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SiC-SiC CMCs Using BN powder coated silicon carbide fibers

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SiC – SiC CMCs using BN powder coated silicon carbide fibers

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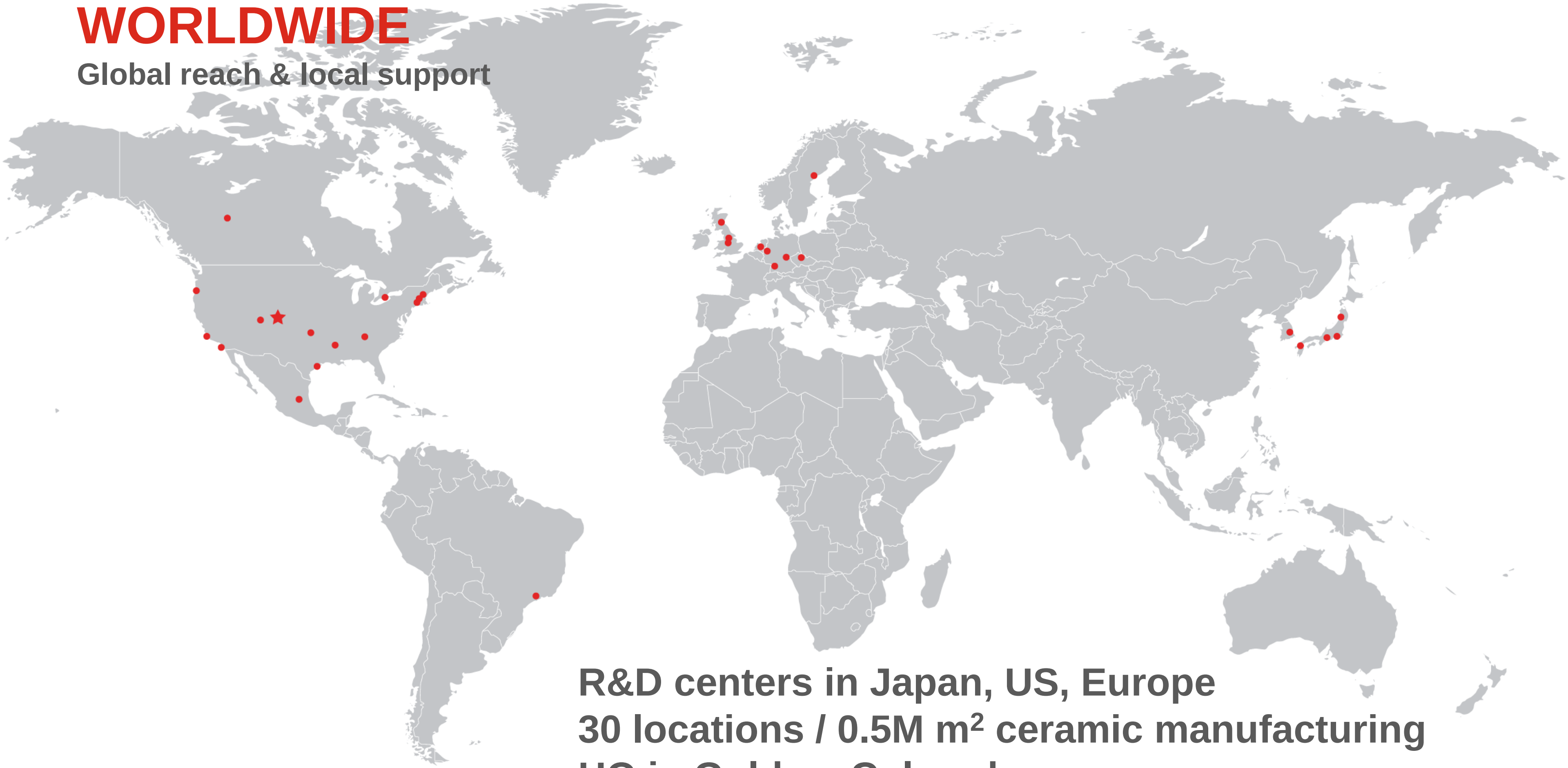
November 8, 2017

CoorsTek

WORLDWIDE

Global reach & local support



R&D centers in Japan, US, Europe

30 locations / 0.5M m^2 ceramic manufacturing

HQ in Golden, Colorado

Silicon Carbide Processing at CoorsTek K.K.

Direct Sintered SiC
- CMP Plates
- Stage platforms

Reaction Bonded SiC
- Wafer boats
- Radiant tubes

SiC CMC

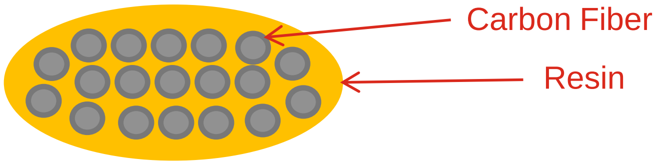
CVD SiC
- Process rings
- Showerheads

Carbon Fiber CMC
- Disc brakes
- Roller hearth

BN Powder Motivation

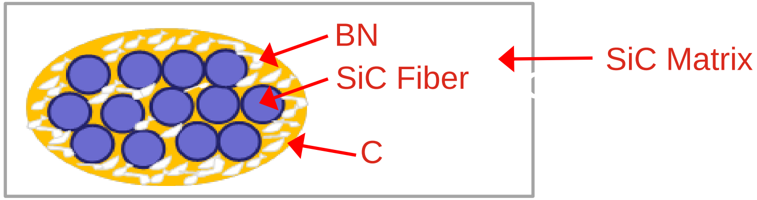
1. Performance

- Lessons from C-SiC composite experience
- Resin/slurry penetrates fiber bundles >
  BN slurry should be able to do the same



2. Process

- Melt Infiltration (MI) production
- Available BN powder & SiC fiber options
    - Denka, Showa Denko, Maruka
    - Ube Tyranno SA fiber
      (coat tows woven into fabric)



3. Value

- CVD alternative
- Lower cost / less hazardous

Mechanical Evaluation | SENB

Samples from ~ 16 layer composite

- ~45 mm x 15 mm x 5 mm
- 2 test bars per sample

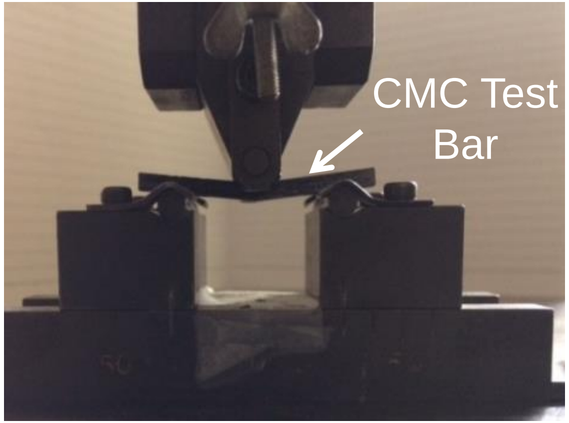
JCRS 201

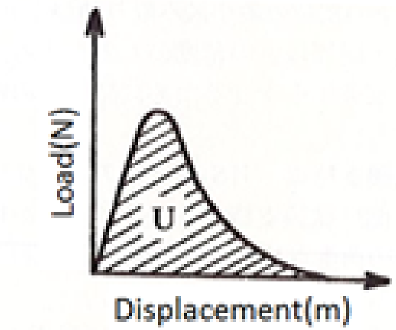
- 40 mm L x 3 mm W x 4 mm D (ideal)
- Notch depth is half the height: typically 2mm
- 3-point flexure   (30 mm span)
- 0.01 mm/min

Fracture energy from crosshead position

$$\gamma = U / 2A$$

- Trapezium software (Shimadzu)





First Trial

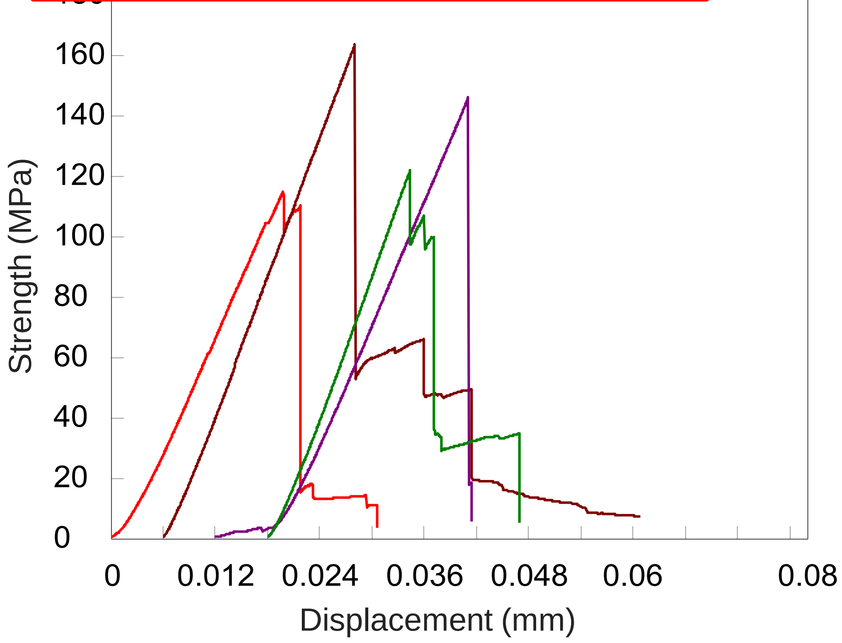
SiC Fabric Sheets	Tyranno SA 8HS weave
Interphase Coating & Drying	BN (Φ0.7 μm） UHP in alcohol + resin
Matrix Infiltration & Drying	Matrix slurry (SiC + phenolic resin)
Prepreg Layout	
Thermoforming	
Preforming	← Cured body
Si infiltration	
Evaluation / analysis	SENB (JCRS 201) SEM, cross-sections

First Trial | Results

Density is high; porosity is low

	Vol.% Fiber	Density (g/cc)	Porosity (%)
Preform	43	2.19	15
Si Infiltrated	---	2.77	2

Fracture energy Average: 40 J/m^2
Strength Average: 140 MPa



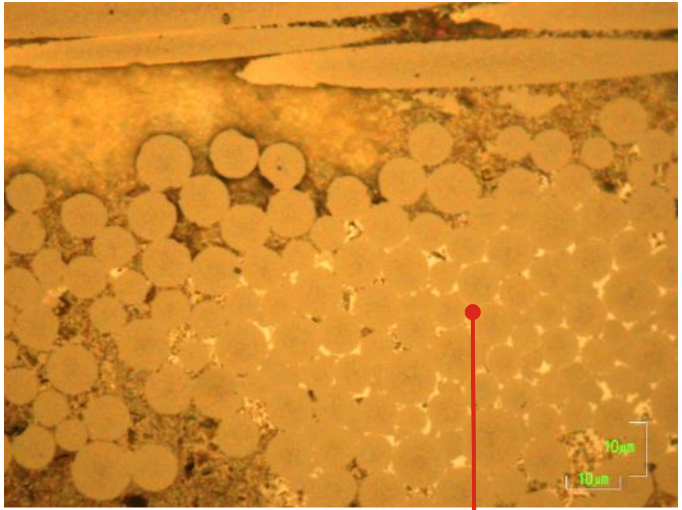
Both strength and breaking energy are low

First Trial | Observations & Conclusions

1. Silicon penetrated the fiber bundle
2. BN appears to be displaced or reacted with the silicon
   > no fiber protection from the BN particles

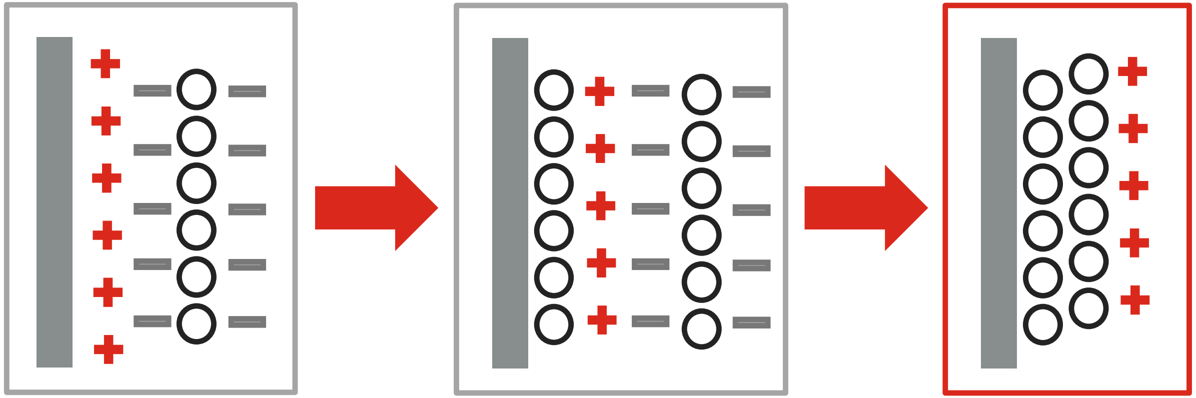
Strategies for improvement:

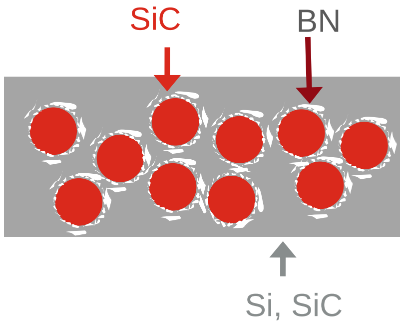
a. Improve adhesion of BN to fibers
b. Electrolyte solutions
c. Thicker BN layers



Bright areas indicate silicon penetrated to interior of bundle.

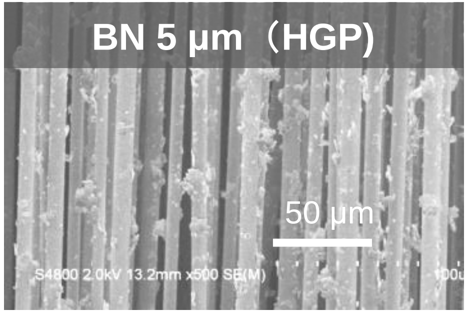
Electrolyte Concept | Heterocoagulation



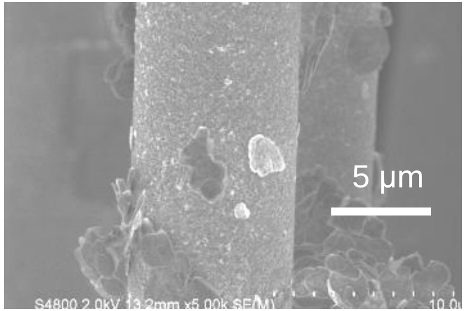


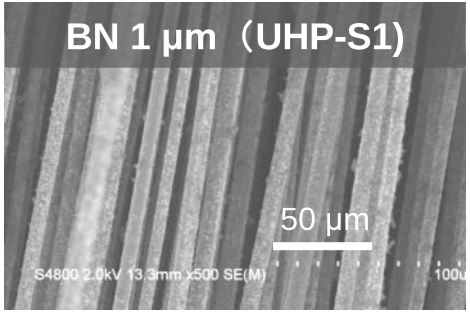
- Poly-electrolyte aqueous solutions: A- & B+
- Powder suspension made with A- electrolyte
- Fabric dipped in B+ electrolyte then in A-
- Coated fabric then rinsed with solution C

Particle Size Limitation

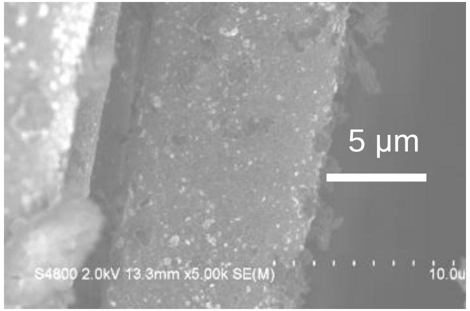


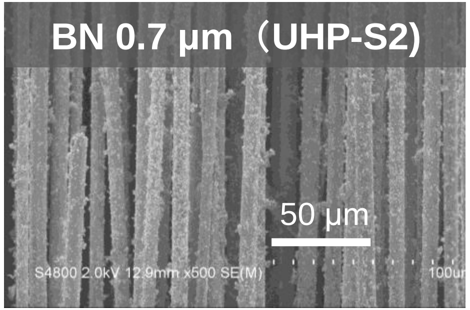
Not effective



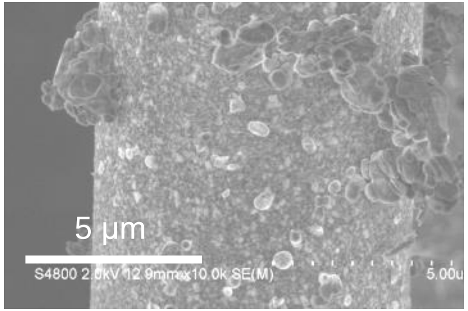


Not effective at 1µm



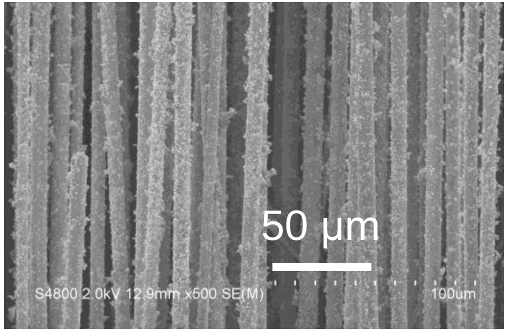


Very effective

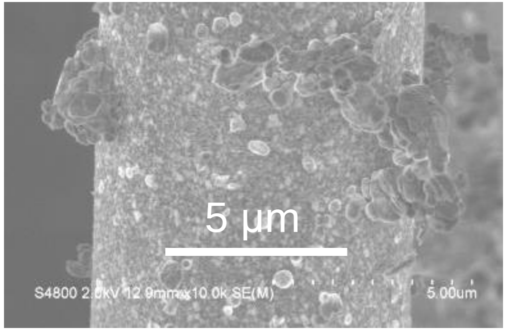


Improving Solution B

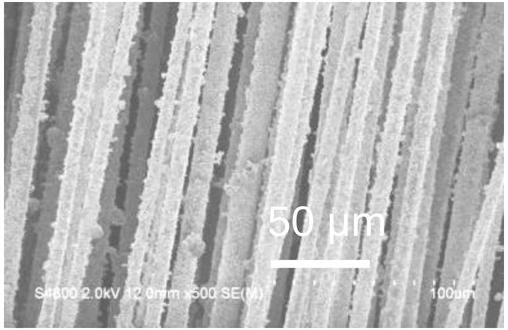
BN 0.7 µm (UHP)



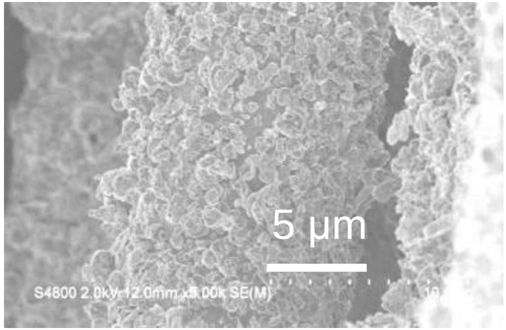
Single layer coat



BN 0.7 µm (UHP)



Improved process



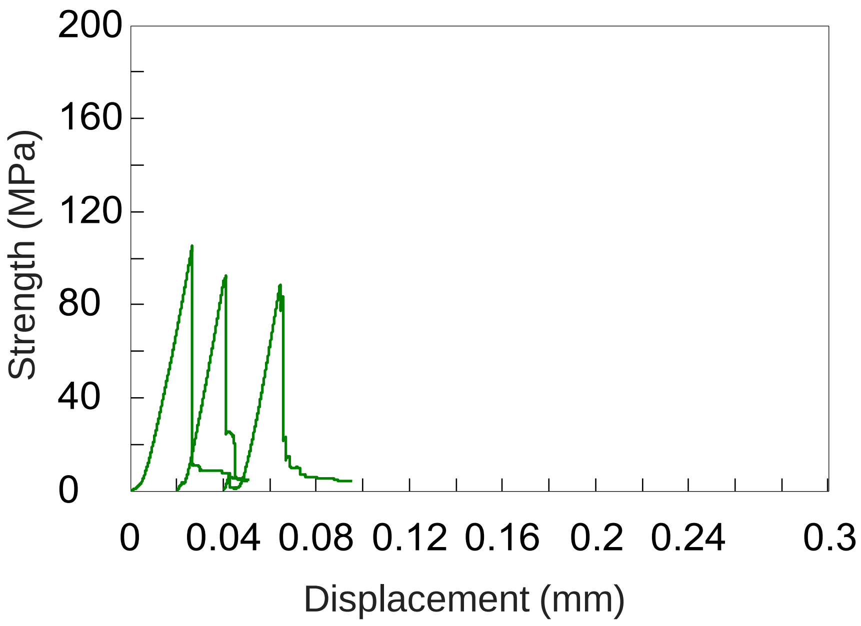
Heterocoagulation | Results

Parameter	Value
Vol % fibers	55
Density (g/cm^3)	2.88
Porosity (%)	3

Heterocoagulation trial

- All failures are brittle
- No improvement in strength
- No improvement in fracture energy

Fracture Energy, average	30 J/m^2
Strength, average	100 MPa



Modifying the MI Process

Issue

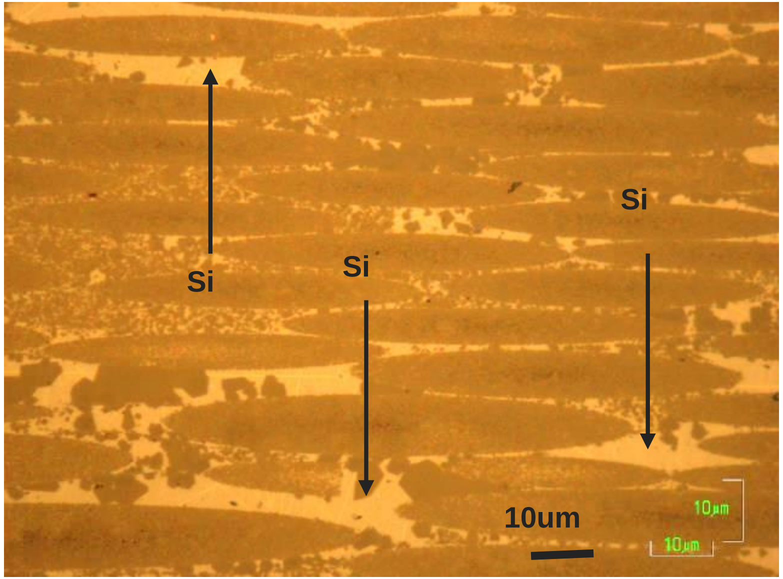
Excess silicon inside and in between fiber bundles

Goals

- Less interaction between Si(l) and BN coating
- Less fiber and interphase degradation
- Better mechanical properties

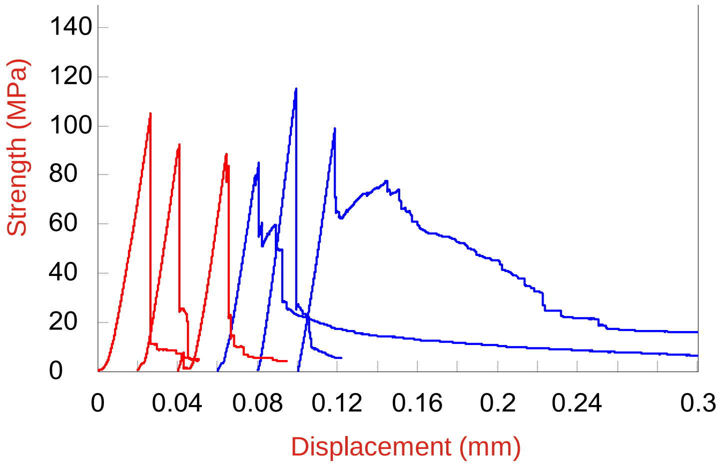
Approach

Reduce excess silicon by providing additional carbon for reaction during infiltration



Combining the Processes

PARAMETER	NO EXTRA RESIN	EXTRA RESIN
Vol.% Fibers	55	58
Density (g/cc)	2.88	2.82
Porosity (%)	3	2



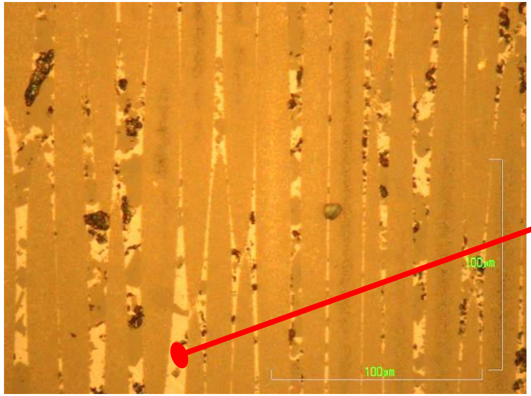
NO EXTRA RESIN
Fracture Energy Average 30 J/m^2
Strength Average 100 Mpa

EXTRA RESIN
Fracture Energy Average 200 J/m^2
Strength Average 100 MPa

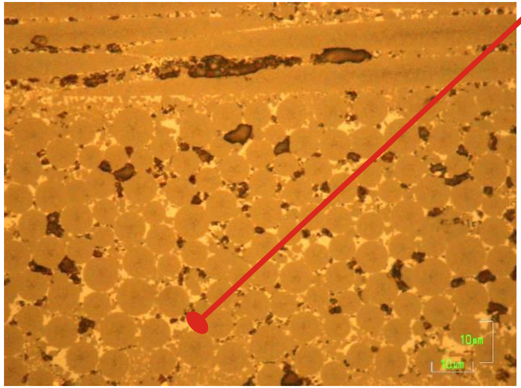
Extra resin (carbon) added

1. Significant increase in fracture energy ... but high variation
2. No strength improvement

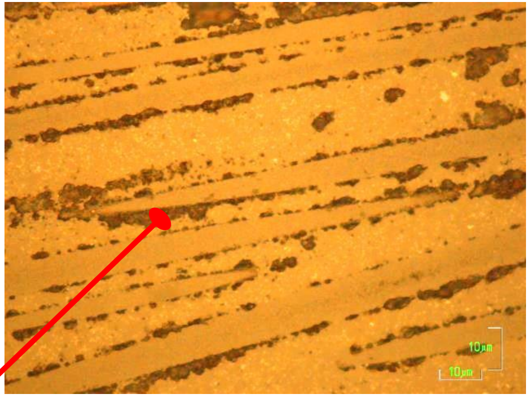
Microstructures | With & Without Extra Carbon



No extra carbon

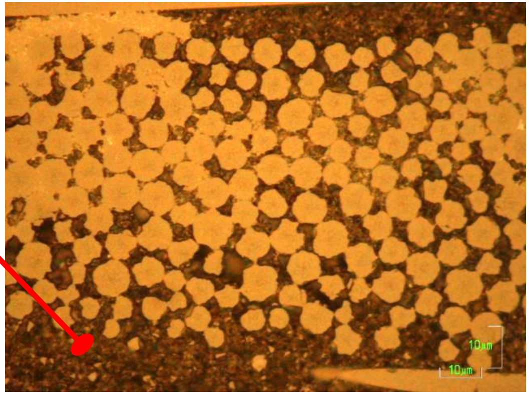


Bright areas are silicon bonded to the fibers.



With extra carbon

Dark areas are silicon carbide next to the fibers.



Modifying Particle Coating Process

Issue

Fibers coated unevenly

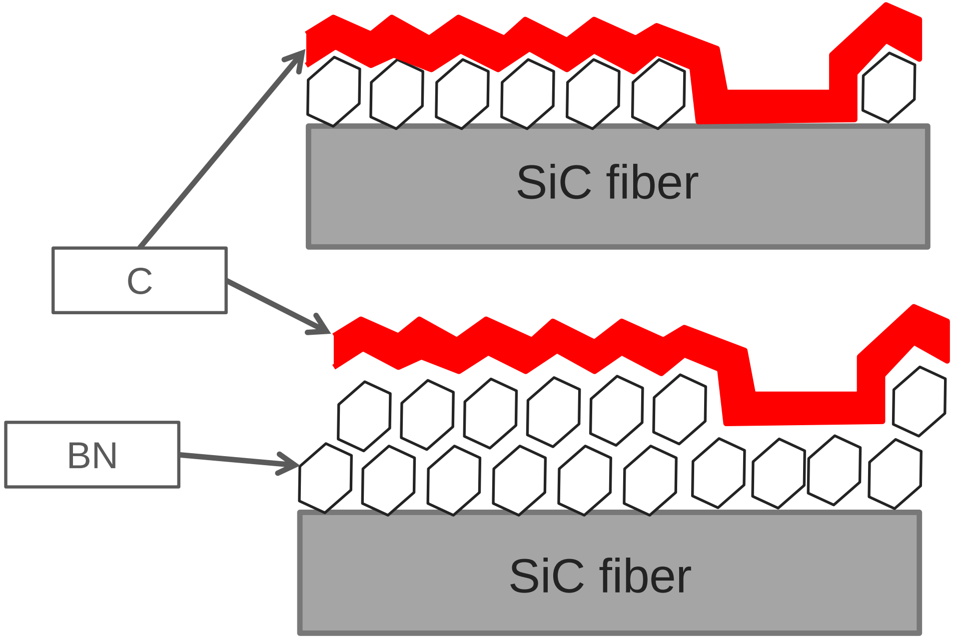
- Multiple layers in some areas
- No coating in some areas
- No fiber protection/weak interphase

Goals

- Improve coating uniformity
- Produce fiber/BN/C structure

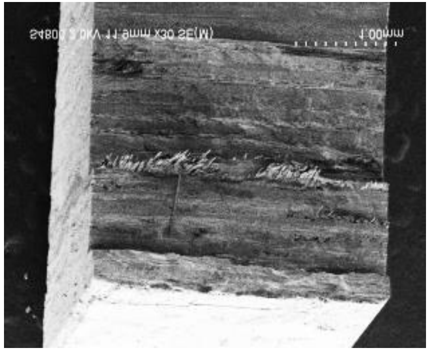
Approach

- Nano-sized BN particles
- Multiple BN coats

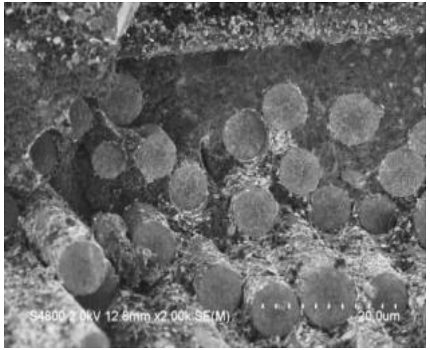


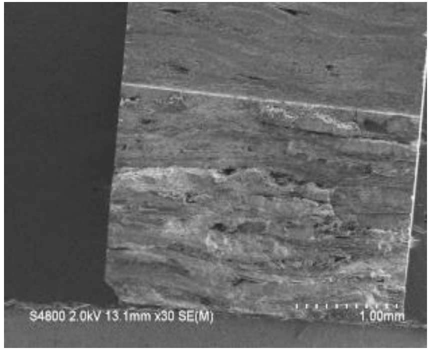
Individual fibers

Fiber Pullout | Improves With Multiple Coats

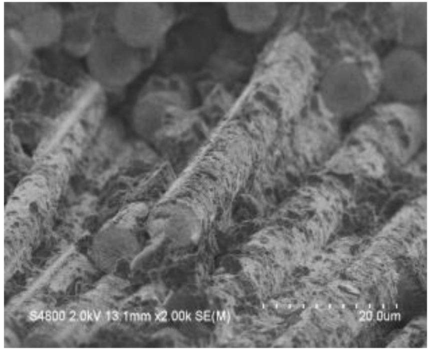


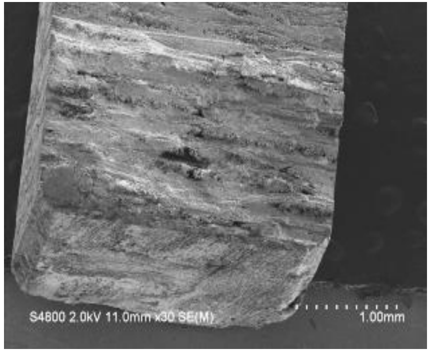
0.7 µm, 1X



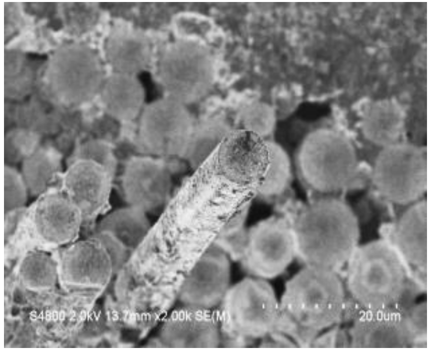


0.05 µm, 5X

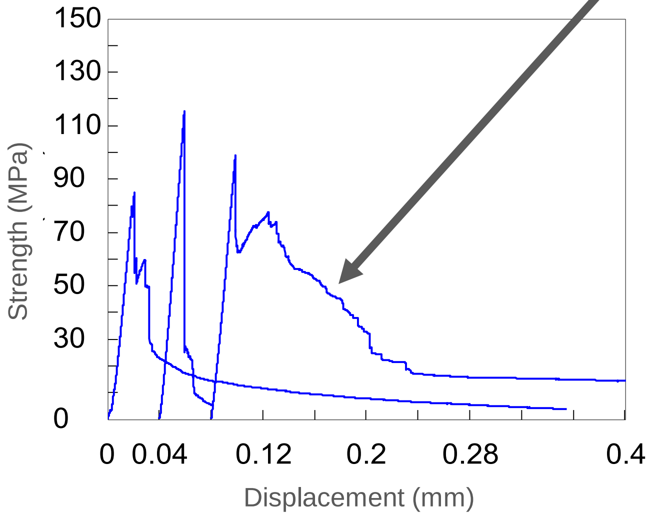


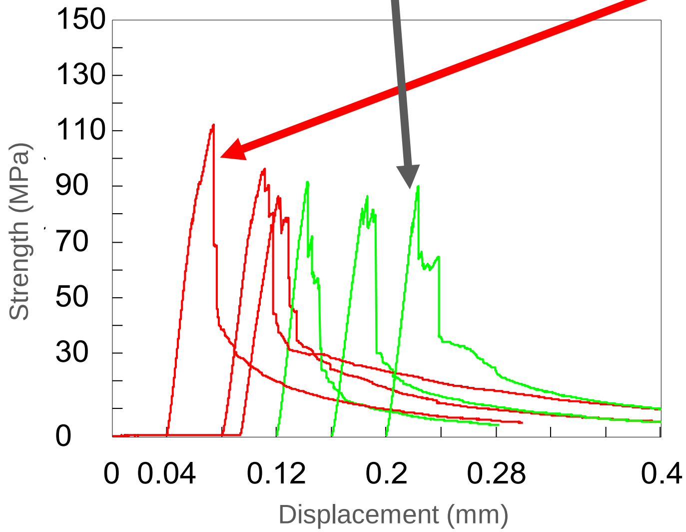


0.05 µm, 7X



PARAMETER		1X BN (UHP) 0.7 µm	1X BN 0.05 µm	5X BN 0.05 µm	7X BN 0.05 µm
Vol. % Fibers		58	49	43	43
Bulk Density	(g/cm^3)	2.82	2.80	2.49	2.53
Porosity	(%)	2	5	11	12
Flex Strength	(MPa)	100	100	90	100
Fracture energy	(J/m^2)	200 (MAX 450)	70 (MAX 90)	100 (MAX 140)	150 (MAX 200)

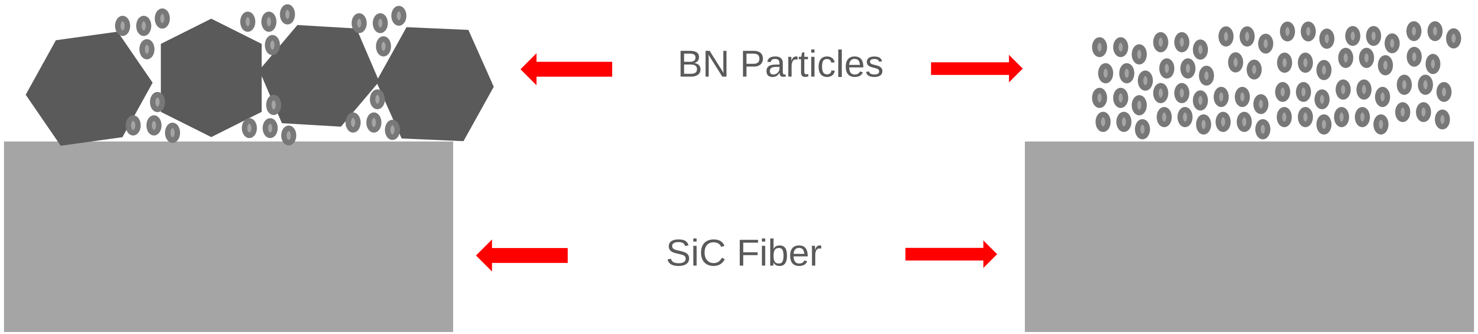




Scenarios to Increase Thickness

- Optimized particle packing; bimodal distribution
- Minimize the number of layers

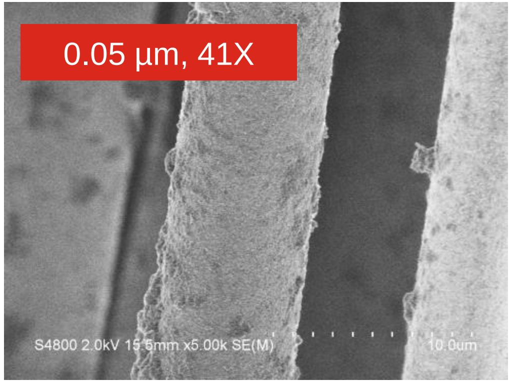
Multiple layers for thickness



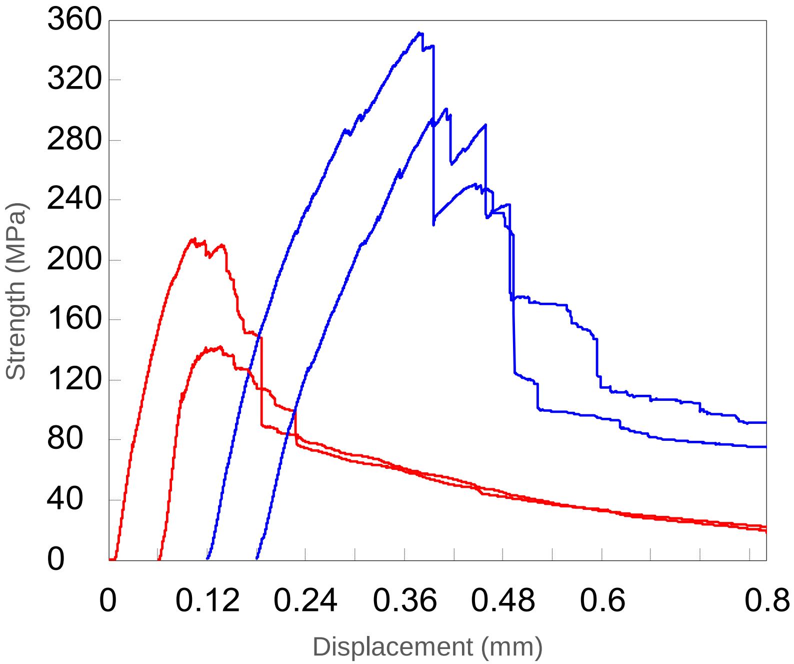
Coating Schematic
1X 0.7 µm + 9X 0.05 µm

Coating Schematic
41X 0.05 µm

Increase Number of Coatings



ITEMS	1 X 0.7 µm 9 X 0.05 µm	41 X BN 0.05 µm
Vol.% Fiber	48	49
Density (g/cm³)	2.60	2.62
Porosity (%)	8.05	7.77
Strength (MPa)	178	326
Avg Energy (J/m²)	1400	4000



Conclusions

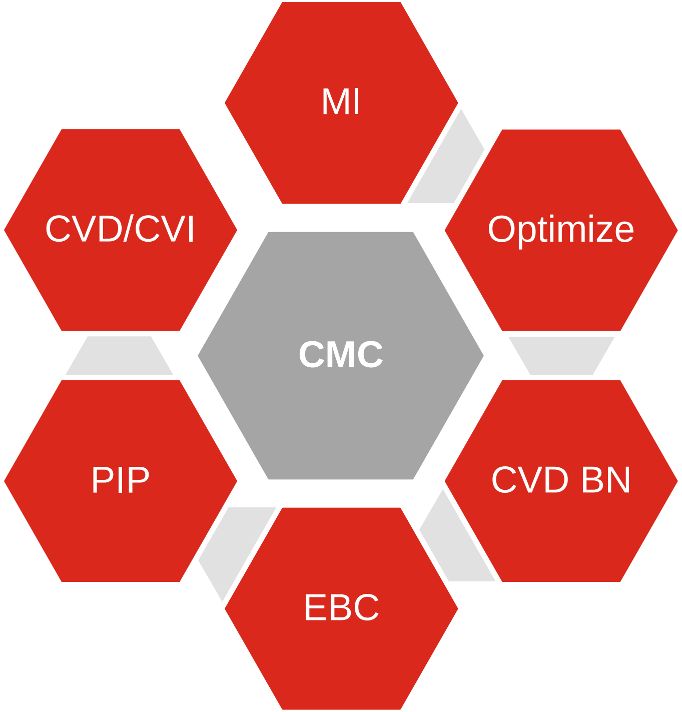
1. BN powder adhesion to SiC fiber by heterocoagulation
    - Coat with fine BN particles (< 0.7 µm)
    - Multiple coats applied

2. MI process improvement
    - Extra carbon added via resin
    - Coating over BN particles

Excellent mechanical properties achieved

- Strengths > 300 MPa
- Fracture energy > 4000 J/m^2

Next Steps



Thank You

November 8, 2017

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