

# HISTORY& FUTURE

The Center for Ceramic Matrix Composites (CCMC) was established within TUT on April 1, 2017. Professor Yutaka Kagawa became the Director of the Center and it started in two divisions with Professor Mitsuhiko Sato at first. Professor Chikara Fujiwara joined in 2018 and Professor Yoshihisa Tanaka in 2019, and the Center expanded to the current four divisions. Professor Michio Takeda joined in 2021. The Center receives support from experts at universities, national research institutes, and companies that have outstanding achievements in the field of CMCs throughout Japan. At present, the Center invites 20 experts as visiting professors.

Since its establishment, the Center has conducted valuable research and development in the practical



application of CMCs. Currently, the Center participates in various national projects and conducts joint research with several companies. The Center is also contributing to academic research. It has hosted a variety of international symposia, workshops and closed meetings, while also giving plenary and invited lectures at international and domestic conferences. Several members of the Center have received awards from academic societies.

The Center will continue to operate as an important base for CMC research and development in cooperation with various universities, companies, and institutes, both inside and outside Japan. It is also taking on the challenge of emerging technologies that will be important in the field of CMCs, including their application in virtual tests, digital twin, Al, CbA, and IoT. Looking into the future, it plans to add new Divisions in response to R&D requests from industry. The Center will further conduct various R&D activities in close cooperation with the Ceramic Matrix Composites Consortium, which was established in 2018 within the Japan Fine Ceramics Association (JFCA) in cooperation with companies, institutes, and universities.

An exhibition of the CCMC was held at the entrance lobby of the Ministry of Education, Culture, Sports, Science and Technology (MEXT) from April 1 to May 8, 2020 as a result of the adoption of TUT's application by MEXT. Several samples of CMCs were exhibited as the achievements of the research and development conducted at the Center. They are currently on display on the 1st floor of the Katayanagi Advanced Research Institute.

Liquid Si melt infiltration system (Fig. A) Fiber bundle re-winding system Ceramics coating equipment Ceramics machining and polishing machines Electric furnaces, Ultra high temperature furnaces

High-temperature mechanical test machine (MTS: maximum 1500 °C) (Fig. B)

High-temperature mechanical test machines (MTS: maximum 1500 °C, standard) (Fig. C)

High-temperature surface observation and digital image correlation strain measurement system, with universal testing equipment (maximum 1500  $^{\circ}$ C) (Fig. D) Screw-driven type universal mechanical test machine (Fig. E)

High-speed laser heating system (up to 1500 ℃ in 30 seconds) (Fig. F) Loading axis adjusting system

Talbot-Lau X-ray observation system with a mechanical loading system (Fig. G)

X-ray CT attachment for a Talbot-Lau X-ray imaging system (Fig. H)

Laser holography for a vibration measurement system (maximum 1500  $^{\circ}\mathrm{C}$  ) (Fig. I)

Micro-Luminescence stress/strain measurement system

Multiscale digital image correlation system

Micro-Raman high temperature stress/strain measurement system (maximum 1400 ℃) (Fig. J) Near-field nano-scale strain measurement system High-temperature FT-IR (maximum 1400 ℃) Optical microscope with a liquid tunable band pass filter

Scanning electron microscopes Fiber-matrix interface mechanical test machine Mechanical test machine for ceramic fiber

Computer simulation tools (FEM, Virtual test, Al, etc.) Library for CMCs (Books, Proceedings, CDs, etc.) (Fig. K)



The Center for Ceramic Matrix Composites Katayanagi Advanced Research Institute

Address 1404-1 Katakuramachi, Hachioji City, Tokyo 192-0982, JAPAN Phone +81-42-632-1566 URL : https://www.teu.ac.jp/karl/cmc/ E-mail : kl-cmc@stf.teu.ac.jp

## **EQUIPMENT** AT THE CCMC













Tensile strain, a Composite Fiber Fabrication Application SIC/SIC C/SIC Power-generation engine (Carrotor etc.)

THE CENTER FOR **CERAMIC** MATRIX COMPOSITES



# **OVERVIEW**

#### The Center for Ceramic Matrix Composites

Fiber-reinforced Ceramic Matrix Composites (CMCs) are important structural materials for elevated-temperature applications. The mission of the Center for Ceramic Matrix Composites (CCMC) is to promote research and development of advanced CMCs, such as, SiC/SiC, C/SiC, and Ox/Ox, with an emphasis on their application to high-performance engineering components. To this end, the CCMC focuses on important R&D fields covering fibers, coatings, processing, tests and analyses, reliability and databases, and the use of advanced computer technologies.

Composed of four research divisions representing major fields of ceramics and associated technical support and administrative offices, the CCMC is committed to play a central role in CMC research and development in Japan. With support from the Ministry of Economy, Trade and Industry (METI) and the Government's Cabinet Office (CAO), the CCMC serves as Japan's hub of CMC research. The Center conducts research activities with industry partners including major Japanese heavy industries, materials companies, national research institutes, and universities. Specially-developed processing and characterization equipment for CMCs can be accessed at the CCMC.

The CCMC provides high level education and research activities through advanced research and development of CMCs in cooperation with professors at Tokyo University of Technology (TUT), world-wide cooperative universities and institutes. Graduate and undergraduate students can find solutions to challenges through training in advanced technologies at the CCMC. Through the process, students are expected to become engineers and experts with practical competence in diverse fields in the near future.



# 04.

# ADVANCED MATERIALS AND PROCESS DIVISION

#### The ADVANCED MATERIALS AND PROCESS

**DIVISION** conducts research in a wide range of areas such as high performance ceramic fiber, new generation fiber coatings, and matrix for advanced CMCs. The goal is to develop high-performance CMCs that will play a key role in the materials industry through tensile strength prediction of SiC fibers in the SiC matrix, including residual stress measurement. Recently, the Division has focused on elevated-temperature properties of advanced SiC fibers and their improvement methods, and on the development of the next generation BN, "S<sup>2</sup>-BN" coating on the SiC fiber surface. At the same time, the Division is developing a process technology that will reduce the cost of manufacturing SiC/SiC components in the near future.

#### THE MAIN THEME OF RESEARCH

- 1. Performance improvement of SiC fibers
- Improvement of strength and creep resistance of SiC fibers through nano-structure control
- Methods of fiber tensile strength prediction using a fractographic approach, including residual stress measurement by Raman spectroscopy
- Evaluation and analysis of mechanical properties of SiC fibers and BN-coated SiC fibers

### 2. New S<sup>2</sup>-BN fiber coating for interphases of SiC/SiC

- Development of Smart System of precursor CVD, S<sup>2</sup>-BN coating process for third-generation BN coating
- Understanding of S<sup>2</sup>-BN coating effects on mechanical properties of SiC fibers and SiC/SiC fabricated by the reactive melt infiltration process
- Development of an inexpensive process route of the S<sup>2</sup>-BN coating on SiC fibers and a procedure for scaling up the coating process for industrial applications



SiC fibers still need to be improved both in strength and in creep resistance at elevated temperatures. However, the improvement of strength requires smaller grain sizes or an improvement of creep resistance. This research aims to achieve a breakthrough in the improvement of both properties simultaneously.

**Fig. A** : One promising approach to improve mechanical properties at elevated temperatures is to control the nano-crystalline structure of the SiC fiber. The photograph shows a TEM image of Tyranno Fiber SA3<sup>TM</sup> micronized in the surface region structure. The dimensions of the SiC crystalline are:  $d_A = 85$  nm,  $d_B = 83$  nm,  $d_C = 104$  nm,  $d_D = 287$  nm, and  $d_E = 335$  nm. This grain size of the order of nano-meters helps to improve the creep resistance of the fiber.

**Fig. B**: Development of the S<sup>2</sup>-BN coating has been conducted and its potential has been evaluated. The S<sup>2</sup>-BN coating realizes the well-controlled nano-structure of the BN layer that is expected to function as a high-performance coating layer. In addition, the S<sup>2</sup>-BN process is an environmentally friendly system.

#### The ADVANCED MANUFACTURING DIVISION

conducts research in the process of fabricating advanced non-oxide ceramic matrix composites. The Division conducts applied as well as basic research on elemental technologies that are crucial in developing new manufacturing technologies for the practical use of CMCs in the near future. It is also exploring the use of computer simulation to determine important parameters of future manufacturing technologies. Current activities focus on the preparation process for the SiC/SiC preform body, the optimization of the process of infiltrating molten Si into the SiC/SiC preform body through computer simulation and experimentation, and the effect of processing variables on the microstructure of SiC/SiC. Fabrications of the international standard SiC/SiC for nondestructive evaluation is another important mission of the Division.





There is still a lack of research on how to obtain high-quality parts using inexpensive manufacturing methods that are considered for industrial use. The reactive melt infiltration "RMI" process is expected to be an inexpensive manufacturing method for SiC/SiC components. However, in order to set processing variables, it is necessary to solve outstanding problems by applying the knowledge gained from past research.

**Fig. A** : Infiltration process of molten Si into the preform body is successfully observed directly, making it possible to control the process using specially-designed melt infiltration equipment. Processing variables can be optimized through a minimum set of experiments. Computer simulation of the infiltration process also facilitates the optimization of variables. The simulation allows us to estimate temperature rises and associated fiber degradation during the molten Si infiltration process. The method will be extended to determine the near-net shape fabrication route of the high-performance SiC/SiC components. This result is based on collaborative research between CCMC and Professor T.Yoshikawa, IIS, the University of Tokyo.

**Fig. B** : A typical example of SiC/SiC to be provided to joint research institutes internationally as a standard specimen for reliability research. This SiC/SiC is named "PteranoSiC."

#### TOKYO UNIVERSITY OF TECHNOLOG

#### THE MAIN THEME OF RESEARCH

#### 1.Inexpensive fabrication process of SiC/SiC using reactive melt infiltration process

- Simple preparation process for laminate-type SiC/SiC preform through slurry infiltration and filament winding process
- Understanding of the infiltration mechanism of molten Si into preform, modeling and quantitative analysis of the infiltration process of molten Si into the preform
- Computer simulation of the infiltration process of molten Si into the preform and understanding the relationship between process variables and performance of SiC/SiC

#### 2. Net shape component fabrication process

- Optimum melt infiltration process for the SiC/SiC component and reliability assessment of the component
  Total design of fabrication process for SiC/SiC, including fiber coatings, preform fabrication, compositing, and reliability assessment
- Optimization of processing variables for designing an inexpensive industrial fabrication process of near-net shape components

# 05.

# ADVANCED MANUFACTURING DIVISION

# 06.

MECHANICAL PROPERTIES AND RELIABILITY DIVISION The MECHANICAL PROPERTIES AND RELIABILITY DIVISION conducts research in the performance of CMCs. The research efforts are directed towards the evaluation and design of high-performance CMCs for elevated-temperature applications. Design and application of Environmental Barrier Coatings (EBCs) are included. Development of new experimental tools, e.g., a new type of X-ray CT, and the direct measurement of strain at elevated temperatures is conducted to bridge cutting-edge technologies and industrial applications. The Division is also conducting research on important nondestructive evaluation techniques necessary to ensure the reliability of SiC/SiC. It uses a wide range of CMCs with special focus on SiC/SiC and Ox/Ox composites.

#### THE MAIN THEME OF RESEARCH

#### 1. Reliability/performance evaluation of SiC/SiC and Ox/Ox from room to elevated temperatures

- Development of a new experimental technique for evaluation of mechanical properties at elevated temperatures. Standard elevated-temperature tests of SiC/SiC and Ox/Ox in accordance with ASTM/ISO standards
- Development of advanced direct strain measurement techniques at elevated temperatures of up to 1500 °C in ambient-air environments
- Study of time-dependent properties of SiC/SiC and Ox/Ox under creep and fatigue conditions

## 2. Analysis/evaluation of mechanical properties of CMCs and EBCs

- Analysis of unique mechanical behavior of CMCs, including fracture mechanics approaches, damage mechanics approaches, and simulation base approaches
- Development of new hardware for nondestructive evaluation (NDE) of advanced CMCs
- Study of physical and chemical degradation behaviors of SiC/SiC+EBC systems and life prediction of EBC systems based on interface fracture mechanics

#### Fig.A $\mathcal{E}_{xx}$ $\mathcal{E}_{yy}$ $\mathcal{E}_{xy}$ 0.020 0.015 0.010 0.005 0.005 0.000 0.005 0.005 0.005 0.000 0.005 0.005 0.005 0.000 0.005 0.005 0.005 0.000 0.005



Understanding of the deformation and fracture behaviors of CMCs at elevated temperatures is important. However, because of elevated temperatures, only limited methods are available. The new method developed at the Center enables direct observation and strain measurement at 1500  $^{\circ}$ C in ambient-air conditions,

with/without mechanical loading. By using the newly developed Talbot-Lau X-ray CT method, it is possible to obtain an image that utilizes scattering and interference behavior between X-ray and CMCs.

**Fig. A** : Surface strain distributions of a particle-dispersed composite have local stress distributions, which result from thermal expansion mismatch between particles and matrix. Direct strain measurement technique allows direct observation of local strain components from room temperature to 1500  $\degree$  with or without an external loading condition.

**Fig. B** : Talbot-Lau X-ray scattering image of a SiC/SiC near fracture portion vividly shows a multiple through-crack formation (B-1). In a low-magnification image, fiber bundle alignment, voids, and matrix cracks are shown (B-2). It is also possible to obtain the image under the test using the loading device installed in the Talbot-Lau X-ray system.



The COMPUTER ASSISTED TECHNOLOGIES

**DIVISION** conducts research in the application of advanced computational methodologies to the processing, evaluation, and analysis of CMCs. The focus of this Division is on the development of advances simulation methodologies and application to a multiscale virtual test to realize digital twin for CMCs. The application of Artificial Intelligence (AI) to reliability assessment of CMCs, especially the application of AI in the integrated judgement from multiple nondestructive tests is also considered as an important subject. The Division is also looking for ways to apply the latest digital technology to research and development for CMC component production: improving the efficiency of component design, manufacturing, inspection, etc. to find effective ways to further increase values in manufacturing. The Division is considering application procedure of the computational approaches to Certification by Analysis (CbA) in collaboration with other Divisions at the CCMC.





The computer is essential to make a valuable contribution to component design, analysis and evaluation, fabrication and related application of CMCs. Computational modeling and simulation have consequently become indispensable tools in helping to predict the damage behavior of CMCs across a range of use conditions.

**Fig. A** : A three-dimensional X-ray CT image is directly modeled into an XFEM model using an adequate image analysis procedure. XFEM analysis is conducted using the real microstructure of SiC/SiC. Matrix cracking behavior well demonstrates formation of through matrix cracks and multiple matrix cracks.  $\sigma$ : applied tensile stress,  $\sigma_{ss}$ : steady state matrix through cracking stress.

**Fig. B** : A new research effort aims to apply AI for NDE of C/SiC in combination with laser holography resonance analysis of C/SiC. Using a set of surface displacements, damage can be detected without a big-database. This result is based on collaborative research between the CCMC and Professor N. Shichijo, Hitotsubashi University.

#### THE MAIN THEME OF RESEARCH

- 1. Development of advanced simulation methodologies
- Development of virtual test codes for advanced CMC components, including long-term degradation behavior under harsh high-temperature environments
- Understanding of the process of characteristic damage evolution using an actual three-dimensional microstructure of CMCs
- Application of AI and IoT technologies for reliability analysis and CbA of advanced CMCs and its application to the judgement of levels of important properties

### 2. Development of New NDE methods for CMCs using advanced AI technology

- Automated damage detection of CMCs using vibration modes with deep neural networks
- Development of image analysis and extraction procedures for constituent phases from 3D X-ray CT images and building an analytical model
- Simulation of mechanical behaviors in CMCs under mixed loading conditions using eXtended FEM (XFEM) and determination of important mechanical properties

# COMPUTER ASSISTED TECHNOLOGIES DIVISION