Effects of F/M Interface Properties on Performance of SiC/SiC Composites by DEMO-NITE Process

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Introduction

NITE-SiC/SiC composites are potential candidate materials for fission and fusion reactor components. R & D toward industrialization of NITE-SiC/SiC composites is currently ongoing by OASIS, Muroran Institute of Technology. As the key facility of "DEMO-NITE" process line, a new chemical vapor deposition (CVD) continuous furnace for ceramics fibers coating has been installed in March 2014 by Project "FEEMA", MEXT program. This facility is open-end type linear CVD furnace, which is able to process flow control type CVI and quasi-isothermal type CVD, continuously, the fiber/matrix (F/M) interface properties are important factor for controlling performance of SiC/SiC composites. However, in the past, batch type CVD process had been used causing many technological issues which degrade performance of SiC/SiC composites. The new CVD furnace is designed and is installed for solving these issues.

F/M interface coating process

- There are two kinds of the F/M interface coating process route.

Route	Example	Quality	Cost
Gas	Batch type CVD, CVI	High	High
Liquid	Polymer pyrolysis	Low	Low

- The high quality F/M interface by CVD process is needed in the nuclear grade SiC/SiC composites.
- The technological issues of batch type CVD process ;
  - Quality (Residual deformation of fiber bundles)
  - Long process time
  - High cost

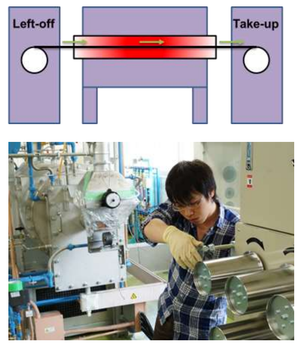
A new CVD continuous furnace for ceramics fibers coating

- This facility is open-end type linear CVD furnace.

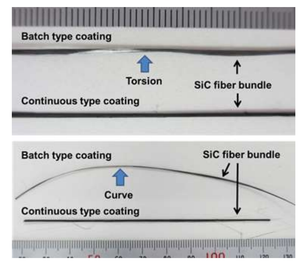
Specifications of CVD continuous furnace

- Isothermal heat zone : 800 mm
- Processing line cross-section : ϕ40 mm
- Temperature : ~ 2000℃
- Atmosphere :
  Ar and/or N_2 + Process gas

Schematic image



●Comparison of batch and continuous type coating



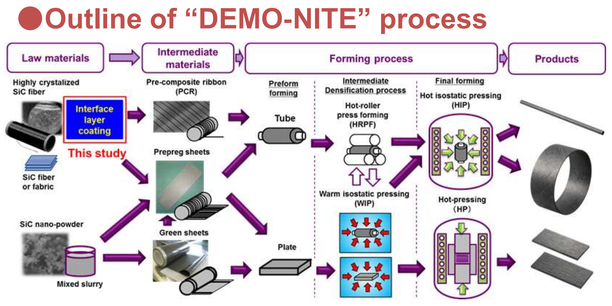
- The twisted and curved fiber bundles for batch process are suppressed by our process to make straight fiber bundles

Objective

- This paper provides the first results from the new CVD furnace under operation of quasi-isothermal type CVD process for continuous C coating on SiC fibers.
- The aim of this study is optimization of the CVD coating process applied, where F/M interface properties for NITE-SiC/SiC composites and production efficiency should be well balanced. In this presentation, the correlation between F/M interfacial structure and mechanical properties of SiC/SiC Composites is also concerned.

Experimental

●Outline of "DEMO-NITE" process



- "DEMO-NITE" process is a new NITE process toward industrialization, which was developed by OASIS, Muroran Institute of Technology, Japan.
- The feature of this process is to use polymer based slurry for making dry type inter-mediate materials, such as green-sheets, prepreg sheets and pre-composites ribbon.
- In the OASIS, construction of pilot plant for production of SiC/SiC composites are currently ongoing.

●Materials

	PyC-300	PyC-500	PyC-1200
Reinforced fiber	Cef-NITE		
PyC interphase thickness [nm]	300	500	1200
Fiber orientation	UD (0^o)		
Matrix densification process	NITE (Nano-Infiltration and Transient Eutectic-phase) Process		
Density [g/cm^3]	2.95	2.82	2.61
Fiber volume fraction [%]	39	48	60

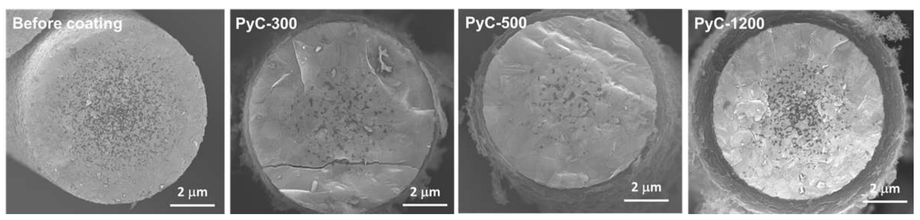
● Evaluation;

- Density ;
  Archimedes' method
- Microstructure ;
  Optical microscope
  FE-SEM
- Mechanical property ;
  Three-point bending test
  Specimen size :
  26L x 3W x 1.2T mm
  Cross-head speed :
  0.5 mm/min
  Support span : 16 mm

Results

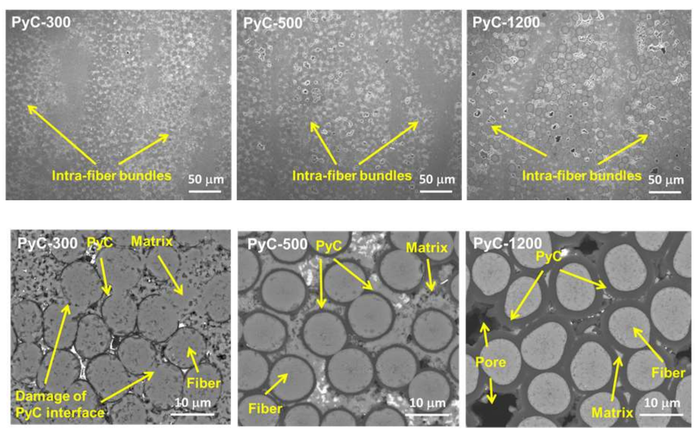
●PyC coated SiC fibers by CVD continuous furnace

- The SiC fibers with various PyC interface thickness were obtained by the controlling of process conditions.
- In the all conditions, the PyC layer is formed on the surface of SiC fiber.



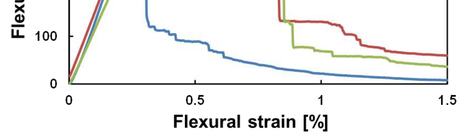
- No gaps are observed in between of fiber and PyC layer.

● DEMO-NITE-SiC/SiC composites prior to process optimization for different interface thickness



- Pores are mainly distributed in the intra-fiber-bundles regions. Matrix in the inter-fiber bundles regions has very well sintered.
- In the case of PyC-300, damage and loss of PyC Interface is observed. This might be due to the reaction between PyC interface and the sintering additives.
- In the case of PyC-500, maintenance of PyC interface is confirmed. The matrix in the intra-fiber bundles has well densified.
- In the case of PyC-1200, maintenance of PyC interface is confirmed. On the other hands, many pores (pore size : ~10 μm) are observed in the intra-fiber-bundles. The matrix densification in the intra-fiber bundles is poor. This is considered to due to decreasing of space for infiltration of SiC powders in the intra-fiber bundles by the increase of interface thickness.

● Mechanical property of DEMO-NITE-SiC/SiC composites with various PyC interface thickness

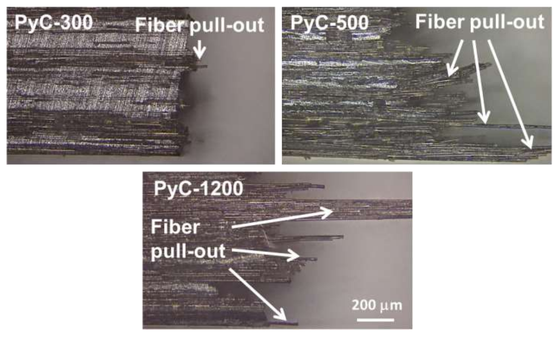


	Elastic modulus [GPa]	Flexural strength [MPa]	Fracture energy [J/m^3]
PyC-300	137±5	336±126	1.0±0.7
PyC-500	136±1	522±11	2.7±0.3
PyC-1200	119±16	543±64	3.0±0.7

- The fracture energy is calculated from the flexural stress - displacement curves.
- The elastic modulus tends to decrease with the increase of PyC interface thickness. This is considered to due to decreasing of density and increasing of PyC interface volume fraction.
- The flexural strength of PyC-500 and PyC-1200 is higher than that of PyC-300.
- The PyC-500 and PyC-1200 clearly displayed a pseudo-ductile fracture behavior. The fracture energy of these composites is higher than that of PyC-300. This is supposed to due to maintain of PyC interface layer.

● Fracture surface of DEMO-NITE-SiC/SiC composites with various PyC interface thickness

- In the case of PyC-300, fracture surface is smooth in comparison with PyC-500 and PyC-1200, A few short fiber pull-outs are observed.
- In the case of PyC-500 and PyC-1200, many crack deflections and long fiber pull-outs are observed.



Summary

- ✓ In order to solve the process issues of the batch type CVD coating, a new CVD continuous furnace at OASIS was designed and installed. The effectiveness of CVD continuous coating was confirmed through suppression of residual deformation of fiber bundles after coating process, which reduces fiber damage through whole fabrication process.
- ✓ The SiC fibers with various PyC interface thickness was obtained under the well controlled process conditions.
- ✓ Although DEMO-NITE process optimization for different coating thicknesses is underway, the composites with PyC-500 indicated the high flexural strength and a pseudo-ductile fracture behavior. This is mainly due to the formation of dense matrix in the intra-fiber bundles, reduced fiber damage during the process.