# Char removal strategy for fire-forensic analysis of mechanically-failed aerospace carbon/epoxy composites





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#### JAMS Project Title:

Post-Crash Fire Forensic Analysis on Aerospace Composites





#### Background

#### **Motivation**

- 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

#### **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









## **Char Removal Experiments: Nitric Acid Oxidation**



Acid Oxidation: Liquid-phase thermo-oxidative char removal experiments were performed on mechanically failed composites ranging from coupon-scale (NCAMP: UNC0, SBS) to structural-scale (open-hole) samples burned in small-scale bunsen burner fires and pool fires

- Solution type: Sulfuric/nitric solution
- Composition:
  - Sulfuric acid: [82 93] wt.%,
  - Nitric acid: [0.9 9.32] wt.%
  - Water: [5.32 8.33] wt.%
- Volume:
- Immersion Time:
- Temperature:
- Stirrer:
- 90 175 °C 60 rpm

45 s – 10 min

30 – 75 ml

#### **Experimental Objectives**

Select suitable temperature/time/acid compositions to:

- Oxidize char while preserving fracture surface morphologies
- Maintain solution acidity to improve char oxidation rates
- Minimize immersion times to maintain structural integrity of the samples





#### **Project Experimental Overview**





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### Fractography of Fire-Damaged UNC0 Specimens





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#### **Temp/Time/Conc Studies on UNC0 Samples**







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#### **Temp/Time/Conc Studies on UNC0 Samples**





#### **Temp/Time/Conc Studies on UNC0 Samples**



#### Effect of Fire-damage & Acid Oxidation on Matrix Fracture Features



Remnants of Interlaminar fracture features (examples: fiber imprints and cusps) were partially
retained in a fire damaged <u>UNC0</u> sample after acid oxidation in ONE test case



### **Fractography of Fire-Damaged SBS Specimens**





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### **Nitric Acid Oxidation of Unburned SBS Specimens**



Typical fracture morphologies observed after acid oxidation

Specimen	N (ml)	S (ml)	W (wt.%)	Time (min)	Immersion Temp (°C)
1	1.75	28.25	6.13	3	135

 Comparison of Fiber Cross-sections Before and After Acid Oxidation



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#### Nitric Acid Oxidation of Fire-Damaged SBS Specimens





#### **Fractography of Fire-Damaged SBS Samples**



#### Cytec Graphite/epoxy SBS Specimens S (ml) W (wt.%) Time (min) Specimen N (ml) Immersion Temp (°C) 2 1.75 28.25 3 6.13 135 Microbuckled Terrace Typical Fracture Morphologies Observed After 5 µm 2,000x 350x Extensive Char Removal After Acid Oxidation 30 µm Nitric Acid Oxidation С С T Microbuckled Fiber 1 µm 15,000x After Acid Oxidation After Acid Oxidation 2 µm 10,000x 13 Matthew Priddy - Mississippi State University JAMS Technical Review - April 19, 2023

# Conclusions: Nitric Acid Oxidation Experiments (UNC0 & SBS)



- Char successfully removed from coupon-scale Cytec UNC0 and SBS specimens burned during small scale fire tests without degrading fracture surface morphologies
  - Remnants of matrix-related intralaminar fracture morphologies were retained in UNC0 samples after fire exposure and nitric acid oxidation
- UNC0 char removal experiments: suitable temperature/time/concentration window determined
  - Temperature: 125-135 °C
  - Nitric acid: ~ 3 8 wt.%
  - Water: ~ 6 8 wt.%
- SBS char removal experiments: temp/conc window for char oxidization
  - Temperature: 135 °C
  - Nitric Acid: 3.18 wt.%
  - We will see these parameters used in the next set of experiments for open-hole (partscale) samples burned for large exposure times (up to 10 min)

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#### **Structural Scale Samples**

 <u>Pultruded Rod Stitched Efficient Unitized Structure (PRSEUS)</u> assemble dry polyester warp-knit AS4 carbon fabric skin stacks, stringers, and frames with through-thickness Vectran stitching prior to VRM-34 resin infusion and cure



PRSEUS components (Material)	Layup	Thickness (in)
Skin stacks (AS4/VRM-34)	[±45/0 <sub>2</sub> /90/0 <sub>2</sub> /∓45] <sub>2</sub>	0.104
Tear strap (AS4/VRM-34)	[±45/0 <sub>2</sub> /90/0 <sub>2</sub> /∓45]	0.052
Stringer wrap (AS4/VRM-34)	[±45/0 <sub>2</sub> /90/0 <sub>2</sub> /∓45]	0.052
Open hole	[±45/0 <sub>2</sub> /90/0 <sub>2</sub> /∓45] <sub>9</sub>	0.47











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### **Pre-Fire Fractography of Open-Hole Specimens**





Hexcel Carbon/epoxy Open-hole Sample  $[\pm 45/0_2/90/0_2/\mp 45]_9$ 



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#### **Post-Fire Fractography of Open-Hole Specimens**





#### Nitric Acid Oxidation of Fire-Damaged Open-Hole Specimens



Specimen	Burning	N (ml)	S (ml)	W (wt.%)	Time (min)	Immersion Temp (°C)
PH# 2C	V- 300 s	4.5	71.5	6.15	1.25	135



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#### Fractography of Fire-Damaged Open-hole Specimens





#### **Post-Fire Fractography of Open-Hole Specimens**





### Nitric Acid Oxidation of Fire-Damaged Open-Hole Specimens



#### Fractography of Fire-damaged Open-Hole Specimens





PH# 5C (V-600 s)

Typical Fracture Morphologies Observed after Nitric Acid Oxidation



#### Evolution of the Fracture Surface Morphologies Due to Fire Exposure and Nitric Acid Oxidation





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## **Conclusions: Nitric Acid Oxidation on Open-Hole Specimens**



- Vertically oriented structural scale Hexcel open-hole specimens failed in tension were exposed to longer duration Bunsen burner tests (5 and 10 mins)
- Nitric acid oxidation experiments on these samples effectively removed char from fiber ends and exposed the underlying fracture morphologies
- Upon fire exposure, the sample burned for 10 min exhibited:
  - Fiber thinning in plies directly submerged in the flame, but not prevalent over the entire cross-section
  - Char formation was also substantial on fiber ends in plies without fiber thinning
- Future work will focus on:
  - Char removal from coupon-scale samples subjected to pool fires at higher exposure durations
  - Char removal from structural-scale samples subjected to pool fires

## **Ongoing Work: Pool Fire Applications**







Schematic of Next-gen oil burner setup [1]

- The oil-fired burner was configured in accordance with Title 14 Code of Federal Aviation Regulations 25.853(c) Appendix F Part II to simulate pool fires.
- Mechanically damaged (Flexure and UNC0) cross-sections of samples were held vertically, 4 inches away from the burner. The flame spread is 11 inches wide. Test durations varied between 12 – 60 s.

## Fractography of Pool Fire-Damaged UNC0 Specimens





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#### Nitric Acid Oxidation of Pool Fire-Damaged UNC0 Specimens





## Fractography of Pool Fire-Damaged UNC0 Specimens





Acid Oxidation

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#### Nitric Acid Oxidation of Pool Fire-Damaged UNC0 Specimens





## Fractography of Pool Fire-Damaged UNC0 Specimens





Typical Fracture Morphologies Observed after Nitric Acid Oxidation

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## **Fractography of Pool Fire-Damaged Flexure Specimens**





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#### Nitric Acid Oxidation of Pool Fire-Damaged Flexure Specimens





## **Fractography of Pool Fire-Damaged Flexure Specimens**





Typical Fracture Morphologies Observed after Nitric Acid Oxidation

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#### Nitric Acid Oxidation of Pool Fire-Damaged Flexure Specimens





## **Fractography of Pool Fire-Damaged Flexure Specimens**





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## **Conclusions: Nitric Acid Oxidation on Pool Fire Specimens**



- Vertically oriented Hexcel UNC0 and Flexure specimens (coupon-scale) were exposed to shorter duration pool fires (36 and 60 s)
- Fire-damaged samples still retained char residues on sample cross-sections and fiber ends, obscuring salient fracture surface morphologies
- Fiber thinning was not observed for lower duration tests even at high heat fluxes (> 75 kW/m<sup>2</sup>)
- Nitric acid oxidation experiments on these samples effectively removed char from fiber ends and exposed the underlying fracture morphologies
- Future work will focus on:
  - Char removal from coupon-scale samples subjected to pool fires at higher exposure durations
  - Char removal from structural-scale samples subjected to pool fires

# **Overall Conclusions**

- Char removal methodology with a broad temp/time/conc window to treat firedamaged composite samples was developed as part of this work
- Nitric acid oxidation effectively removed char and exposed underlying fracture surface morphologies (as shown with fractography):
  - <u>Compression samples</u>: evidence for microbuckling and the direction of crack propagation were preserved after char removal
  - <u>SBS and Flexure samples</u>: compressive and tensile failure features were preserved after char removal
  - <u>Open-Hole samples</u>: Burned for longer exposure durations (5 and 10 min) at very low treatment times (45 to 75 s)
- Future work will focus on fire damage characterization and char removal of structural scale samples exposed to higher duration pool fires





#### **Thank You!**





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Nitric Acid

#### Oxidation



#### **Publications**



#### **Technical reports**

Lacy Jr., T. E., Kundu, S., Pittman Jr., C. U., Priddy, M., Boushab, D., Ouidadi, H., Righi, H., Madabhushi, A. (2021). Post-Crash Fire Forensic Analysis of Aerospace Composites-Phase I. Technical Report for Federal Aviation Administration. Mississippi State University, Starkville, MS. (Submitted to the FAA)

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