

Advanced Energy Materials Research Section

T. Hinoki, Associate Professor

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.

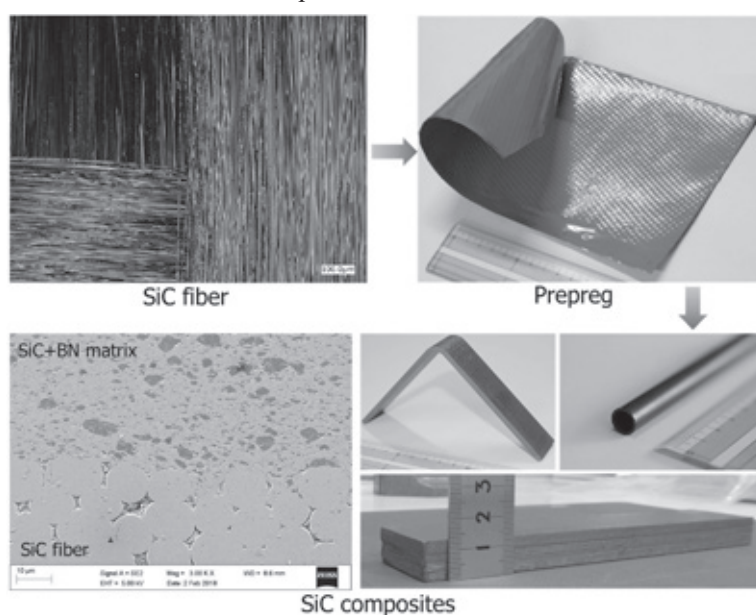


Fig. 1 Silicon carbide fibers, prepreg and BN particle dispersion SiC composites.

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.

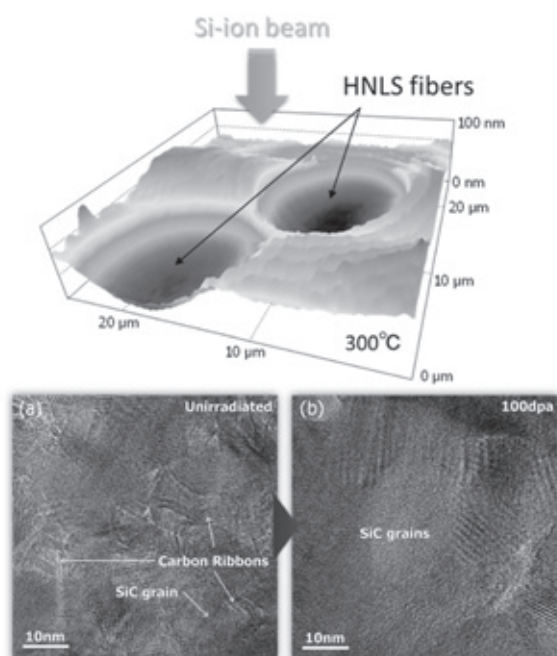


Fig. 2 Irradiation effect on irradiation induced swelling and microstructure of SiC composites.

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 μm 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.

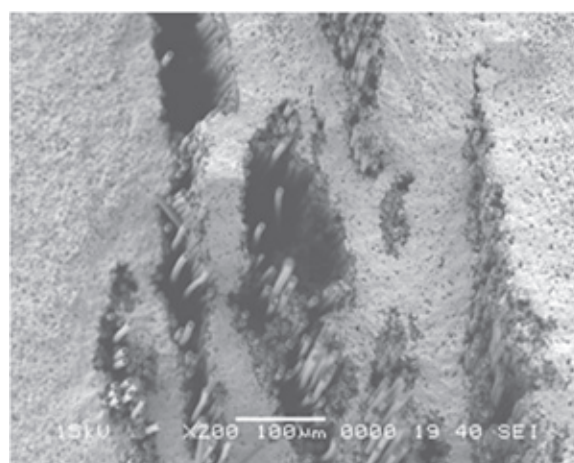


Fig. 3 Fracture Surface of SiC Fiber Reinforced W Composites.

Collaboration Works

檜木達也, Politecnico di Torino (イタリア), Oak Ridge National Laboratory (アメリカ), セラミックス材料の接合強度評価技術開発

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)

檜木達也, VUJE (スロバキア), Project Coordinator, Safety of GFR through innovative materials, technologies and processes -SafeG- (Horizon 2020)

Financial Support

檜木達也, 科学技術振興機構, 7-1. 材料特性評価を通じた極限環境下での材料挙動の理解

檜木達也, (株) フジミインコーポレーテッド, (研究題目非開示)

檜木達也, (株) フジミインコーポレーテッド, (研究題目非開示)

Publications

T. Koyanagi, Y. Katoh, T. Hinoki, C. Henager, M. Ferraris, S. Grasso, Progress in development of SiC-based joints resistant to neutron irradiation, Journal of the European Ceramic Society, 40, 4, 1023- 1034, 2020

F. Sun, M. Nakata, S.E. Lee, M. Zhao, T. Wada, S. Yamazaki, A. Koike, S. Kondo, T. Hinoki, M. Hara, Y. Oya, Synergistic effects of high energy helium irradiation and damage introduction at high temperature on hydrogen isotope retention in plasma facing materials, Journal of Nuclear Materials, 533, 152122, 2020

檜木達也, 金山拓夫, 扇谷聡, 福原俊介, 鴨志田圭吾, 下田一哉, 鶴井宣仁, 帆加利翔太, 耐酸化 BN 粒子分散 SiC 複合材料の開発, 日本セラミックス協会学会誌, 55, 6, 423-426, 2020

T. Miyazawa, L.M. Garrison, J.W. Geringer, J.R. Echols, M. Fukuda, Y. Katoh, T. Hinoki, A. Hasegawa, Tensile

properties of powder-metallurgical-processed tungsten alloys after neutron irradiation near recrystallization temperatures, Journal of Nuclear Materials, 542, 152505, 2020

K. Shimoda, T. Hinoki, Effects of fiber volume fraction on the densification and mechanical properties of unidirectional SiCf/SiC-matrix composites, Journal of the European Ceramic Society, 41, 2, 1163-1170, 2021

J. Wang, Y. Hatano, T. Hinoki, V. Kh. Alimov, A.V. Spitsyn, N.P. Bobyr, S. Kondo, T. Toyama, H.T. Lee, Y. Ueda, T. Schwarz-Selinger, Deuterium retention in W and binary W alloys irradiated with high energy Fe ions, Journal of Nuclear Materials, 545, 152749, 2021

Presentations

千田雅也, Yang Subing, 柴山環樹, 中川祐貴, 林慶知, 檜木達也, Si イオン照射された 4H-SiC における残留弾性ひずみの非破壊評価, 日本金属学会 2020 年秋期第 167 回公演大会, オンライン開催, 2020.9.15-18

Y. Du, B. Huang, F. Shinoda, K. Kawasaki, T. Hinoki, Effect of tungsten matrix on mechanical properties of SiC fiber reinforced W composites, 日本原子力学会 2020 年秋の大会, Online, 2020.9.16-18

檜木達也, プラズマ対向材料/構造材料界面の反応ダイナミクスと照射効果, スクレイブ・オフ層及びダイバータ物理サブクラスター 炉材料サブクラスター、ブランケットサブクラスターダイバータサブクラスター (令和 2 年度第 1 回) 合同会合, オンライン開催, 2020.11.9

檜木達也, 大矢恭久, 照射試料準備状況, 核融合科学研究所一般共同研究研究会「原型炉プラズマ対向機器のための界面制御技術の現状と課題」日米科学技術協力事業核融合分野 FRONTIER 計画国内研究者会議合同研究会「原型炉プラズマ対向機器のための界面制御技術の現状と課題」日米科学技術協力事業核融合分野 FRONTIER 計画国内研究者会議合同研究会, オンライン開催, 2020.11.13

檜木達也, 橋本直幸, Task 1 2019 年度および 2020 年度の成果および 2021 年度の予定, 核融合科学研究所一般共同研究研究会「原型炉プラズマ対向機器のための界面制御技術の現状と課題」日米科学技術協力事業核融合分野 FRONTIER 計画国内研究者会議合同研究会「原型炉プラズマ対向機器のための界面制御技術の現状と課題」日米科学技術協力事業核融合分野 FRONTIER 計画国内研究者会議合同研究会, オンライン開催, 2020.11.13

檜木達也, タングステン及び SiC 複合材料開発 (日米協力を中心に), 炉工学クラスター炉材料サブクラスター会合 (令和 2 年度第 2 回), オンライン開催, 2020.11.24

檜木達也, 国際共同研究における海外炉を用いた材料照射研究の課題から考える国内炉の必要性, 新たな照射試験炉の建設に向けたワークショップ, FUKURACIA 丸の内オアゾ (ハイブリッド開催), 2020.12.22-23

檜木達也, イオン照射材に対する様々な評価技術の開発, 日本原子力学会 2021 年春の年会, オンライン開催, 2021.3.17-19

LEE Junyab, 檜木達也, 無加圧液相焼結 SiC 材料の緻密化と機械的特性に及ぼすプロセスパラメーターの影響, 日本セラミックス協会 2021 年年会, オンライン開催, 2021.3.23-25

堺弘行, 檜木達也, 表面改質による SiC 焼結材の耐水蒸気特性の獲得, 日本セラミックス協会 2021 年年会, オンライン開催, 2021.3.23-25