Post-Crash Fire Forensic Analysis on Aerospace Composites

Federal Aviation Administration

Joint Centers of Excellence for Advanced Materials

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JAMS Technical Review

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Introduction

- Post-Crash Fire Forensic Analysis on Aerospace Composites
- Project Participants:

  Principal Investigators: *Matthew W. Priddy*, *Thomas E. Lacy Jr.*, *Santanu Kundu*, *Charles U. Pittman Jr.*, *Jaime Grunlan*

  Postdoctoral Researcher: *Thomas Kolibaba*

  Graduate Students: *Abhijith Madabhushi (PhD), Aniket Mote (PhD), Dounia Boushab (PhD)*, *Hasnaa Ouidadi (MS)*

  Undergraduate Students: *Dalton Lovitt, Keri Sullivan, Hagan Dalton*

- FAA Technical Monitor: *Dave Stanley*
- Industry Partnerships/Other Collaborations: *NIAR, Aurora Flight Sciences*
- Source of matching contribution for the current award: *Aurora Flight Sciences, MSU, and TAMU*

Background

Motivation and Key Issues

- In-flight aircraft fires may result in severe degradations in composite material performance and in overall flight safety
- Non-fire related aircraft crashes can result in major post-crash fires on the ground
- Char formation due to post-crash fires can mask relevant aspects of the structural damage morphology necessary to identify the underlying failure mechanisms

[1]

[2]

[3]

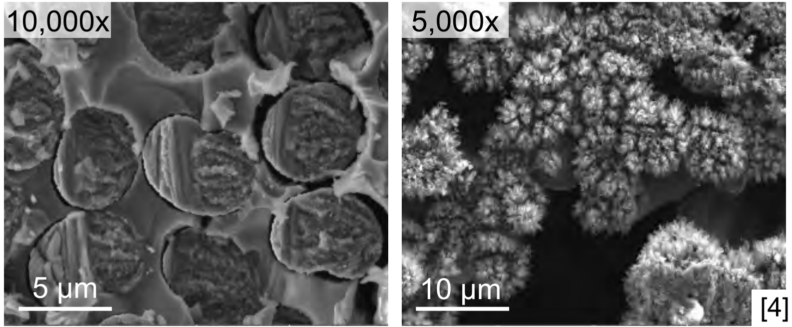
Objective and Scope

1. Develop method(s) for removing char from fire-damaged surfaces of carbon fiber reinforced epoxy composites
2. Assess the viability of these methods for determining root cause of composite mechanical failure after post-crash fire damage

Approach

- Examination of the fracture surfaces
- Fire application
- Post-fire examination
- Char removal application

UNC0 Before burn test | UNC0 After V 36 s burn test



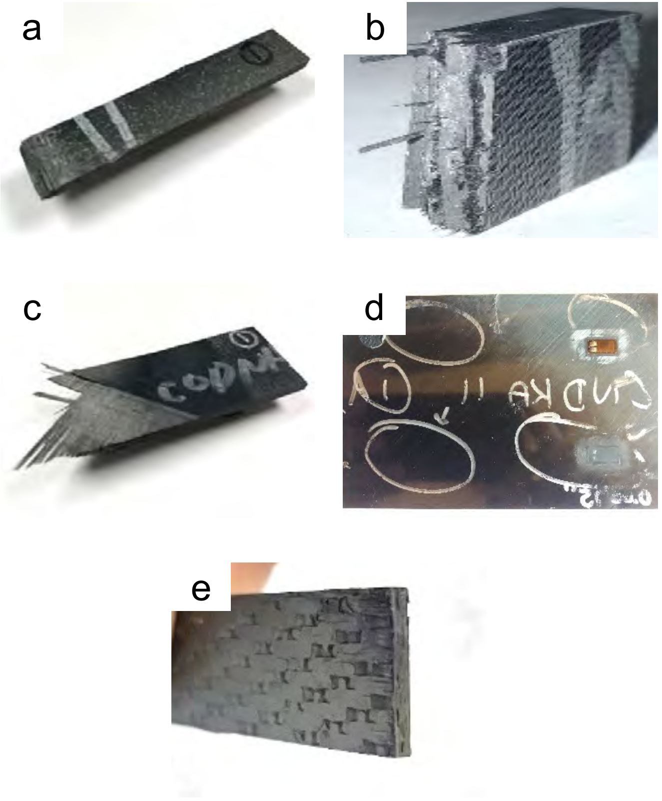
[4]

Material Systems: Coupon Level Specimens

- Cytec T40-800 Cycom 5215 graphite/epoxy composite
- HexForce™ SGP370-8H/HexPly® 8552 woven-fabric carbon/epoxy

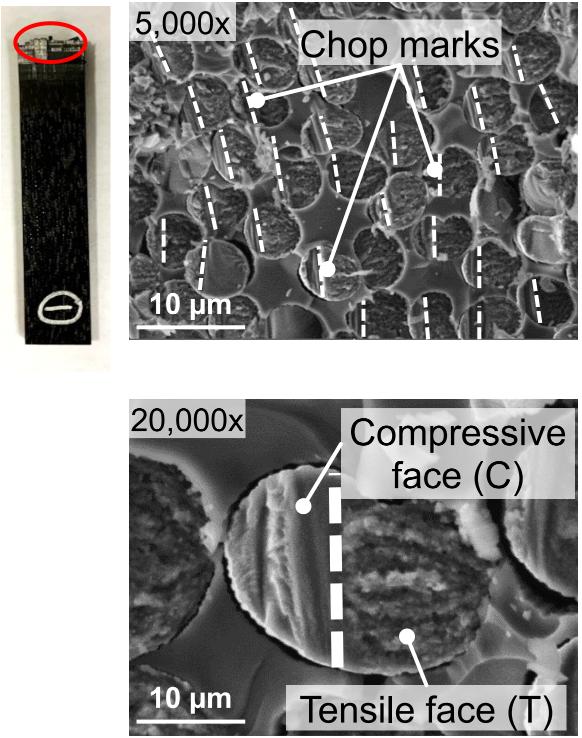
Specimen Type	Number of Plies	Layup
Cytec Unnotched Compression[a] (UNC0)	21	$[90/0/90]_7$
Cytec Short Beam Strength[b] (SBS)	45	$[0]_{45}$
Cytec In-Plane Shear[c] (IPS)	16	$[45/-45]_{4S}$
Cytec Compression After Impact[d] (CAI)	32	$[45/0/-45/90]_{4S}$
Hexcel Carbon/Epoxy[e] (Pristine*)	4	[0/90/90/0]

** Pristine: Specimens not subjected to mechanical testing*

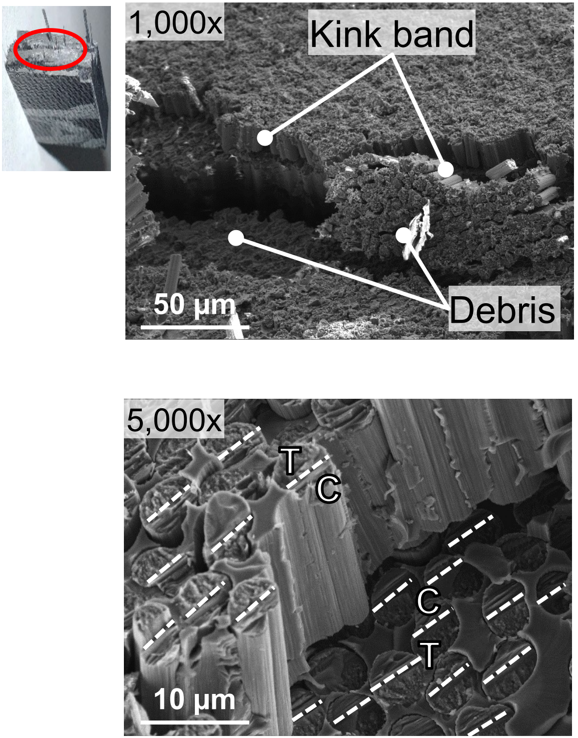


Pre-Fire Exposure Fractography of Mechanically Failed Cytec T40-800 Cycom 5215 Graphite/Epoxy Specimens

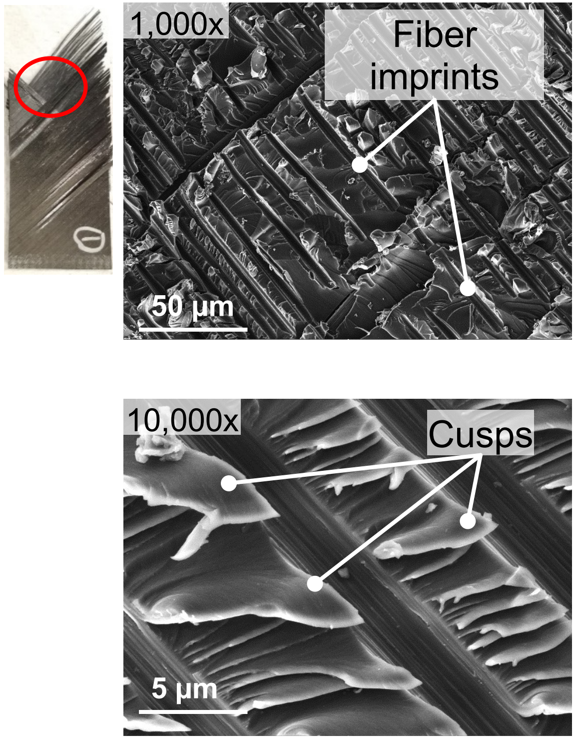
UNC0



SBS

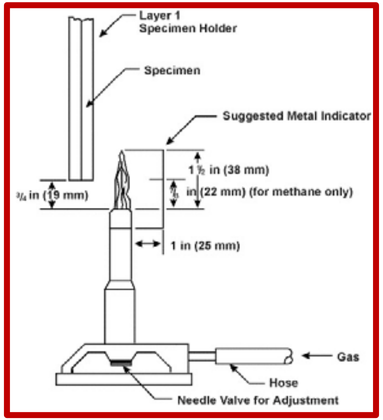


IPS



Fire Application Approach

- The FAA has defined vertical and horizontal Bunsen burner test protocols to address fire tests as specified in the Federal Aviation Regulation (FAR) 25.853 and FAR 25.855
- Draft-free cabinets that meet the FAA fire test requirements were used to conduct *vertical* and *horizontal* Bunsen burner tests on composite specimens



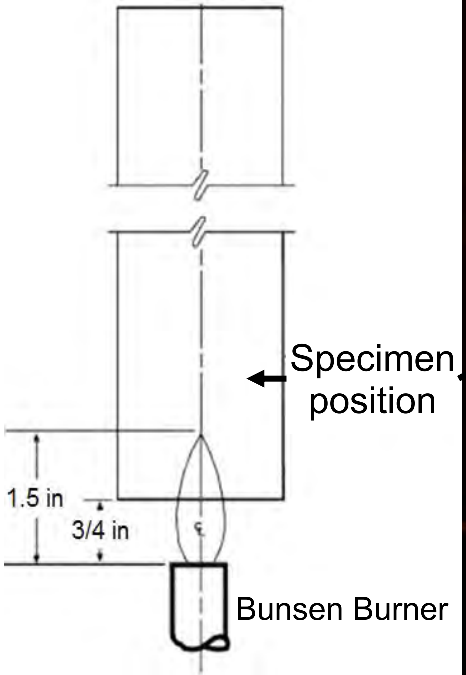
Schematic for Burner Plumbing and Burner Flame Height Indicator [5]

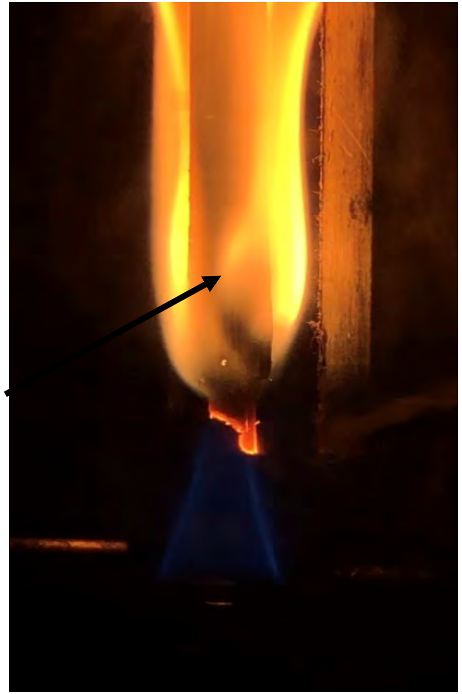
Draft-free ***Vertical*** *Bunsen* Burner Test Cabinet [4]

Draft-free ***Horizontal*** *Bunsen* Burner Test Cabinet [4]

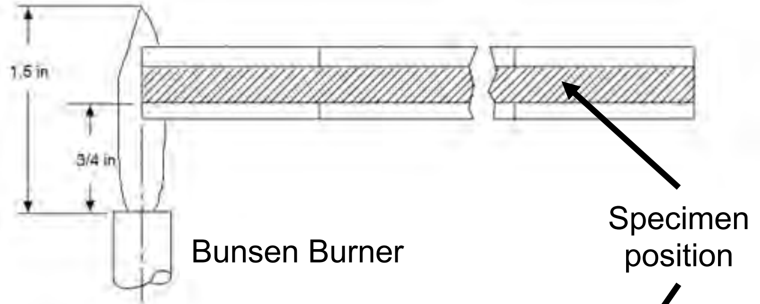
Fire Application: Specimen Orientation with Respect to Burner Flame

- Vertical Burn Test Setup





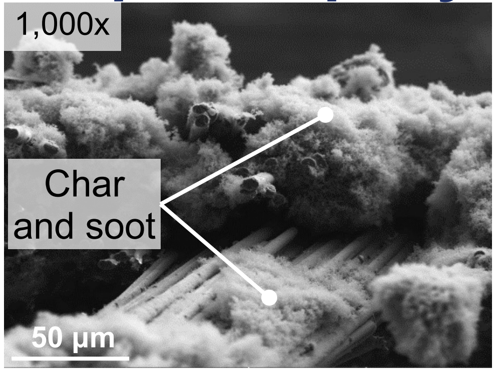
- Horizontal Burn Test Setup



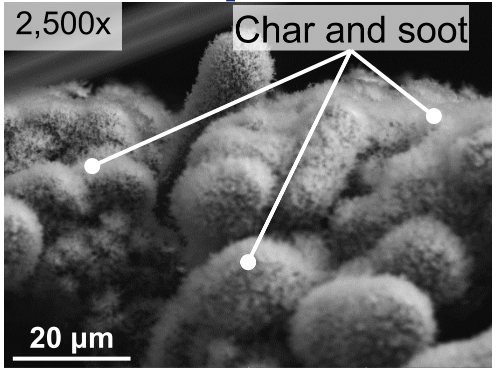
Fire Application: Test Matrix

Cytec Mechanically-Failed Specimens Subjected to Fire Testing				
Burning configuration	**Exposure time**	**UNC0**	**SBS**	**IPS**
Vertical Burning	6 s	N/A	N/A	3
	12 s	3	3	3
	36 s	3	3	3
	60 s	3	3	3
Horizontal Burning	15 s	3	3	3
	45 s	3	3	3
	75 s	3	3	3

Post-Fire Exposure Fractography of Graphite/Epoxy *UNC0* Specimen Fracture Surfaces



V-12 s fire exposure



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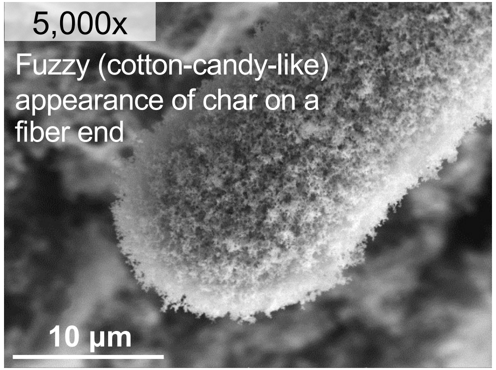

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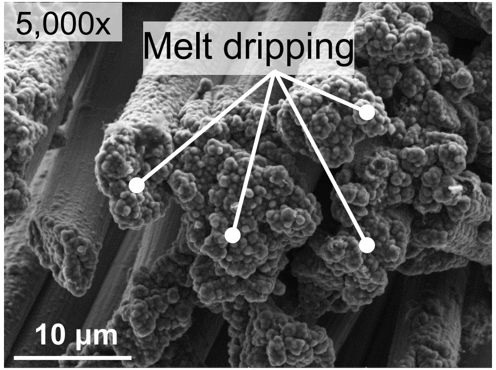
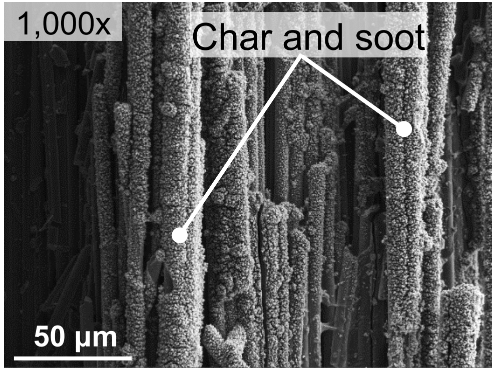
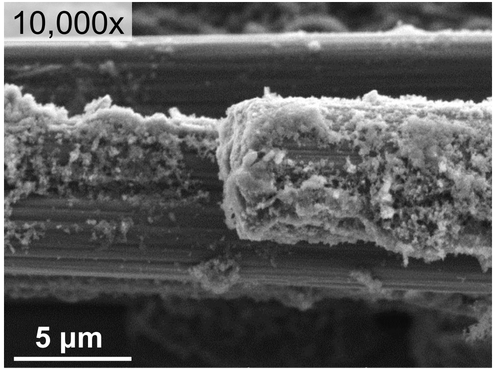


V-36 s fire exposure



V-60 s fire exposure

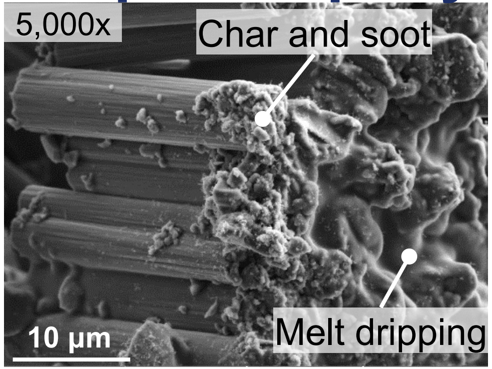


H-15 s fire exposure

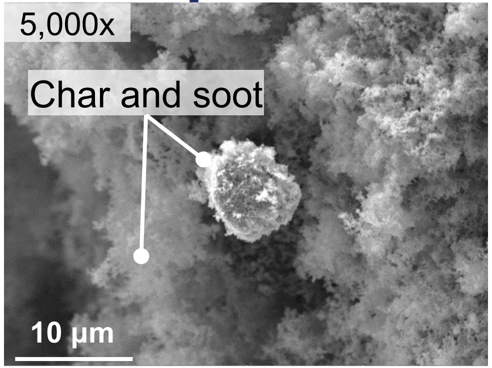
V: Vertical

H:Horizontal

Post-Fire Exposure Fractography of Graphite/Epoxy *SBS* Specimen Fracture Surfaces



V-12 s fire exposure



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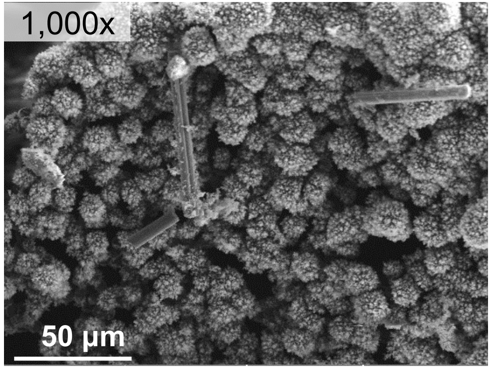

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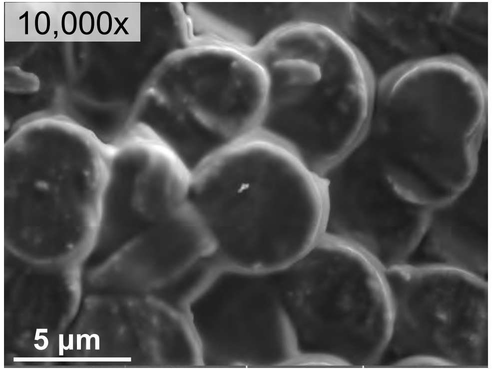

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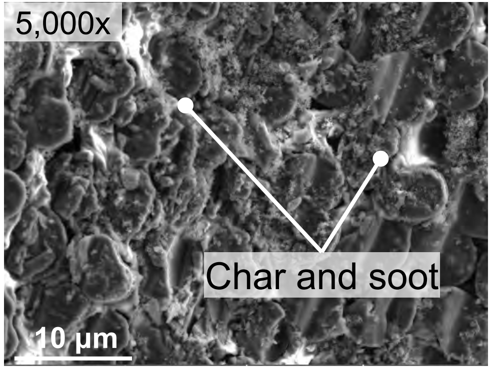
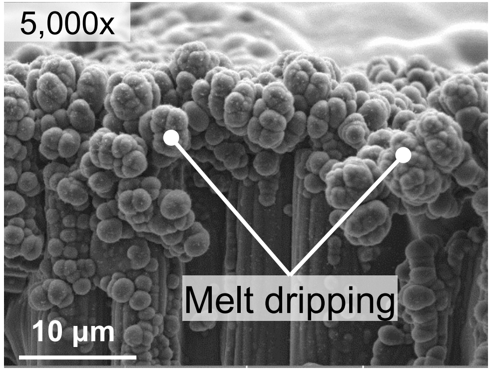
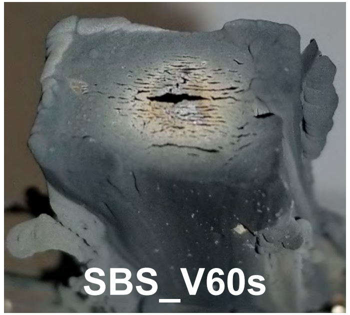


H-45 s fire exposure



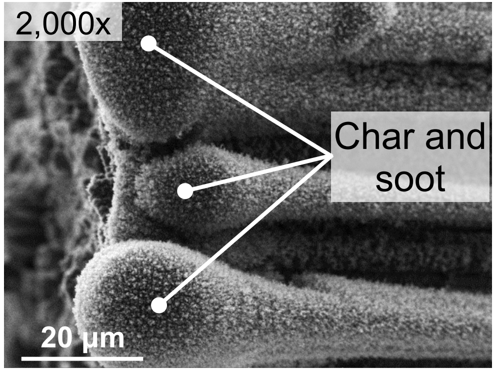
H-75 s fire exposure



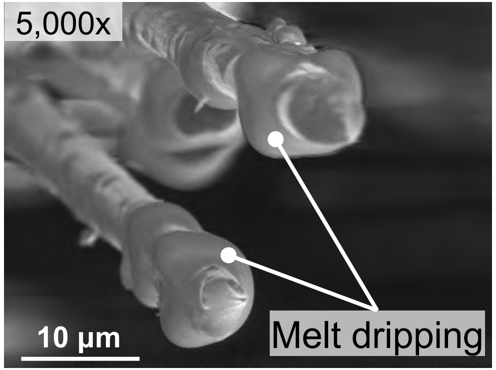
V: Vertical

H:Horizontal

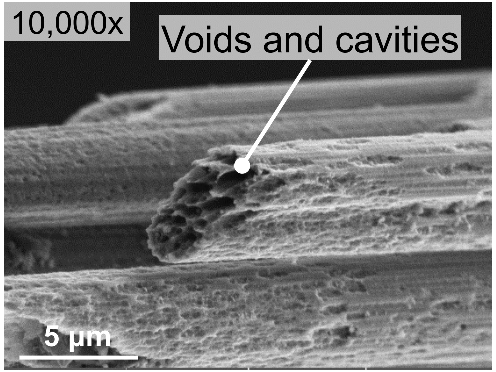
Post-Fire Exposure Fractography of Graphite/Epoxy *IPS* Specimen Fracture Surfaces



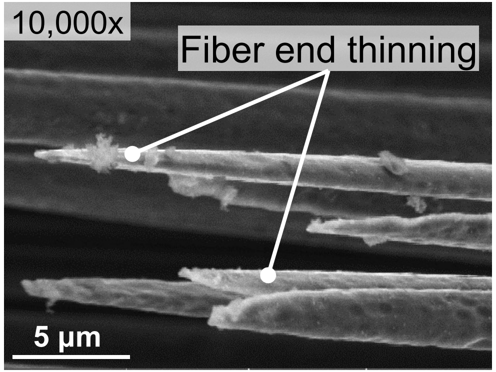
V-6 s fire exposure



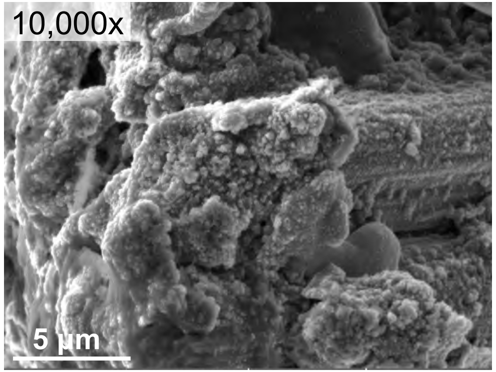
V-12 s fire exposure



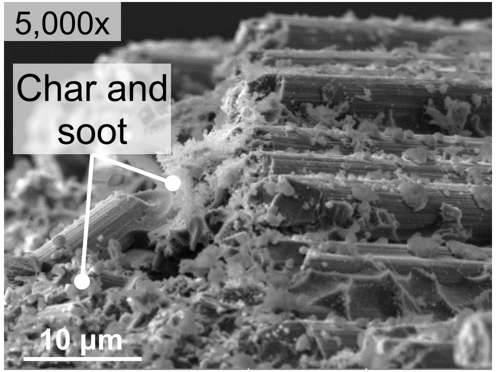
V-36 s fire exposure



V-60 s fire exposure



H-15 s fire exposure



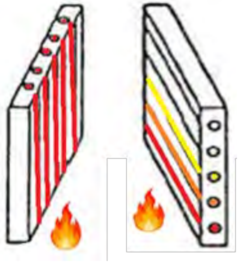
H-75 s fire exposure

V: Vertical

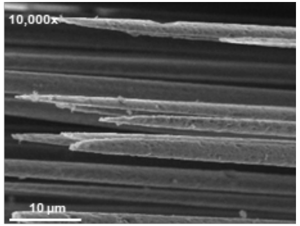
H:Horizontal

Key Observations

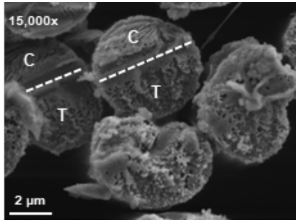
- Thermal damage was ***highly dependent on the ply orientation relative to the flame***:
  - Fibers oriented ***perpendicular*** to the heat-exposed surface *conducted heat into the interior of the composite*
  - Fibers oriented ***parallel*** to the heat-exposed surface acted like a ***thermal protection layer***



- Thermal damage by the presence of ***different ply groupings*** & the ***total available free surface area***:
  - ***More free surface area*** *results in* far ***greater thermal degradation*** for a given fire exposure
  - Exposed fiber bundles were susceptible to severe thinning and thermal oxidation which ***destroyed key fractographic features***



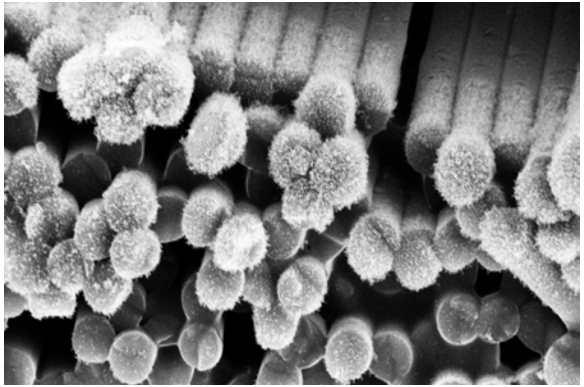
- Recessed fibers may be ***relatively unaffected*** by fire exposure, which may permit limited post-fire forensic analysis



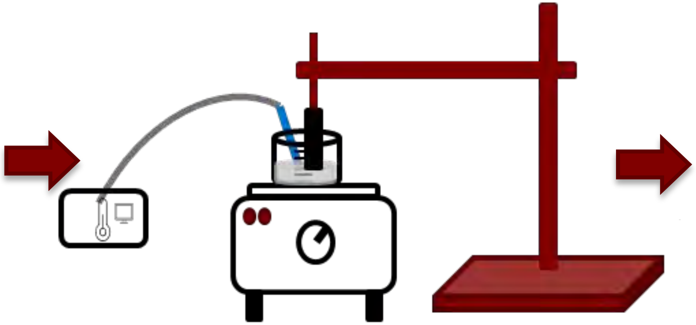
- The total number of plies also ***affects the degree of damage*** for a given fire exposure

Char Removal: Sulfuric/Nitric Acid Immersion

Burned carbon/epoxy surface

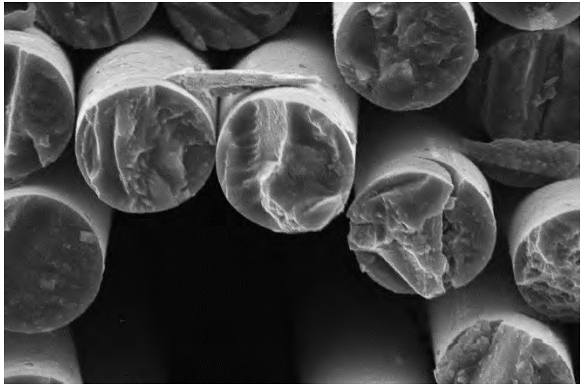


Acid immersion setup



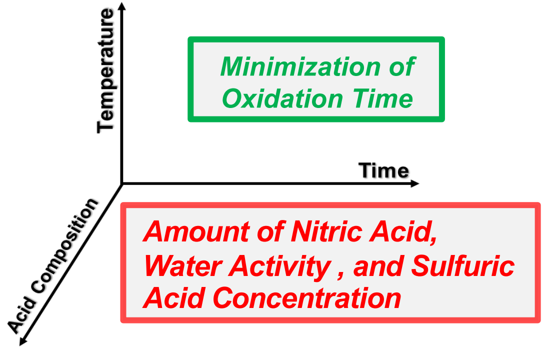
T = 100 - 250 °C, t = 5 - 60 min

Surface after acid immersion

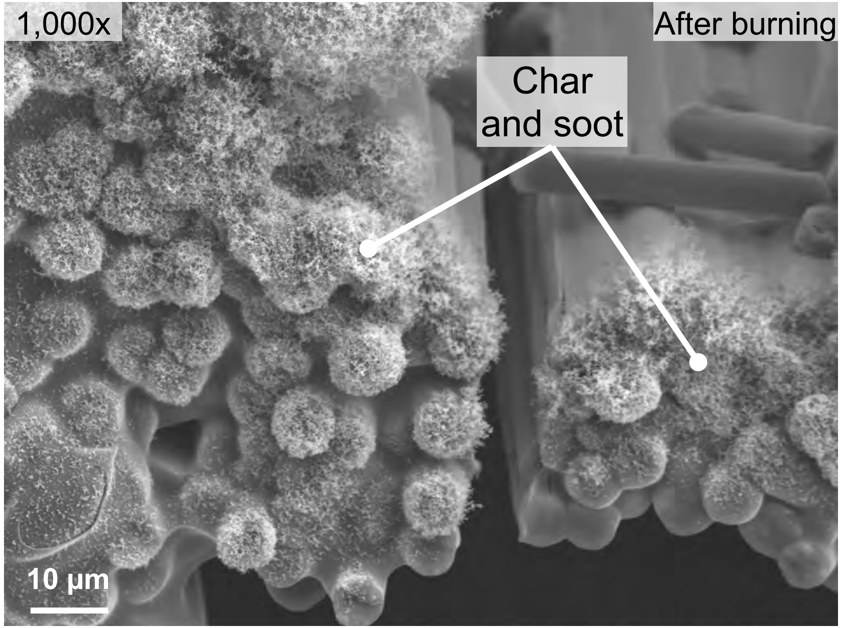


<u>**Acid compositions**</u>:

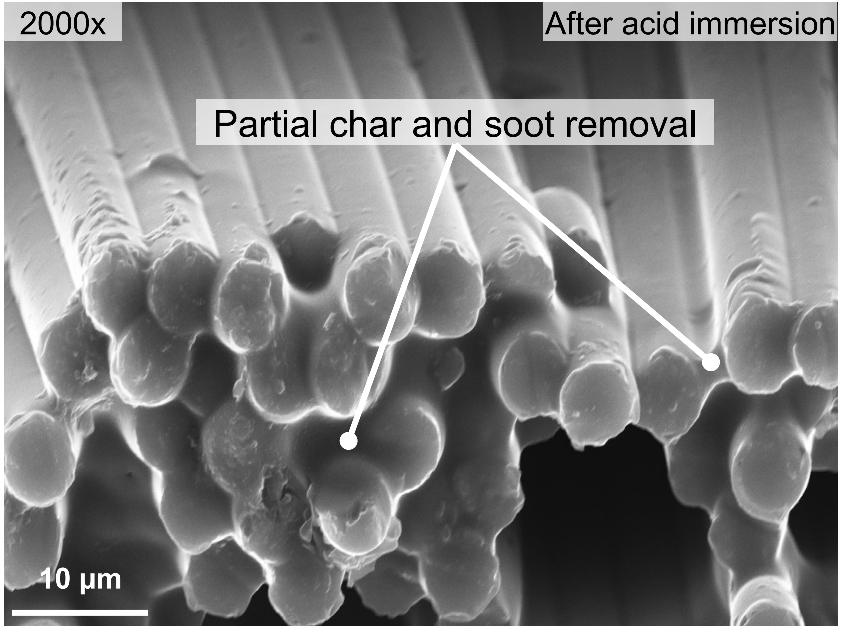
- conc.{96-98%} H_2SO_4 + 2 drops of 30%vol. HNO_3
- 95 wt.% conc.{96-98%} H_2SO_4 + 5 wt.% {45%} HNO_3
- 95 wt.% conc.{96-98%} H_2SO_4 + 5 wt.% {60%} HNO_3



Sulfuric/Nitric Acid Immersion: Pristine Burned Hexcel Carbon/Epoxy Specimen



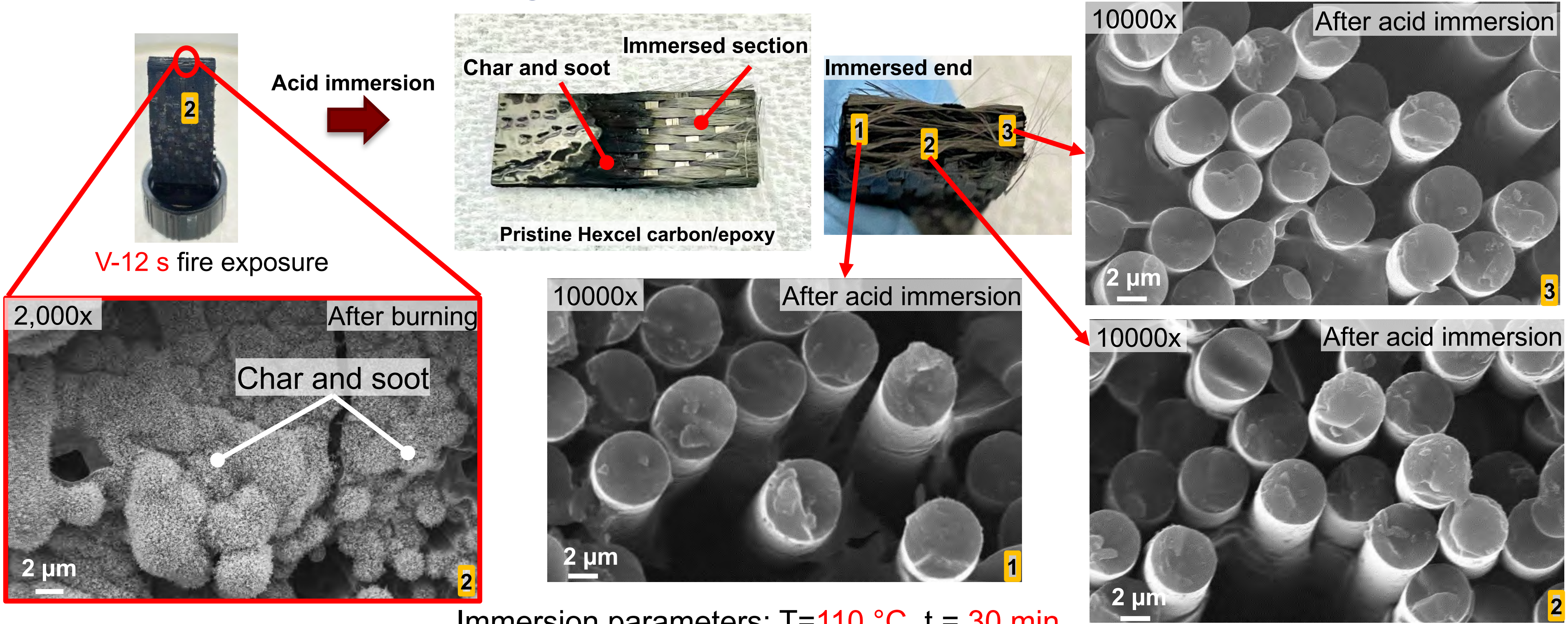
V-12 s fire exposure



Immersion parameters: T=75 °C, t = 60 min

***Acid volume:* 30 ml, *and acid composition:* conc.{96-98%} H_2SO_4 + 2 drops of 30%vol. HNO_3**

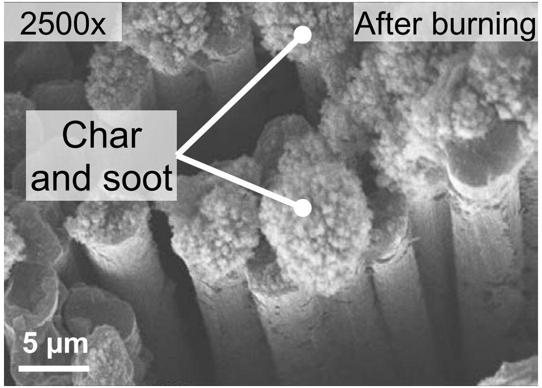
Sulfuric/Nitric Acid Immersion: Pristine Burned Hexcel Carbon/Epoxy Specimen Total Char Removal

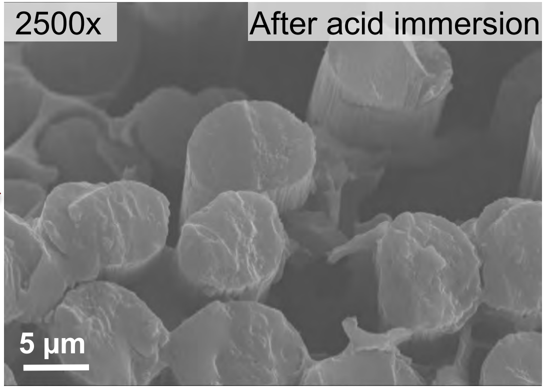


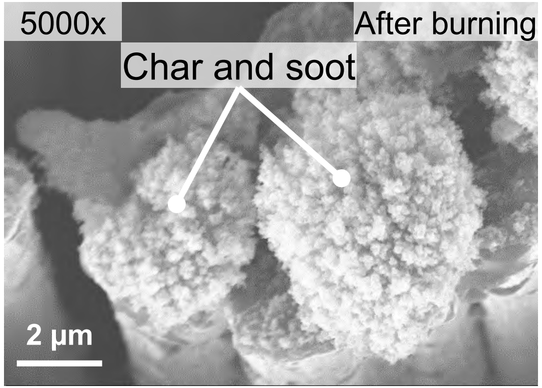
Immersion parameters: T=110 °C, t = 30 min

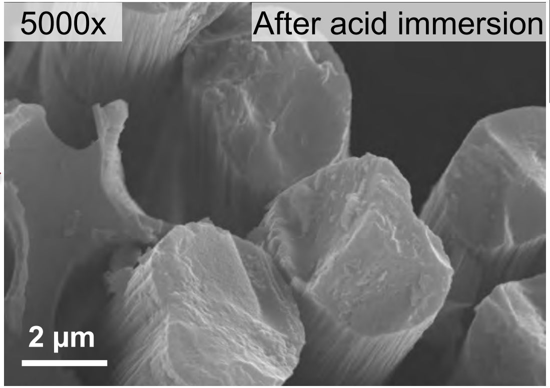
Acid volume: 30 ml, and acid composition: conc.{96-98%} H_2SO_4 + 2 drops of 30%vol. HNO_3

Burned Cytec Graphite/Epoxy *UNC0* Specimen Before and After Sulfuric/Nitric Acid Immersion







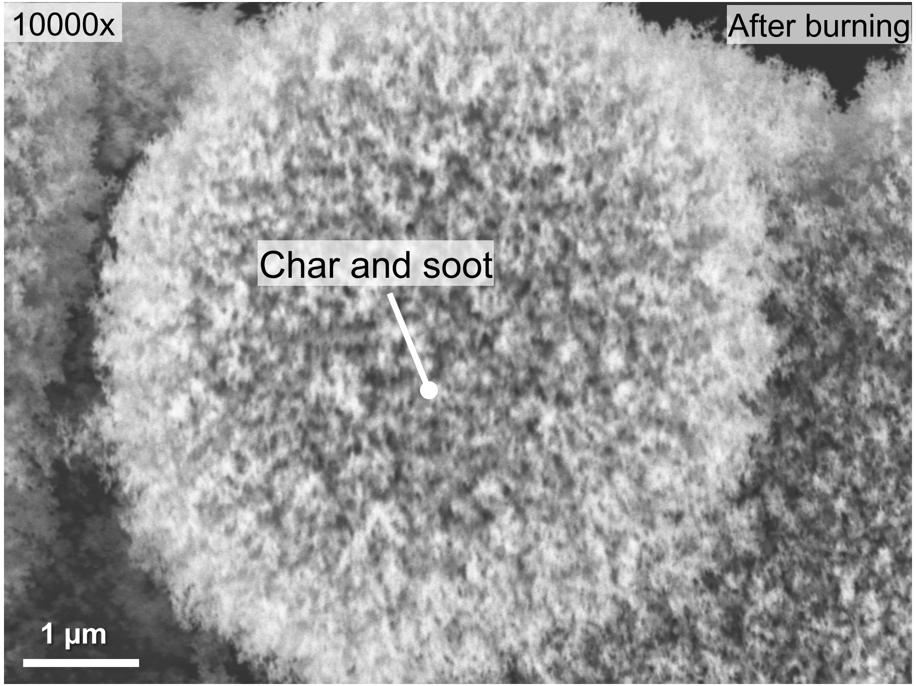


V-12 s fire exposure

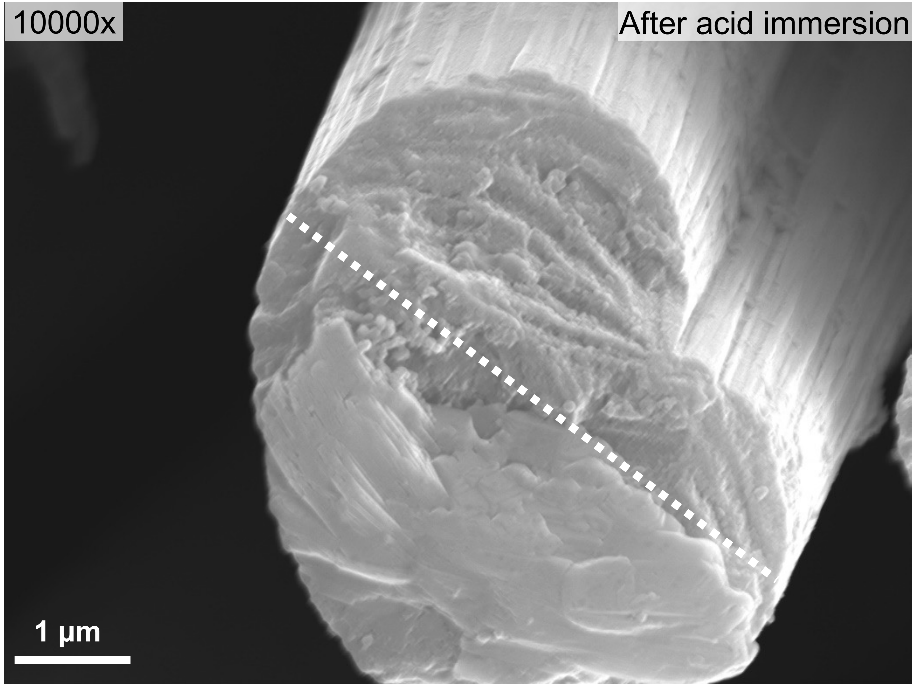
Immersion parameters:
T=200 °C, t = 30 min

Acid volume: 30 ml, and acid composition: conc.{96-98%} H_2SO_4 + 2 drops of 30%vol. HNO_3

Burned Cytec Graphite/Epoxy *UNC0* Specimen Before and After Sulfuric/Nitric Acid Immersion



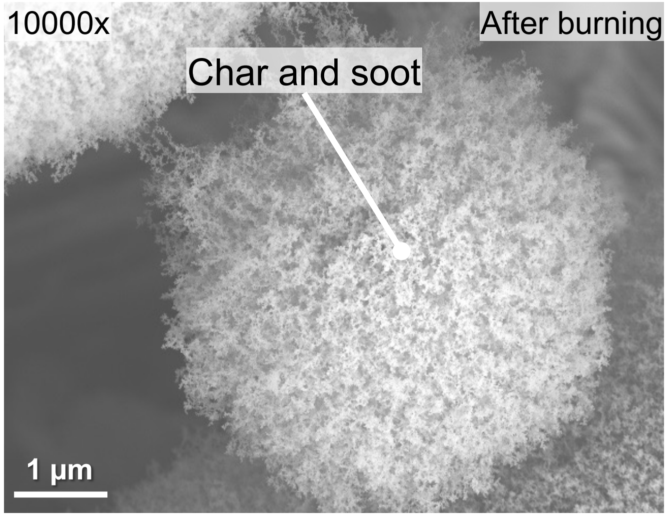
V-12 s fire exposure



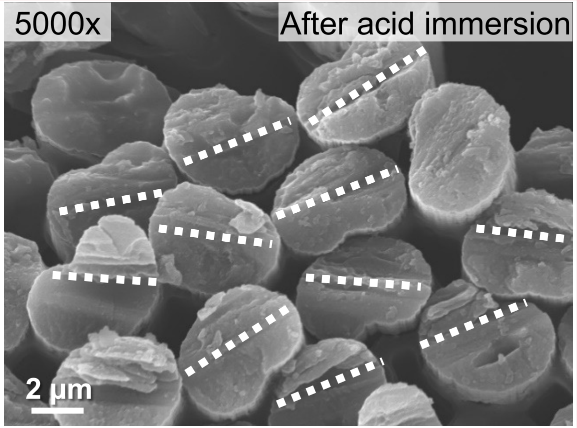
Immersion parameters: T=200 °C, t = 30 min

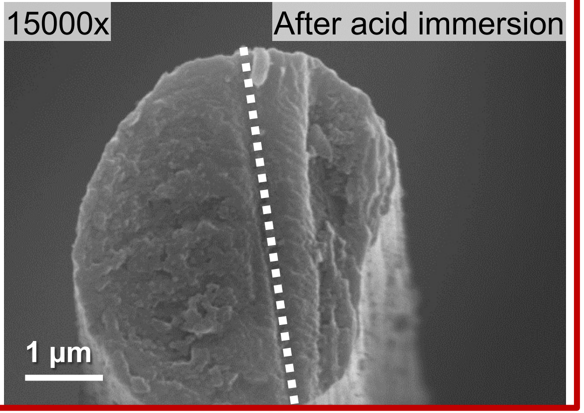
Acid volume: 30 ml, and acid composition: 30 ml of conc.{96-98%} H_2SO_4 + 2 drops of 30%vol. HNO_3

Burned Cytec Graphite/Epoxy UNC0 Specimen Before and After Acid Immersion- {45%} HNO_3



V-12 s fire exposure

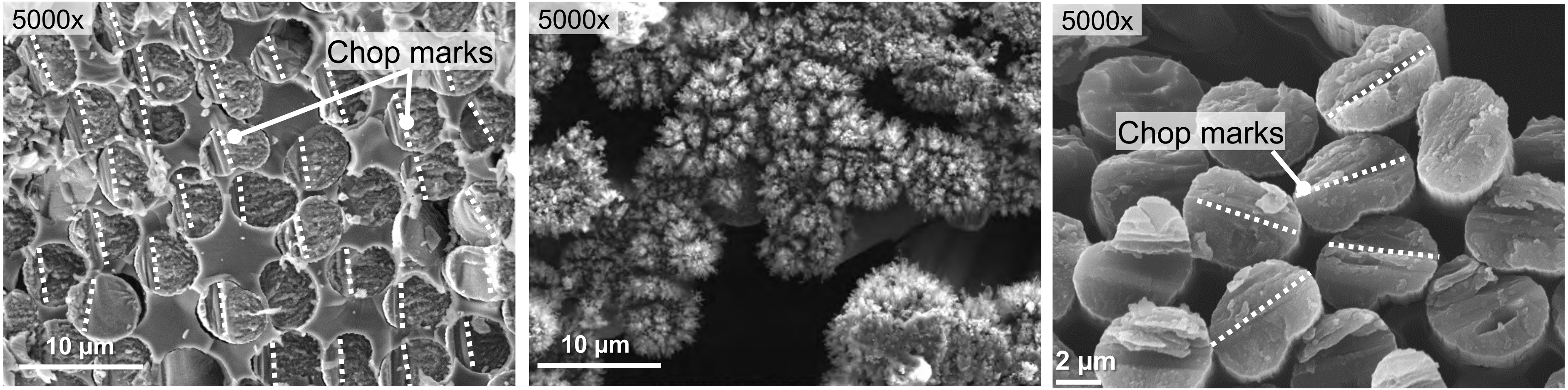




Immersion parameters: T=200 °C, t = 5 min

Acid volume: 30 ml, and acid composition: 95 wt.% conc.{96-98%} H_2SO_4 + 5 wt.% {45%} HNO_3

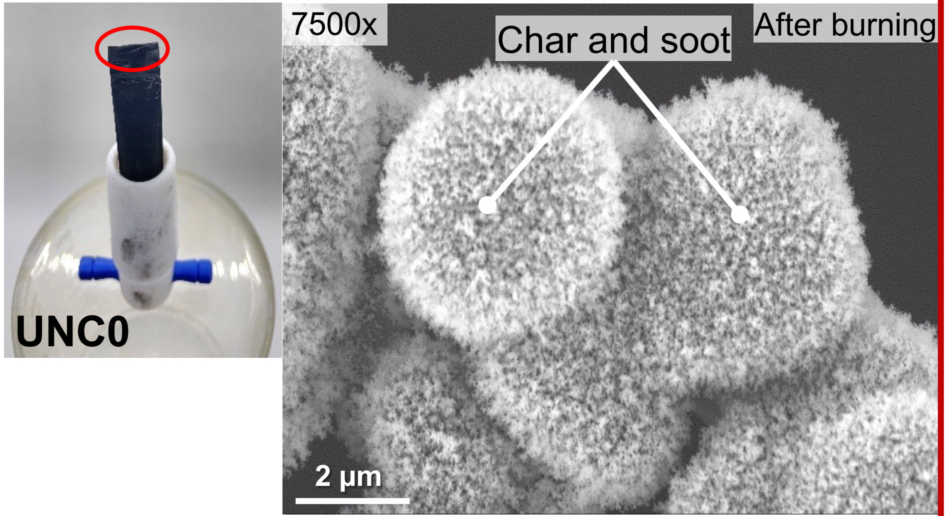
Surface Characterization of Cytec Graphite/Epoxy UNC0 Specimen: Before/After Burn Test and Char Removal



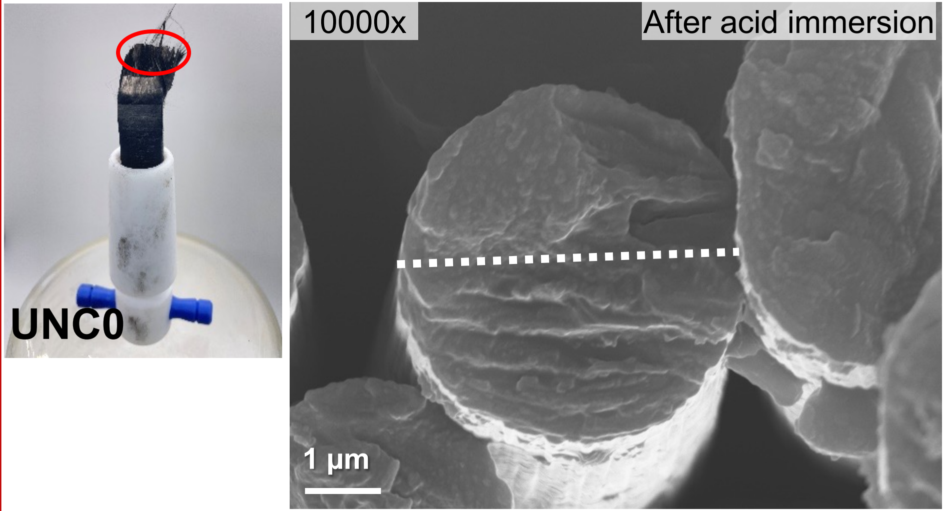
Before fire exposure | After V-12 s fire exposure | After Immersion T=200 °C, t = 5 min

Acid volume: 30 ml, and acid composition: 95 wt.% conc.{96-98%} H_2SO_4 + 5 wt.% {45%} HNO_3

Burned Cytec Graphite/Epoxy UNC0 Specimen Before and After Acid Immersion- {60%} HNO3



V-12 s fire exposure



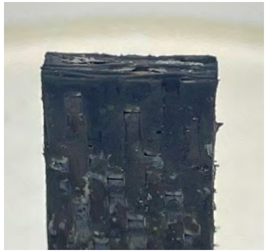
Immersion parameters:
T=150 °C, t = 5 min

Changing acid composition and amount of nitric acid allowed complete char removal after *5 min* immersion

acid composition: 95 wt.% conc.{96-98%} H_2SO_4 + 5 wt.% {60%} HNO_3

Key Observations

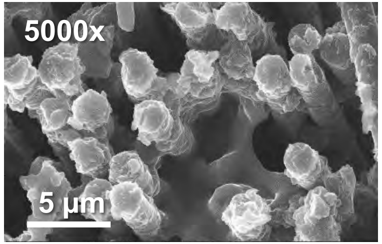
- Acid immersion experiments resulted in a near total removal of char, epoxy, melt-dripping, *etc.* across the immersed specimen surface ***in all specimens***



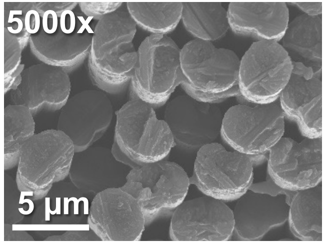
- *Nitric acid (HNO_3) concentration and quantity greatly influenced the removal of epoxy, char and other thermal by products from the immersed region*

- *Adjustment of HNO_3 content in the acid mixture and can allow for optimization of immersion times and temperatures*

- *Minimization of immersion temperatures and times is crucial for safety considerations*



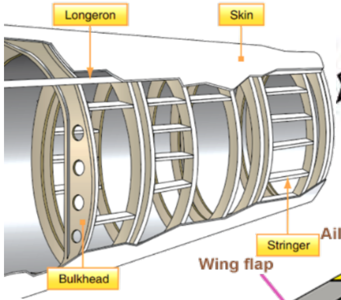
T=75 °C, t = 60 min

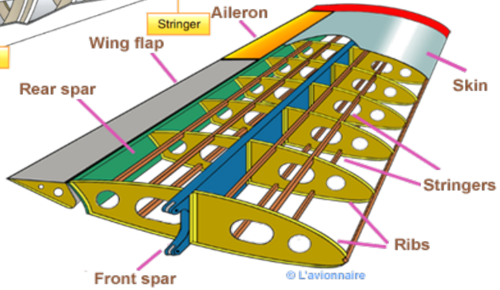


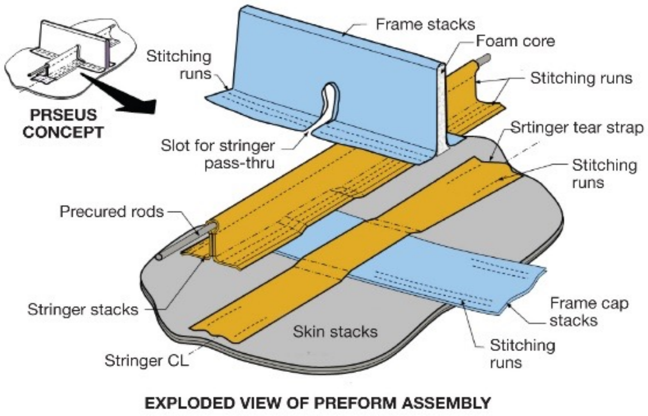
T=200 °C, t = 5 min

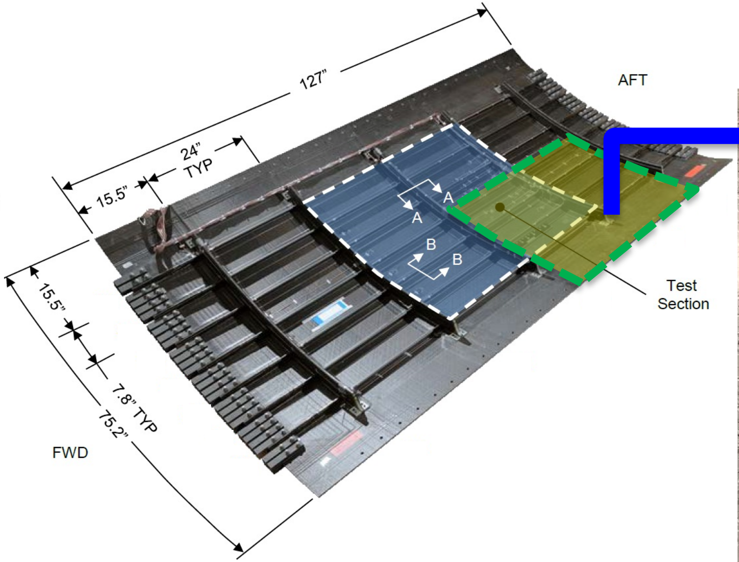
Principal Structural Elements (PSEs): Large-Scale Specimens

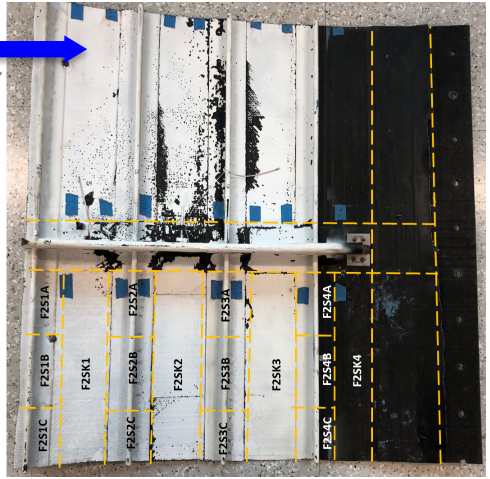
- Post-crash forensic analysis of composite aircraft structures typically focuses on PSEs
- Pultruded Rod Stitched Efficient Unitized Structure (PRSEUS) is made of warp-knit multiaxial AS4 carbon fiber fabric stacks, stitched together with Vectran stiches, and infused with Hexcel’s Hexflow VRM-34 epoxy-resin.
- A single warp-knit carbon fiber stack has a layup of [-45/+45/90/0/90/+45/-45]





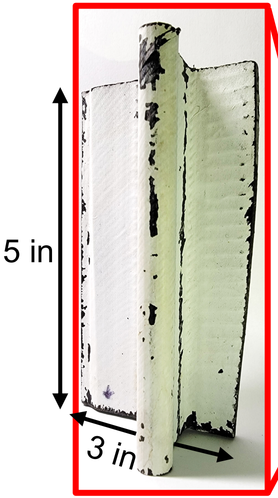


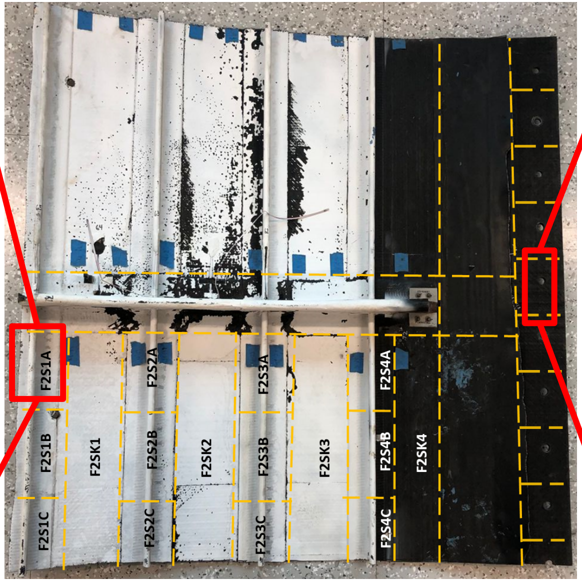




PRSEUS

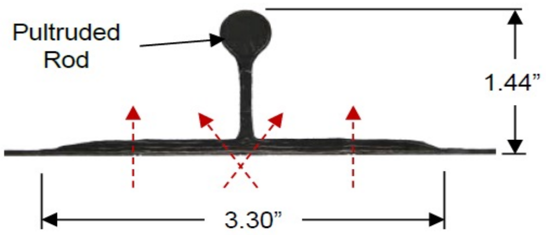
Stringer



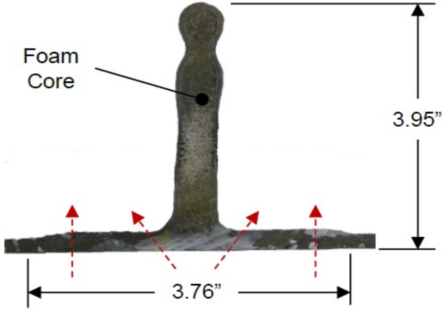


Open Hole

Stringer cross-section

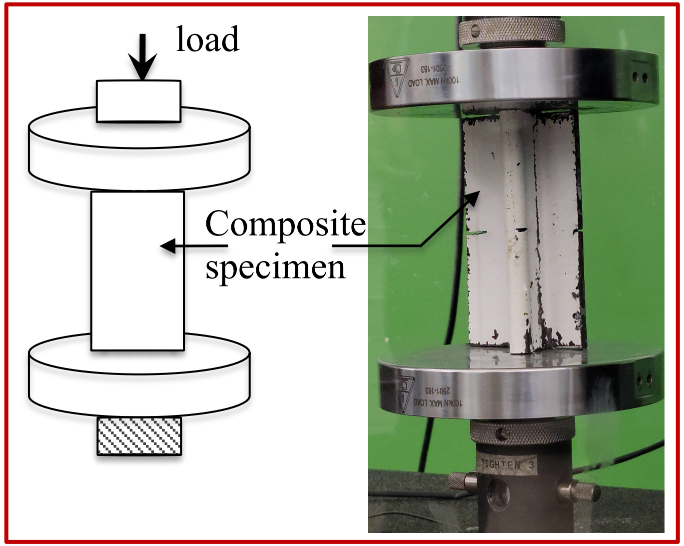


Frame cross-section



Flat Platen Compression Tests on PRSEUS Carbon-Epoxy Stringer Specimens

- Mechanical test setup



- Mechanically failed stringer elements

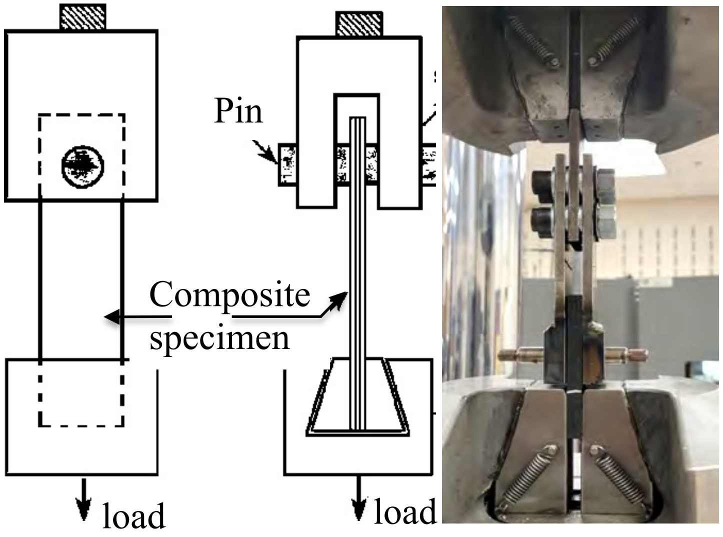
Stringer-1

Stringer-2

Stringer-3

Pin Load Tension Tests on PRSEUS Carbon-Epoxy Open Hole Specimens

- Mechanical test setup



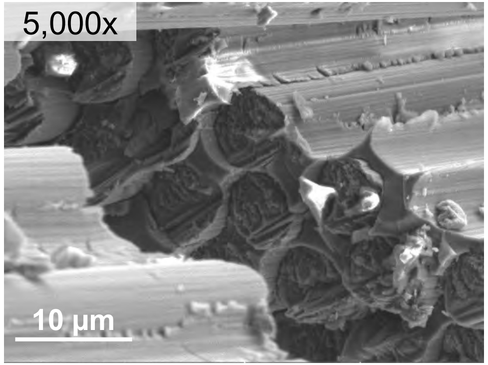
- Mechanically failed open hole specimens

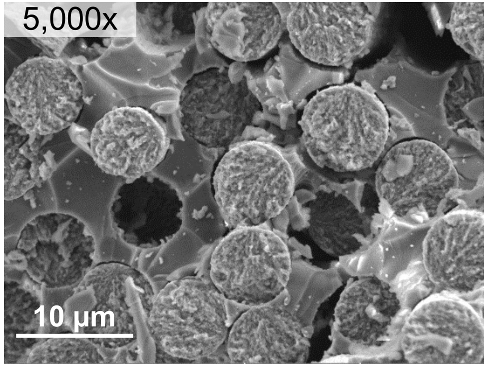
Open hole-1

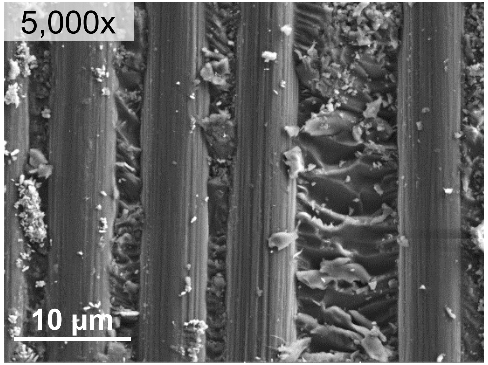
Open hole-2

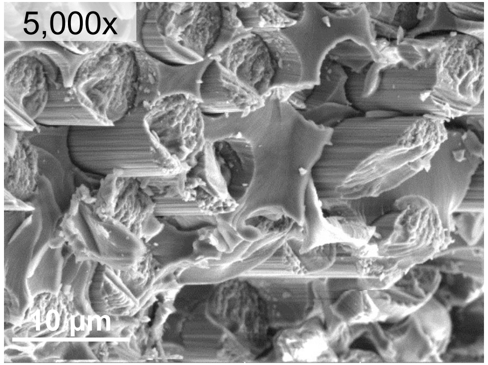
Open hole-3

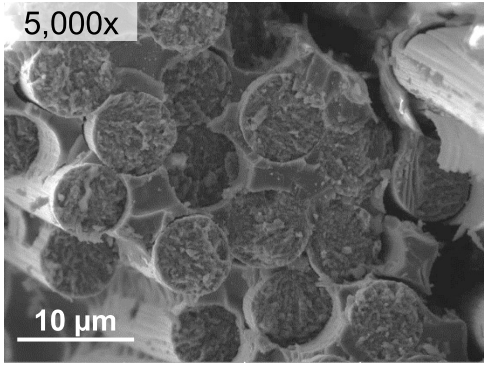
Pre-Fire Exposure Fractography of Mechanically Failed PRSEUS Carbon-Epoxy Open Hole Specimen

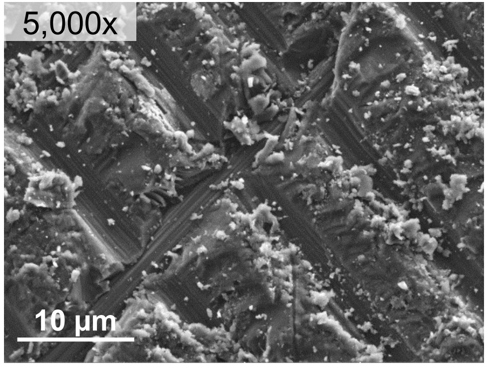




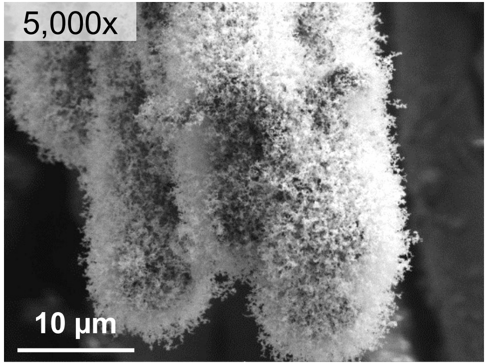


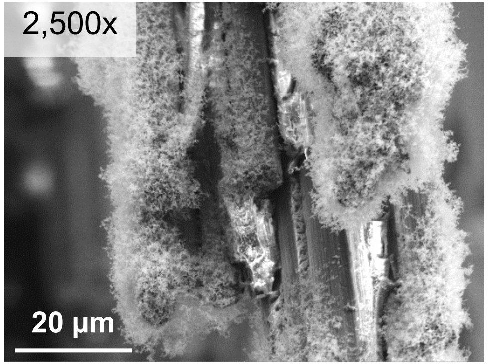


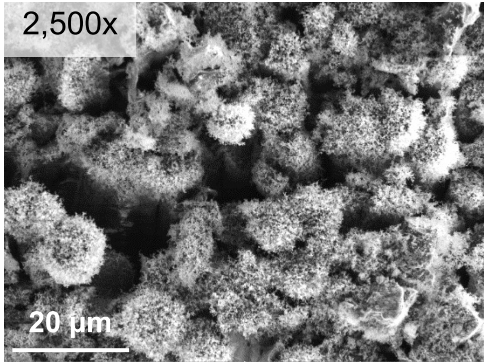


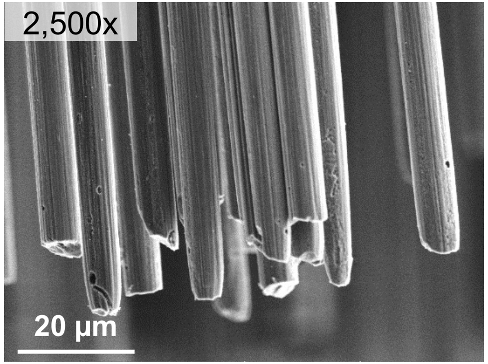


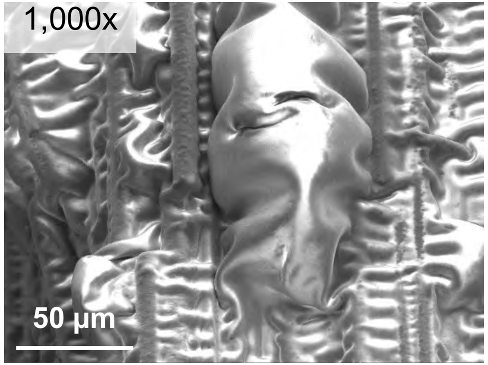
Post V-60s Fire Exposure Fractography of Mechanically Failed PRSEUS Carbon-Epoxy Open Hole Specimen

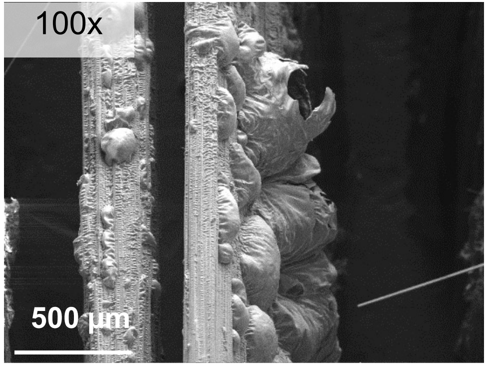






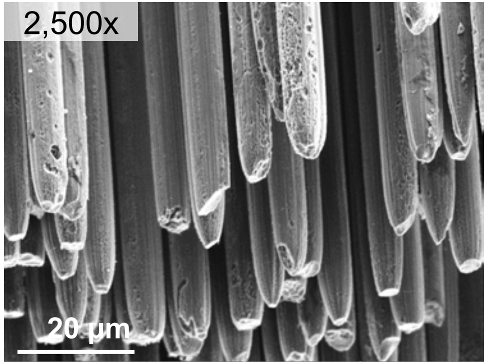


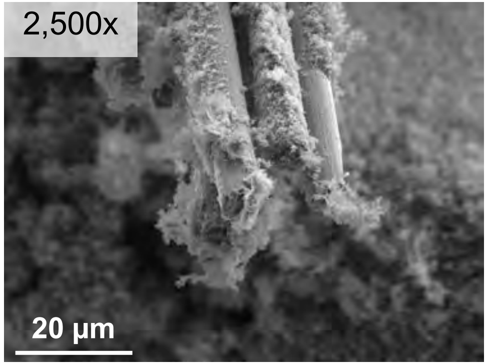


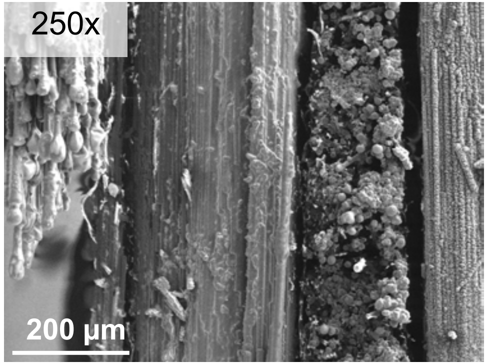


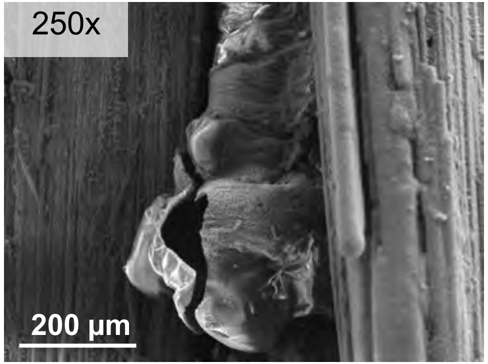
OH V-60s

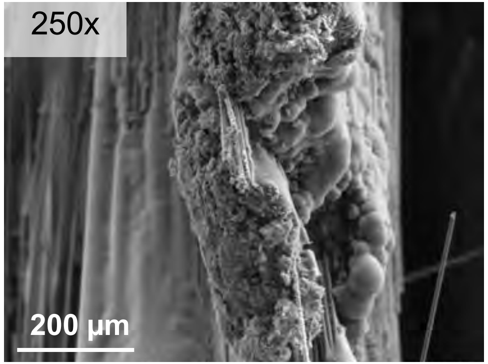
Post V-120s Fire Exposure Fractography of Mechanically Failed PRSEUS Carbon-Epoxy Open Hole specimen

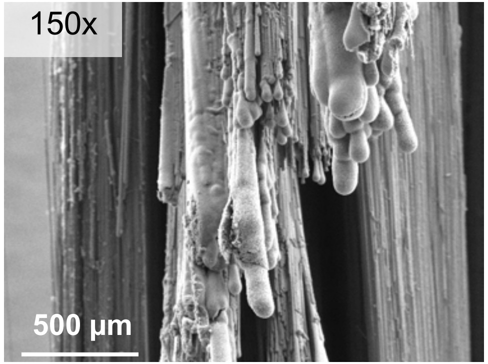






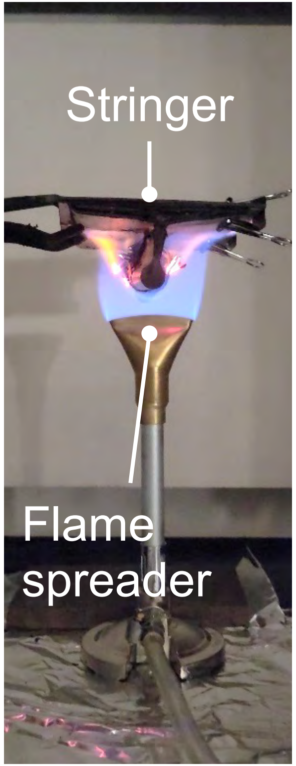






OH V-120s

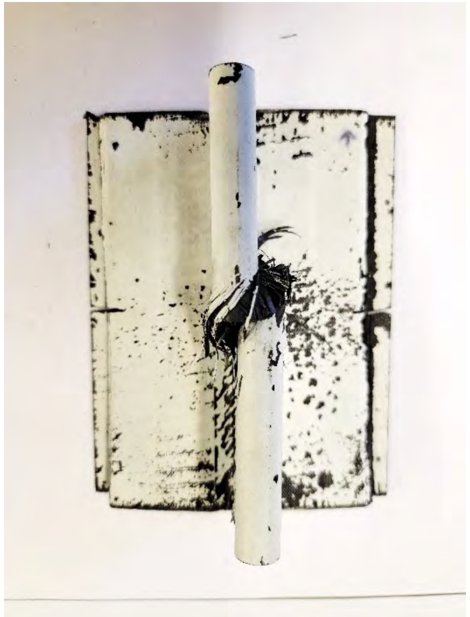
Fire Damage in a Mechanically-Failed PRSEUS Carbon-Epoxy Stringer Specimen Subjected to 60 s Burn Test



Before burn test

After 60 s burn test

Before burn test



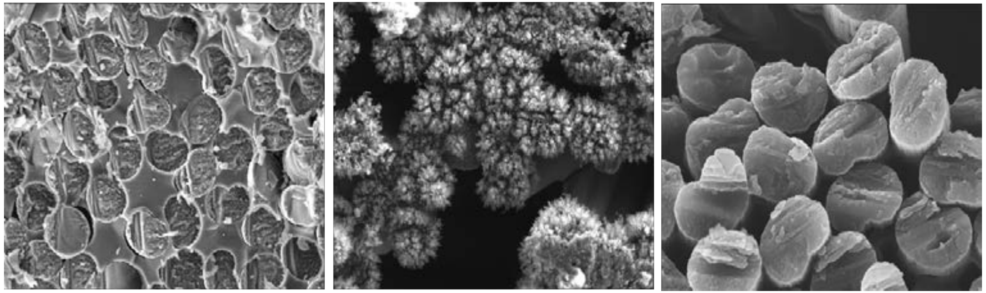
After 60 s burn test

Conclusions and Future Work

- Developed a protocol for repeatable char formation on coupon specimens
- Sulfuric/nitric acid immersion effectively removes char from pristine and mechanically failed coupon specimens

- Optimize acid concentration, exposure temperature, and exposure times as a function of specimen geometry and material system
- Study the extent of oxidation due to acid immersion on the exposed fibers
- Scale char removal techniques to *large-scale* aircraft structural members & PSEs

Before burning | After burning | After char removal



Technical Publications

- Priddy, M., Lacy T., Kundu, S., Pittman. C., Madabhushi, A., Righi, H., Ouidadi, H., Boushab, D., Mote A., 2021, “Post-Crash Fire Forensic Analysis of Aerospace Composites,” FAA Year 1 Final Report.
- Priddy, M., Lacy T., Kundu, S., Pittman. C., Madabhushi, A., Righi, H., Ouidadi, H., Boushab, D., Mote A., 2020, “Post-Crash Fire Forensic Analysis of Aerospace Composites,” FAA Literature Review Report.

Thank you!

Federal Aviation Administration

Joint Centers of Excellence for Advanced Materials

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[3] Charpentier. A., 2014, "The odds of a cluster of airplane accidents" Open Edition. Available in: https://freakonometrics.hypotheses.org/16093

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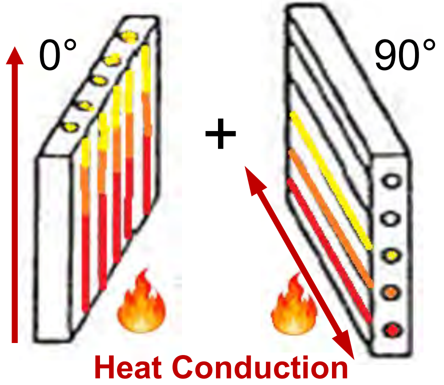
Appendix

Influence of Ply-orientation Relative to the Flame on Fire Damage During Vertical Burn Tests

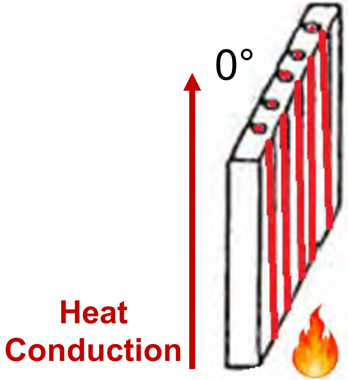
UNC0 Specimens: 0° Plies Burned *Parallel* to the Fibers, 90° Plies Burned *Perpendicular* to the Fibers

- 0° Plies: Exhibit similar mechanisms as mentioned above
- 90° Plies: Act like a ***thermal protection layer*** that can ***impede (slow)*** heat transfer to the interior of the specimen.
  - Conduct heat ***parallel to the fire-exposed surface*** (along fiber axis)
  - Promote decomposition and combustion of the epoxy matrix ***parallel to the fibers***
  - Increase melt dripping and char deposition at the lateral edge of composite
- Difference in ply-orientation increases thermally induced-strain and temperature gradients
  - More delamination, ply-splitting, matrix cracking
    - Provide pathway for outgassing
    - ***Less*** residual thickness increase



SBS Specimens: 0° Plies burned *Parallel* to the fibers

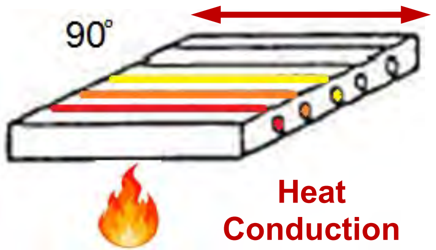
- Conduct heat ***perpendicular to the fire-exposed surface*** to the interior of the composite
- Promote formation and deposition of:
  - Melt dripping
  - Internal pockets of matrix decomposition
  - Surface char deposition
- Promote development of new matrix cracks and fissures to accommodate explosive outgassing resulting in ***significant*** residual thickness increase



Influence of Ply-orientation Relative to the Flame on Fire Damage During Horizontal Burn Tests

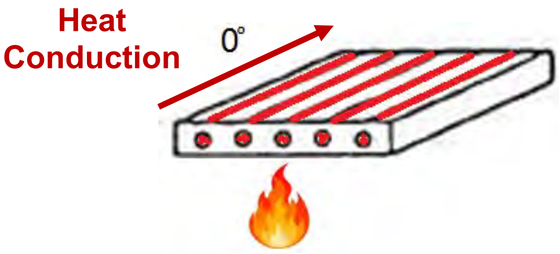
UNC0 Specimens: Outer 90° Plies Burned *Perpendicular* to the Fibers

- 90° Plies: Act like a thermal protection layer that can impede (slow) heat transfer to the interior of the specimen.
- Conduct heat parallel to the fire-exposed surface (along fiber axis)
- Less heat conduction and thermal damage through-the-thickness



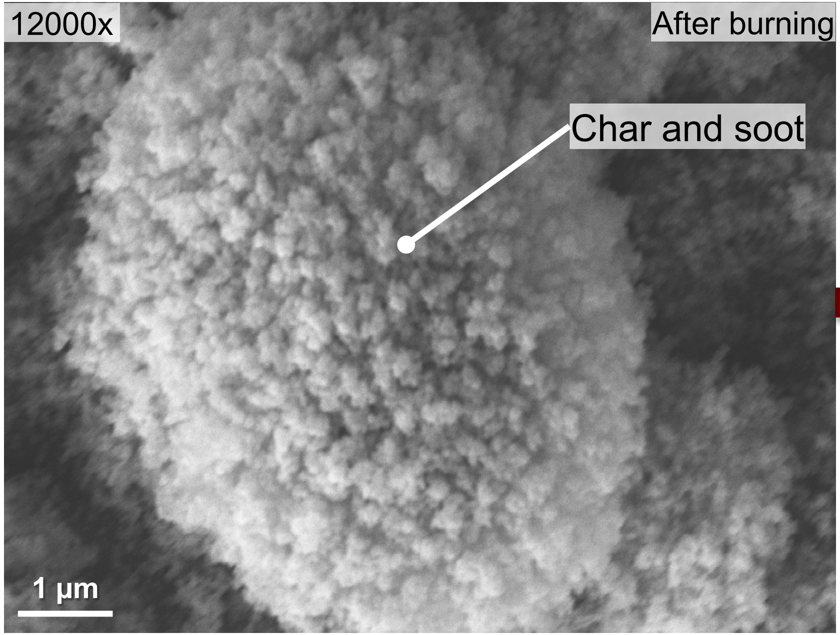
SBS Specimens: Outer 0° Plies burned *Perpendicular* to the fibers

- Conduct heat perpendicular to the fire-exposed surface to the interior of composite
- Promote formation, decomposition and combustion of the epoxy matrix along the primary heat conduction path (parallel to the fibers)
- Less heat conduction and thermal damage through-the-thickness

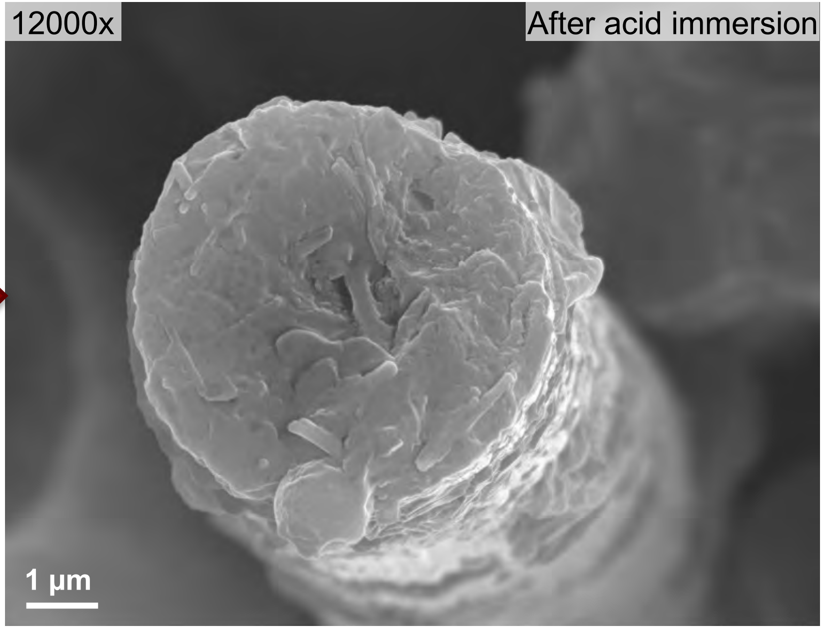


Heat transfer due to convection of hot gasses and smoke bypass the specimen causing less severe fire damage in both specimens

Pristine Burned Cytec Graphite/Epoxy Specimen Before and After Sulfuric/Nitric Acid Immersion



V-12 s fire exposure

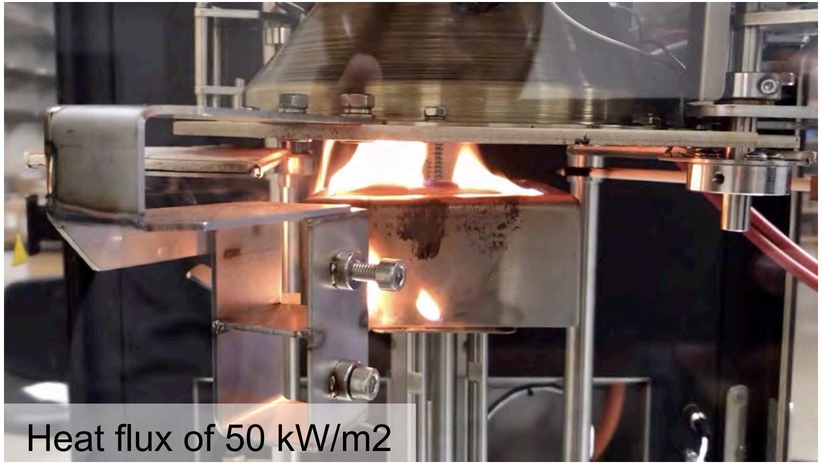


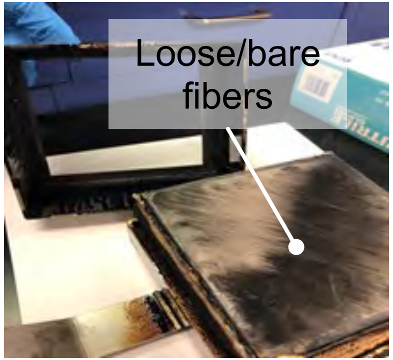
Immersion parameters: T=200 °C, t = 30 min

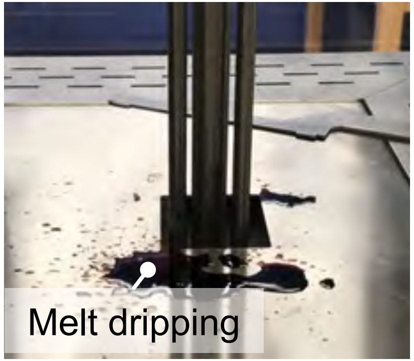
Acid volume: 30 ml, and acid composition: 30 ml of conc.{96-98%} H_2SO_4 + 2 drops of 30%vol. HNO_3

Cone Calorimeter Test of CAI Specimen

- ***Large-scale matrix decomposition*** throughout the entire specimen
  - No epoxy matrix or char were visible
  - Thermal-based approach may be efficient at removing char from burned specimens
- Broken and micro-buckled fibers at the CAI failure plane virtually indistinguishable from the surrounding fibers
- ***Large-scale multi-ply-delamination***
- Large amounts of ***melt dripping*** from specimen edges
  - Viscous tar-like substance that solidified after cooling







Fire Damage in a Mechanically-Failed PRSEUS Carbon-Epoxy Stringer Specimen Subjected to 60 s Burn Test

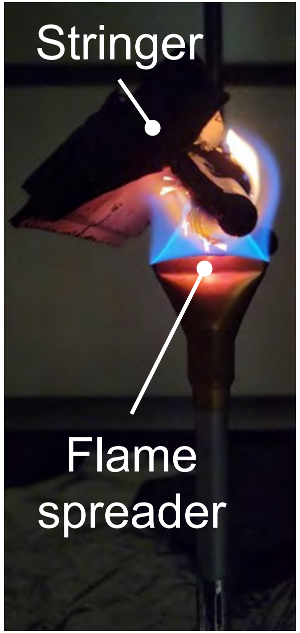
Before burn test

After 60 s burn test

Before burn test

After 60 s burn test

Fire Damage in a Mechanically-Failed PRSEUS Carbon-Epoxy Stringer Specimen Subjected to 60 s Burn Test



Before burn test

After 60 s burn test

Before burn test

After 60 s burn test