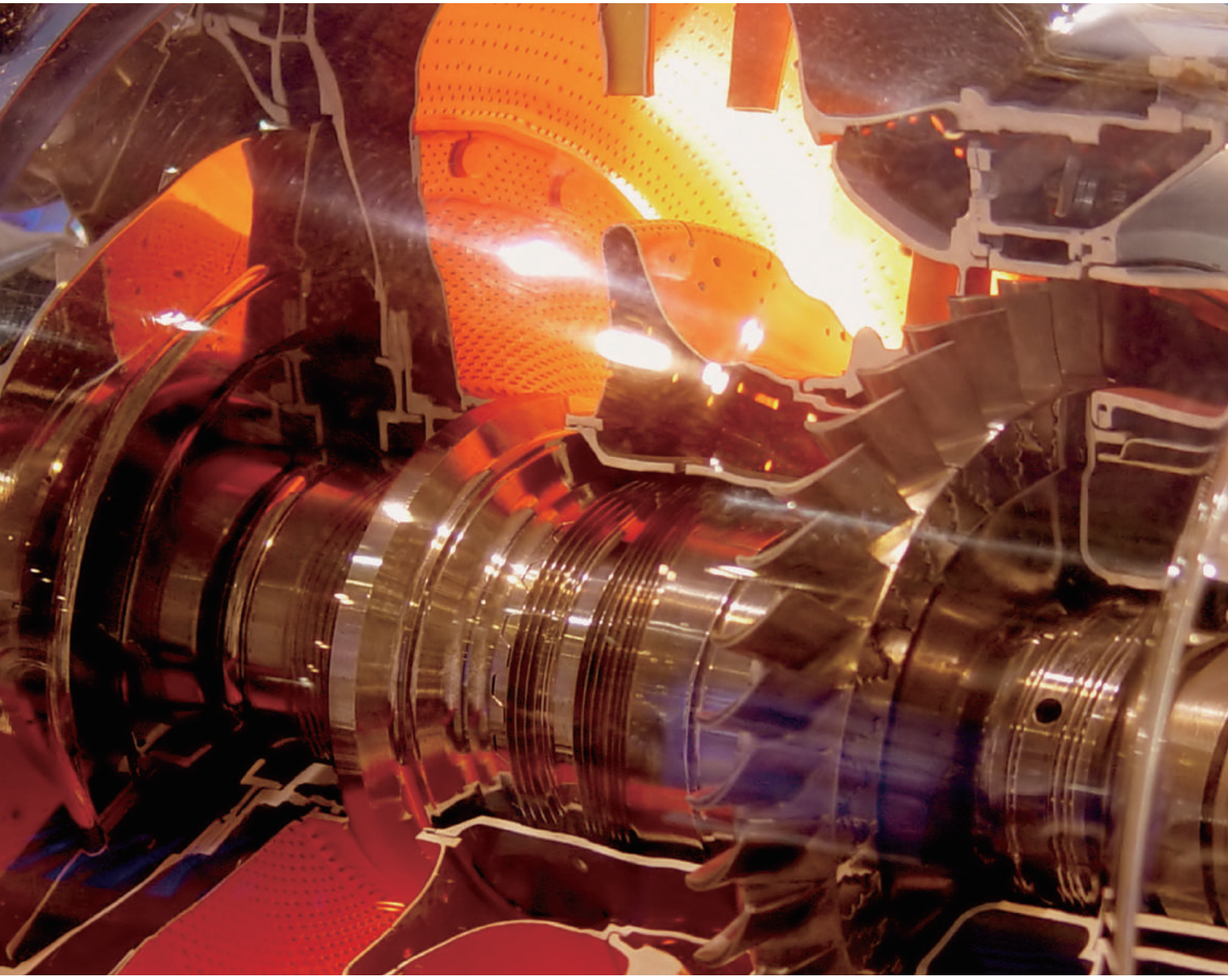
Ceramic Coating

Development of Ceramic Environmental Barrier Coating

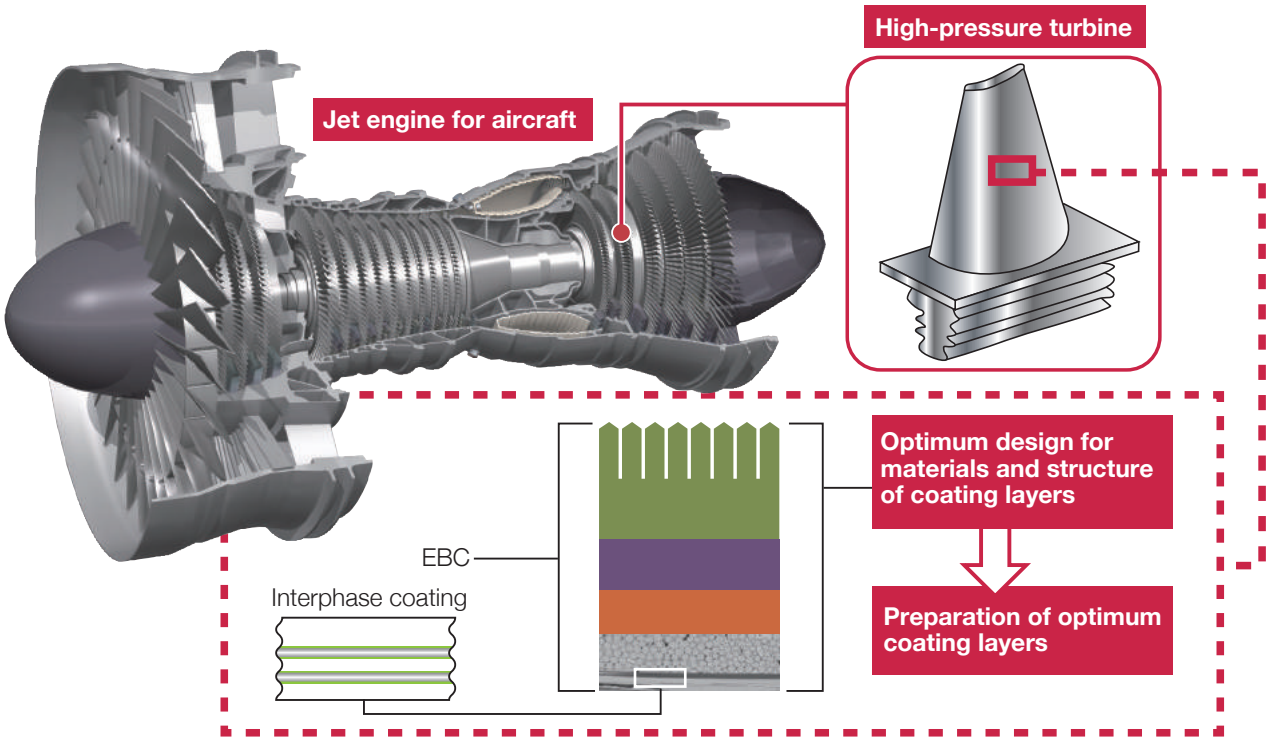
SIP-SM'4
Structural Materials for Innovation



- Development of environmental barrier coating (EBC) is necessary for the practical application of light-weight ceramic components in order to improve fuel efficiency and reduce CO_2 emissions for aircraft jet engines. We are developing excellent EBC and interphase coating technology with optimum structural and material design.
- EBC technology applicable to the production of the light-weight ceramic components promotes the commercialization of advanced aircraft engine technology in Japan, and enhances the domestic supply chains, from raw materials to finished products.

SIP Cross-ministerial Strategic Innovation Promotion Program

Concept & Approach



We are developing light-weight ceramic components that maintain their EBC and interphase structures and thermomechanical durability even after a burner rig test at 1400°C under an atmosphere including oxygen and water vapor. The following innovative technologies and methods will be developed:

(1) Optimum material design of multilayer ceramic coatings that show excellent heat resistibility and anti-permeability against oxygen and water vapor.
(2) EBC processing technologies to realize optimum multilayer ceramic coatings.
(3) Interphase ceramic coating technologies on surfaces of SiC-fibers, which give damage tolerance effect to the substrate under ceramic coatings.
(4) A method to predict critical conditions of EBC fracture and delamination, and demonstrate excellent properties of the components developed in this research.

Research Themes and Participating Laboratories

Framework for R&D in Ceramic Coating Area

Theme 1

We are developing EBC and interphase coating technologies, based on the optimum design of the coating layers.

Theme 2

Collaboration between industries, universities, and public laboratories is sufficient to evaluate EBC and interphase coatings developed in the SIP program, as well as actual applicability under a combustion gas environment.

Theme 1: Coating Technology

EBC

JFCC (Japan Fine Ceramics Center)

Development of optimum design of high temperature resistant coatings and preparation of EBC by EB-PVD method

Yokohama National University

Development of oxygen barrier coatings by aerosol deposition method

Tohoku University

Development of binding layer coatings by chemical vapor deposition method.

Interphase Coating

Development of interphase coatings by chemical solution deposition method

Yokohama National University

Development of interphase coatings by chemical vapo deposition method.

R&D of Ceramic Environmental Barrier Coating

Optimum design of EBC coating

■EBC

①Material design for environmental shielding — JFCC

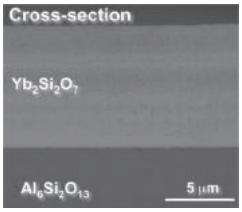
Mass transfer within EBCs, such as oxygen permeation through a mullite layer, was studied and analyzed to obtain an optimized EBC structure. Therefore, a robust EBC structure with environmental shielding and thermo-mechanical functions is proposed.

Processing technology of coatings

■EBC

①Double electoron beams PVD — JFCC

An Yb-silicate layer with dense and/or porous structure was successively formed on substrates using a double electron beams PVD apparatus.



Layers coated by DEB-PVD

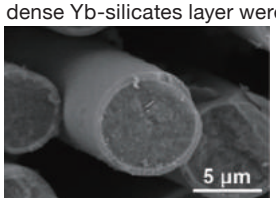
②Aerosol deposition — Yokohama Natl. Univ.

Dense crystalline monolithic layers of mullite were formed at room temperature without heating by Aerosol Deposition (AD) method.

■Interphase coating

①Laser-CVD — Yokohama Natl. Univ.

SiC fibers can be covered with Yb-silicates by a laser-assisted CVD process. Conditions for forming a dense Yb-silicates layer were obtained.



A layer coated on a SiC fiber by laser CVD

②Solution deposition — JFCC

Optimization of a coating solution, a coating process,and heat treatment conditions were studied. A dense and/or porous Yb-silicates coating later was obtained on the surface of a SiC fiber.

Evaluation of coating performance

■EBC

①Evaluation of applicability of EBC — IHI

Preliminary heat cycle tests using a burner-rig were carried out at 1350 °C for 90 cycles after preparation of jig to hold the specimen and study the test conditions.

②High Temperature Corrosion Test — JUTEM

Apparatuses were improved to carry out a corrosion test in water vapor at high temperature and the test using EBC materials was started under a high-pressure atmosphere with water vapor at 1400°C

③Soundness evaluation — NIMS

Construction of an evaluation method targeting interface toughness for multilayer coatings is being developed. The method will be standardized as a new toughness test.

④Mechanical property evaluation of EBC — TIT

A high temperature nano-indentation system attached to a laser heating apparatus has been installed and the temperature dependency of elastic modulus of constituent materials is being measured.

⑤Simulation of EBC fracture under mechanical and thermal loading — Univ. Tokyo

A FEM model to obtain energy release rates for EBC materials has been developed. We have obtained energy release rates for various crack propagation and loading directions.

■Interphase coating

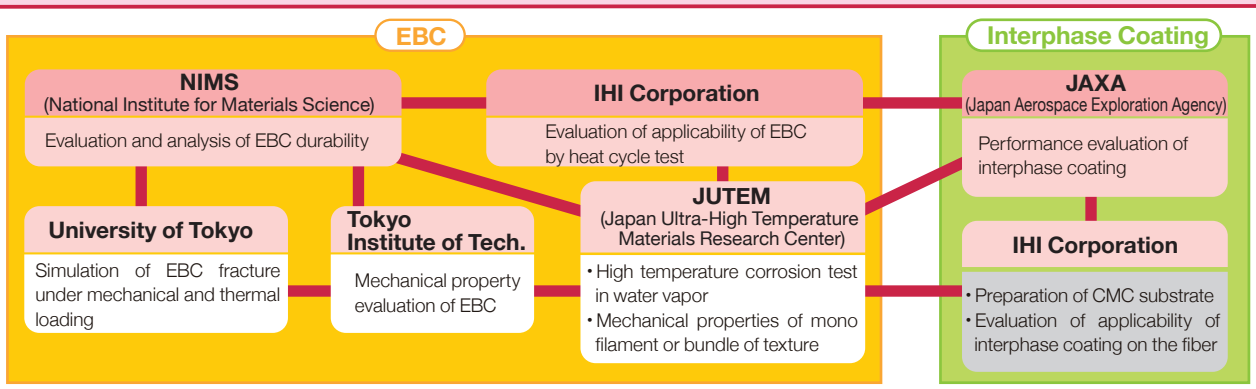
①Soundness evaluation — JAXA

Fiber-bundle mini-composites have been prepared and the performance is being evaluated. The fiber push-out method was studied to evaluate the interface properties between a fiber and matrix.

②Evaluation of applicability of interphase coating — IHI

Specially designed furnace for the MI process has been successfully constructed. A small-scale process optimization is now in progress.

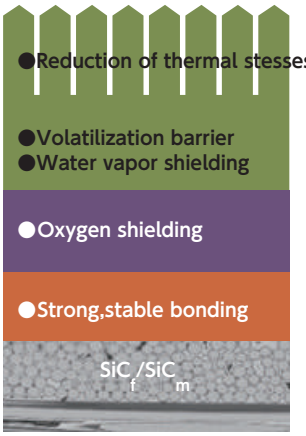
Theme 2：Evaluation of coatings for applicability to aircraft engines



Breakthrough in Advanced Aircraft Engine Applications at 1400°C

Advanced structural design for optimization of environmental shielding and thermomechanical properties

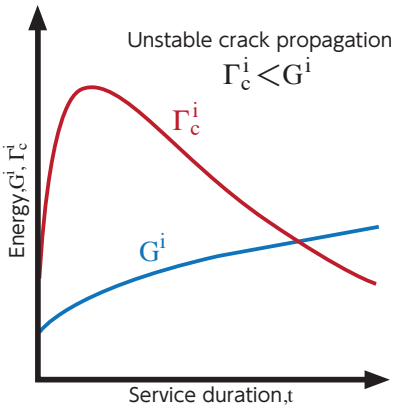
It is crucial to reduce the weight of engine components and improve heat resistance for decreasing both fuel consumption and CO_2 emissions. In order to use ceramic components that are lighter in weight and more heat resistant than Ni-based super alloy, an environmental barrier coating (EBC) is necessary to cover the surface of component for a long period of time.
Therefore, an advanced design for both environmental shielding and thermomechanical durability is being developed on the basis of mass-transfer mechanism through the EBC under high temperature atmosphere including oxygen and water vapor. We are newly developing the coating technologies to fabricate multi-layered EBCs under development with the goal of achieving exceptional performance by the overall EBC system through the use of layers with specific characteristic functions.



■Multilayer coating system of EBC

Novel method to evaluate fracture toughness of EBC

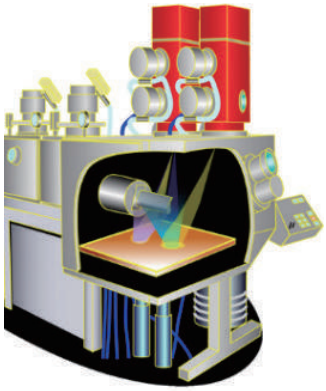
No method has been established thus far to accurately estimate the conditions where EBC is safely used. The establishment of a new method is essential to enhance the reliability of aircraft engines.
This study measures fracture toughness of EBC after a durability test under a mock environment of operation. The energy release rate is also calculated, taking into account changes in properties of component materials. We will establish a method to predict critical conditions of EBC fracture and delamination from temporal changes in these values due to damage development and structural change. We will optimize EBC structure using the established method.
We will develop a test that can evaluate realistic fracture modes with a compact and simple specimen so that it can be used for actual coating materials. The application of the test for supporting future certification and standardization is within our scope.



■Evaluate usage limits of EBC

Scheme of double electron beam PVD apparatus

Based on the EBC design guidelines, we are developing robust EBCs to allow longer periods of practical use under combustion environments at 1400 °C by an electron beam-PVD (EB-PVD) technique, which is commonly applied to form a thermal barrier coatings (TBC) on Ni-based superalloys. The latest double electron beam (DEB)-PVD is able to form the EBC constituent complex oxide layers containing components with very different vapor pressures, chemically gradient composites, and so on.



■Scheme of Double Electron Beam PVD apparatus

Japan Science and Technology Agency

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Japan Science and Technology Agent (JST)
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Members of Research Domain C: Ceramic Coating

Expectations for the development of environment -resistant ceramic coatings

TERUO KISHI
Program Director

Developing heat-resistant and environmental barrier coatings (EBCs) and applying them to lightweight ceramic components greatly reduces the weight of turbine rotor and stator blades as components of aircraft engines, and making the components dramatically more heat-resistant, durable, and reliable in operating environments. By researching SIP, we expect to develop technologies for fully using, as coatings, new ceramic materials having oxygen and water vapor shielding properties in an operating environment in the order of 1400°C which has yet to be achieved in the world. Beyond material development, our results should lead to the rapid application of such materials to actual machinery for industrial purposes.

Area Director in the Domain for Ceramics Coating
MASASUKE TAKATA
Japan Fine Ceramics Center (JFCC)

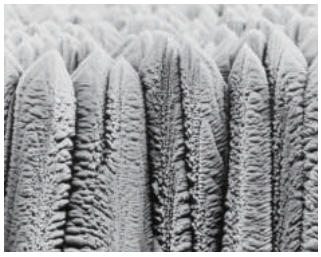
As a nation, Japan does not have sufficient natural and energy resources, and must therefore make wise choices in order to survive and thrive. Fortunately, the Japanese are diligent and well-educated people. When the EBC (the objective of this research) is developed, it will be play an important role in conserving energy, and a substantial reduction in CO_2 can be expected. EBC development can also contribute to the advancement of Japan's airplane industry. As area director, I strive to develop the first advanced EBC in Japan by working with highly skilled and motivated partners in industry, government, and academia.

Development of advanced ceramic coating processes

Keywords : Coating, Diffusion, Grain boundary, Surface, Electron-beam PVD, Chemical solution deposition

SATOSHI KITAOKA
Japan Fine Ceramics Center

An advanced multilayer environmental barrier coating (EBC) exhibiting excellent environmental shielding properties, heat-cycle resistance, and structural stability has been developed based on a quantitative analysis of mass transfer in complex oxides in a simulated high-temperature environment. The EBC is formed on a ceramic matrix composite (CMC) using electron-beam physical vapor deposition (PVD). In addition, in order to improve the resistance of the CMC to damage during long-term exposure to a high-temperature environment, interphase coating of the fibers was carried out using chemical solution deposition. This figure shows the microstructure formed by electron-beam PVD technique. The feather-like texture and nanometer-scale voids provide excellent heat-cycle resistance and thermal barrier properties.

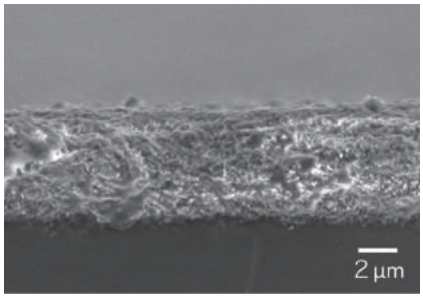


Development of oxygen barrier coatings by aerosol deposition method

Keywords : Aerosol deposition (AD), Coating, Oxidation, Microstructure, Thermal stability

MAKOTO HASEGAWA
Yokohama National University

Mullite is one of the component materials for the environmental barrier coating. As for the coating process used generally for the mullite coating, it has a possibility to oxidize the substrate due to the exposure at high temperatures. Aerosol deposition (AD) is a coating process that can produce the coating by collision of the ceramics powders in a sonic velocity under vacuum at room temperature. It produces dense and crystalline coating at room temperature without oxidation of substrate. Further, there is no change of chemical composition between the coating and processed powder. AD is effective to produce the coating in a few μm ordered thick. However, due to the low deposition rate, it is not realistic to produce the thick coating about ~100 μm. In this study, mullite coating by AD is considered as an oxygen barrier "pre-coating." The figure shows a typical example of the mullite coating by AD. Dense mullite coating without oxidation of substrate is formed. From the XRD analysis, the mullite coating is crystallized.



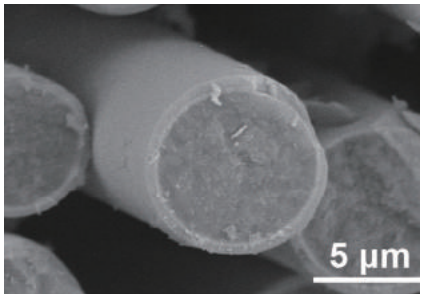
Dense mullite coating processed by AD

Development of interphase coatings by laser chemical vapor deposition

Keywords：Interphase coating, Chemical vapor deposition (CVD), Rare-earth silicate

AKIHIKO ITO
Yokohama National University

Interphase coatings on fibers, which reinforce ceramic composites, secure damage tolerance of ceramic matrix composites (CMCs). Current coating materials poseissues of degradation due to damage tolerance as a result of oxidization by a high-temperature water vapor. Thus, a stable interphase material and its coating process should be developed. A laser-assisted chemical vapor deposition can produce coatings with the control of microstructure and orientation. An ytterbium silicate layer, which has a dense structure with crystal orientation, would enhance the environmental barrier properties of interphase coatings and damage tolerance of CMCs. We have demonstrated the orientation control of ytterbium silicate coatings on a model substrate and the preparation of dense coatings on SiC fibers. We will optimize the coating process through collaborative works in the SIP program. The achievement can be a part of the high-performance environmental barrier coating system for the next-generation gas turbine engine.

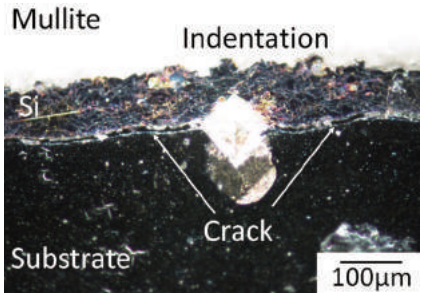


Evaluation and analysis of EBC durability

Keywords：EBC, Lifetime limitation, Interface, Fracture toughness

HIDEKI KAKISAWA
National Institute for Materials Science

We aim to show the lifetime limitation of EBC by analyzing the key factors causing EBC damage: investigating chemical (structure collapse by reaction and material transfer) and physical (delamination and through-thickness crack) damages. Therefore, we are developing an interface fracture test for EBCs damaged in simulated environmental conditions in order to obtain the change of interface toughness as a function of thermomechanical exposure time. The test under development allows us to reproduce the fracture mode of EBC with a specimen as small as possible cut out of a real component, targeting the new standardized toughness test. The figure shows an example of pre-crack introduction by an indentation technique at any interface in the multilayer benchmark specimen.

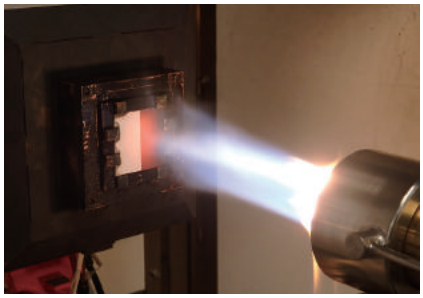


Evaluation of applicability of EBC and interphase coating on the fiber

Keywords : CMC, Evaluation, Applicability

TAKESHI NAKAMURA
IHI Corporation

We have extensive experience of aero-engine development and CMC research, and we have a role to supply CMC plates to the collaborative research groups. A furnace to manufacture and supply CMC plates has already been introduced and the preliminary thermal cycle tests at over 1000°C have been conducted.
The CMC materials developed in this research project should have good anti-recession properties in a combustion gas including water vapor at 1400°C. We evaluate the applicability of interphase coating on the fiber and EBC on CMC. As for an interphase coating, we evaluate fatigue and creep properties of the material after a matrix infiltration process. As for EBC, we evaluate a durability and adhesiveness of the coating by thermal cycle tests, using the burner rig facility shown here.



Members of Research Domain C: Ceramic Coating

Simulation of EBC fracture under mechanical and thermal loading

Keywords : Finite element method, Coupled thermal-stress analysis, Crack

YOSHITAKA UMENO
The University of Tokyo

As EBC is exposed to severe temperature gradients and thermal cycle conditions during operation, fracture is likely to occur due to strong thermal stress in the coating layers. To assure a high reliability and support the design of EBC, the estimation of criteria of interface and transverse fracture by finite element method (FEM) simulation is demanded. We conduct simulations by taking into account actual thermal environments around EBC with the aim to improve the design of the EBC structure. To clarify the mechanism and criteria of EBC fracture, we evaluate the strength of EBC in operation through mechanical analyses under thermal and mechanical loading conditions. Simulation results will lead to sophisticated guidelines for designing the EBC structure.

Stress distribution near crack tip

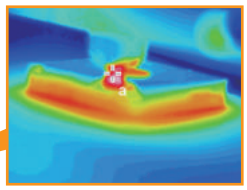
Mechanical property evaluation and structural design of EBCs

Keywords : Nanoindentation,Temperature dependence, Young's modulus, Internal stress

TAKASHI AKATSU
Tokyo Institute of Technology

The energy release rate of EBCs is a key parameter to safely use turbine blades with EBCs during a fixed period of time, where the rate is designed to be smaller than fracture energy. The energy release rate is given as a function of crack size, applied stress, the elastic modulus of each EBC layer, residual stress, etc. In this project, the elastic modulus of each EBC layer is measured from room temperature to elevated temperatures to quantitatively evaluate stresses on each EBC layer and estimate the energy release rate. Nanoindentation technique is adopted to measure the elastic modulus. The elastic modulus of EBC layers heated at elevated temperature for an extended amount of time is also measured to examine the durability of EBCs.

High-temperature nanoindentation system



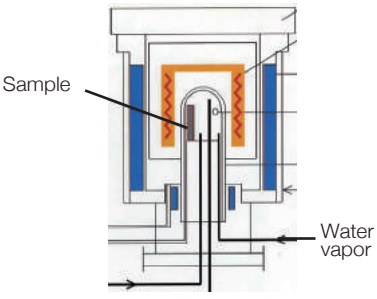
Thermography around an indenter at elevated temperatures

High Temperature corrosion test in water vapor and Mechanical properties of mono filament or bundle of texture

Keywords : Mechanical properties, Severe environment, High temperature, Thermodynamics

YUKIHIKO SUGIURA
Japan Ultra-High Temperature Materials Research Center (JUTEM)

EBC for CMC materials is evaluated by measuring mass change and optical observation after exposure under pressurized vapor at 1400°C for an extended amount of time. The degradation of texture after the coating process and the resistance to the environment of interphase coating are evaluated by measuring the tensile strength changes before/after the exposure test of interface coated texture.
JUTEM is the only institute in Japan that has been evaluating the high temperature properties of CMC for more than 10 years. The technology developed at JUTEM supports the improvement of CMC materials and EBC; therefore, the results of evaluation are applicable to real engine systems.

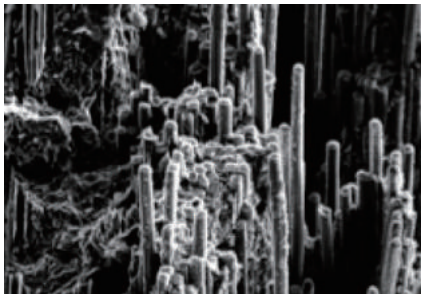


Performance evaluation of interphase coating

Keywords : Interface, Fracture, Mechanical properties

KEN GOTO
Japan Aerospace Exploration Agency (JAXA)

In order to make damage-tolerant SiC fiber-reinforced SiC composites, the interface between fiber and matrix needs to possess a function to deflect matrix cracks to fiber axis directions. This section evaluates the performance of interphase coating. The evaluation methods undertaken are techniques using fiber bundle mini composites and the single fiber push-out. The evaluation will include change of interface micro-structures, residual thickness of interphase coating, and/or a chemical reaction of fibers after SiC matrix processing. Finally, the interphase coating performance evaluation will provide feedback to the coating process unit to obtain a proper interphase coating system for advanced SiC/SiC.



Coordinator in Domain C
for “Ceramic Coating”
MINEO MIZUNO

My name is Mineo Mizuno, and I am a coordinator in Domain C. I remember the days of conducting research on ceramic matrix composite (CMC) materials. There was a government-funded 7-year research project starting in 1992 on the R&D of 100 kW ceramic gas turbines for automobiles (so-called PEC-CGT). I was responsible for the development of evaluation methods for CMCs, including SiC/SiC materials. In addition to publishing more than 10 papers, 14 pieces of the PEC standard on evaluation methods for CMC properties were issued in 1997. Several JIS and ISO standards for CMCs are based on the PEC standard. In those days, the SiC/SiC seemed to be too expensive for aircraft engine components. However, at present, their application will soon be realized. I hope to develop new SiC/SiC materials with excellent EBC in this research.

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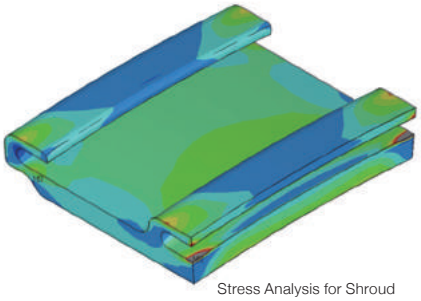
Members of Research Domain C: Ceramic Coating

Development of oxide ceramics matrix composite coating sheets

Keywords : Oxides, Toughness, Affordable, Abradable, Shroud

MASANORI USHIDA
MITSUBISHI HEAVY INDUSTRIES AERO ENGINES, LTD. Engineering Department

The concept of the coating of oxide ceramic matrix composite sheets adds toughness to a monolithic ceramic substrate and prevents crack propagation into the ceramic, resulting in suppression of catastrophic fracture. Additional thermal barrier coating layers on the surface protect bare oxide CMC sheets from hot gas and expands applications to higher usable temperatures. This coating structure with slight cooling could have a similar heat resistance capability as SiC/SiC CMCs, but may reduce the manufacturing cost to about one-tenth the cost of SiC/SiC.
This group is developing CMC coating sheets, improved heat resistance oxide fibers, and thermal barrier coating layers on CMC coating sheets. Manufacturing trials and tests have been conducted to evaluate the mechanical properties in this research.



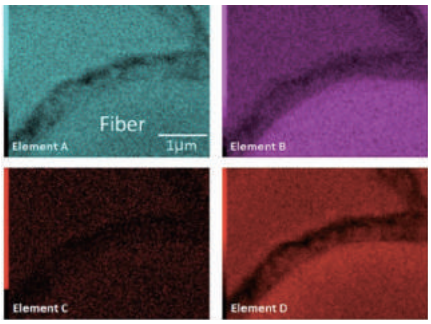
Stress Analysis for Shroud

Design and evaluation of oxide ceramics matrix composite coating sheets

Keywords : Oxides, Toughness, Design, Evaluation

HIDEKI KAKISAWA
National Institute for Materials Science, Research Center for Structural Materials

We are conducting research on the measurement of physical properties, structural analysis, and characterization of fracture behavior of ceramic matrix composite (CMC) coating sheets. The effect of raw materials and processing conditions on the microstruture of CMC coating sheets has been investigated, and the microstructure has been correlated with fracture behavior in bending tests. We revealed that the fracture of the CMC coating sheets is affected by adhesion between the fiber and matrix; pores between the woven fabrics. The conditions under which non-brittle behavior occurs were discussed based on the results. We have also developed a system for microstructural observation and strain measurement at high temperatures. With these achievements, we support the optimization of process conditions and the acquisition of material data required for design.



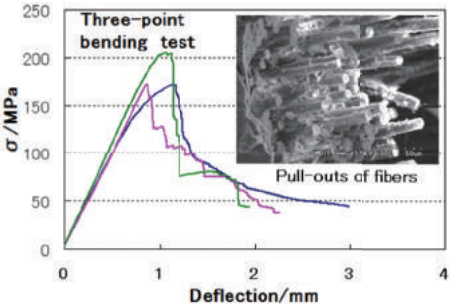
Example of element analysis near fiber

Development of low-cost processing of oxide ceramics matrix composites

Keywords : Oxide ceramics matrix composites, Toughness, Low-cost, PIP, Interphase

YOSHIO HASEGAWA
ARTKAGAKU Co., Ltd. Research and Development Division

Oxide ceramic matrix composite (CMC) sheets for adding toughness to a monolithic ceramic substrate have been developed. The CMC sheets are used as an intermediate layer between a substrate and a thermal barrier coating layer (TBC) which needs heat-resistance of more than 1000°C. Oxide CMC with high strength controlling fiber-matrix interphases are being developed by the polymer infiltration and pyrolysis (PIP) method because of the low-cost and flexibility to conform to CMC.
We are currently optimizing a polymer precursor for use as a matrix; the surfaces of oxide fibers; a method for sticking CMC sheets to substrate materials; and the thermal barrier coating layer on CMCs.



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Development of oxide ceramics matrix composite coating sheets

Keywords : Continuous alumina fiber, Improvement

KAZUHIRO KUMETA
NITIVY CO., LTD. Technical Division

Continuous fiber materials with sufficient heat resistance are equired as materials for the development of coating sheets for oxide ceramic matrix composites.
Improvements in the properties of a heat resistant fiber were achieved by adjusting the materials and manufacturing process, based on our manufacturing experience with continuous alumina fiber. The woven fabric is provided for experimental production of oxide CMC. We are continuing to develop continuous alumina fiber with sufficient mechanical and heat resistance properties based on the test results of oxide CMCs.

Woven fabric
of continuous alumina