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Establishments of Fabrication and Evaluation Methods for Innovative SiC Fiber Reinforced SiC Matrix Composites

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Abstract. Based on the improvement in reinforcing SiC fibers and the utilization of very fine nano-SiC powders, the well known 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 microstructure stability which made them attractive for not only energy application but many other industrial applications. For the real deployments of these materials, mass production system and evaluation methods, together with the design code and safety assurance systems are essential. The current efforts to establish these bases were introduced.

Introduction

The development of ceramic matrix composites has been driven by the expectation of substantial economic and environmental benefits if these materials are used in transportation and energy-related industrial applications. Many of the potential applications for Ceramic Matrix Composites (CMC) components are characterized by long service lives that are measured in tens of thousands of hours, and service conditions that involve elevated temperatures and aggressive environments. Some of these applications include filters and heat-exchangers for coal-fired power plants and combustor liners for gas turbine engines.

SiC/SiC composites are considered for use in extremely harsh environments at high temperature primarily due to their excellent thermal, mechanical and chemical stability. In particular, recent improvement in the crystallinity and purity of SiC fibers, the developments and improved composite processing have improved physical and mechanical performance under harsh environments.

The novel processing called Nano-powder Infiltration and Transient Eutectic-phase (NITE) processing has been developed based on the liquid phase sintering (LPS) process modification. The NITE processing can achieve both the excellent material quality and low processing cost.

For the practical application of advanced SiC/SiC composite, it is necessary the establishment of the mass production system as well as the stable supply of raw material including high performance SiC fiber and nano-phased SiC powder.

Despite of the importance of comprehensive evaluation at service environment, the limited basic properties of the limited material have been evaluated for SiC/SiC composites. In order to understand its anisotropic mechanical properties at high temperature, development of various testing methods for different fracture mode is also one of important issues.

The objective of this paper is to introduce challenging for the establishment of mass production facilities and the characterization methods for advanced SiC/SiC composites in Japan.

Efforts for Mass Production of Advanced SiC/SiC composites

Mass Production System for Advanced SiC fiber. SiC/SiC composites is mainly consisted of SiC fiber, fiber/matrix interphase (usually, Carbon or Boron nitride have been used.), and SiC matrix. SiC fiber is most important constituent of SiC/SiC composites, because the mechanical properties and allowable temperature limits of SiC/SiC composites were generally dominated by the kinds of SiC fiber. SiC fibers produced via polymer pyrolysis especially have the advantage of flexible, fine-diameter form over those from chemical vapor deposition (CVD) or powder sintering process. SiC fiber, such as Nicalon® (Nippon Carbon Co., Ltd., Japan) and Tyranno® (Ube industries, Ltd., Japan), synthesized from organosilicon polymers, have been produced industrially and applied widely to heat-resistant materials and to the reinforcements for composite materials.[1] Among convention SiC fibers, Hi-Nicalon Type-S and Tyranno-SA show superior allowable temperature limits due their inherent properties including extremely low content of oxygen and high crystallinity. Fig. 1 shows conventional Tyranno-SA and its microstructure.

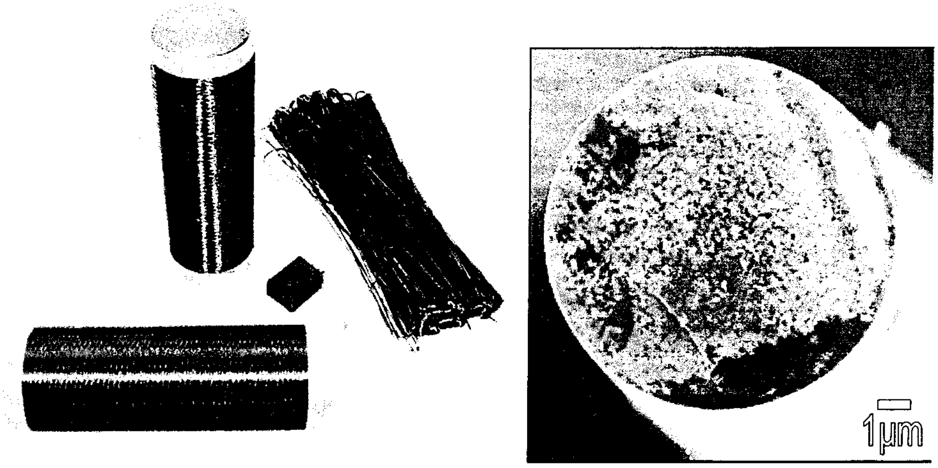


Fig. 1 Conventional Tyranno-SA fiber and its microstructure.

However, low productivity of these high performance SiC fibers is still remained as a technical key issue must be solved. Fortunately, the construction of new continuous production line for the new version of Tyranno-SA is challenged by Institute of Energy Science and Technology Co., Ltd. (IEST) under strong cooperative ties with Ube industries, Ltd. and Japan Ultra-High Temperature Materials Research Institute (JUTEMI). It is expected to produce 0.5~1 ton of highly homogeneous SiC fiber annually, when the construction of new continuous production is finished.

Pilot Grade Production of NITE-SiC/SiC composites

Based on the improvements in reinforcing SiC fibers and very fine nano-SiC powders, 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 microstructure stability which made them attractive for not only nuclear application but many industrial applications [2]. Under strong collaboration between Kyoto University and IEST, 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 wises. To indicate the feasibility of very high temperature helium gas cooled fast reactor by utilizing newly developed NITE-SiC/SiC composite materials, R & D on NITE process with near net shape foaming having been carried out. Fig. 2 and 3 show the large scale production scheme of NITE process and NITE-SiC/SiC composites with various size and shapes, respectively.

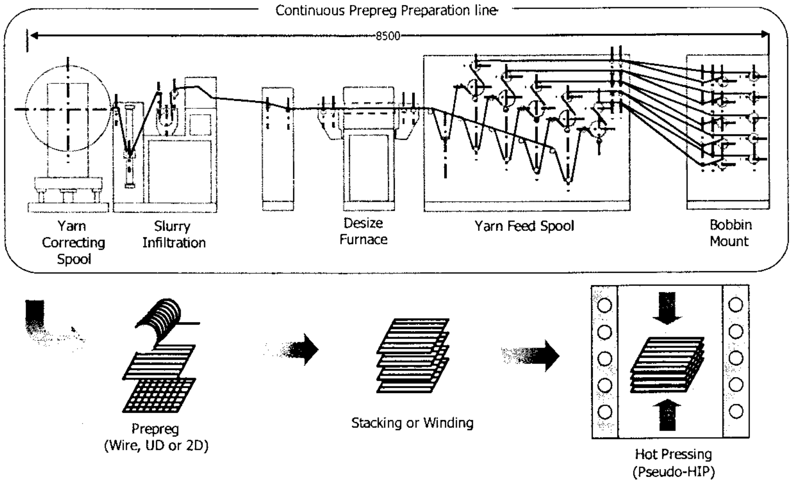


Fig. 2 Large scale production scheme of NITE process

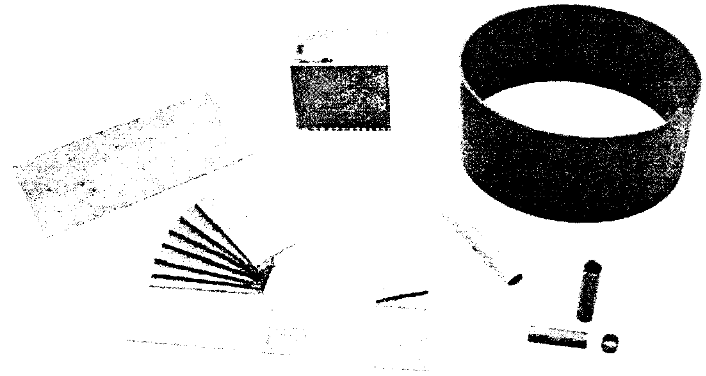


Fig. 3 Pilot grade NITE-SiC/SiC composites with various size and shapes

Efforts for Establishment of Evaluation Method for Advanced SiC/SiC composites

Miniature Tensile Specimen Test Technique. Small specimen test technique (SSTT) has been developed to meet several demands in advanced energy industries. Statistical analysis will be available as the number of specimens increasing. Moreover, what is most important is to make it possible to evaluate mechanical properties of very small "lab-scale" products for practical applications. Therefore, this technique has long been strongly required. For the establishment of this technique, miniaturization of test specimen has been considered the critical issue and related size effect study has also been recognized as very important. Fig. 4 exhibits two types of miniature tensile specimens; a face-loaded specimen and an edge-loaded specimen. Both provide high accuracy and a good repeatability. In particular, simple rectangular geometry of the face-loaded specimen can reduce unexpected flaws during machining.

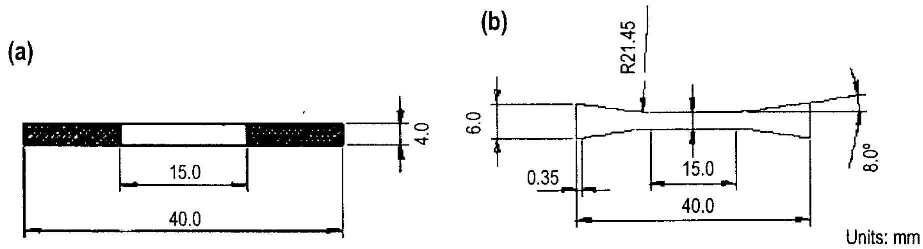
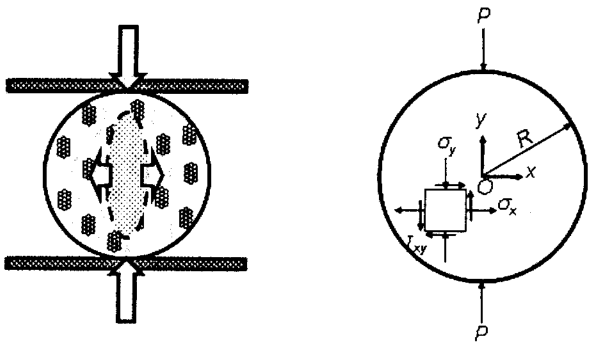


Fig. 4 Schematic illustrations of (a) a face-loaded miniature specimen and (b) an edge-loaded miniature specimen

In contrast, the edge-loaded specimen enables the high-temperature operation because of no requirement of metallic support tabs. However, it is not suitable for weak inter-laminar shear strength composite. Only face-loaded specimen is applicable to such a weak material.

Diametral compression test. The most common methods used to evaluate off-axis properties of CMCs are the shear tests. Although shear test methods are used to evaluate shear interlaminar strength in advanced ceramics, there is significant difficulty in test specimen machining and testing. Improperly prepared notches can produce non-uniform stress distribution in the shear test specimens, and can lead to ambiguity of interpretation of strength results. Such difficulties are identified in and American Society for Testing and Materials (ASTM) test standard titled, "Standard Test Methods for Shear Strength of Continuous Fiber Reinforced Advanced Ceramics at Ambient Temperatures," and identified as C1292-95. Shear test specimens also rarely produce a gage section that is in a state of pure shear. Recently, the transthickness tensile strength of CMCs has been successfully developed and defined at ASTM C1468. Uniaxially-loaded transthickness tensile strength tests measure the tensile interlaminar strength, avoid the complications listed above, and provide information on mechanical behavior and strength for a uniformly stressed material.

However, it seems that the application of conventional transthickness tensile test method to high temperature test environments is limited due to its fixturing. Test specimen must be bonded to the fixture by adhesives. In this case, the bonding strength between fixture and test specimen must be larger than the transthickness tensile strength of test specimen. Moreover, adhesive must be stand at test temperature. Diametral compression test, so called Brazilian test, has been generally used as an indirect tensile test method for concrete, rock, polymer and ceramics. Disk shaped specimen is compressed by two plates, as shown at Fig. 5. Although, the specimens with small diameter show large data spread in interlaminar strength, but any significant size dependence on average value can not be observed.



$$\sigma_T = \frac{2P}{\pi D t}$$

σ_T : Tensile stress
P : Compressive load
D : Diameter
t : specimen thickness

Fig. 5 Concepts of diametral compression test

Summary

The objective of this paper is to introduce challenging for the establishment of mass production facilities and the characterization methods for advanced SiC/SiC composites in Japan.

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