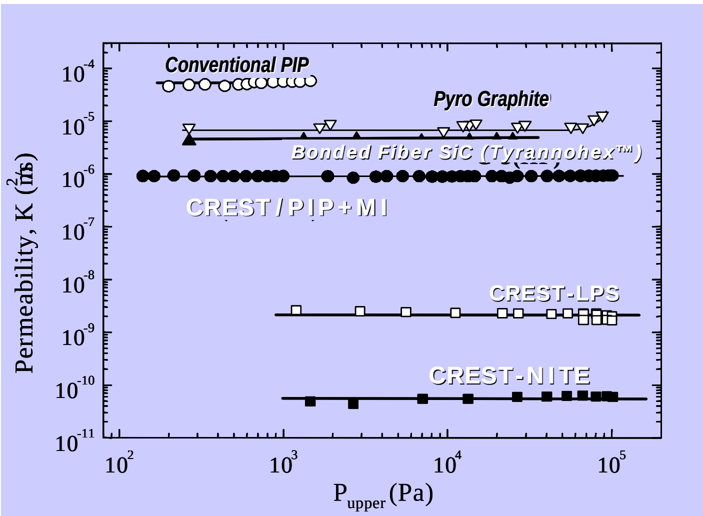

Fig. 4 Improvement in hermeticity-helium permeability-

700 C, but even by the SiC/SiC made by stoichiometry PIP + MI process (another process developed by CREST-ACE Program) the advantage becomes clear at 350 C. For the case of the SiC/SiC by NITE process, in every temperature range, no material has better thermal stress figure of merit value.

Another concern of ceramics composites is gas leak tightness and surface coating is thought to be the only solution to this. Therefore, hermeticity evaluation including helium permeability has been conducted. As shown in Fig. 4, comparing with the composites made with conventional processes and bonding, the SiC/SiC composites from NITE process and micro-crystalline LPS process exhibited helium permeability that is by orders lower than those for conventional materials and isotropic graphite (IG-110) [6]. This is the first result of ceramics composites showing reasonable helium leak tightness, which suggests the applicability of SiC/SiC composites to gas-cooled fusion blanket structures without a hermetic coating.

4.Trial of Large Scale Production

Based on the excellent results obtained from the laboratory scale products, the first trial to produce large scale 2D-SiC/SiC with varieties of shape was performed at Ube Industries Ltd., Japan. The typical shape and size of the two dimensionally (2D) SiC reinforced SiC composites using flat woven fabrics of Tyrano-SA by NITE process were as follows;

1: 97(w) x 97(l) x 70(t) mm block, 2: 195(w) x 195(l) x 2(t) mm plate,
3: 10(d) x 1.5(t) x 150(l) mm tube, 4: 150(w) x 150(l) x 15(t) mm cooling panel,
5: 106(d) x 3(t) x 100(l) mm tube.

As the comparison of thermal shock resistance with the newly developed SiC/SiC composites for the Japanese national project, Advanced Material Gas Generator (AMG) Project (1991-2000), cyclic heating test (combustion cycle test) by burning gas and cooling air was performed at Mitsubishi Heavy Industries. As shown in Fig. 5, SiC/SiC composite

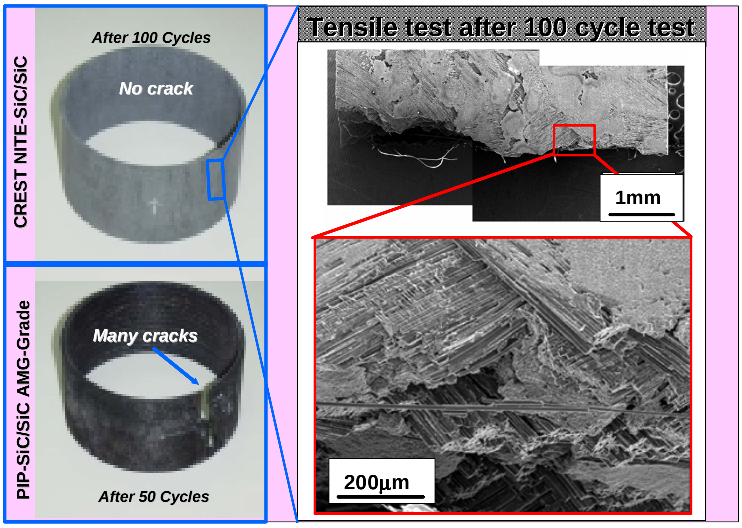

Fig. 5 Combustion test (between 1350C and 20C) results

from AMG project was broken before 50 combustion cycles (between 1350 C and 20 C). Whereas, for the case of the SiC/SiC by NITE process was quite stable, where no visible crack or damage was detected. After the combustion test, tensile test specimens were cut out from the test samples and were tested at room temperature. The two photographs right sides of Fig.5 were SEM fractographs of fractured tensile specimen of NITE composite. There was no degradation in tensile strength for the NITE composites and was about 80% degradation for the AMG materials. These results were closely related with the microstructural behavior by the combustion cycling test through oxidation and cracking, especially inter-laminar cracking only seen in the AMG materials, as shown in Fig. 6.

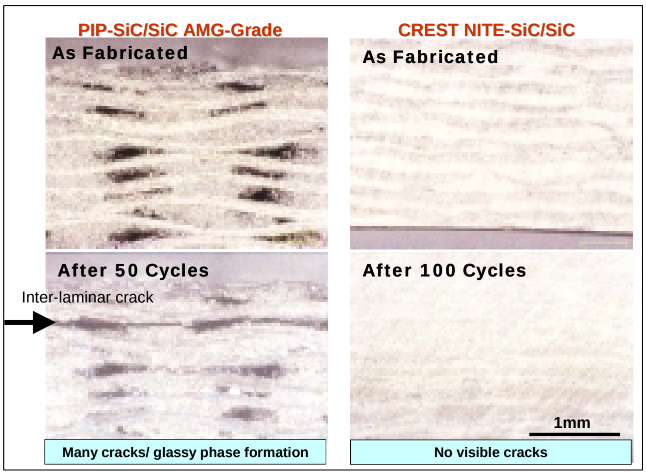

Fig.6 Microstructure change by combustion test

5. The Concept to Optimize NITE Process

As was mentioned, NITE process is the advanced concept of liquid phase sintering process, where fairly large amount of liquid phase was applied to make dense material. Figure 7 is a conceptual diagram indicating boundary lines to eliminate fiber damage and to satisfy sinterability (potentiality to make dense material). The lines for conventional process have no satisfactory condition, but by improvements in SiC fibers and by applying nano-infiltration technique, two lines are coming closer each other and finally the area satisfying these requirements. The shaded area shown in Fig. 7 indicates the condition for NITE process. To summarize the NITE process development, the followings are given;

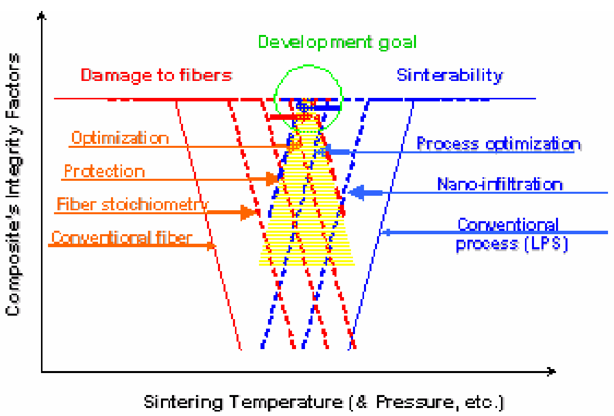

Fig.7 Direction of process development

1: Transient eutectic phase process was successfully applied to SiC/SiC composite production, where nano-powder infiltration benefits in many aspects and PyC interphase is effective against process-induced damages. The enhanced matrix densification is significant

2: The reduction of oxide remnants, mostly YAG, is a remaining issue. Although segregation to the rim of fiber-bundles was detected, grain boundary film formation was not confirmed.

3: Matrix grain size, microstructure/microchemistry appeared important for interface integrity largely affecting thermal conductivity. This might be tailored by raw materials and process temperature selection.

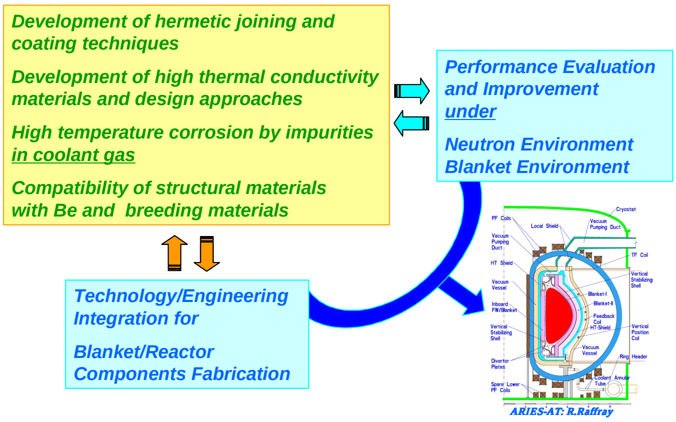

Fig.8 Engineering integration of SiC/SiC R & D

6. Engineering Integration

NITE process developed looks so far quite promising to be applied for the fusion power reactors. The current recognitions to supply electricity from fusion reactors by mid-21 century may require acceleration of materials R & D in time wise and quality wise. For this purpose, it is important to perform engineering integration together with generic materials development. Figure 8 indicates the strategy of the engineering integration where the importance to perform (1) blanket/reactor component fabrication and (2) performance evaluation and improvement under fusion environment is emphasized. The major issues are shown in the column top-left.

7. Summaries

There has been a great improvement in SiC/SiC performance under CREST-ACE. NITE process is a breakthrough which promises the reality of SiC/SiC to be applied for fusion power reactors. Based on the excellent properties obtained from UD-SiC/SiC by laboratory scale production, the first trial to produce large size 2D-SiC/SiC with varieties of shape was done. Even under un-optimized process condition at Ube Industries Ltd., those products presented outstanding performance.

This result is encouraging to go for the next step, where; (1)Elimination of polymer precursor for intra-fiber bundle densification, (2)Improved control of slurry quality, (3)Further reduction in amount of oxide additives, (4)Incorporation of interphase with improved oxidation resistance, (5)Improve net-shaping technique by pseudo isostatic pressing, will be investigated.

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