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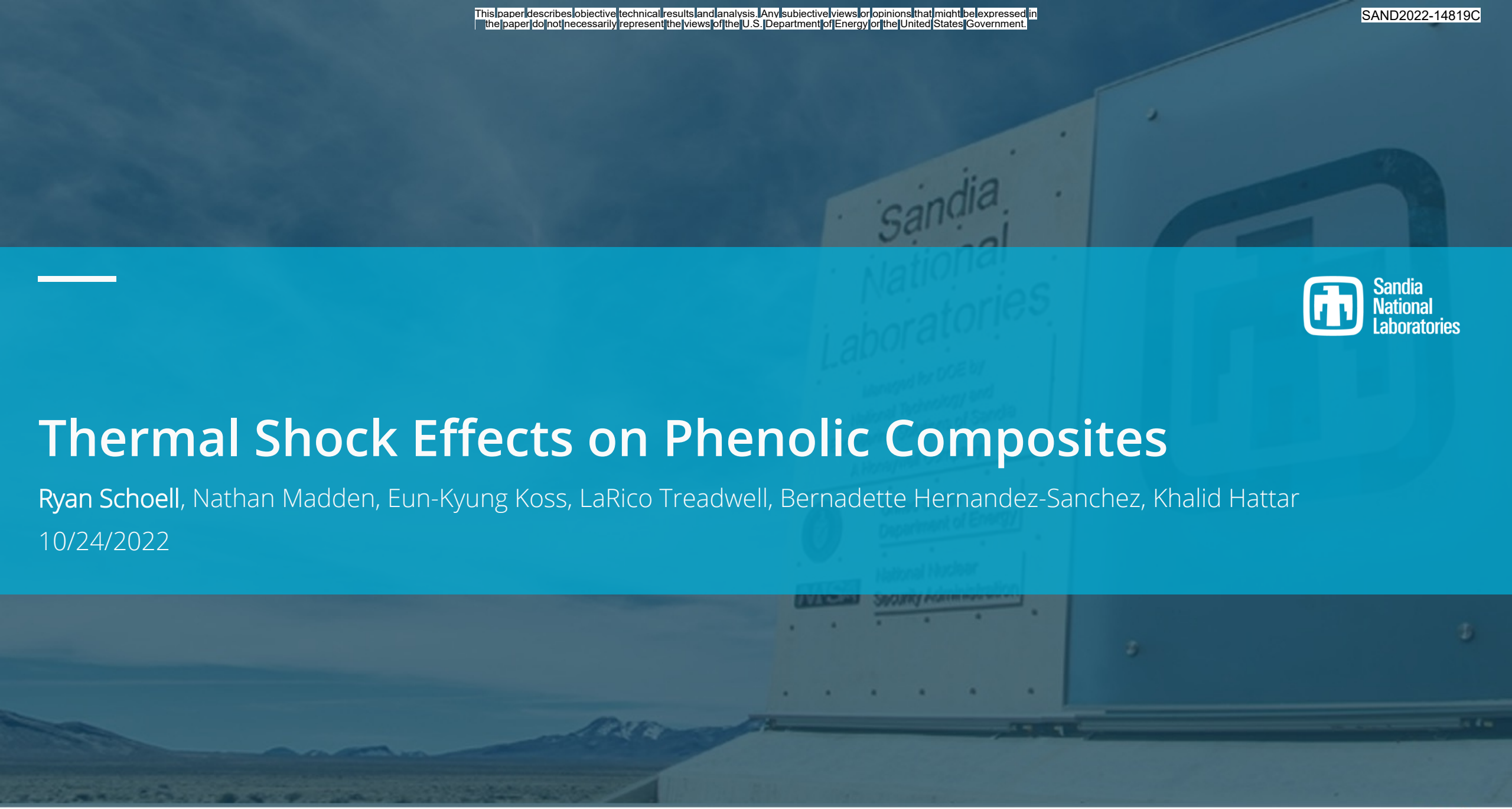

Sandia National Laboratories

Thermal Shock Effects on Phenolic Composites

Ryan Schoell, Nathan Madden, Eun-Kyung Koss, LaRico Treadwell, Bernadette Hernandez-Sanchez, Khalid Hattar

10/24/2022

U.S. DEPARTMENT OF ENERGY | NNSA National Nuclear Security Administration

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Outline

- Introduction
- Methods
- Results
  - Slow Heating with Double Tilt Heating Holder
  - Pulse Laser
  - Continuous Slow Laser
- Conclusions
- Future Work

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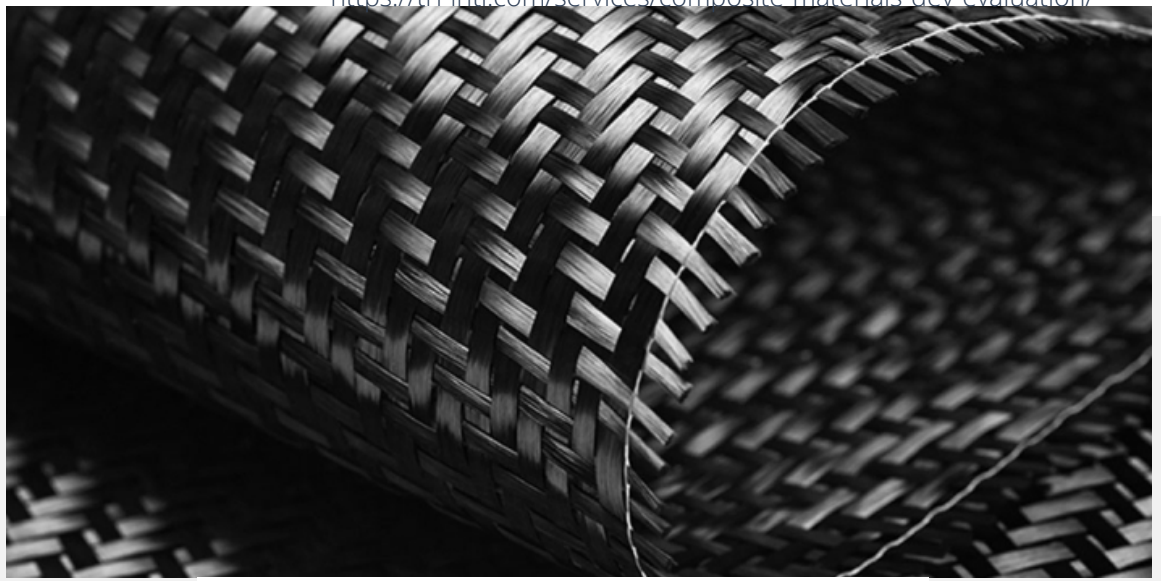
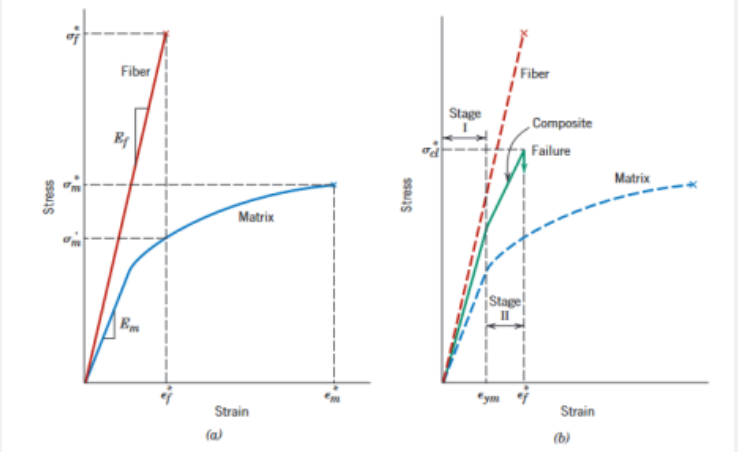
Introduction

https://tri-intl.com/services/composite-materials-dev-evaluation/



- Composites offer unique properties such as high strength, low density, crack resistance, etc.
  - Combination of different materials i.e. fiber reinforced polymers
- Many variables can go into creating fiber reinforced polymers
  - Fiber size
  - Fiber Type
  - Matrix Type
  - Ratio of Fiber to Matrix



Goal: To probe the thermal shock properties of different phenolic based composites i.e. Carbon vs. Silica Fiber using small scale testing

Callister, W.D. (2007) Materials Science and Engineering An Introduction. 7th Edition, John Wiley & Sons, New York

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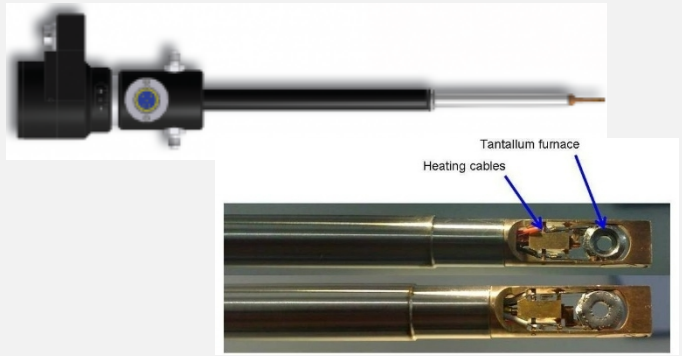
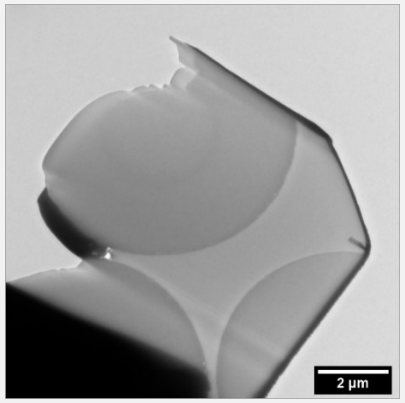
Methods

Gatan Double Tilt Heating Holder

- Slow Heating experiments up to 1000 C
- 15 C/min
- Can track temperature

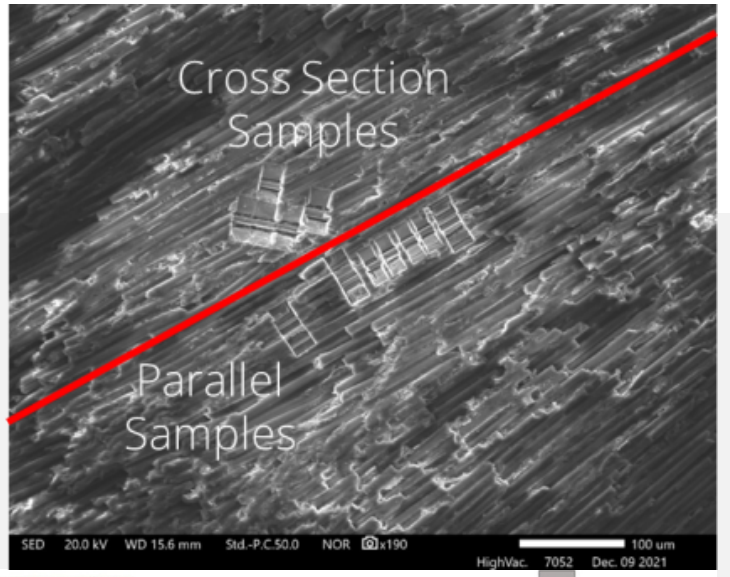


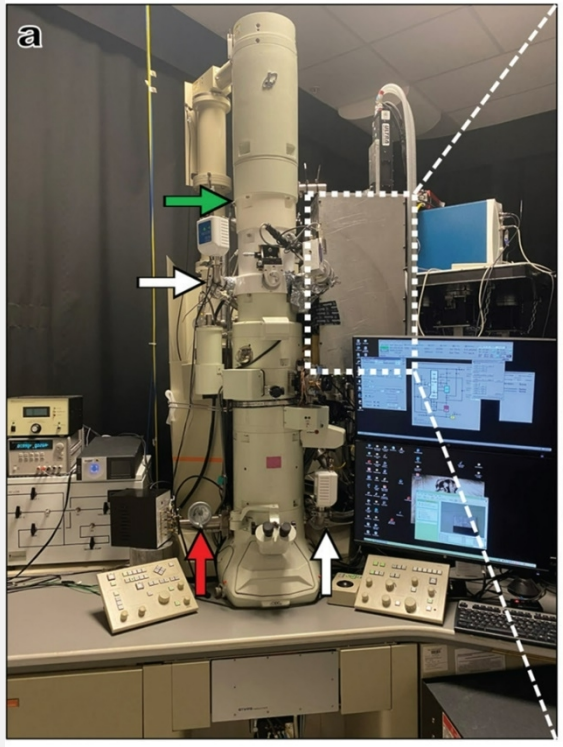


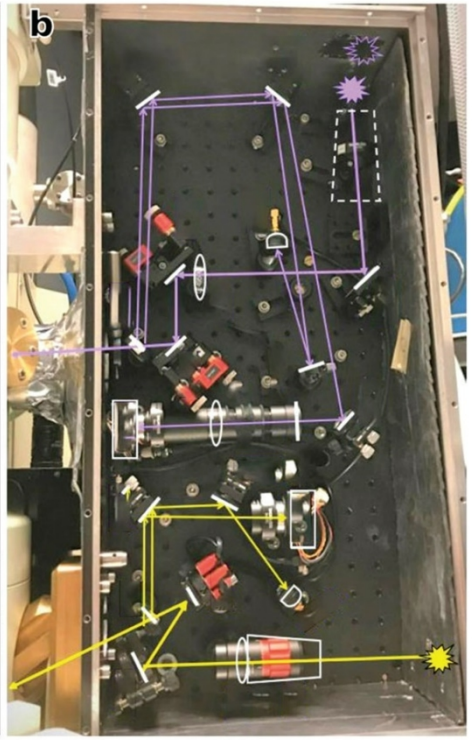
BF TEM

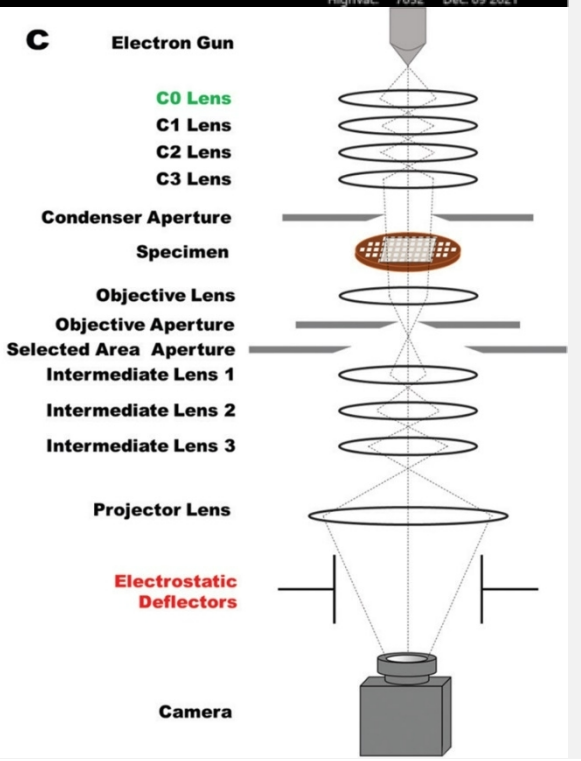
IR (1064 nm) Laser inside TEM

- Quick Heating
- "Constant Laser" vs. Pulsed Laser
- Can't track temperature yet but a solution is in the works (Track Power)
- ~20 um in diameter beam (localized)









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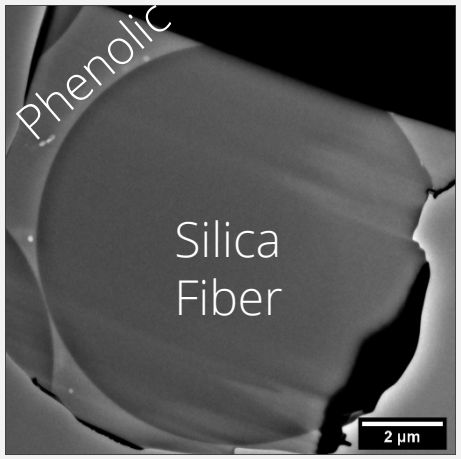
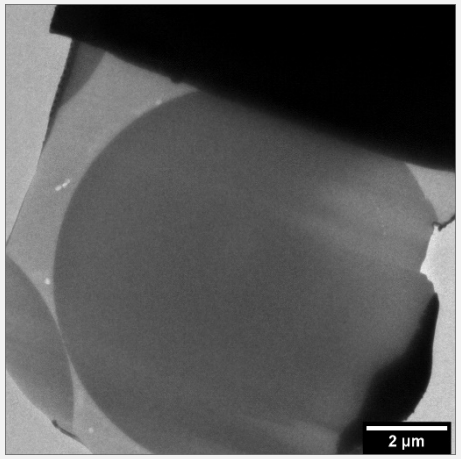
Slow Temperature Ramp with Heating Holder (15 C/min) to 1000 C

Silica Fiber Phenolic

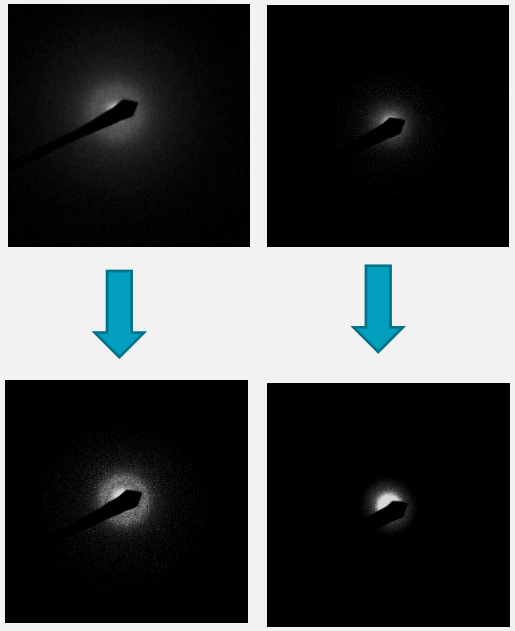
BF TEM



1000 C at 15 C/min



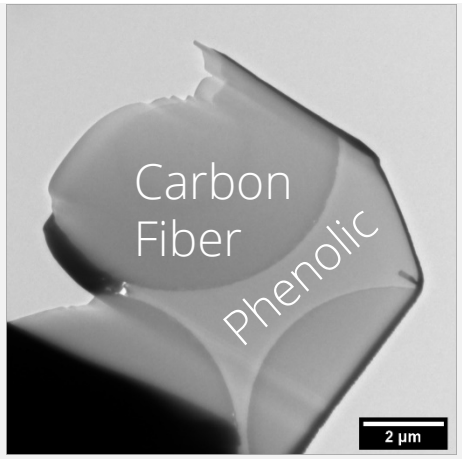
DP - Fiber DP - Phenolic



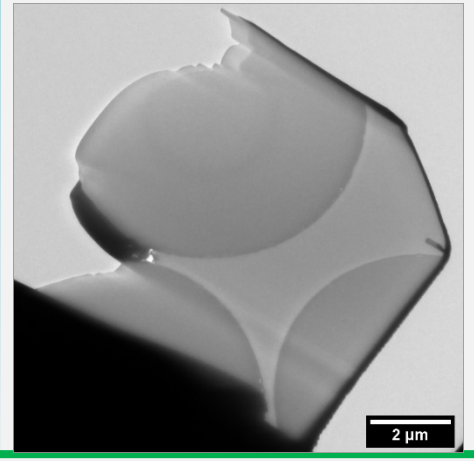
No significant changes

Carbon Fiber Phenolic

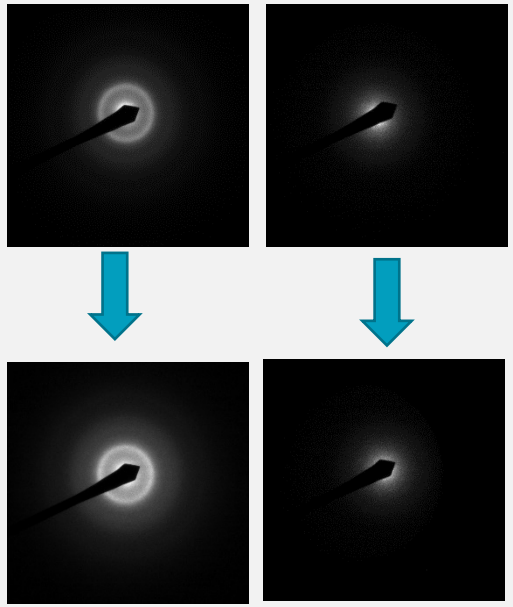
BF TEM



1000 C at 15 C/min

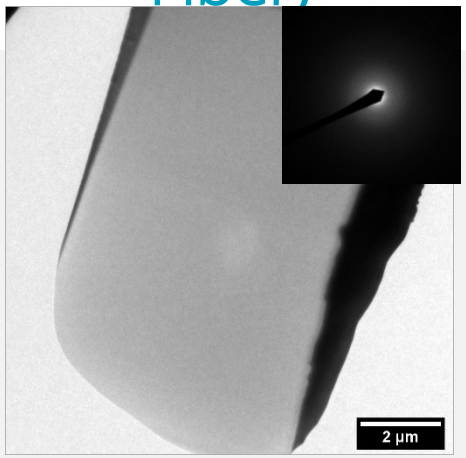


DP - Fiber DP - Phenolic

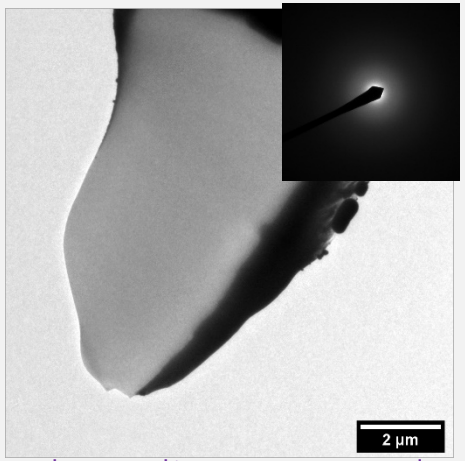


No significant changes

Phenolic (No Fiber)



1000 C at 15 C/min



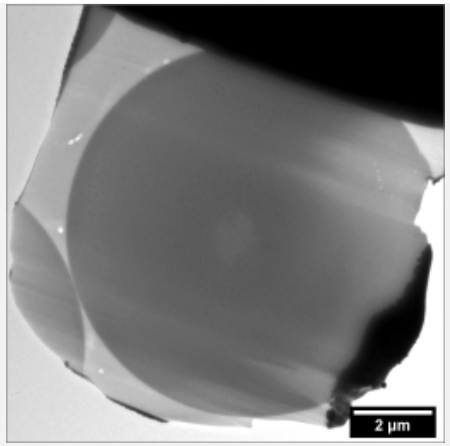
Phenolic appeared to shrink

Both fiber sample appear to withstand temperatures up to 1000 C with slow heating rate and the fibers appear to give structure to sample

Rapid Heating with Laser Pulse (70%, 10 ms)

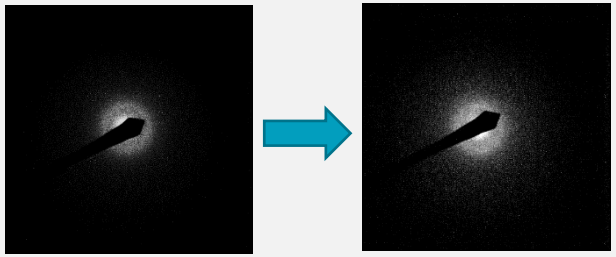
Silica Fiber Phenolic

BF TEM (Video)

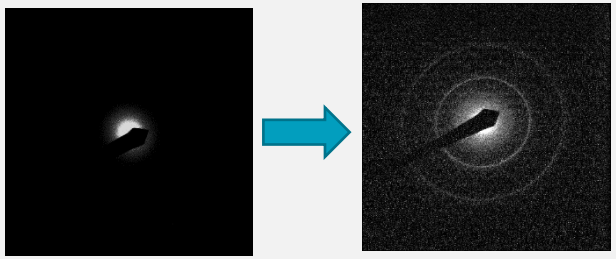


- Part of phenolic matrix was ablated due to quick laser pulse
- Fibers survived and parts of the phenolic around the fiber survived
- Diffraction shows more local crystallinity in phenolic after pulse

DP - Fiber

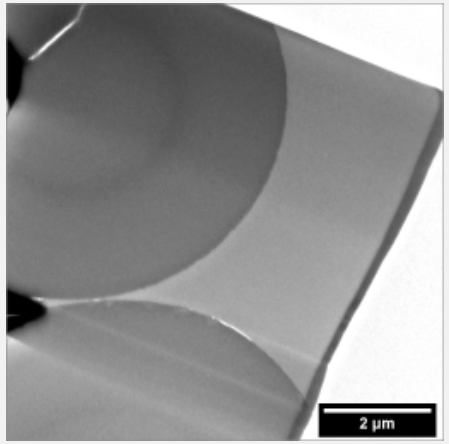


DP - Phenolic



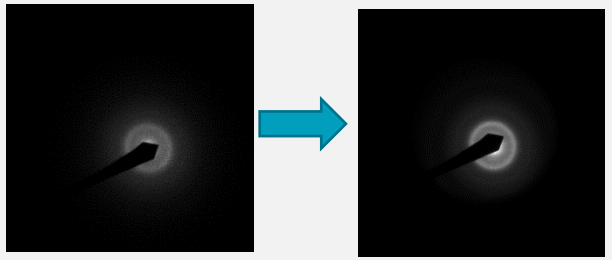
Carbon Fiber Phenolic

BF TEM (Video)

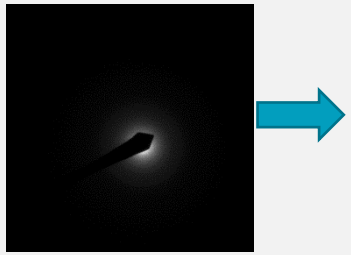


- All phenolic was completely ablated which led to fiber dropping
- No change in diffraction pattern of fiber

DP - Fiber



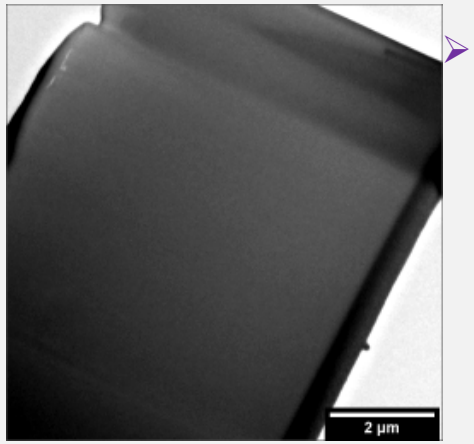
DP - Phenolic



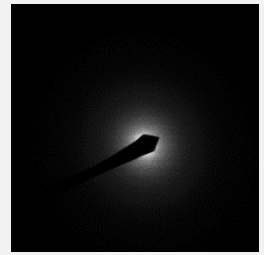
Phenolic containing part was destroyed by laser

Phenolic (No Fiber)

BF TEM (Video)



- All phenolic was completely ablated



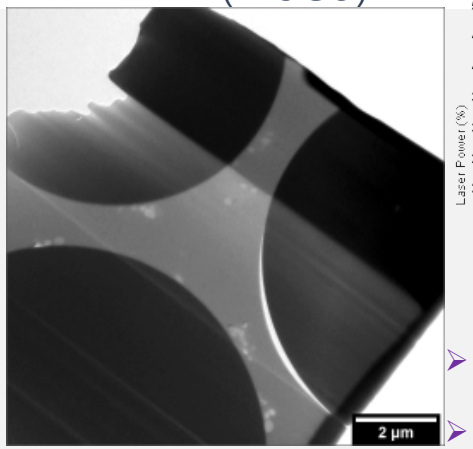
Phenolic was destroyed by laser

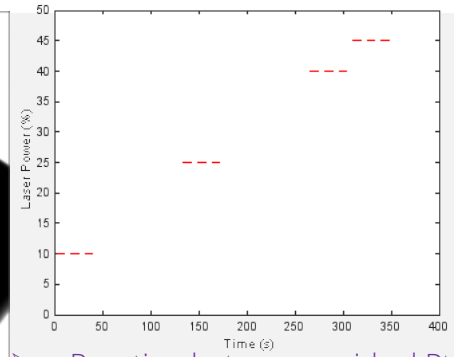
Only Silica Fiber sample contained some phenolic left (Need more analysis into Laser i.e. Percent Absorbed)

Slow Temperature Ramp with Laser

Silica Fiber Phenolic

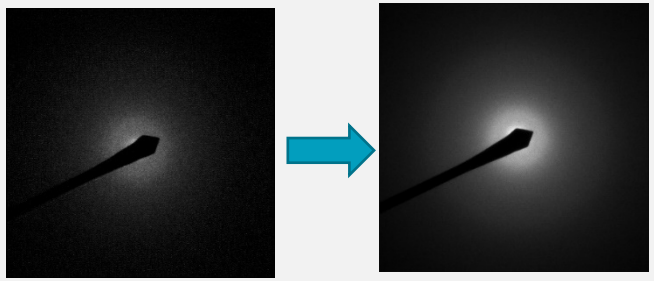
BF TEM (Video)



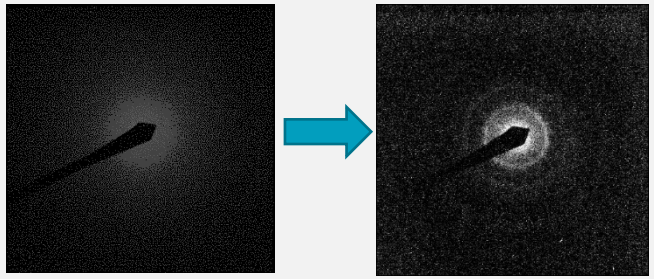


- Reaction between residual Pt and Sample around 20%
- Delamination around 40%
- Complete Sample Failure around 45%
- Local crystallinity in Phenolic

DP - Fiber

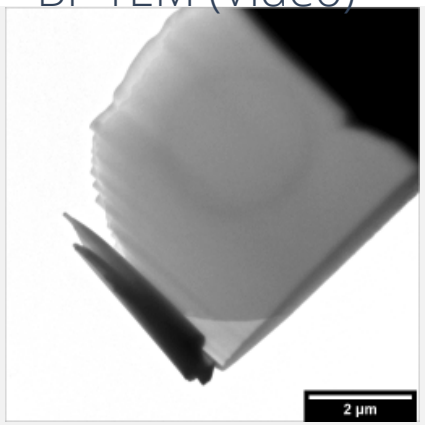


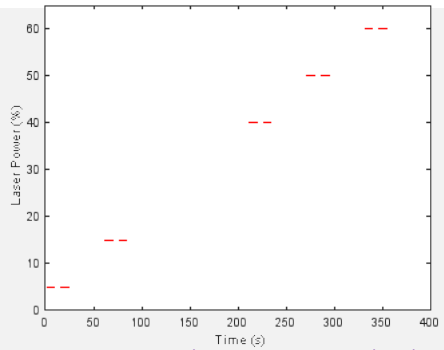
DP - Phenolic



Carbon Fiber Phenolic

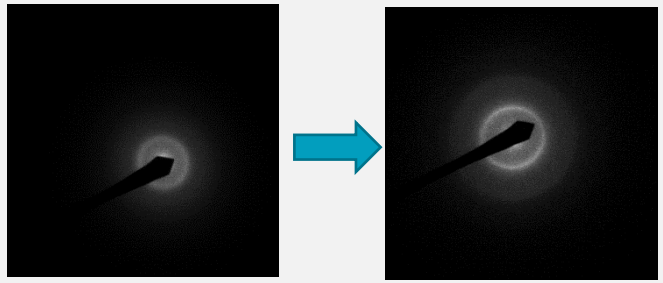
BF TEM (Video)



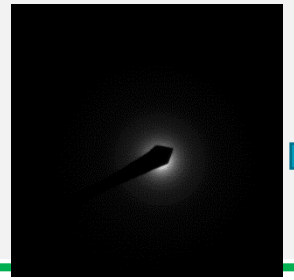


- Reaction between residual Pt and Sample around 25%
- Complete Sample Failure around 55-60%
- No significant changes to crystallinity of fiber

DP - Fiber



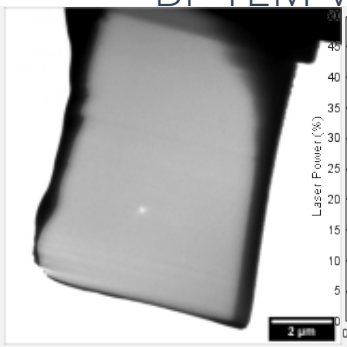
DP - Phenolic

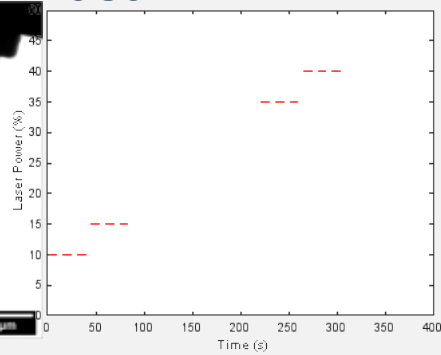


Phenolic containing part was destroyed by laser

Phenolic (No Fiber)

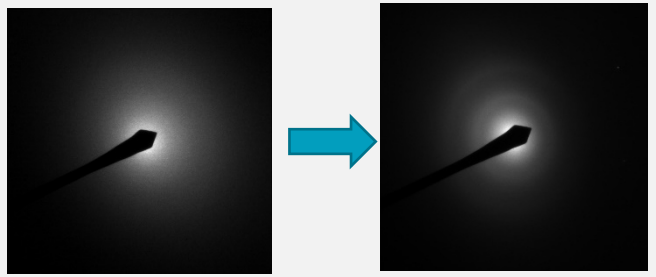
BF TEM Video





- Sample completely failed around 45% Power
- At 35%, sample does the development of some faint rings pointing to localized crystallinity but still mostly amorphous
- Failed around the same power as the silica fiber sample

At 35% Power



Carbon based phenolic survived higher laser power (Need to consider laser absorption)

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Conclusions

Slow Heating

- Both fiber samples were stable up to 1000 C
- Fibers appear to provide structure to phenolic up to 1000 C

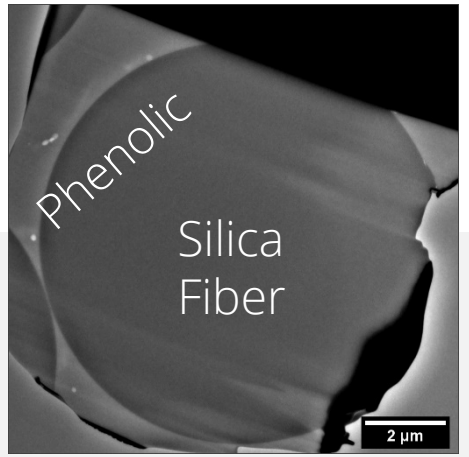
Laser Pulse

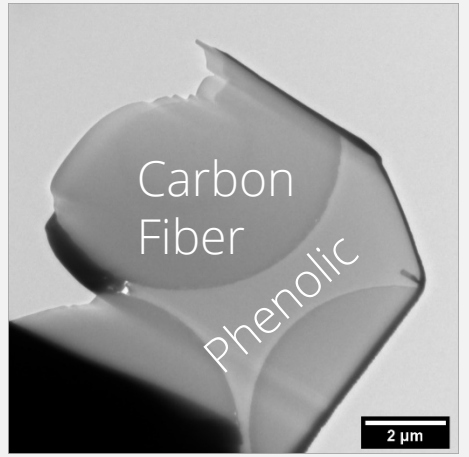
- Silica fiber sample survived laser pulse compared to carbon fiber sample and phenolic
  - Need an idea of how much of the laser was absorbed

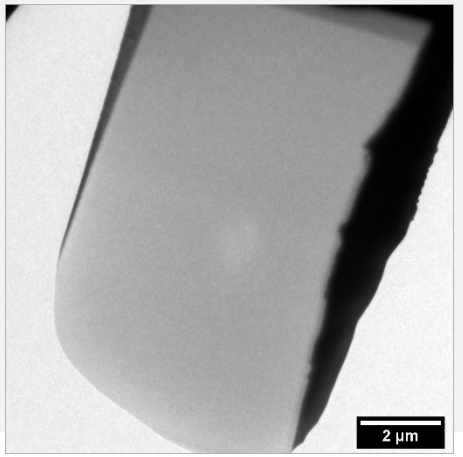
Slow Laser

- Carbon fiber sample was able to withstand highest laser power compared to silica fiber sample and phenolic

Able to show ability to perform small scale testing





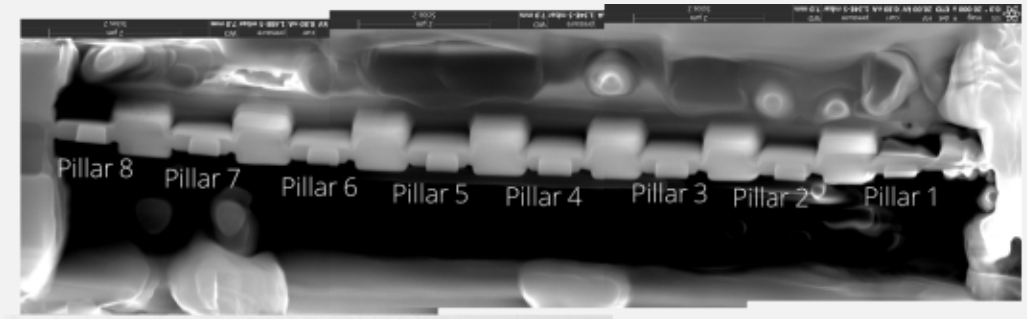


Future Work

Mechanical Testing

[PI 95 Pico-Indenter]

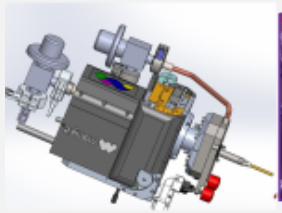
- Used to perform mechanical testing
- Can track load vs. Displacement which can be converted to stress vs strain

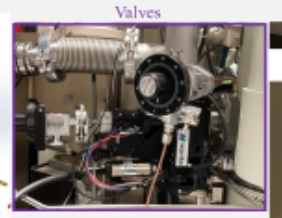


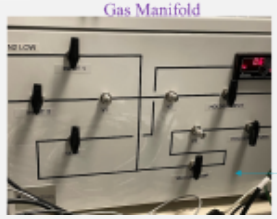
Gas Injection Testing

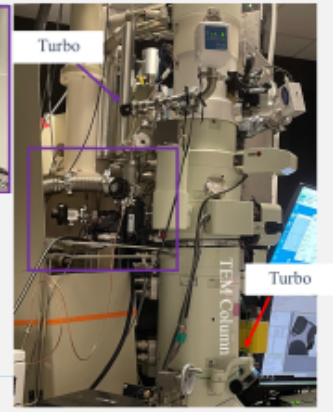
[Gas Injection System (WAVIKS)]

- Can inject gases (Air, H_2, O_2, N_2, etc) into sample area
- Compare with results in better vacuum

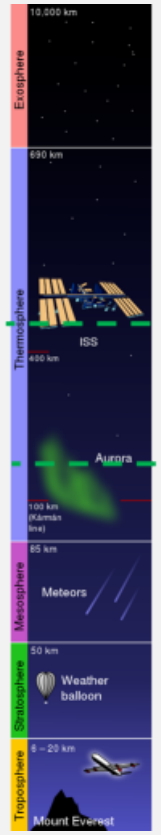








I³TEM Vacuum Limit (~450 km, $2x10^{-5}$ Pa)



I³TEM Vacuum Limit (~210 km, $1x10^{-3}$ Pa)