Synthesis and Testing of Coated Carbon Nanotube Composite Microstructures

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Project Motivation

Aerospace – hypersonic flight

- Materials challenges

Ultra-high-temperature ceramics

- Hafnium diboride (HfB_2) – melting temperature: 3250°C

Chemical vapor deposition

- Composite foams
- Carbon nanotubes (CNTs) base material + HfB_2 coating

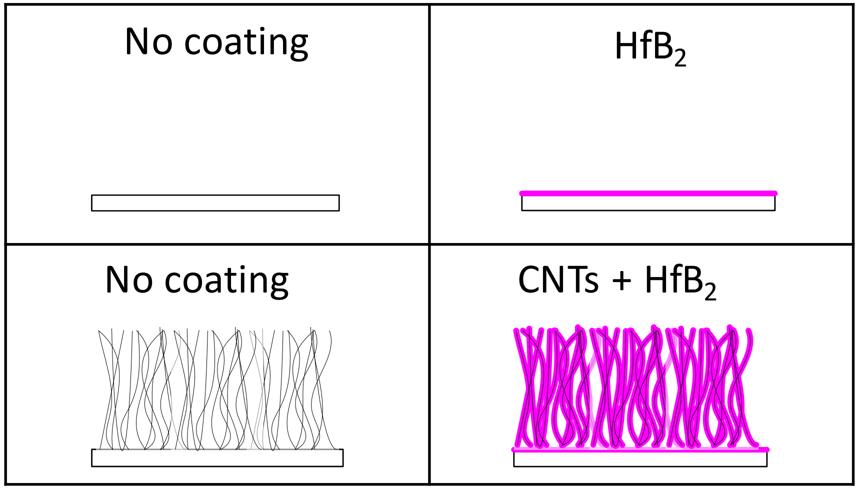


Image source: NASA

Synthesis

Substrate + metal catalyst + gasses = CNTs

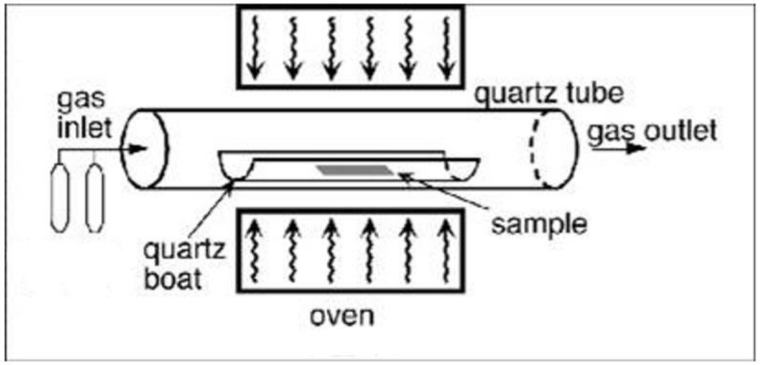
Chemical vapor deposition

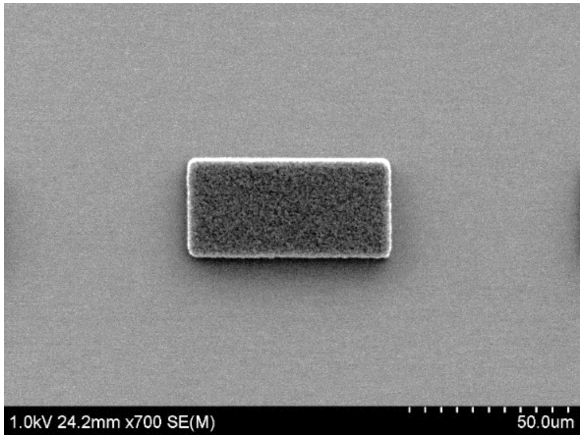
CNT growth recipe

- Pre-bake – air (850°C)
- Growth step – H_2, C_2H_4, He (775°C)
- Flush tube – He (< 775°C)

Coating

- Hafnium diboride precursor of $Hf[BH_4]_4$





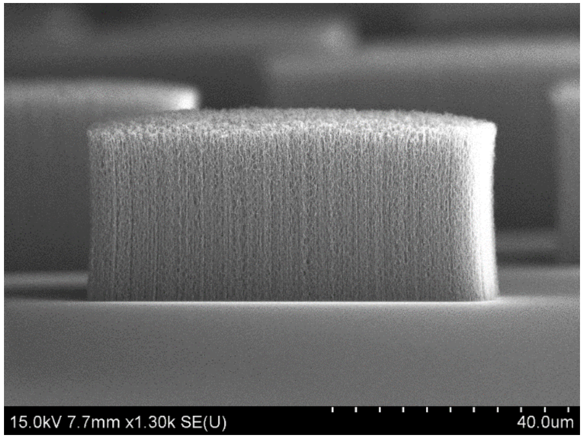
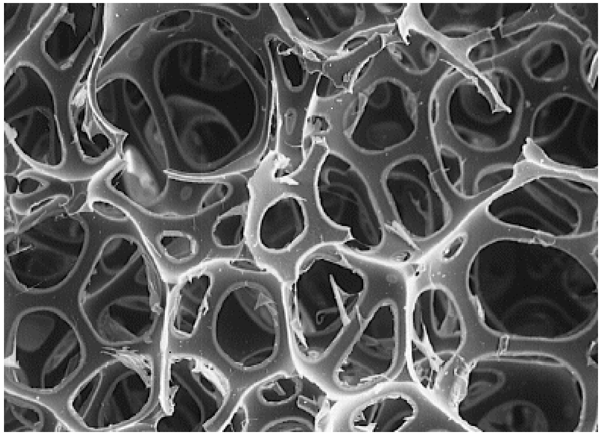


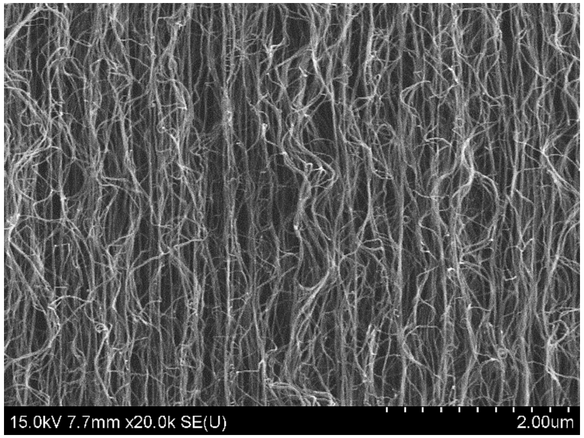
Image source: https://sites.google.com/site/nanomodern/Home/CNT/syncnt/cvd

Coating CNTs

- CNTs have excellent thermal conductivity, mechanical, and electrical properties
- Patterned substrate creates a “forest” of vertically aligned
- Acts like a foam – held together by Van der Waals forces
- Creates a network of “cells” for the gaseous coating to infiltrate



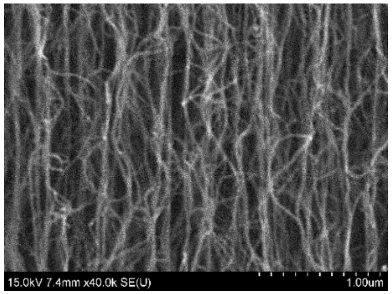
Typical Foam*



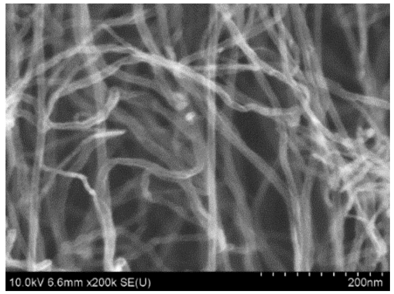
CNTs

*Image source: Dow Polyurethanes - Foam Bubble Initiation

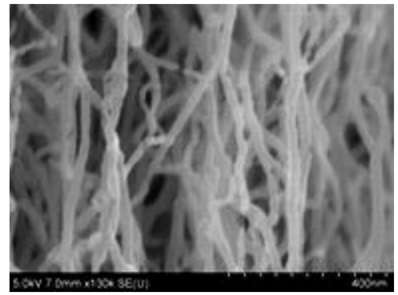
Coating Thickness



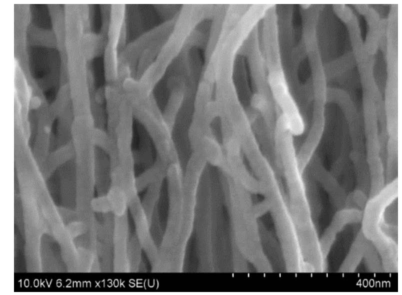
Uncoated



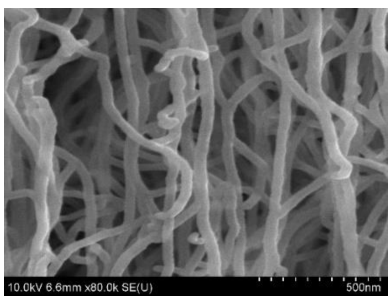
~ 3 nm



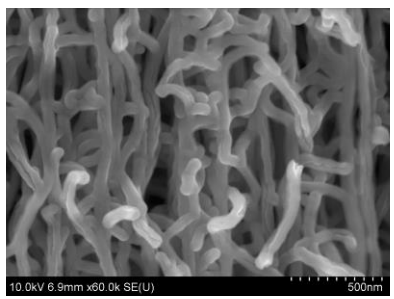
~ 7 nm



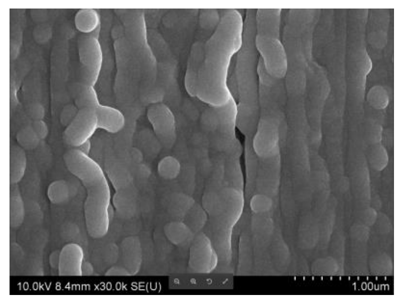
~ 12 nm



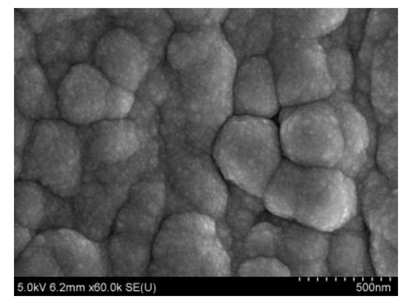
~ 17 nm



~ 28 nm



[2 Torr]



[4 Torr]

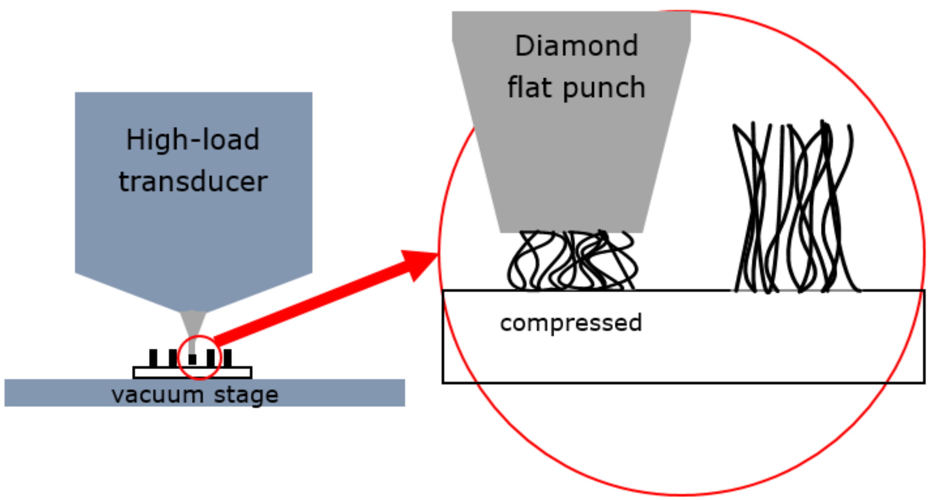
Nanoindentation Testing

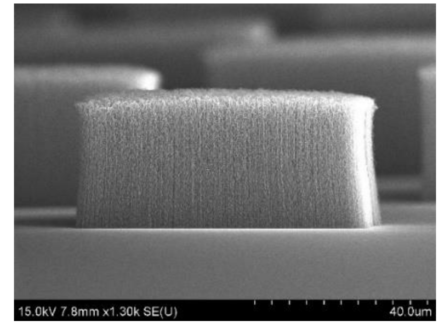
Test Apparatus

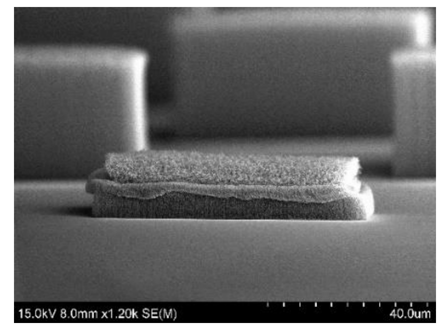
- Hysitron Triboindenter
- 150µm flat punch (tip)
- Load-control vs displacement-control

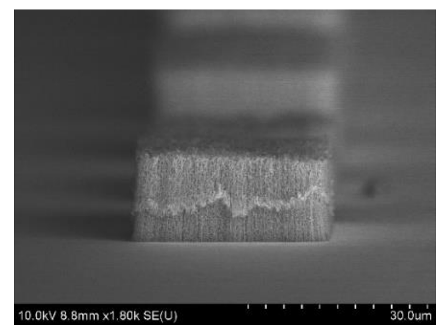
Pillar behavior

- Collapsing – thin coatings
- Fracture – thick coatings

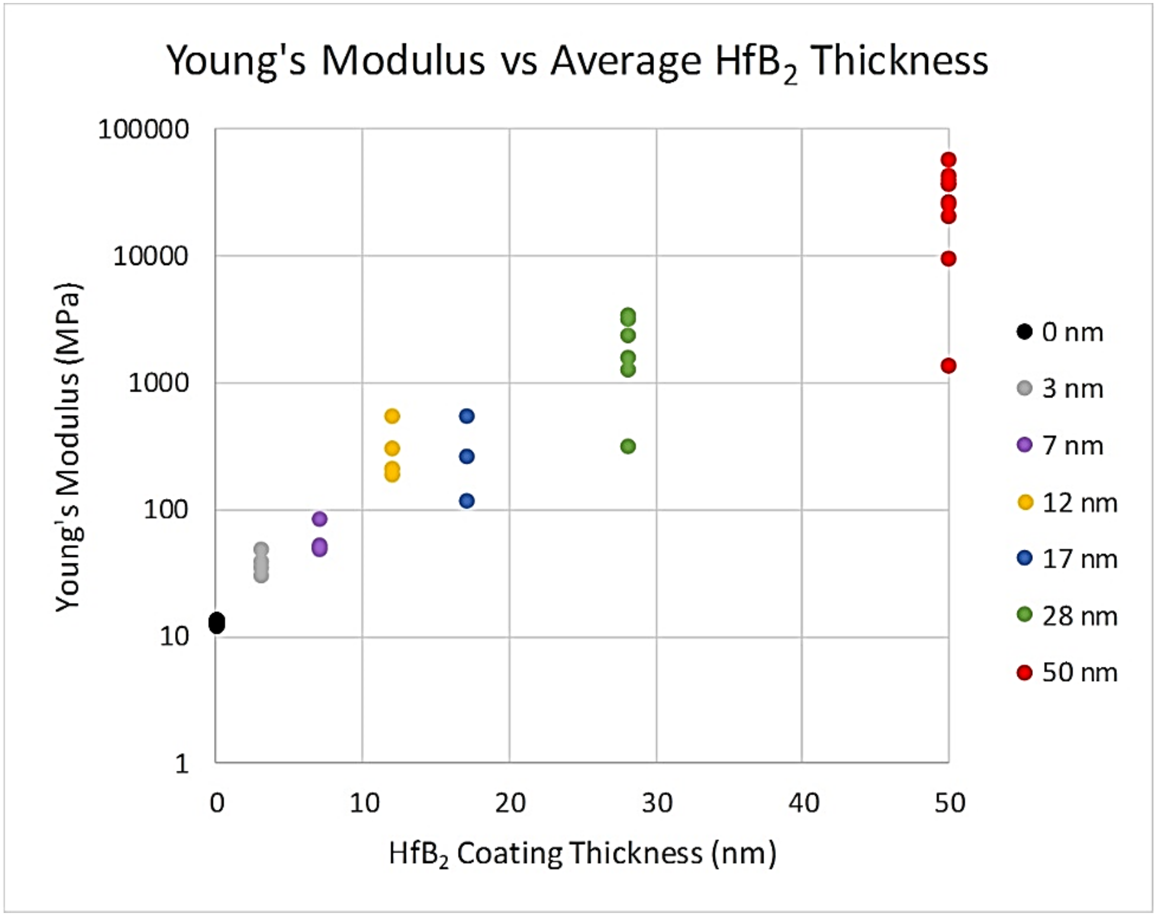








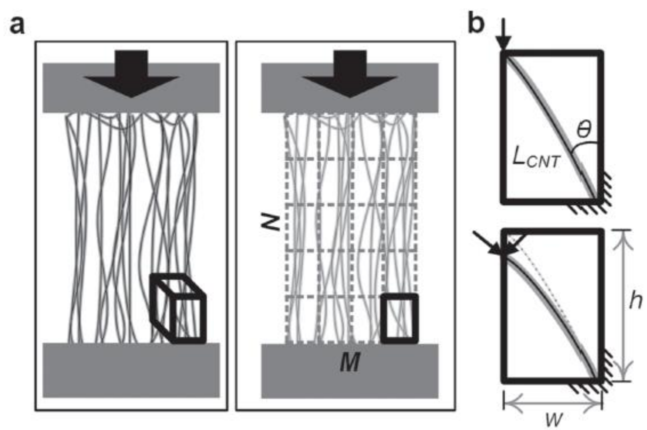
Results



Young's Modulus vs Average HfB2 Thickness
100000
10000
1000
100
10
1
Young's Modulus (MPa)
0
10
20
30
40
50
HfB2 Coating Thickness (nm)
0 nm
3 nm
7 nm
12 nm
17 nm
28 nm
50 nm

Relationship

- Young's modulus was plotted against density to determine a power law relationship
- $E \sim \rho^{1.698}$
- CNT cell size of 115 nm
- CNT density ~ 2,200 kg/m^3
- HfB_2 density ~ 10,500 kg/m^3



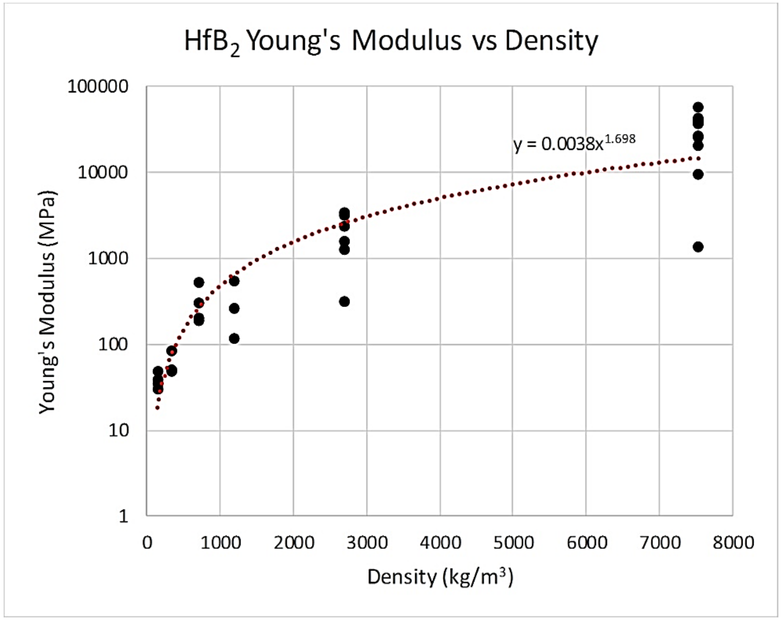
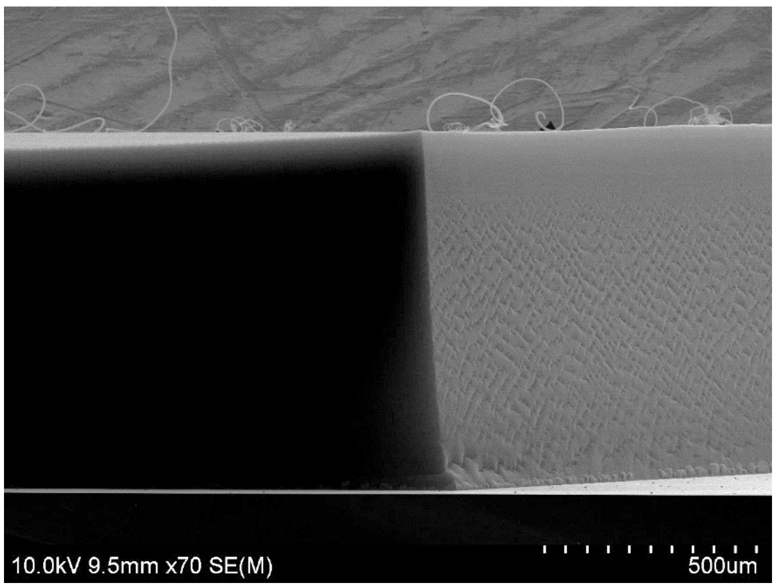
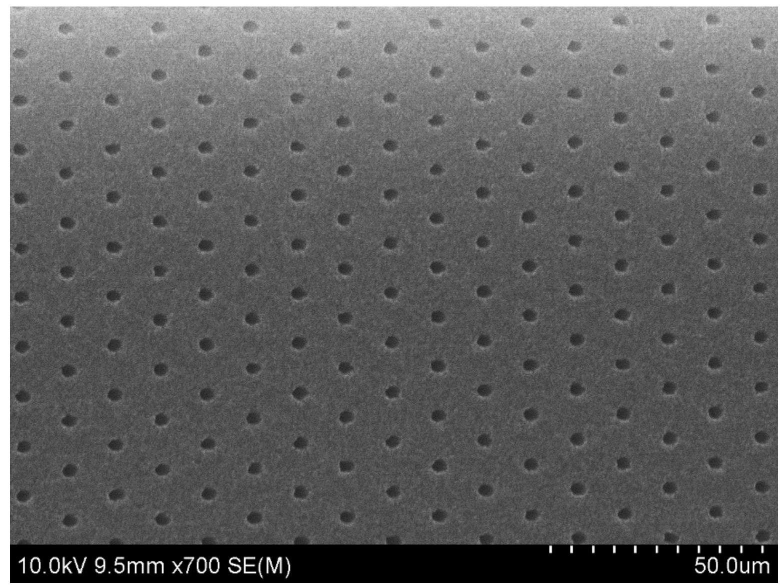


Image source: A. Brieland-Shoultz *et al.*, "Scaling the Stiffness, Strength, and Toughness of Ceramic-Coated Nanotube Foams into the Structural Regime"

Future Work

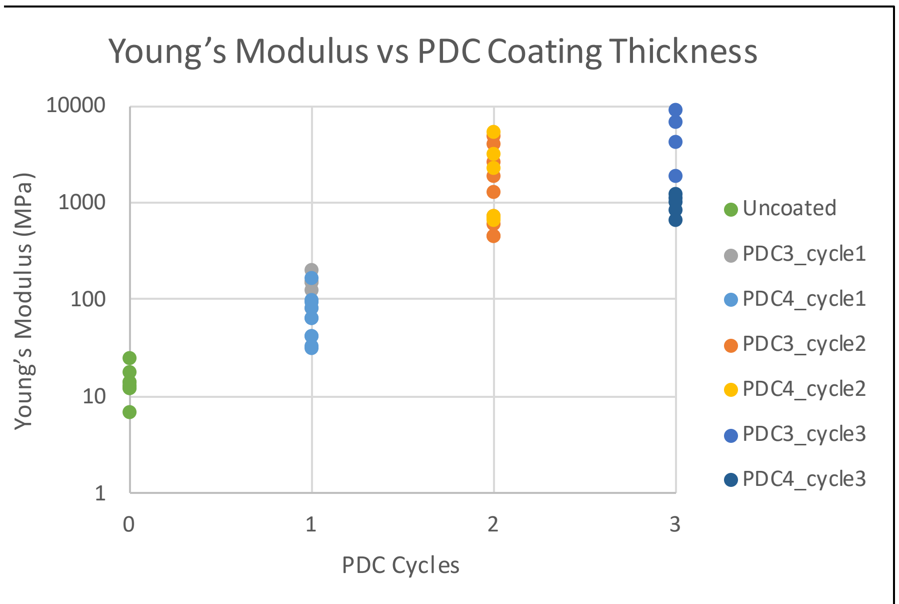
- Infiltration through larger forest – observe coating effects
- Honeycomb perforated – observe coating effects

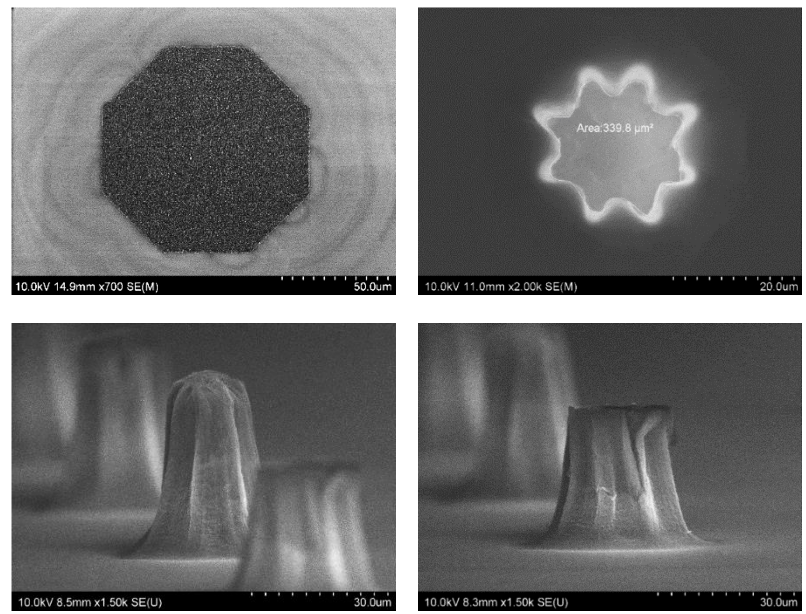




Future Work: PDC

- Polymer derived ceramic (PDC)
- Silicon oxycarbide (SiOC) polymer solution
- Pyrolysis 1000 °C cure in argon resulting in 42% mass loss and 30% linear shrinkage





Summary

Applications

- Applications: Hypersonic, re-entry vehicles, heat shields, leading edges

Results

- Trend in stiffness for the HfB2 coated pillars was $E \sim \rho^{1.698}$
- Highest stiffness for uncoated pillars was about 12 MPa
- Highest stiffness for HfB2 coated pillars approximately 50 nm coated pillars was 56 GPa

Composite foams

- CVD allows to use materials that are difficult to process with conventional methods
- CNTs and coatings to improve properties and develop unique materials

KINETIC MATERIALS RESEARCH GROUP

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