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The Pennsylvania State University

Advanced Manufacturing of Ceramic Matrix Composites (CMC) by Field Assisted Sintering Technology (FAST)

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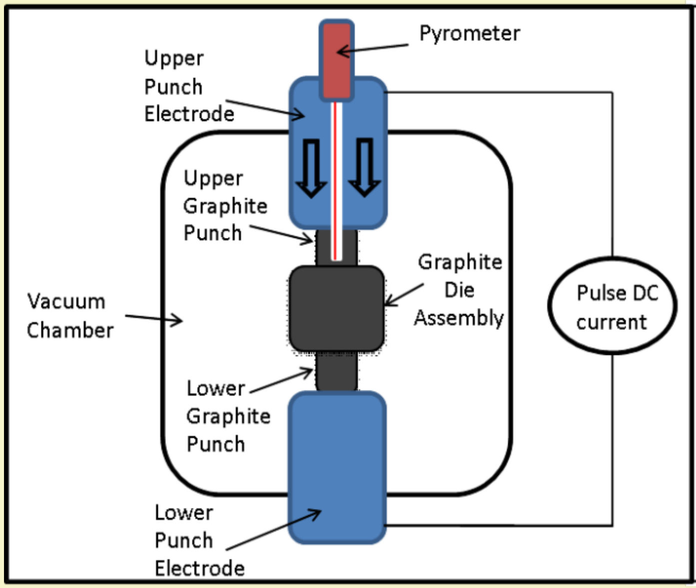
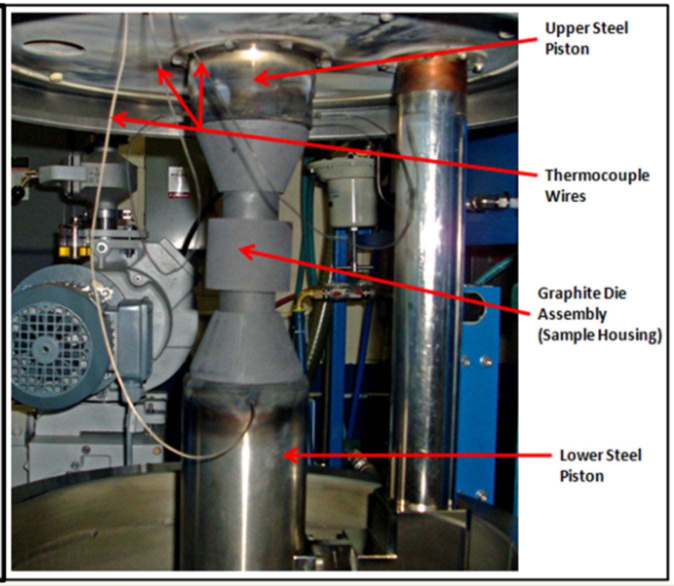
Outline

- Introduction
  - FAST
  - CMC Fabrication
  - LEARN efforts to date
- Results
  - Fiber samples (no matrix)
  - Slurry impregnated CMC
    - Mechanical properties and comparison
  - Post-CVI sintered CMC
- Future Efforts and Summary

Introduction to FAST

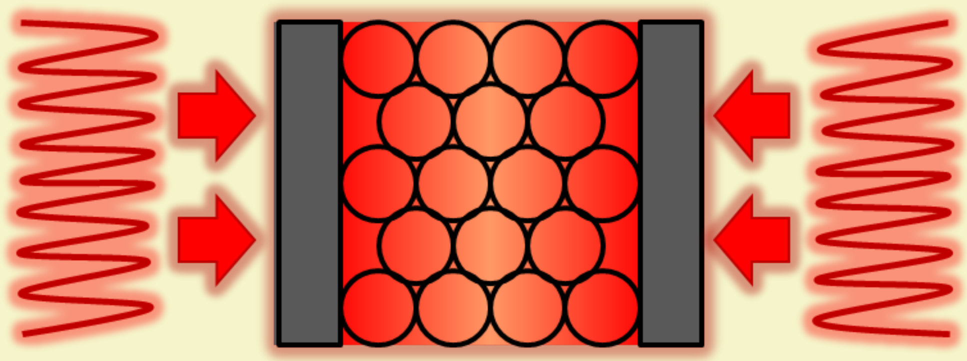
- Field Assisted Sintering Technique (FAST)



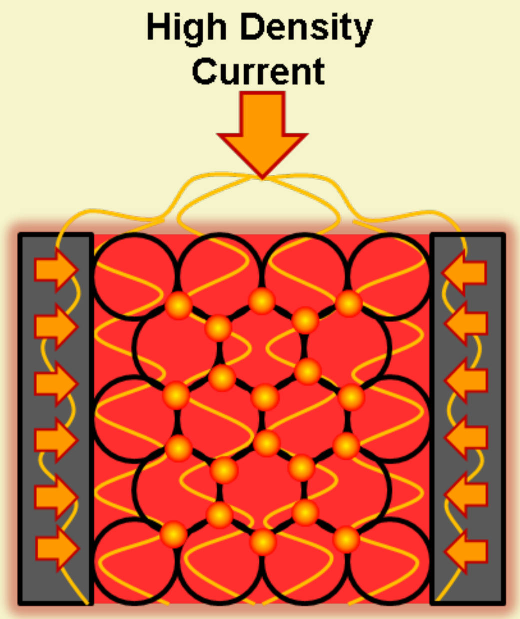


Differences Between Conventional and FAST Processes

- Conventional Sintering
  - HIP, HP, Pressureless
  - Radiative heating
  - Slow heating rates
  - Temperature gradients from outside to inside



- Field Assisted Sintering Technique
  - Resistive Heating
  - Heat generated directly inside the die and material
  - Faster heating rates



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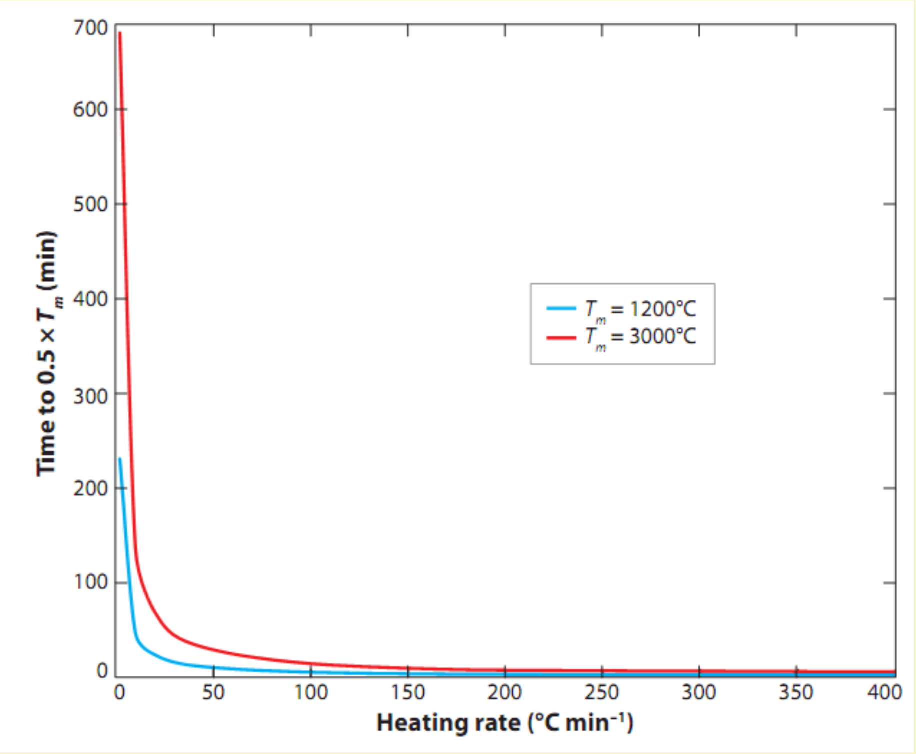
Heating Rate: A key difference

- Heating Rate Comparison:

Conventional	FAST
Typical: 1-10°C/min	*Typical: 50-500°C/min*

- Heating quickly minimizes surface diffusion effects (i.e. non-densifying effects)
- Higher rates occur because of direct resistive (Joule) heating as compared to external radiative heating



J. E. Garay, in "Annual Review of Materials Research, Vol 40", (Annual Reviews, Palo Alto, 2010) p. 445.

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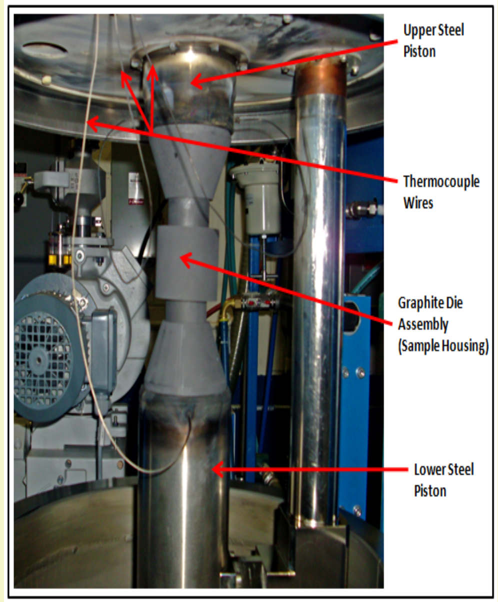
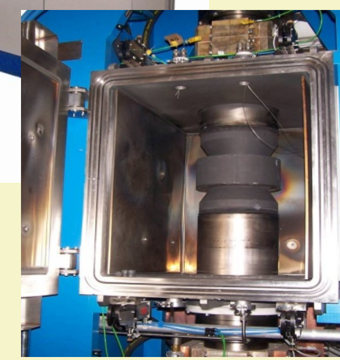
FCT's FAST Machine Capability





PSU's R&D Unit Capability:
- **Equipment capability load : 25Ton**
- **Maximum Diameter: 3.25" (80 mm)**
- **Pulse current: 0 - 10KAmps**
- **Pulse time: 1 to 1000 ms**
- **Pause duration: 0 to 1000 ms**
- **Temperature capability: RT to 2400 ^{0}C**
- **Computerized Process control system**

- **Prototype Large R&D**
- **Equipment capability load : 250 Ton**
- **Maximum Diameter: 300 mm**
- **Pulse current: 0-10KAmps**
- **Pulse time: 1 to 1000 ms**
- **Pause duration: 0 to 1000 ms**
- **Temperature capability: RT to 2400 ^{0}C**
- **Computerized Process control system**

Photographs of Sintering Set up in the FAST system

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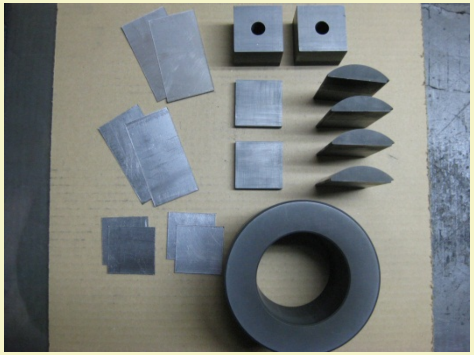

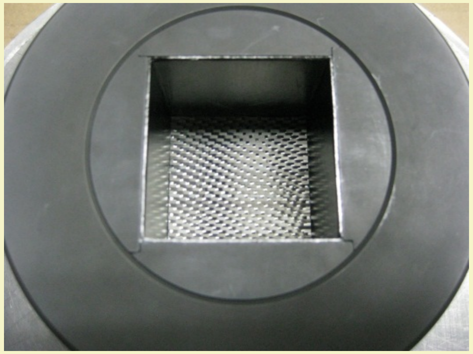

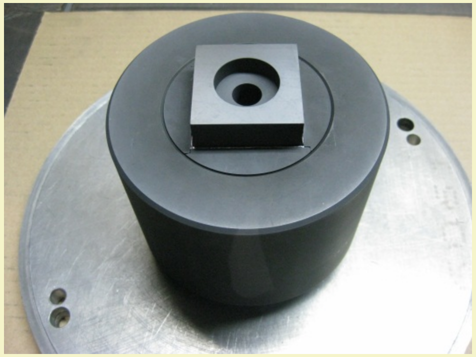

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Current Manufacturing Processes of CMC

1. Chemical Vapor Infiltration (CVI) method:

1. Processing time: hundred of hours
2. Gradient in porosity if thickness is more than 4 mm
3. Porosity : 10-15%
4. Delicate process parameter control

2. Si-Melt Infiltration

1. Si is used as melt media that is converted to SiC matrix by chemical reaction, stoichiometric composition is challenging
2. Residual Si is undesirable, forms Si-oxide, that lowers mechanical properties
3. Processing time: few days
4. Maximum processing temperature ~1300 ^{0}C

3. Polymer Impregnation Pyrolysis (PIP):

- Pre-Ceramic polymer precursor
- Multiple impregnation cycles (8-15)-few days
- Lower purity SiC-matrix
- Matrix often cracks during pyrolysis shrinkage

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LEARN: Development of CMC with Low Porosity

Following sintering efforts were conducted under LEARN program:

- Uncoated Sylramic fiber-8 ply
- Carbon coated Sylramic fiber-8 ply
- Carbon coated Sylramic fiber with nano SiC+B powder infiltration-9 ply
- Explored sintering post processed CVI materials to improve density

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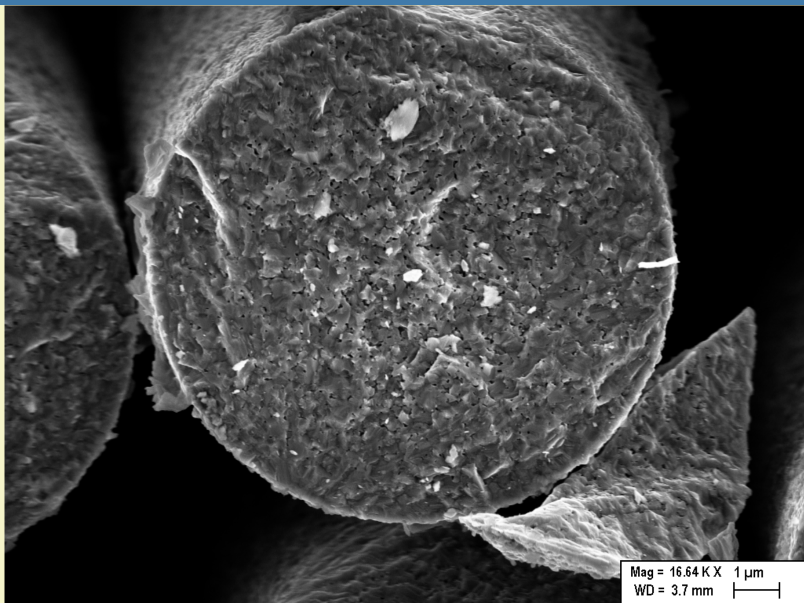
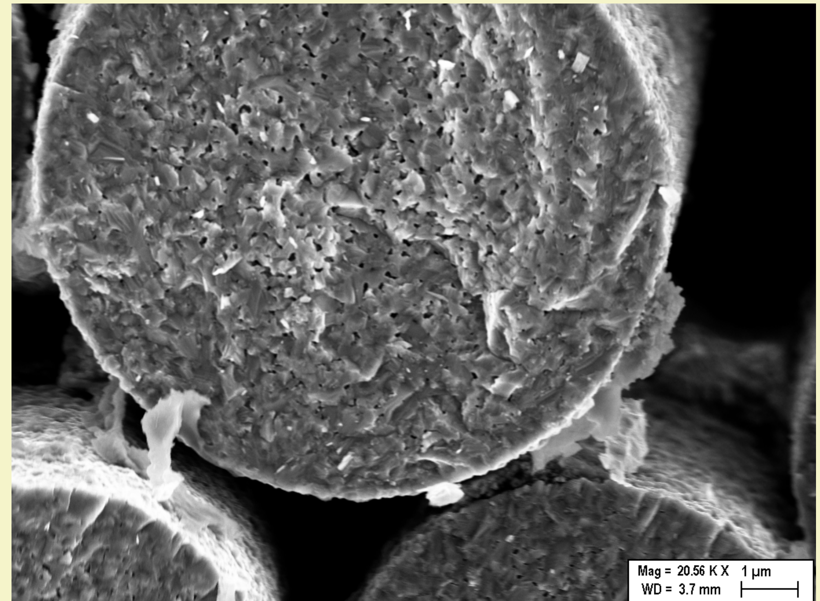
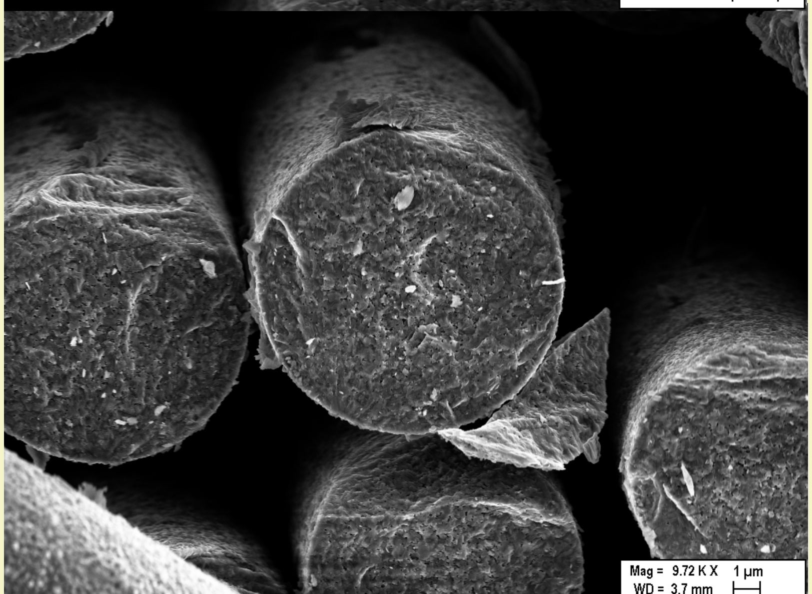
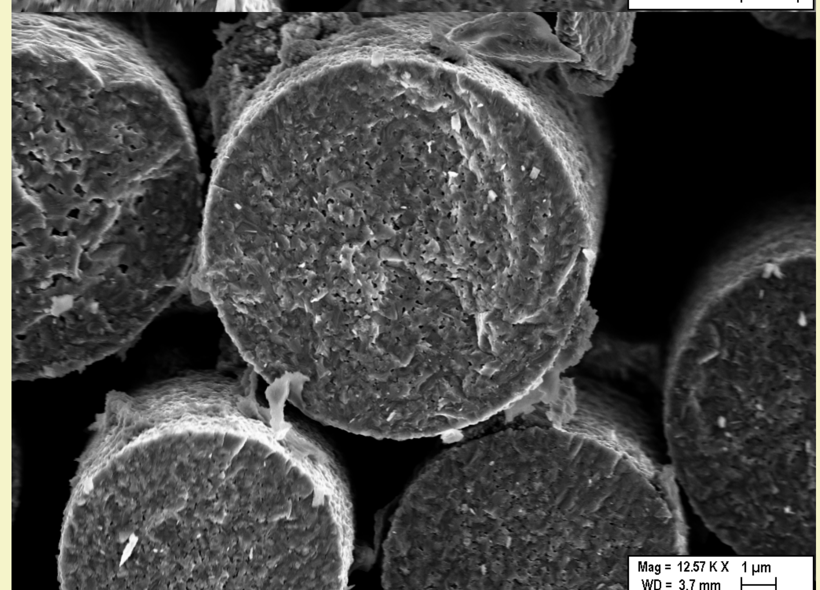
Microstructure of Coated Sylramic Cloth









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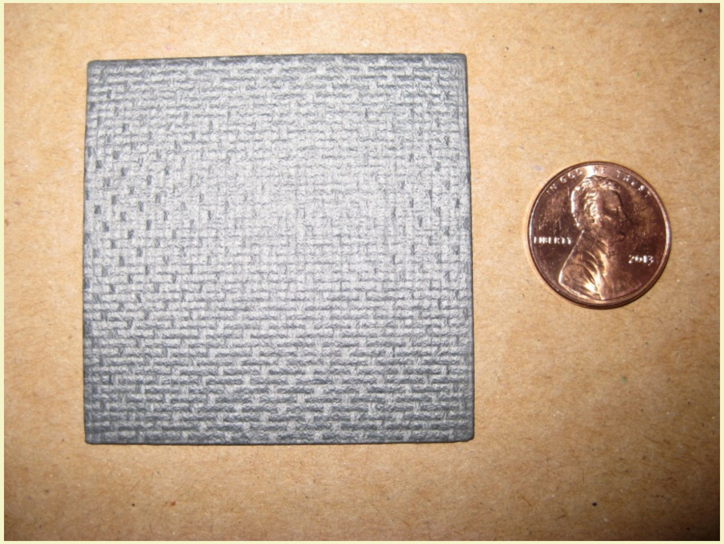
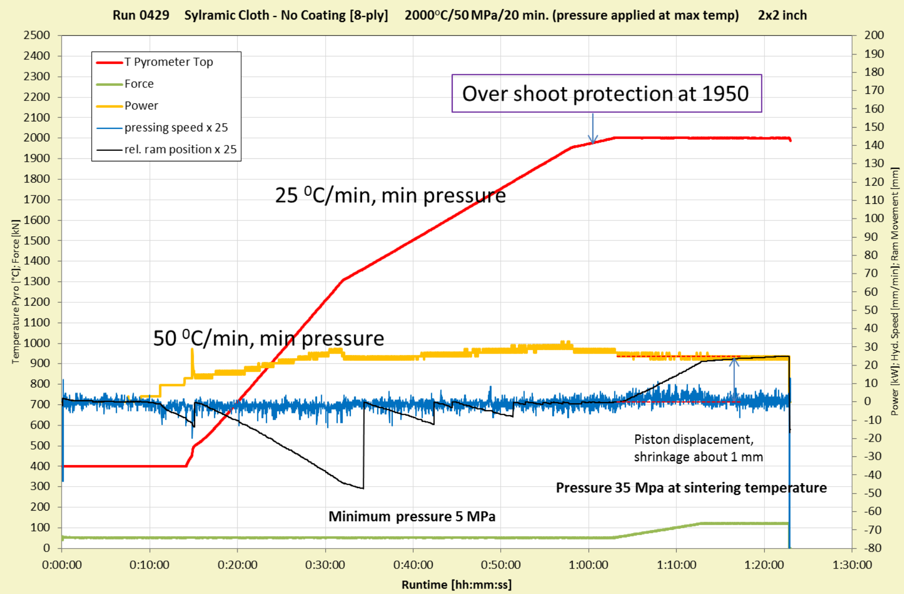
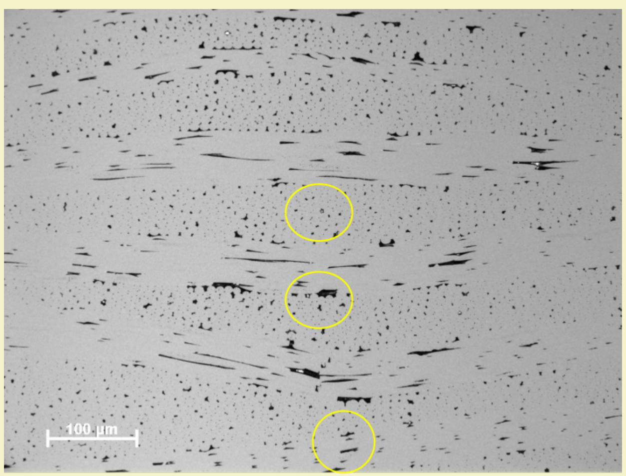
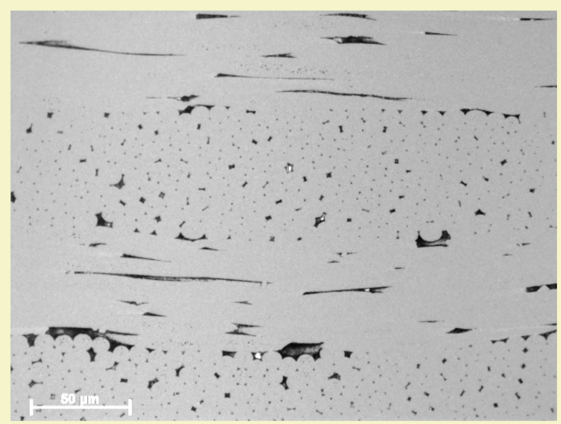
Sintering of Sylramic Cloth









Sylramic Cloth – No Coating, 8-ply: 2000°C/50 MPa/20 min.

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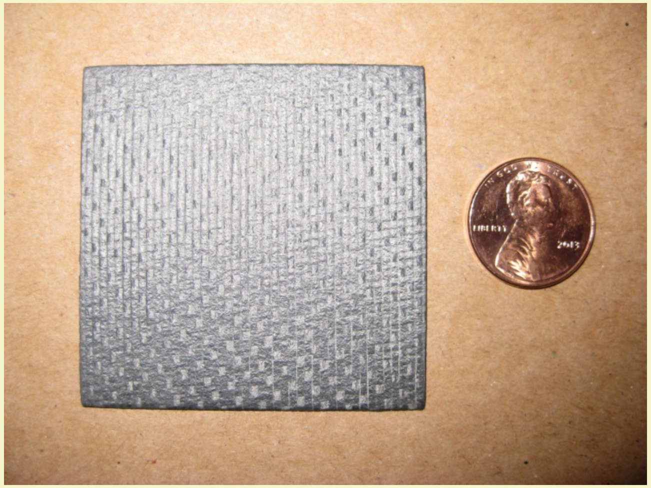
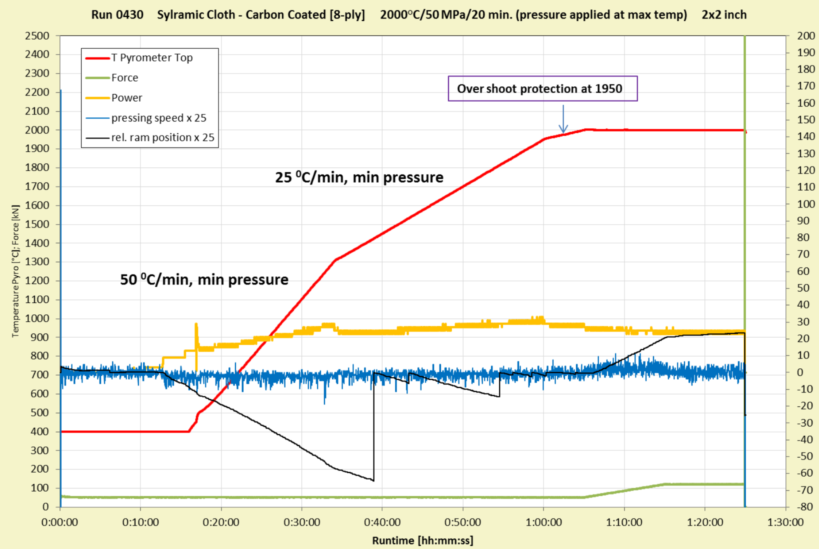
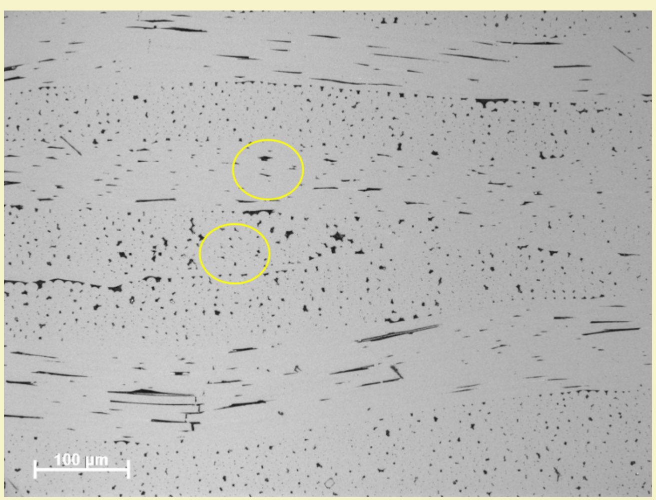
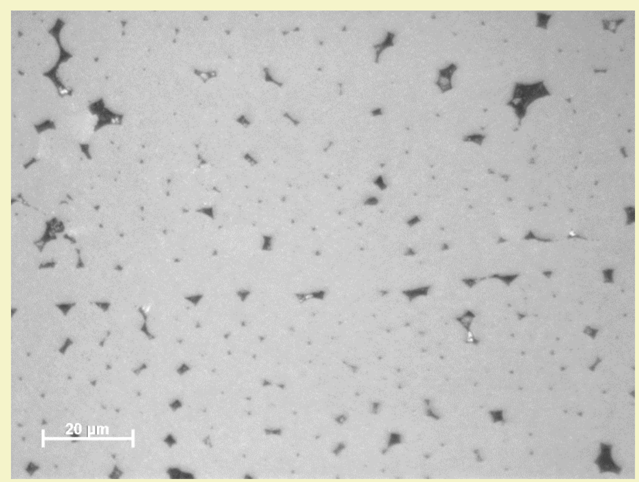
Sintering of Carbon-coated Sylramic Cloth









Run #0430 Carbon-coated Sylramic Cloth 8-ply 2000°C/50 MPa/20 min.

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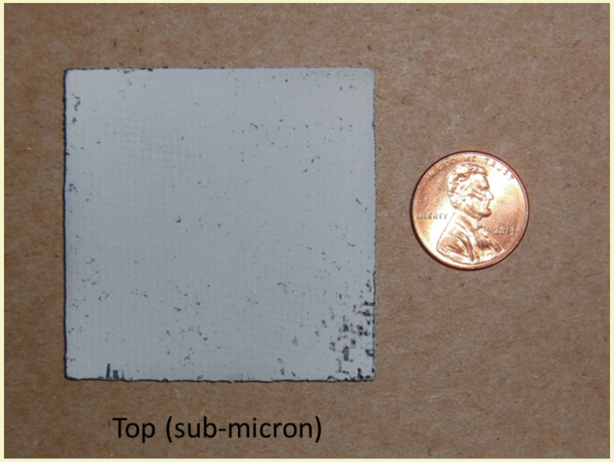
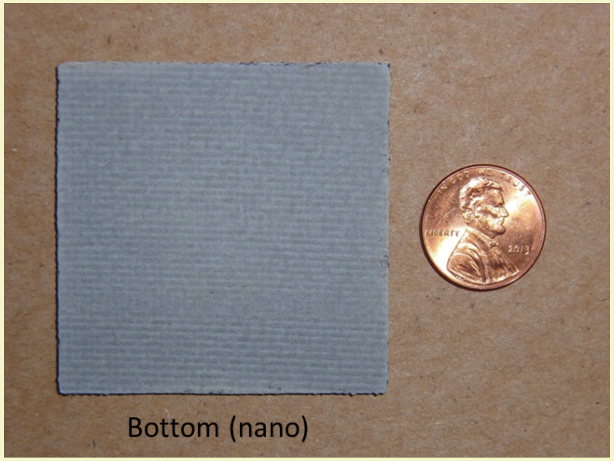
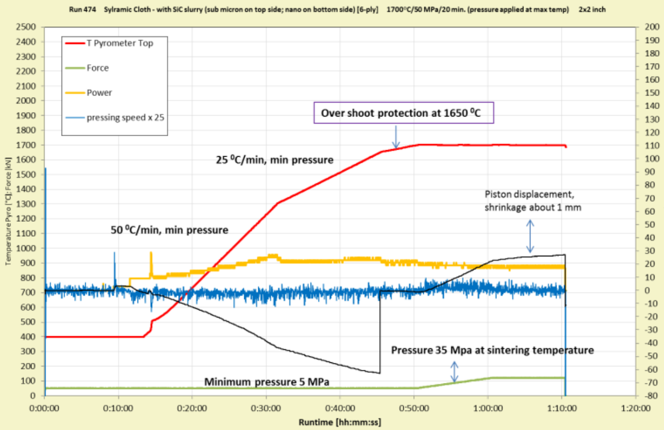
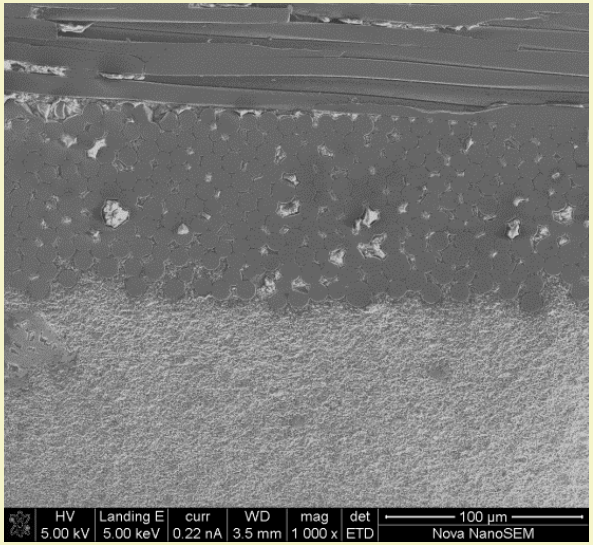
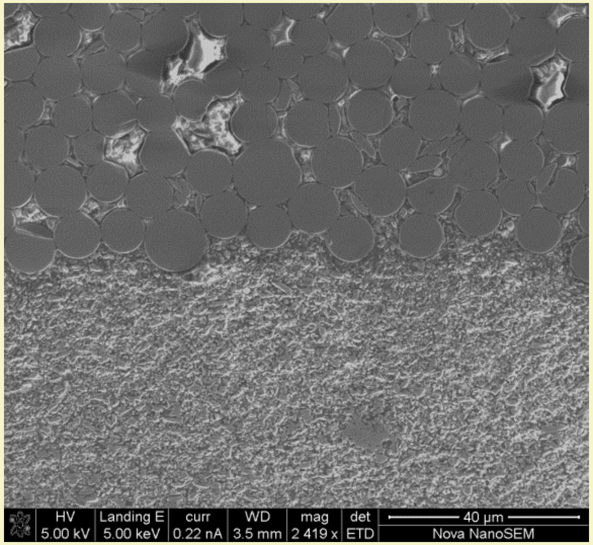
Sintering of Sylramic SiC Cloth with SiC Slurry





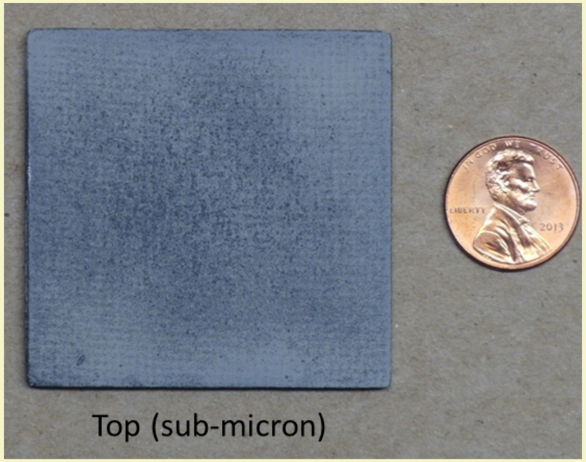


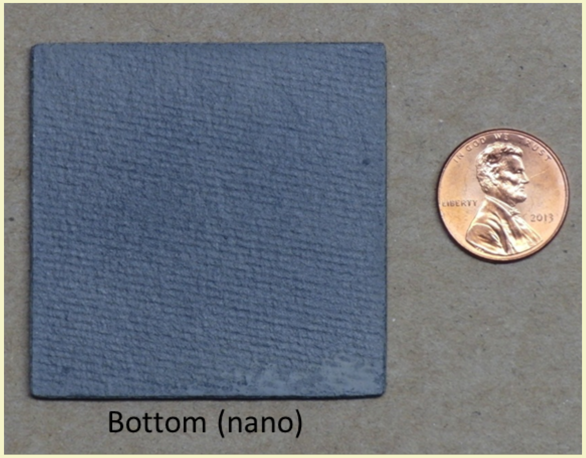


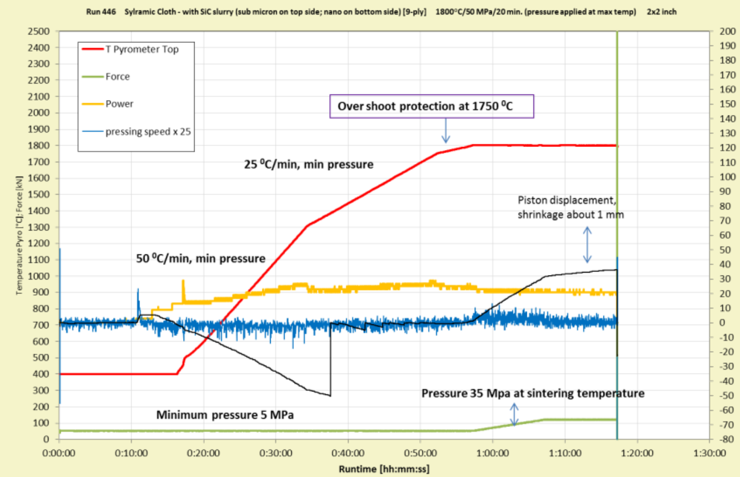


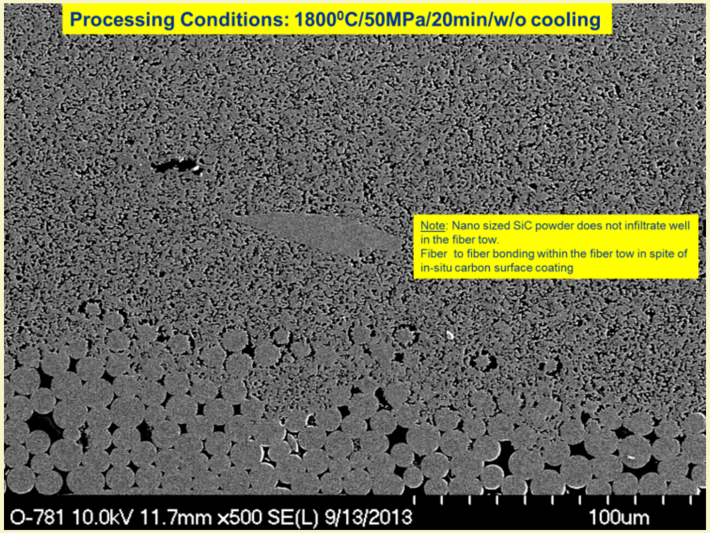
Run #0474 [6-ply]: Submicron top side and nano on bottom; and sintered: 1700°C/50 MPa/20 min, Avg. sample thickness is 0.083 in

Sintering of Sylramic SiC Cloth with SiC Slurry









Run #0446 [9-ply]: 1800°C/50 MPa/20 min, Avg. sample thickness is 0.106 in.

- Carbon coating dissolved in the SiC matrix (coating is too thin)

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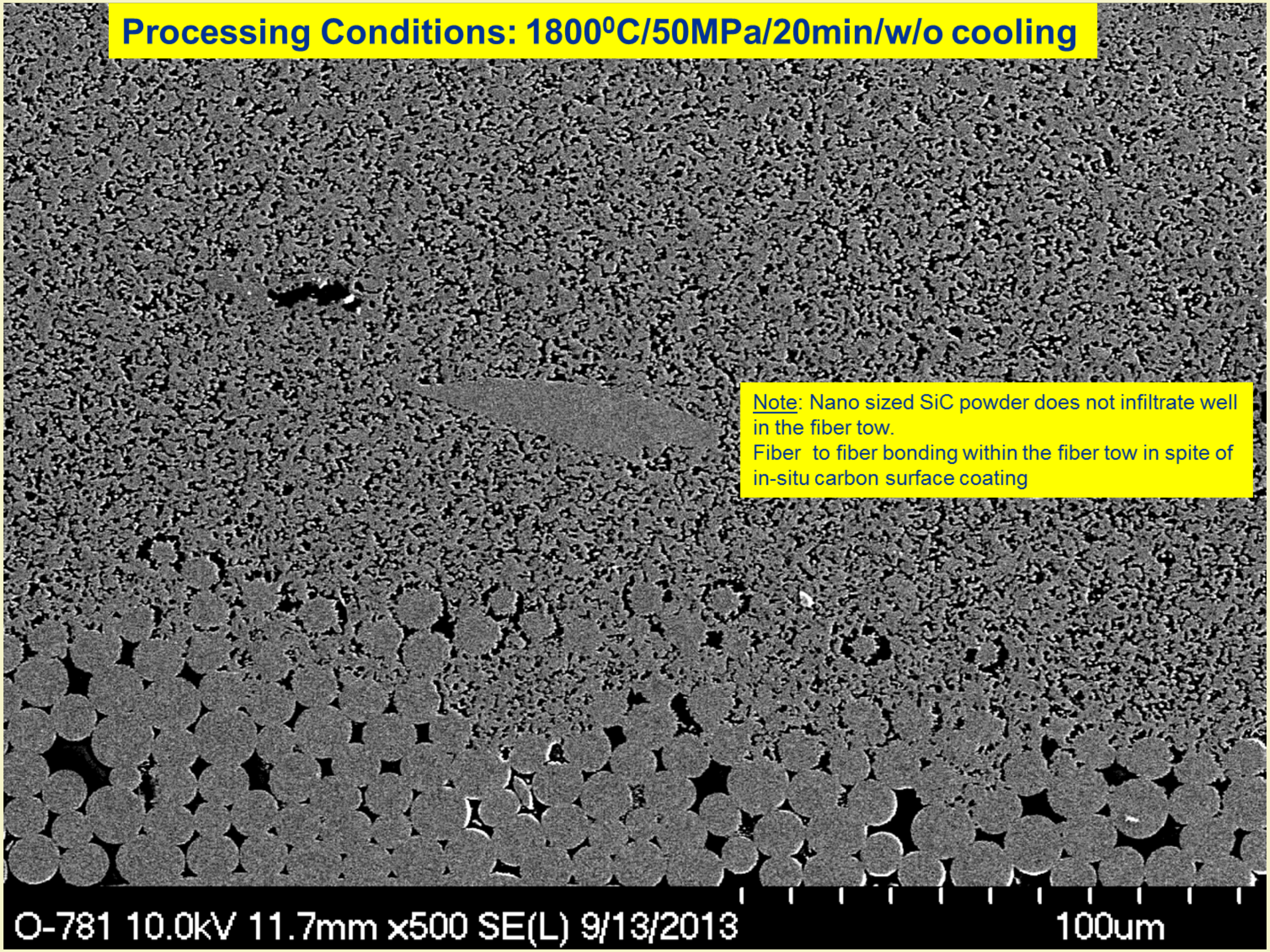
Cross Section SEM of SiC/SiC (nano) Composites Fabricated by FAST



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Bend Properties of SiC/SiC (nano) Composites Fabricated by FAST

Specimen#	Cloth	Slurry coating	Processing conditions	Test temperature	Ultimate bend strength	Ultimate bend strain	Tangent modulus
				^{0}C	MPa	%	GPa
Run 0474-1	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1700^{0}C/50MPa/20min/w/o cooling	25	85	0.09	90
Run 0474-2	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1700^{0}C/50MPa/20min/w/o cooling	25	65	0.10	98
Run 0474-3	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1700^{0}C/50MPa/20min/w/o cooling	25	52	0.05	105
Run 0474-4	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1700^{0}C/50MPa/20min/w/o cooling	1315	91	0.11	94
Run 0474-5	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1700^{0}C/50MPa/20min/w/o cooling	1315	100	0.13	76
Run 0474-6	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1700^{0}C/50MPa/20min/w/o cooling	1315	66	0.08	71
Run 0446-1	Carbon coated Sylramic/9 ply	Submicron:Top Nano: Bottom	1800^{0}C/50MPa/20min/w/o cooling	25	131	0.11	168
Run 0446-2	Carbon coated Sylramic/9 ply	Submicron:Top Nano: Bottom	1800^{0}C/50MPa/20min/w/o cooling	25	152	0.11	175
Run 0446-5	Carbon coated Sylramic/9 ply	Submicron:Top Nano: Bottom	1800^{0}C/50MPa/20min/w/o cooling	25	189	0.18	160
Run 0446-3	Carbon coated Sylramic/9 ply	Submicron:Top Nano: Bottom	1800^{0}C/50MPa/20min/w/o cooling	1315	121	0.08	152
Run 0446-4	Carbon coated Sylramic/9 ply	Submicron:Top Nano: Bottom	1800^{0}C/50MPa/20min/w/o cooling	1315	113	0.08	147
Run 0448-1	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1900^{0}C/50MPa/20min/w/o cooling	25	155	0.06	241
Run 0448-2	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1900^{0}C/50MPa/20min/w/o cooling	25	244	0.10	233
Run 0448-3	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1900^{0}C/50MPa/20min/w/o cooling	1315	171	0.08	198
Run 0448-4	Carbon coated Sylramic/6 ply	Submicron:Top Nano: Bottom	1900^{0}C/50MPa/20min/w/o cooling	1315	151	0.08	192

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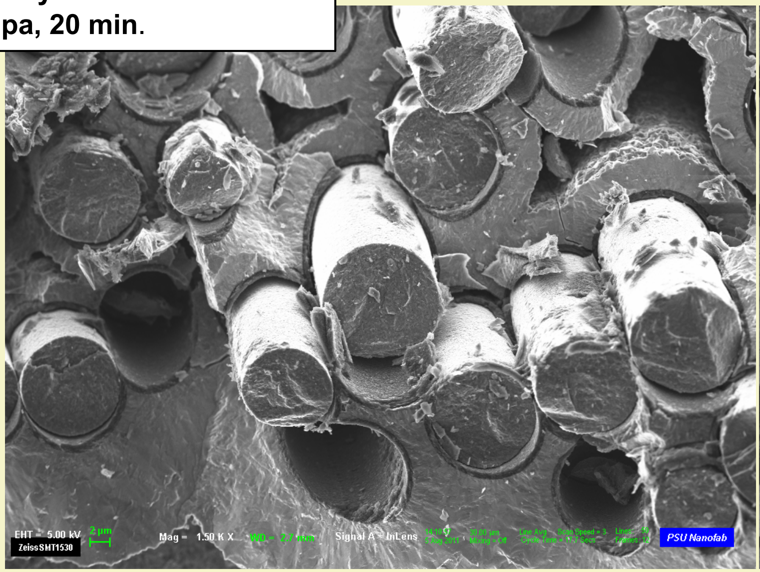
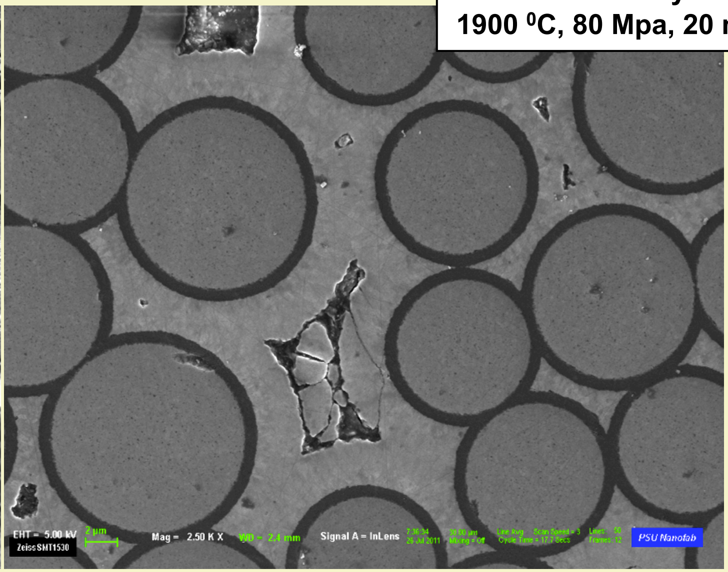
Past Effort

Material	Porosity %	Avg Flexural Strength (MPa)	Average Flexural Strain (%)
SiC-Monolithic	1	607	0
T300/FAST SiC/2000 ^{0}C/ 80MPa/20min	6	416	0.86
Tyranno SA Fiber/ 2000 ^{0}C / 80MPa/2min	8.3	350	0.30
Sylramic iBN/1850 ^{0}C / 80Mpa/20 min	9.7	206	0.24

Fracture
FAST sintered Sylramic iBN SiC/SiC
1900 ^{0}C, 80 Mpa, 20 min.

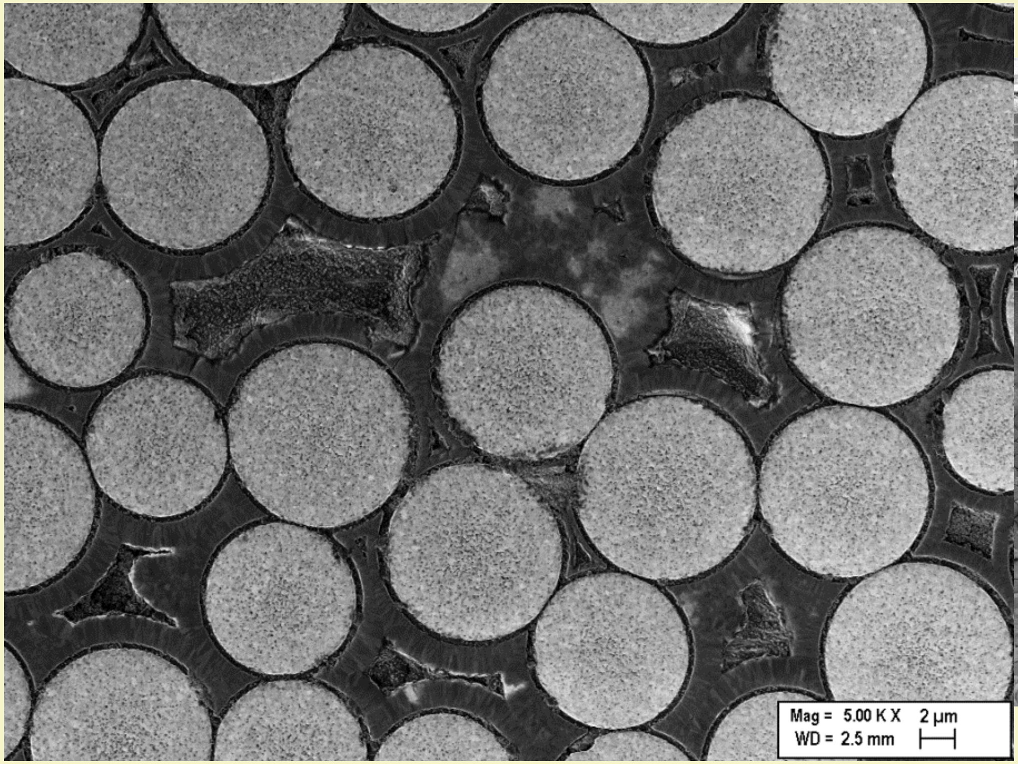


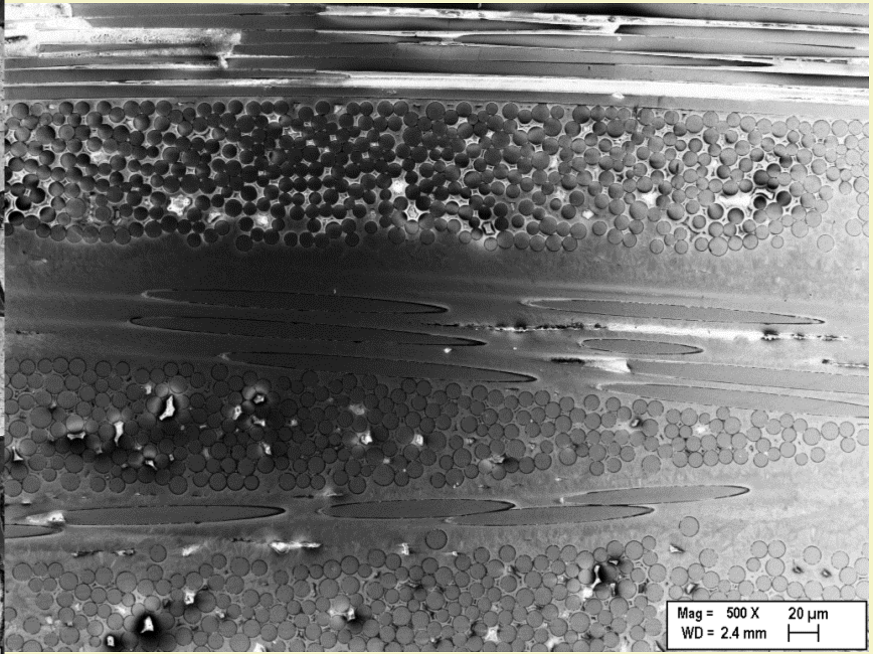
Polished
FAST sintered Sylramic iBN SiC/SiC
1900 ^{0}C, 80 Mpa, 20 min.



Past Effort

CVI SiC-SiC CMC (As received)





FAST Effort

Sintered at following conditions:

- **1800 ^{0}C @ 35 and 55MPa/10 min**
- **2000 ^{0}C @ 35 and 55MPa/10 min**

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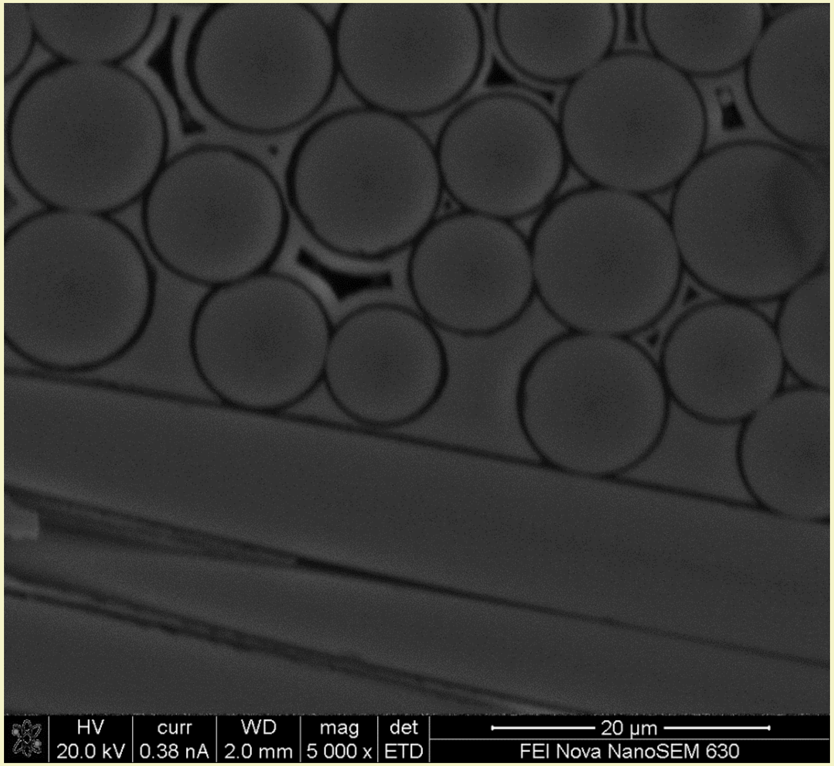
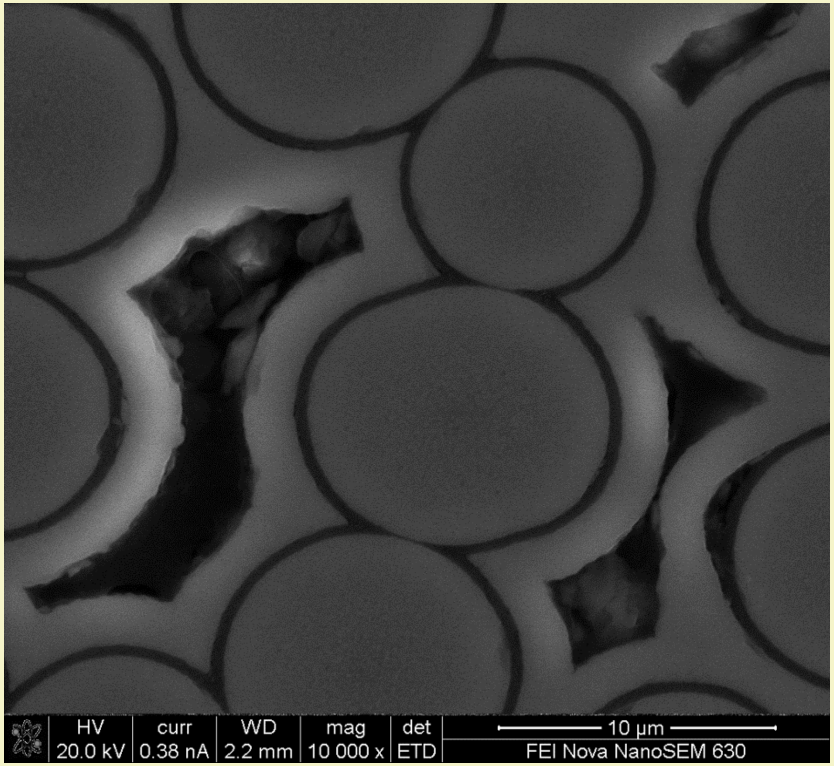
Cross section microstructure of CVI-SiC-SiC CMC-FAST





Sample# 1102-2D(1): 50ºC/min, 1800ºC/ 55MPa/ 10min

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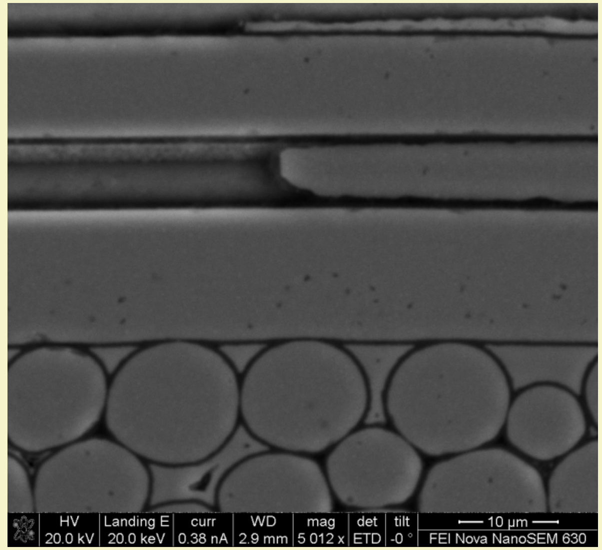
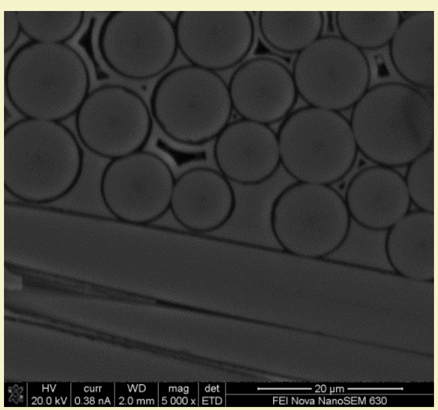
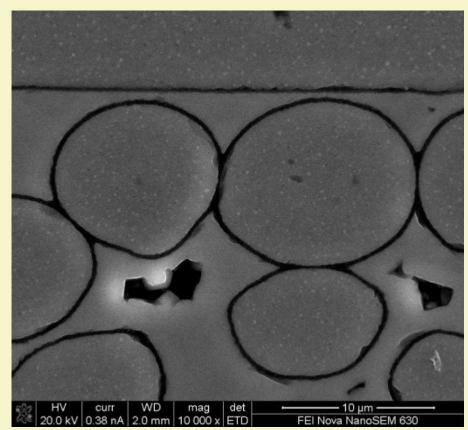
Cross section microstructure of CVI SiC-SiC CMC-FAST



Sample# 1102-2D(2): 50ºC/min, 2000ºC/ 35MPa/ 10min





Sample# 1102-2D(2): 50ºC/min, 2000ºC/ 55MPa/ 10min

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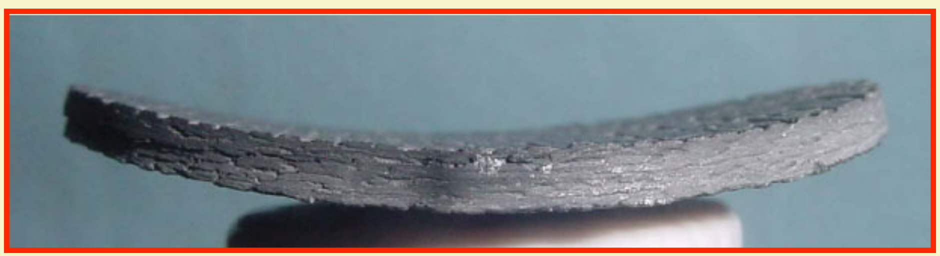
Future Exploration: Contoured CMC by FAST

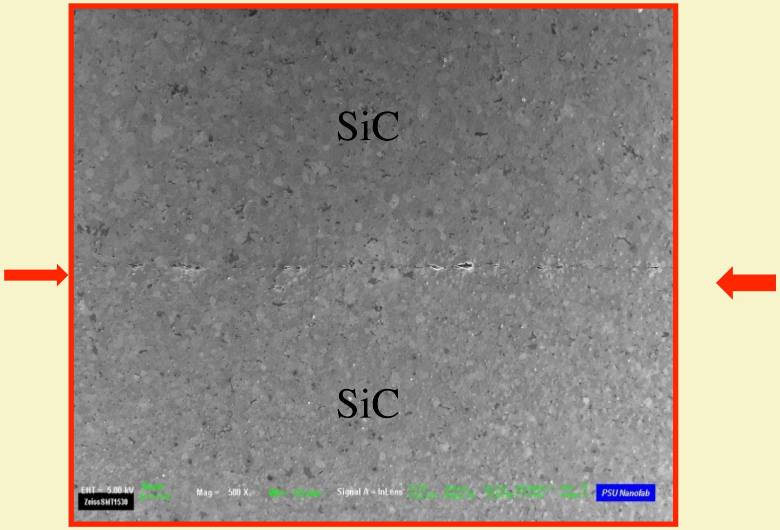
Airfoil



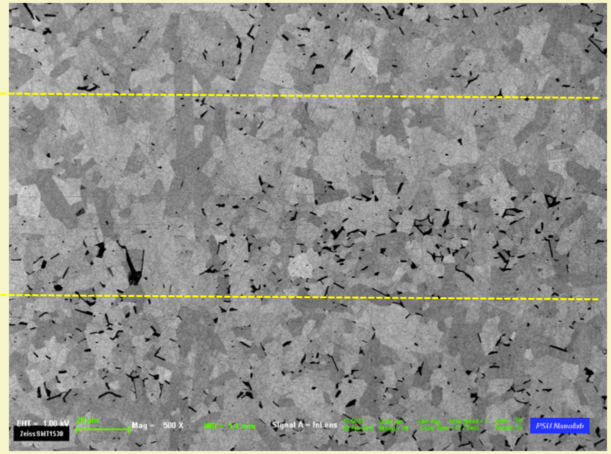
- Contoured shape (Tyranno SA/FAST SiC) formed by FAST starting with rigidized flat panel

Past Effort

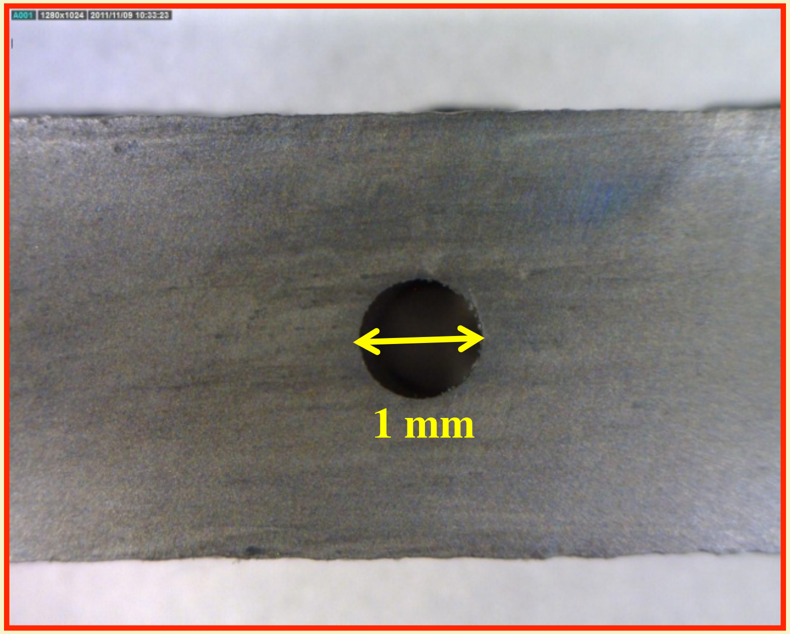
Future Exploration: Joining and Cooling Channels



Joining of SiC-SiC at 2000 ^{0}C, 35 MPa, 5 min



Joining of SiC-SiC at 2200 ^{0}C, 35 mpa, 5 min



Formation of cooling channels holes using removable mandrel

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Summary and Future Effort

Summary:

- **Uncoated SiC woven cloth sintered as monolithic SiC**
- **Sintered carbon coated SiC woven cloth and carbon dissolution was observed in all sintering temperatures (1700 to 1900 ^{0}C)**
- **Nano SiC powder slurry could not be infiltrated in gaps between SiC fibers, resulting in low density and coating breakdown**
- **Micron SiC powder slurry sintered well**
- **Mechanical properties were lower when compared to CVI materials**

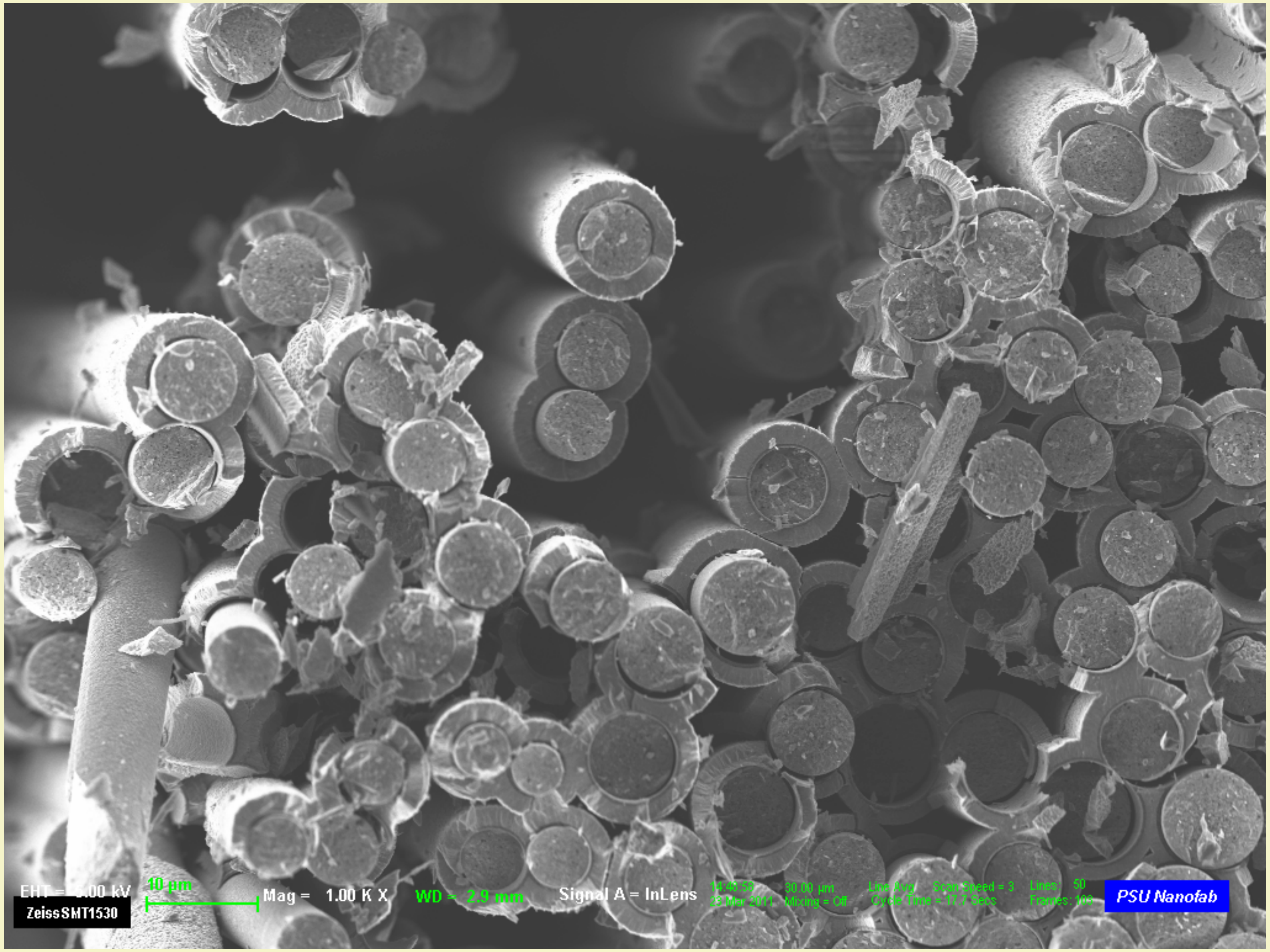
Future Effort:

- **Effort is underway to apply SiC coating on top of carbon coated SiC cloth by CVD**
- **Addition of SiC coating will serve two purposes:**
  - **Improve density within the fiber tows**
  - **Provide additional protection for fiber breakdown**
- **Polycarbo silane will be used to provide SiC matrix in the gap between woven cloth**
- **Net-shaped fabrication of components will be demonstrated at the end of the LEARN-I**

Back up slides

Back up slides

Tyranno SA fibers/Coated with Pyro C/ CVD SiC



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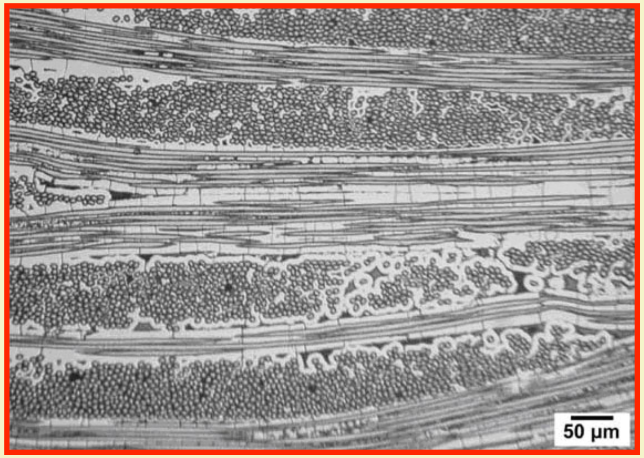
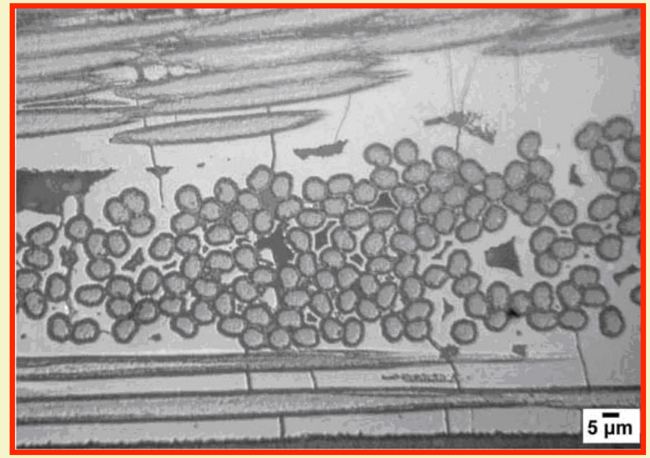
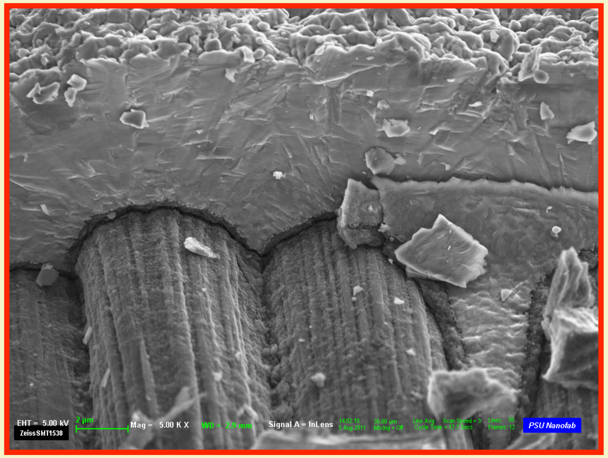
Results on FAST Processing C/SiC



Metallographic cross-section shows low porosity, but also frequent matrix microcracks



Retention of fiber identity and fiber coating



Crenulated fiber morphology maintained

<u>Physical Properties</u>

- **T300/FAST SiC: 2.24 g/cc and 5.9% porosity**

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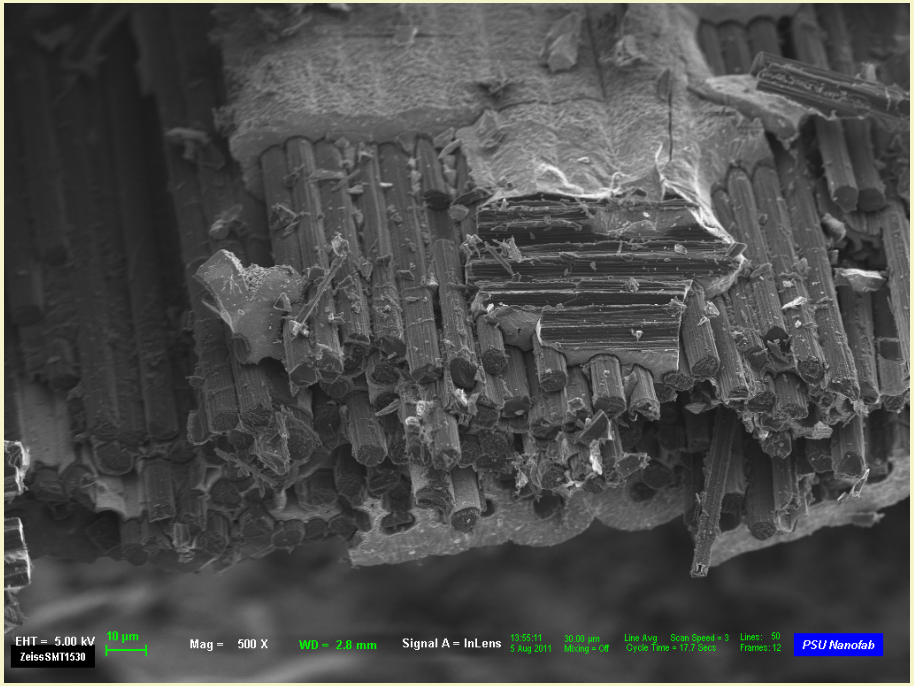
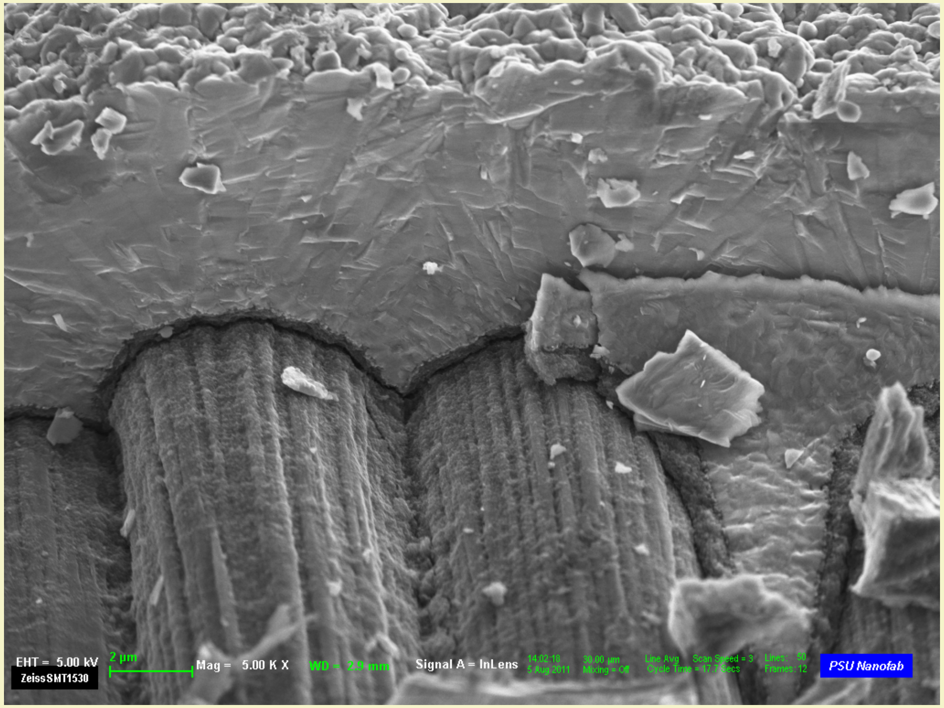
Fractured Surface of C -SiC Matrix Composite





T300 (B5), >1800°C

- Fiber stability is retained after sintering.

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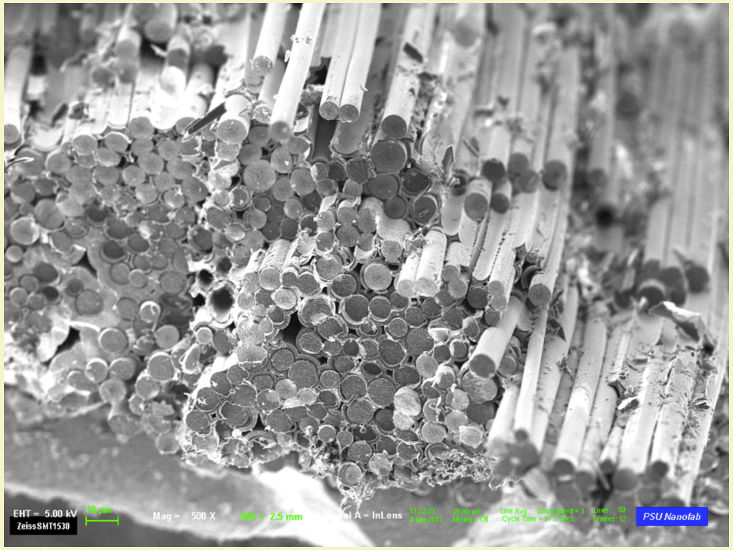
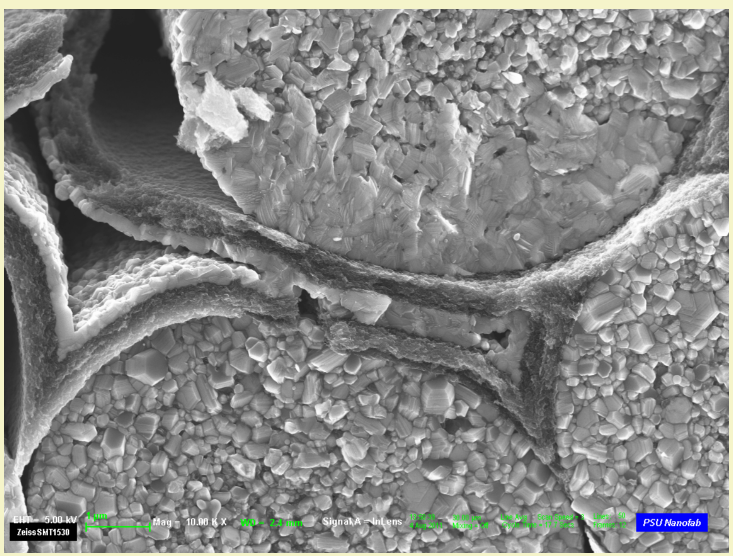
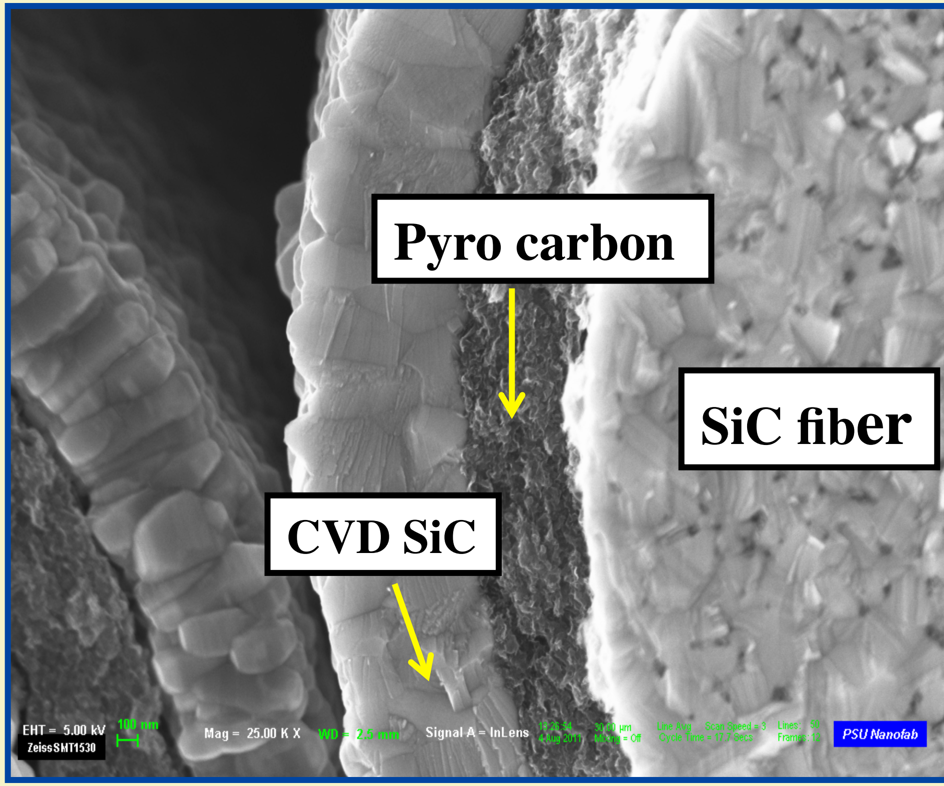
Fractured Surface of Tyranno SA (B5)







Sintered at >1800°C

- Fiber pulled out
- No damage on fiber during sintering

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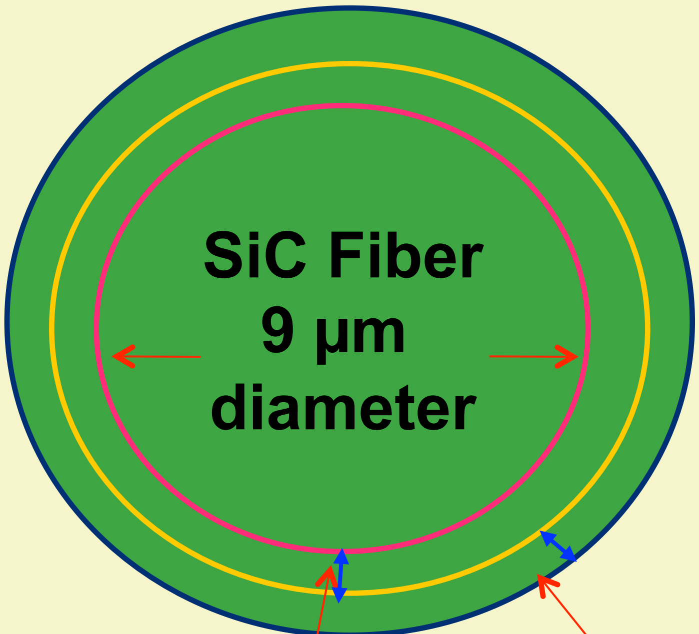
New Concept in Fabrication of CMC by FAST

SiC fiber-ATK/Sylramic fiber- woven → atomic BN coating @GRC → SBIR (Hypotherm company, CA) - BN/SiC or Pyrolitic carbon/SiC coatings



Coating: BN or pyrolitic carbon
Coating thickness: 0.5 μm

Coating: SiC
coating thickness: 0.2 μm

- Powder: SiC-RTP
- Particle size: 1.3 μm
- SiC Fiber: Two types of coated fiber weaved cloth
    - Weaving orientation: 4 over and 1 under configuration
- # film layers : 4 or 8
- Thickness per ply: 15 mils
- Desired Sample Thickness: 120 mils
- Recommended sintering temperature: 1700 and 1900 ^{0}C
- Pressure: 35 and 80 MPa
- NOTE: Flip flop the ply to keep the same orientation

Collaboration: NASA

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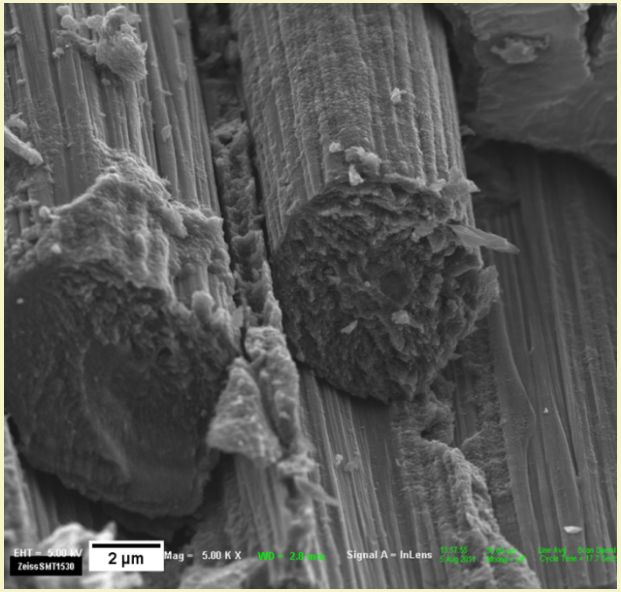
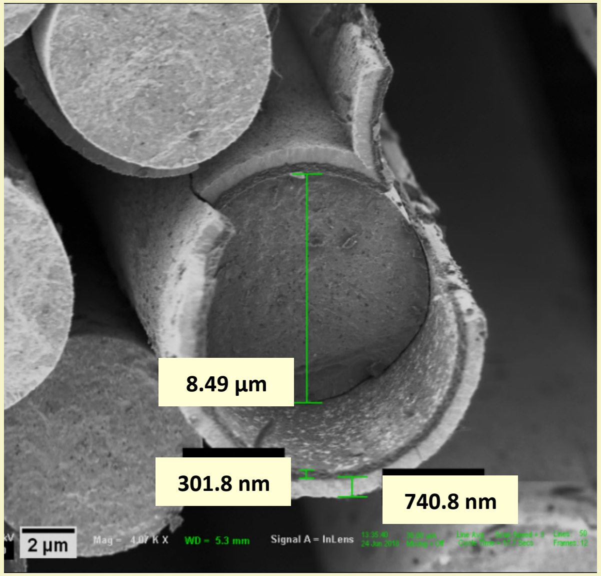
Microstructure of Fiber Cross Fibers

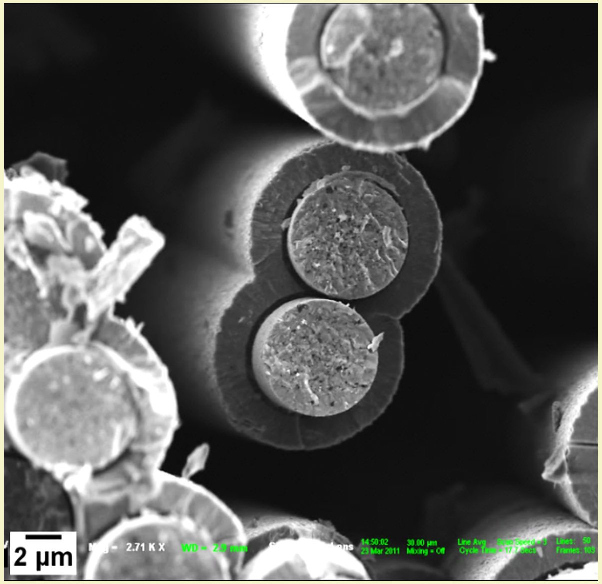
- Three types of fiber were used
- All fibers were coated with pyrolytic carbon and CVD SiC



T300 Carbon Fibers



Tyrano SA SiC Fibers



Sylramic iBN SiC Fibers

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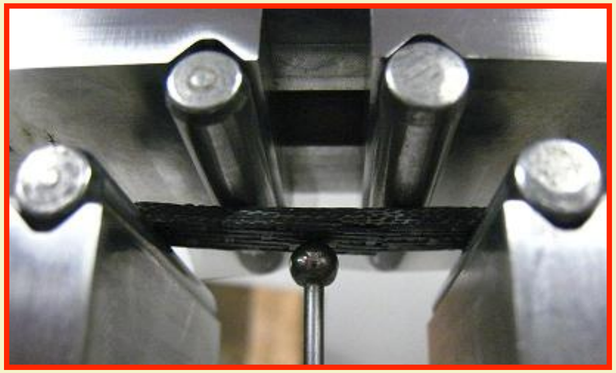
Flexural Properties of CMC After FAST

Material	Porosity %	Avg Flexural Strength (MPa)	Average Flexural Strain (%)	Reported Conventional Tensile Strength (ksi)
SiC-Monolithic	1	607	0	40
T300/FAST SiC/2000 ^{0}C/ 80MPa/20min	6	416	0.86	~40-50
Tyranno SA Fiber/ 2000 ^{0}C / 80MPa/2min	8.3	350	0.30	40-50
Sylramic iBN/1850 ^{0}C / 80Mpa/20 min	9.7	206	0.24	40-50

- Each fiber had CVD SiC/Graphite coating
- Flexural strengths of all materials correlated with density
- Inadequate FAST densification resulted in shear failures of flexural coupons
- Always observed some fiber breakage with Sylramic
  - Possible artifact of bonded Sylramic fibers



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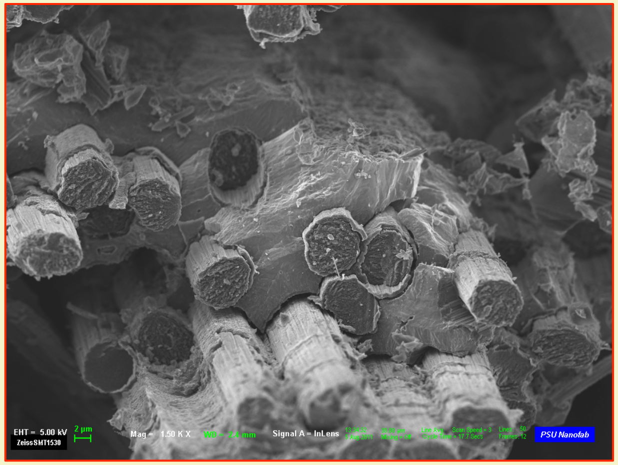
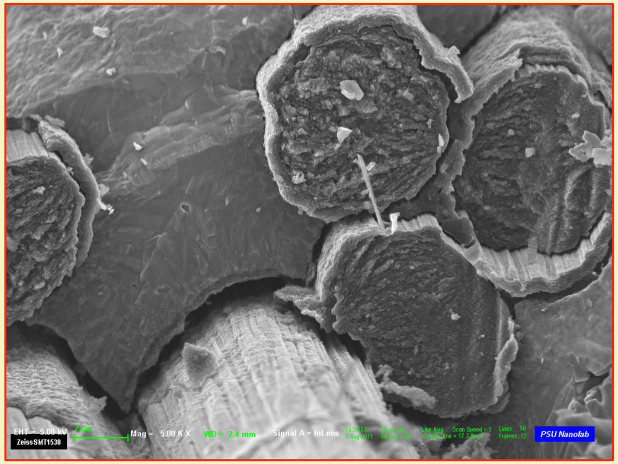
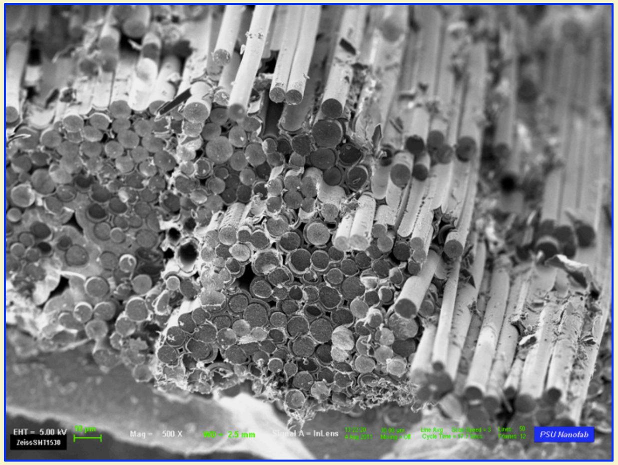
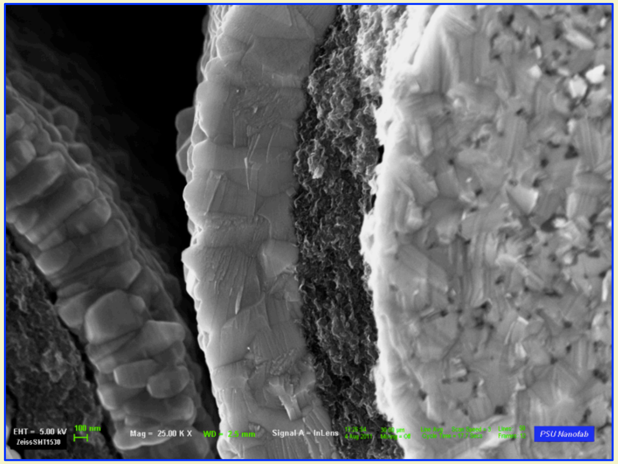
Fracture Surface



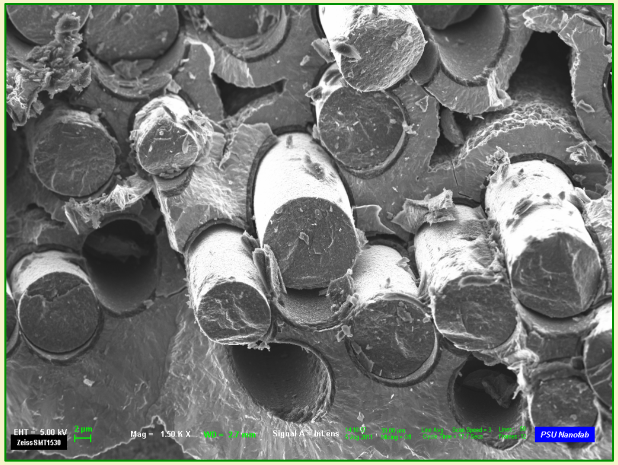


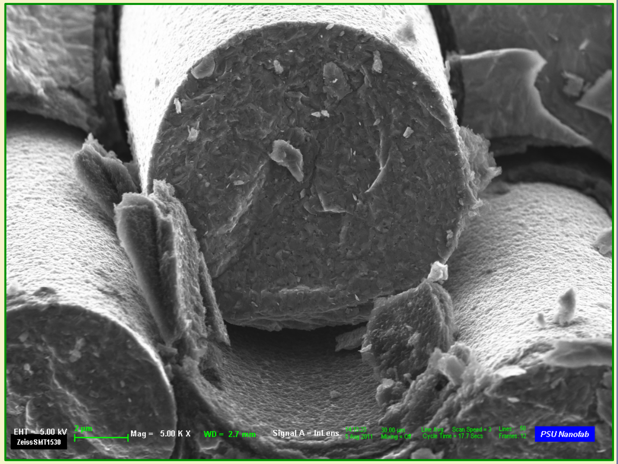
T300 (A1), 2000°C, 80 MPa, 20mins





Tyrano (A1): 1800°C, 80MPa, 20 mins





Sylramic (A1):1900°C, 80MPa, 20 mins

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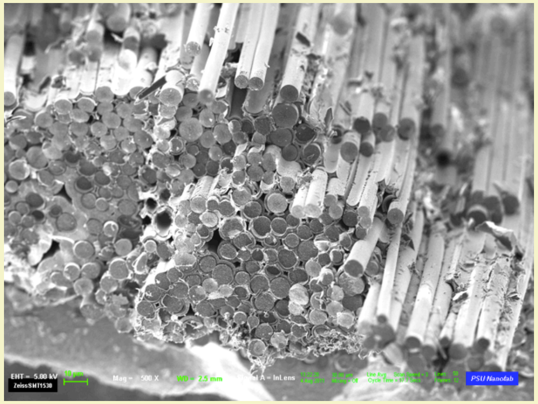
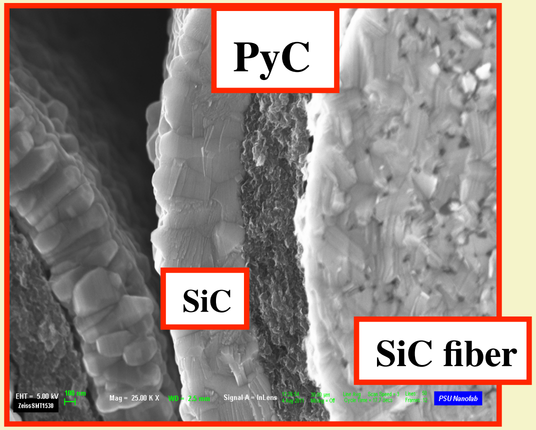
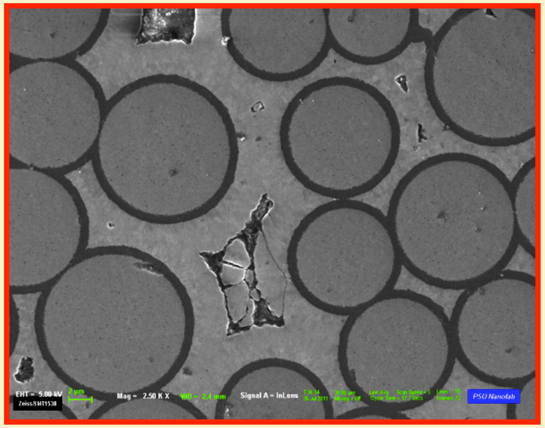
Results on FAST Processing of Tyranno SA SiC/SiC



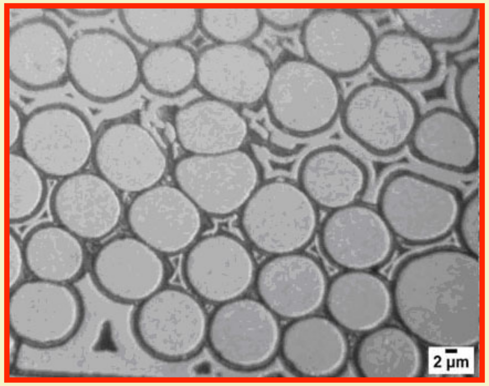
Fractured Surface of Tyranno SA (B5): Fiber put-out (Sintered 1800 ^{0}C, 80 MPa, 20 min)



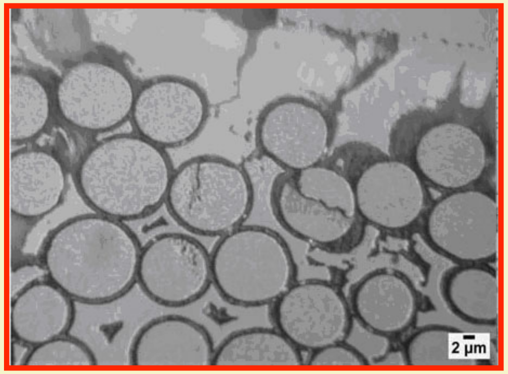
Fracture surface shows fine grain structure of Tyrano fiber is maintained (A1) Sintered: 1800°C, 80MPa, 20 mins)



FAST processing of Sylramic iBN shows retention of fiber coating identity: sintered at 1900 ^{0}C, 80 Mpa, 20 min.



More aggressive FAST deforms fibers



Improper FAST conditions can fail fibers and result in loss of fiber-fiber coating retention

Physical Properties

- **Tyranno SA/FAST SiC: 2.84 g/cc and 8.3% porosity**
- **Sylramic iBN/FAST SiC: 2.80 g/cc and 9.0% porosity**

Past Effort