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# <Advanced Energy Conversion Division> Advanced Energy Materials Research Section

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Advanced Energy Materials Research Section

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1. Introduction

The research activity puts emphases on R&D of the advanced energy materials like SiC composites reinforced with SiC fibers with 10 μm diameter and tungsten composites reinforced with SiC fibers for aerospace, nuclear advanced fission and fusion application utilizing nano-technique. The R&D include development of novel materials, surface modification, and environmental effects including high temperature oxidation and irradiation effects from basic science through engineering. Many collaborative researches are ongoing with domestic and international institutions in US, EU and OECD.

2. Development of Particle Dispersion Silicon Carbide Composites by Liquid Phase Sintering

Silicon carbide (SiC) is one of very attractive engineering ceramics in particular for severe environment. Silicon carbide composites basically require weak fiber/matrix interphase like carbon (C) or boron nitride (BN). The interphase material and its thickness are keys to determine mechanical properties. However the interphase is the weakest link in terms of environmental effect like oxidation. Precise control of the interphase is also the critical issue in particular for large scale production and affects material cost significantly. The objective of this work is to develop oxidation resistant SiC composites without fiber/matrix interphase

by applying particle dispersion in SiC matrix.

Silicon carbide composites were fabricated by liquid phase sintering (LPS) method. Silicon carbide with BN matrix was formed by mixture of SiC powder and BN powder in LPS composites. The prepreg technique was developed for industrial application under collaboration with industry. Mechanical properties were characterized by various methods including tensile test and fatigue test in air up to 1400 C. Microstructures and fracture surfaces were characterized by FE-SEM and FE-TEM.

The BN particle dispersion composites showed excellent high temperature oxidation resistance. Oxidation was limited to near surface. The BN particle dispersion composites don't require fiber/matrix interphase. It decreases material cost significantly. Productivity is also excellent compared to conventional SiC composites. The BN particle dispersion composites didn't break applying 190 MPa following 100,000 cycles at 1400 C of fatigue tests in air without any coating. Machinability of BN particle dispersion composites is good. Stress-strain curves of the material fabricated by LPS seems brittle. However it has reliable matrix cracking stress and lower notch sensitivity like pseudo-ductile composites. Figs. 1 show silicon carbide fibers, prepreg and BN particle dispersion SiC composites.

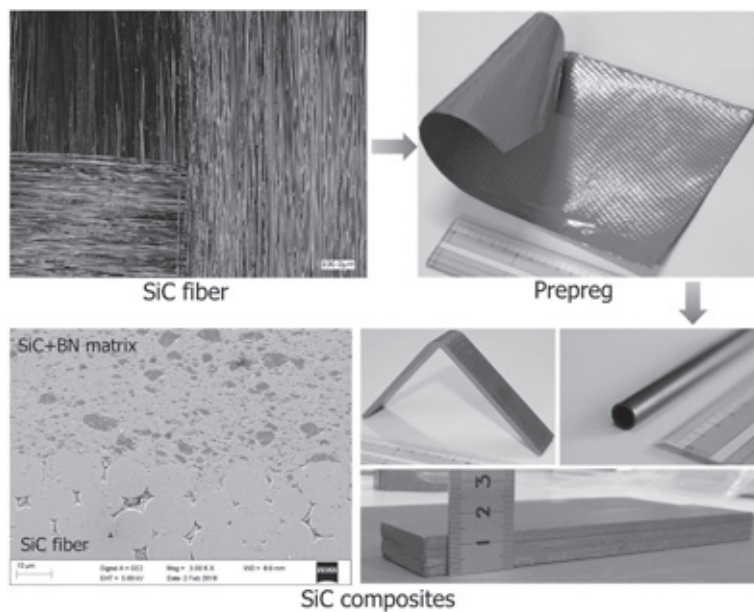


Fig. 1 Silicon carbide fibers, prepreg and BN particle dispersion SiC composites.

### 3. Ion Irradiation Effect on SiC Composites

Silicon carbide composites are expected to apply for nuclear fission and fusion environments due to stable properties under neutron irradiation. Dimensional change of high purity SiC saturated at around 1 dpa in most of temperature range. Microstructure and mechanical properties didn't change above around 1 dpa. Silicon carbide composites consist of SiC fiber, fiber/matrix interphase and matrix. Carbon interphase is a candidate for nuclear application.

It was found that C was unstable at high fluence in particular at relatively low temperature. One of highly crystalline SiC fiber, Hi-Nicalon type-S has small amount of C. Disorder and elimination of C interphase happened following 100 dpa ion irradiation by DuET. Shrinkage of Hi-Nicalon type-S was observed by AFM following 100 dpa ion irradiation as shown in Fig. 2, where high purity SiC matrix swelled. Carbon ribbons observed by FE-TEM for non-irradiated Hi-Nicalon type-S disappeared following 100 dpa irradiation. It is considered that C ribbons were dissolved in SiC grains. The issues of C interphase and C in Hi-Nicalon type-S were discussed under steering committee of Generation IV International Forum (GIF) –Gas Cooled Fast (GFR) Reactor System-, OECD NEA. Evaluation of high dose irradiation using ion irradiation and development of high dose irradiation tolerant SiC composites will be carried out under PROJECT ARRANGEMENT ON FUEL AND CORE MATERIALS under GIF GFR framework.

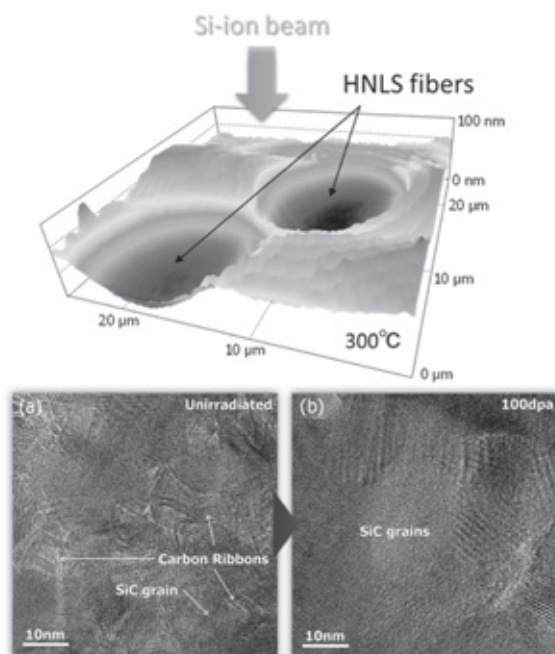


Fig. 2 Irradiation effect on irradiation induced swelling and microstructure of SiC composites.

### 4. Development of Silicon Carbide Fiber Reinforced Tungsten Composites

Tungsten is a primary candidate material for divertor and first wall. A tungsten material is expected to be used at high temperature due to its extremely high melting point. However mechanical properties of tungsten degrade at high temperature due to recrystallization of pure tungsten above 1000°C. Neutron irradiation also affects mechanical properties significantly. Silicon carbide has very close coefficient of thermal expansion with tungsten. Silicon carbide fibers can be used at above 1000°C under neutron irradiation without significant degradation of mechanical properties. The objective of this work is to develop the tungsten material with ductile behavior under high temperature neutron irradiation environment by reinforcement of silicon carbide fibers.

Tungsten foils or tungsten powders were sintered with silicon carbide fibers by various conditions at 1000~1800°C and ~20MPa. Carbon coated fibers and non-coated fibers were used. Mechanical properties were characterized by tensile test. Microstructure was evaluated by FE-SEM.

Very dense composites were fabricated in case of composites sintered with relatively small tungsten powders less than 1  $\mu\text{m}$  and reinforced with non-coated silicon carbide fibers, however pseudo-ductile fracture wasn't observed. The composites showed pseudo-ductile fracture behavior by fiber pull-out attributed to weak fiber/matrix interfacial strength in case of composites sintered with relatively large tungsten powders or tungsten foils, or composites reinforced with carbon coated silicon carbide fibers as shown in Fig. 3. The pseudo-ductile fracture behavior is independent of embrittlement of tungsten above recrystallization temperature or under neutron irradiation. The magnitude of tungsten can be reduced by replacing with silicon carbide fibers. It can contribute to safety in case of severe accident.

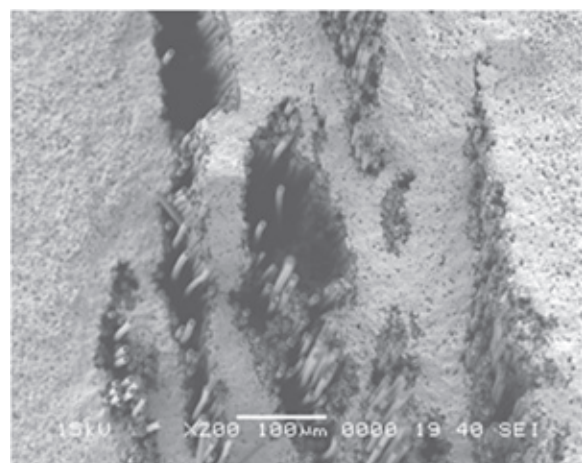


Fig. 3 Fracture Surface of SiC Fiber Reinforced W Composites.

## Collaboration Works

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檜木達也, THE EUROPEAN COMMISSION'S JOINT RESEARCH CENTRE (EU), Generation IV International Forum, GFR system

檜木達也, SCK-CEN (ベルギー), Project Coordinator, 先進事故耐性エネルギーシステムのための革新的被覆管材料

檜木達也, Oak Ridge National Laboratory (アメリカ), FRONTIER 計画 (原型炉ダイバータにおける界面反応ダイナミクスと中性子照射効果)

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