# In Situ Synchrotron Micro-CT of Microballoon-Filled RTV Silicone During Thermal Decomposition

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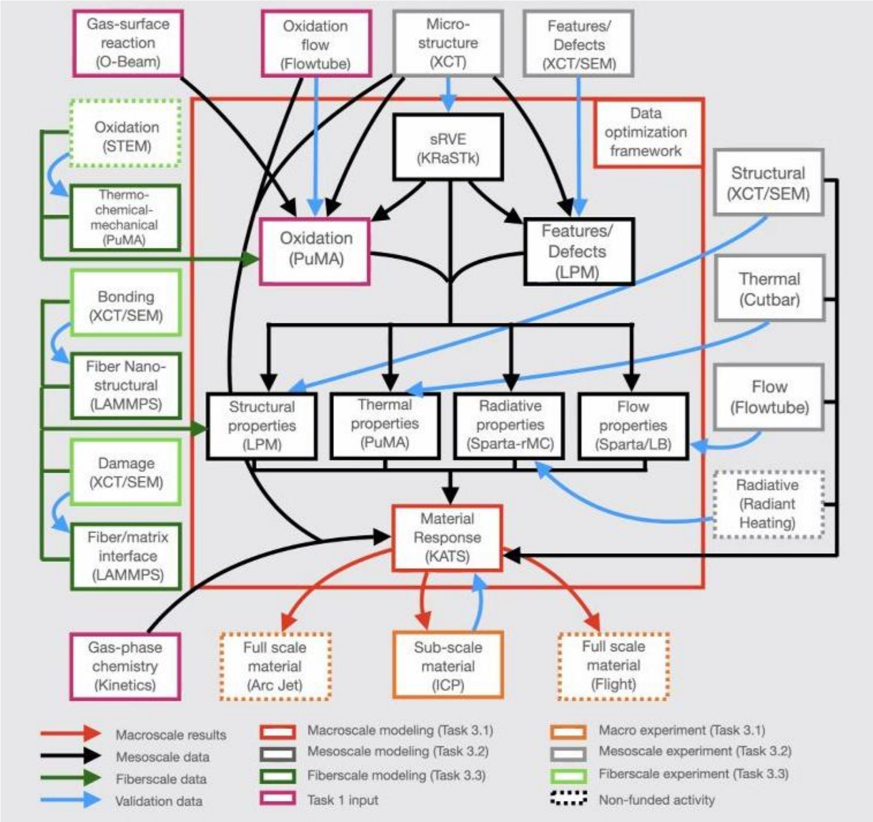
# Presentation Outline

**Executive Summary**

# Advanced Computational Center for Entry System Simulation (ACCESS)

5-year NASA Center for development of multi-physics modeling capabilities for heat shield response on atmospheric interface

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Aerospace Engineering Sciences
UNIVERSITY OF COLORADO **BOULDER**

# Multi-scale, Multi-Physics of PICA Response

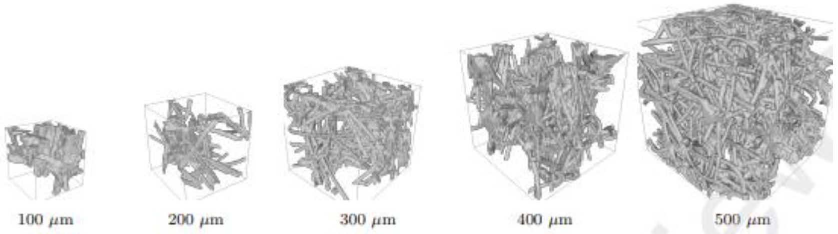


Figure 1: Microstructures constructed from XRCT with five different sizes

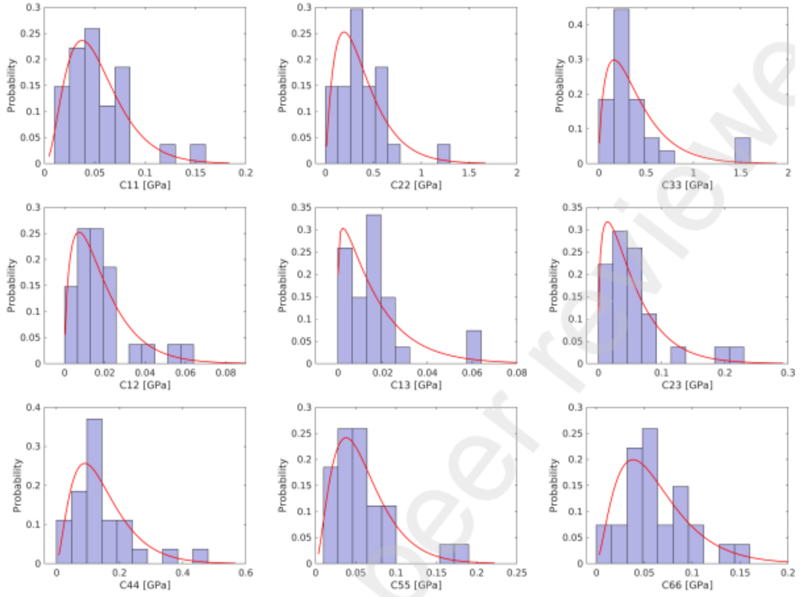
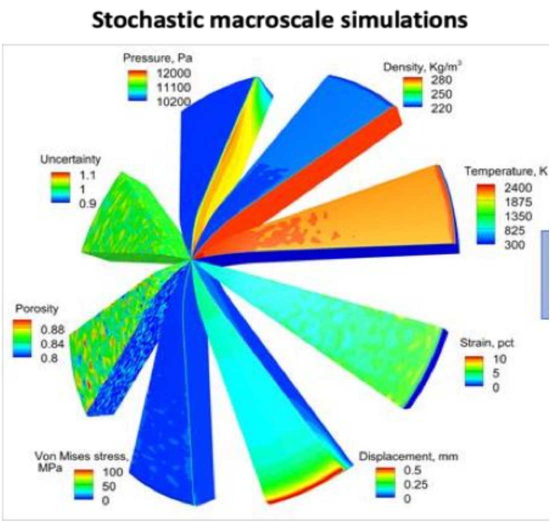
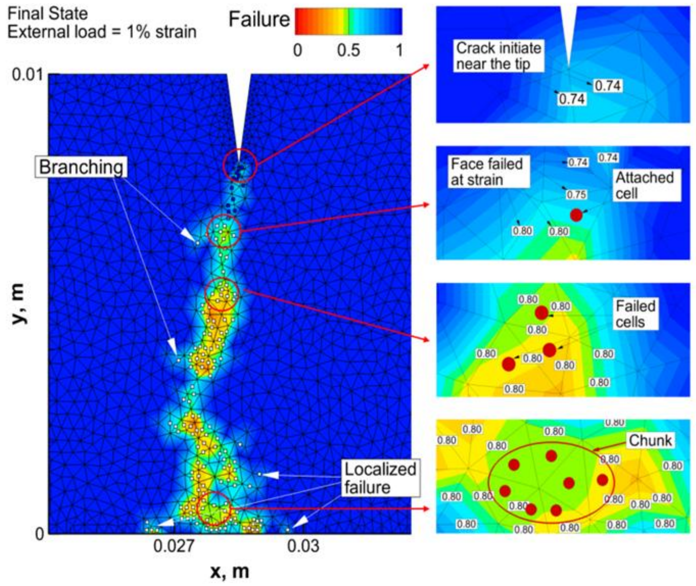


Figure 7: Gamma distribution of effective elastic stiffness components

**Assumptions to challenge:**

1. **FiberForm volumes to produce properties distributions**
2. **Properties are spatially homogenous**



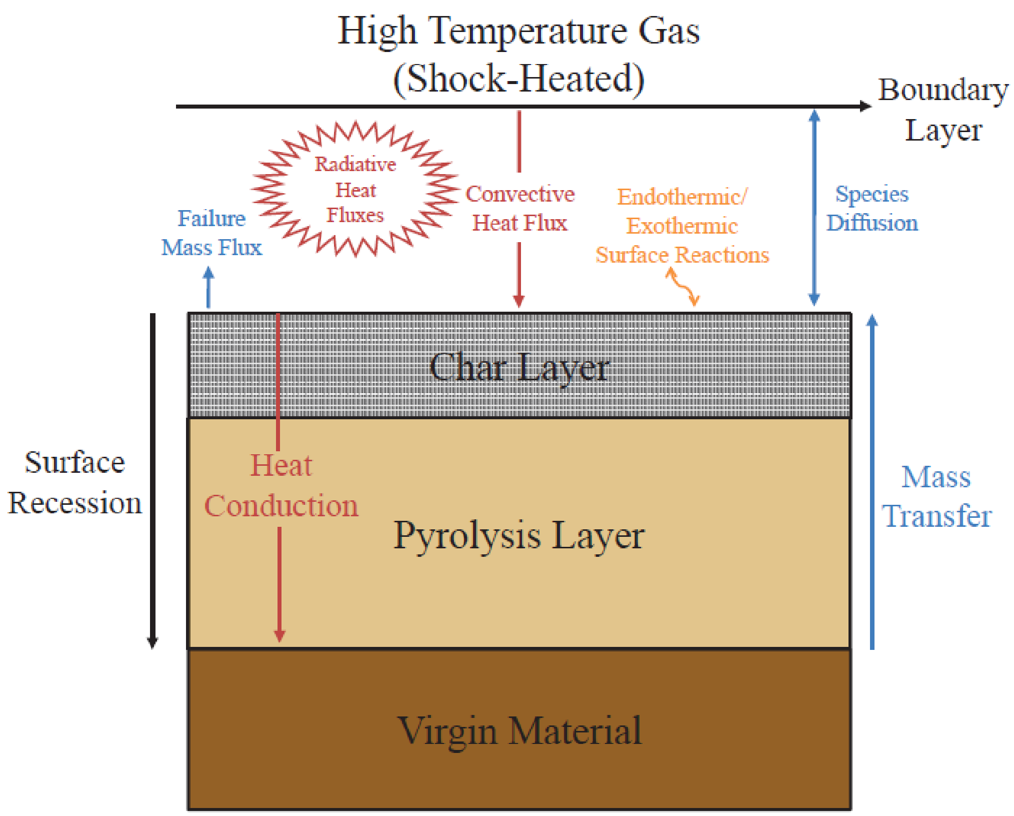





Liu, D., Maya, K. A., Chen, H., Banerjee, A., Chacon, L. A., & Poovathingal, S. J. (2024). The effective thermal and mechanical properties of FiberForm: Computation, microstructure-sensitivity analysis, and epistemic uncertainty quantification. *Composites Part B: Engineering*. Preprint.

Fu, R. (2024, November). *Fracture and Failure Modeling in Ablative Materials*. Presented at the 14th Ablation Workshop, University of Kentucky, Lexington, KY.

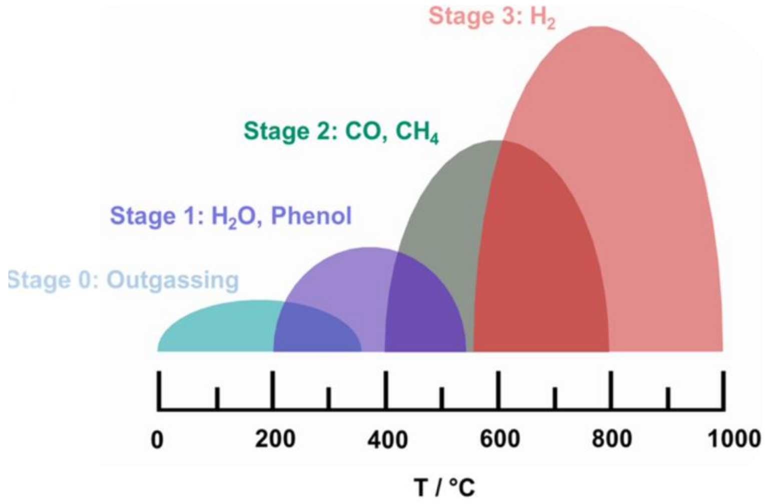
# How Does PICA Protect the Spacecraft?



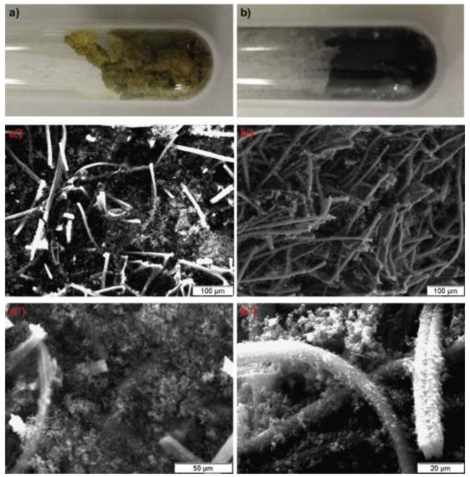

Tran, Huy K., Daniel J. Rasky, and Lili Esfahani. "Thermal response and ablation characteristics of lightweight ceramic ablators." *Journal of Spacecraft and Rockets* 31.6 (1994): 993-998.

**Energy Dissipation: Ideal Pyrolysis & Sublimation**

- Phenolic undergoes pyrolysis (polymer to ceramic reduction)
- Highly porous material allows for pyrolysis gas transport out
- Reduced ceramic residue is sublimated by shock-heated flux




Bessire, Brody K., Sridhar A. Lahankar, and Timothy K. Minton. "Pyrolysis of phenolic impregnated carbon ablator (PICA)." ACS applied materials & interfaces 7.3 (2015): 1383-1395.



Wong, Hsi-Wu, et al. "Detailed analysis of species production from the pyrolysis of the Phenolic Impregnated Carbon Ablator." *Journal of analytical and applied pyrolysis* 122 (2016): 258-267.

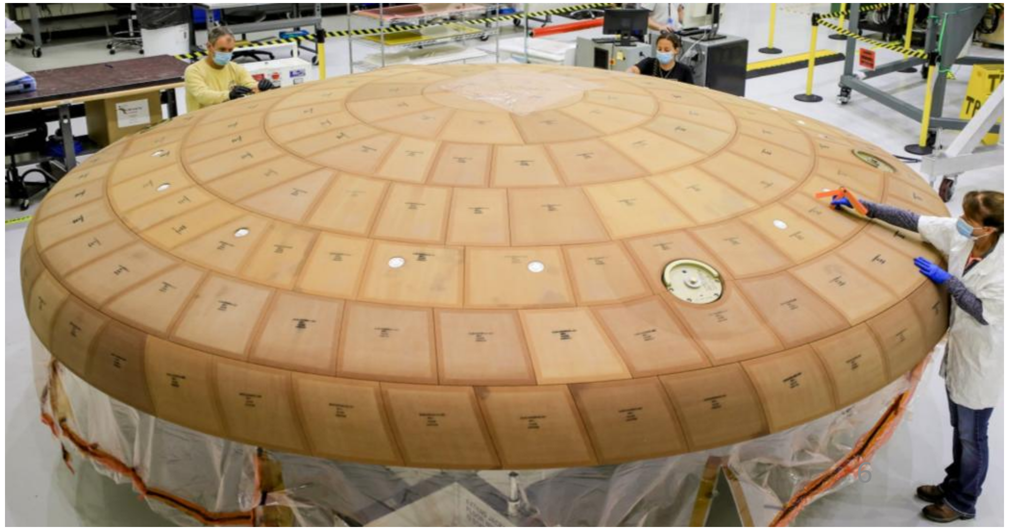
# Background and Motivation

- Room temperature vulcanizing (**RTV**) silicone is used as a bonding agent and gap filler for ablative TPS.
- **Challenge**: Reliability depends on how RTV swells, forms pores, shrinks, and creates a char layer during re-entry.
- **Knowledge Gap:** Pre and post flight analysis is not enough. Need to see the internal microstructural changes as they occur.

How do we understand this?

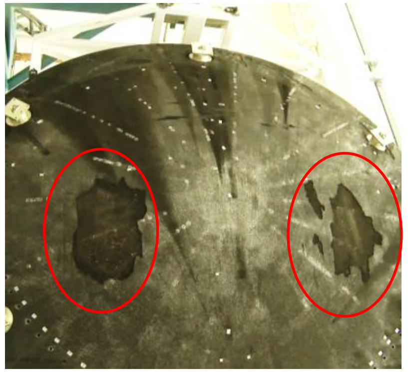
**Conduct Experiments:** Used Synchrotron XRCT at the Advanced Light Source (ALS) to capture the material's decomposition with high temporal resolution.

Pre-test and post-test photographs of a flat-face Avcoat test article with RTV-560 gap filler tested at a nominal EM-1 profile for 660 seconds. Source: NASA



# Examining Mechanical Damage of PICA

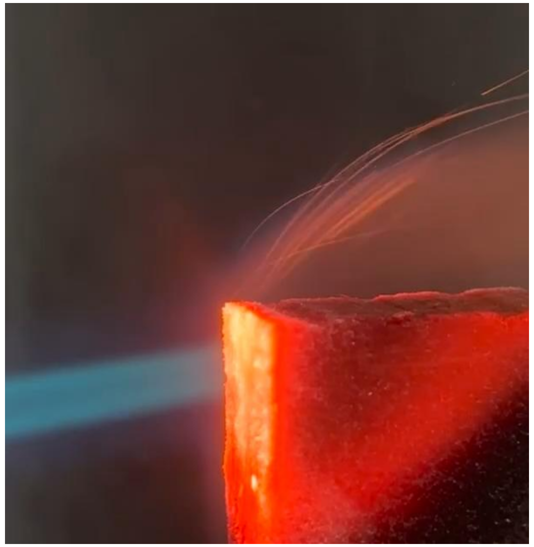
NASA AVCOAT



**Understanding Damage Mechanisms of Spallation-sized and Larger failure is critical**

SpaceX PICA-X

Meurisse, Jeremie BE, et al. "Multidimensional material response simulations of a full-scale tiled ablative heatshield." Aerospace Science and Technology 76 (2018): 497-511.



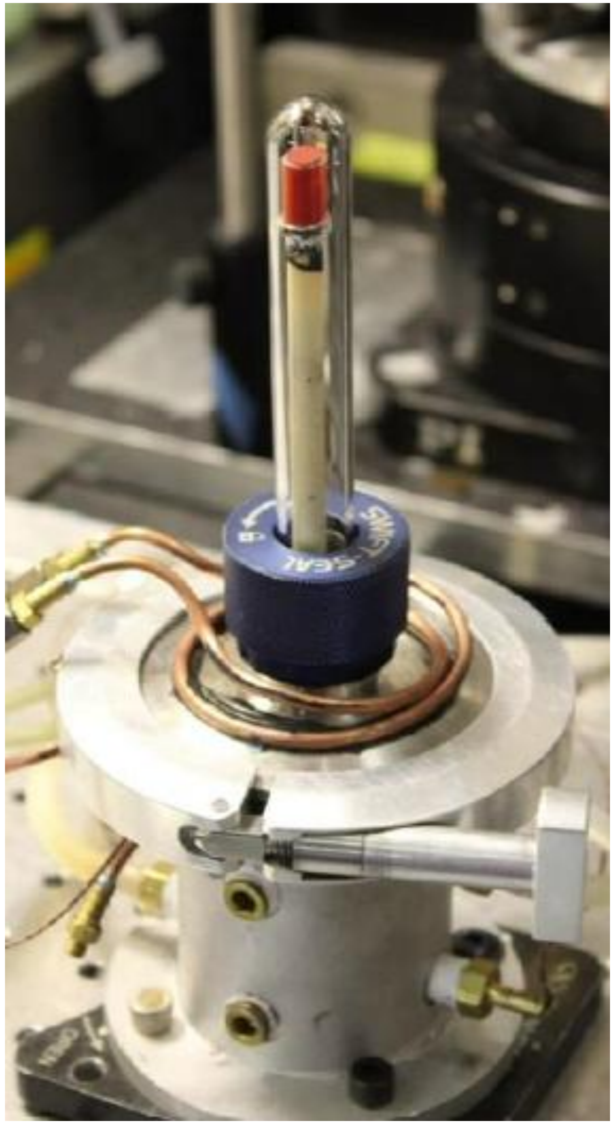
Dansereau, 2024 *Visualization of the spallation of PICA under combustion torch conditions*

*NASA Ames Testing of PICA in Arcjet facility*
Price, Kristen J., et al. "Arc-jet measurements of low-density ablator spallation." *Experimental Thermal and Fluid Science* 133 (2022): 110544.

# RTV Analysis Goals

- Collect data over a range of temperatures, atm composition, dryness
- Investigation of how the sample degrades over heating and time and image the 3D volume, with the goal in mind to track expansion, balloon coalescence, tearing, fracture and ceramic reduction / oxidation change the internal structure.
- Compare to baseline RTV

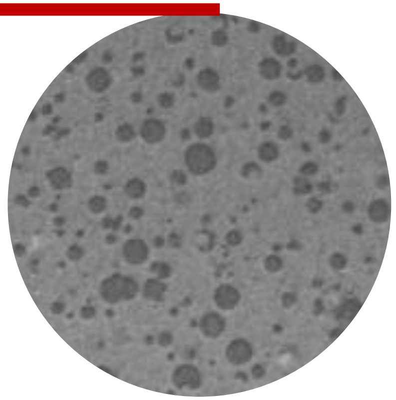
# Experimental Setup



Unconstrained Sample Constrained Sample

Virgin RTV MB samples: 6mm by 12mm cylinder

Side heating with 6 IR lamps



Hollow spheres

Collect raw data ✓

↓

Reconstruction ✓

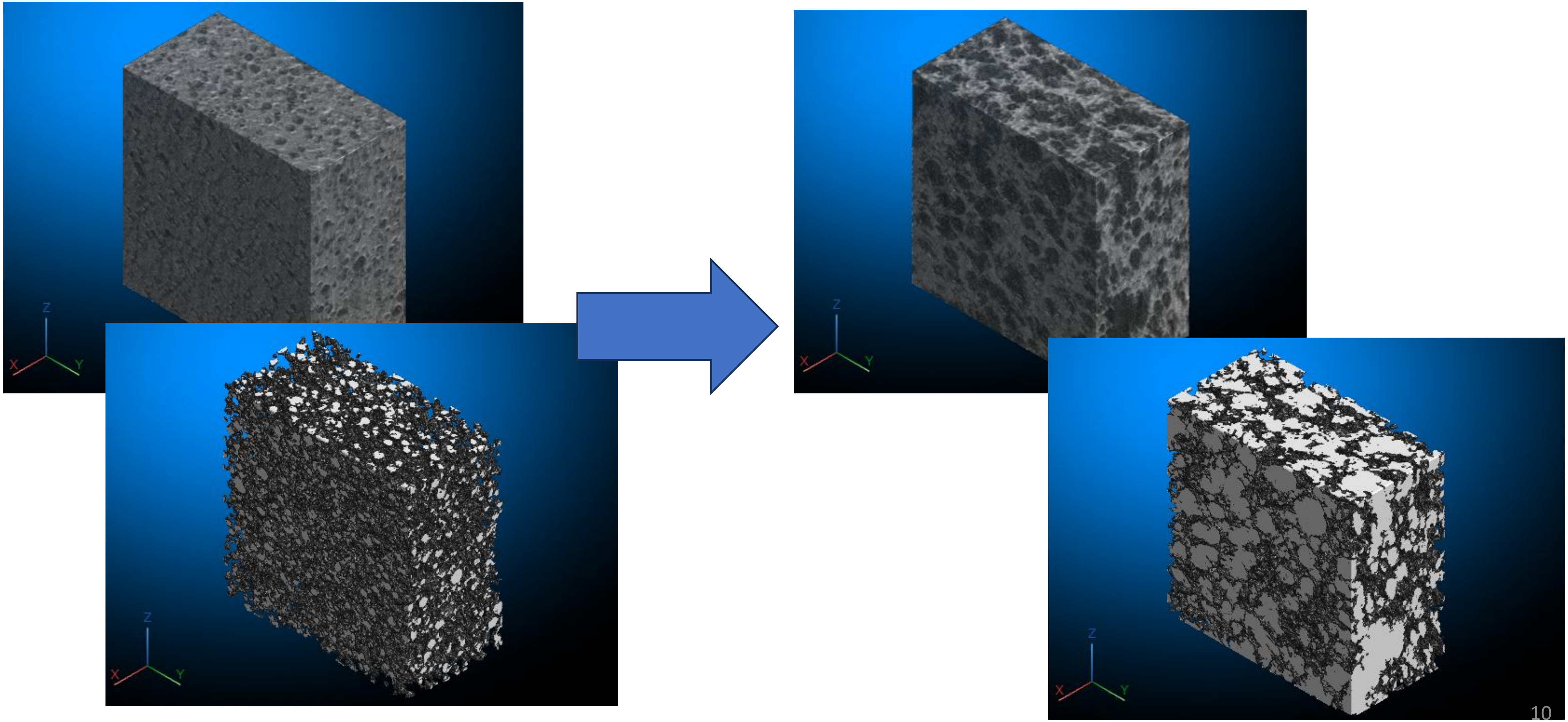
↓

**Segmentation**

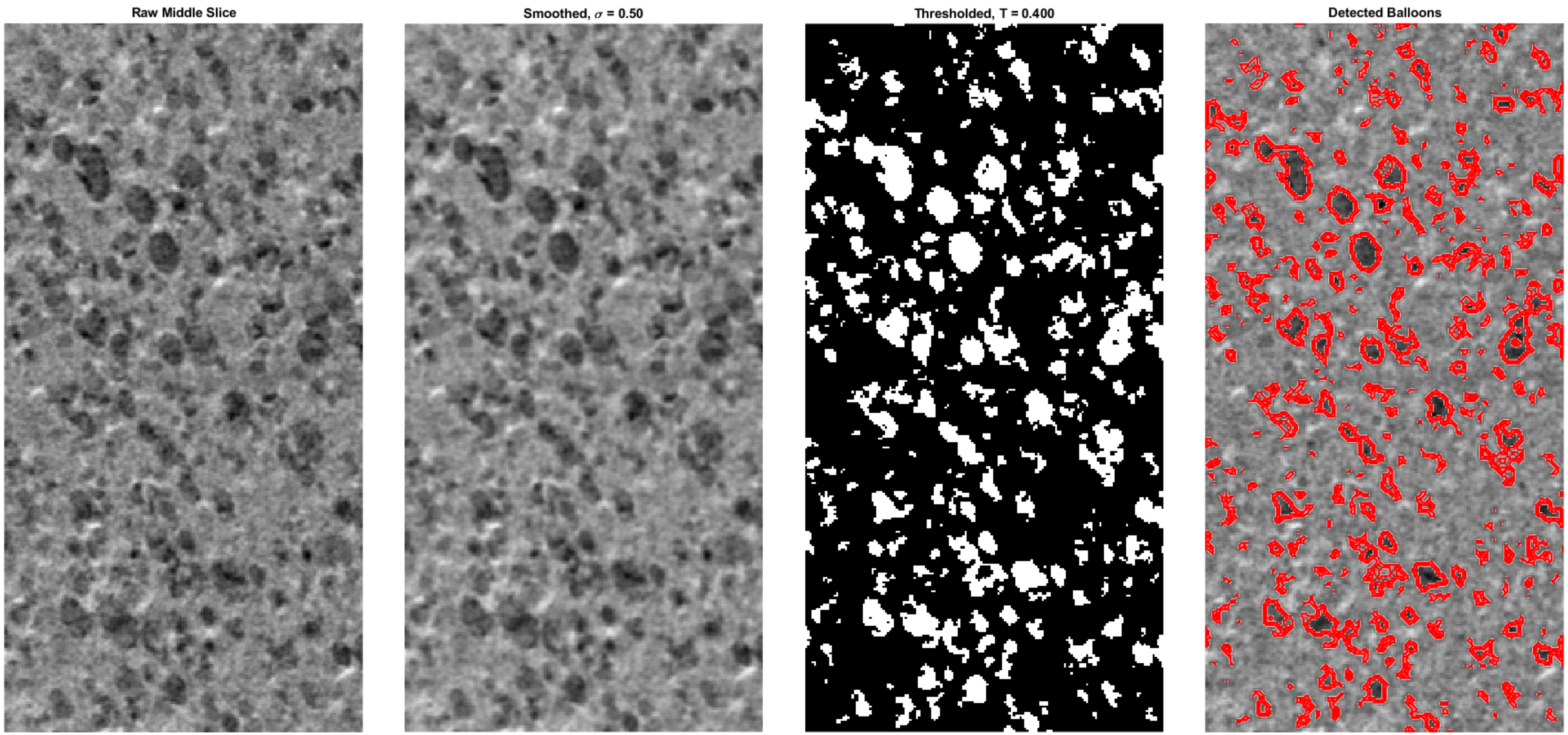
↓

Quantitative Analysis

# Initial visualizations of RTV micro-CT



# Initial visualizations of RTV micro-CT

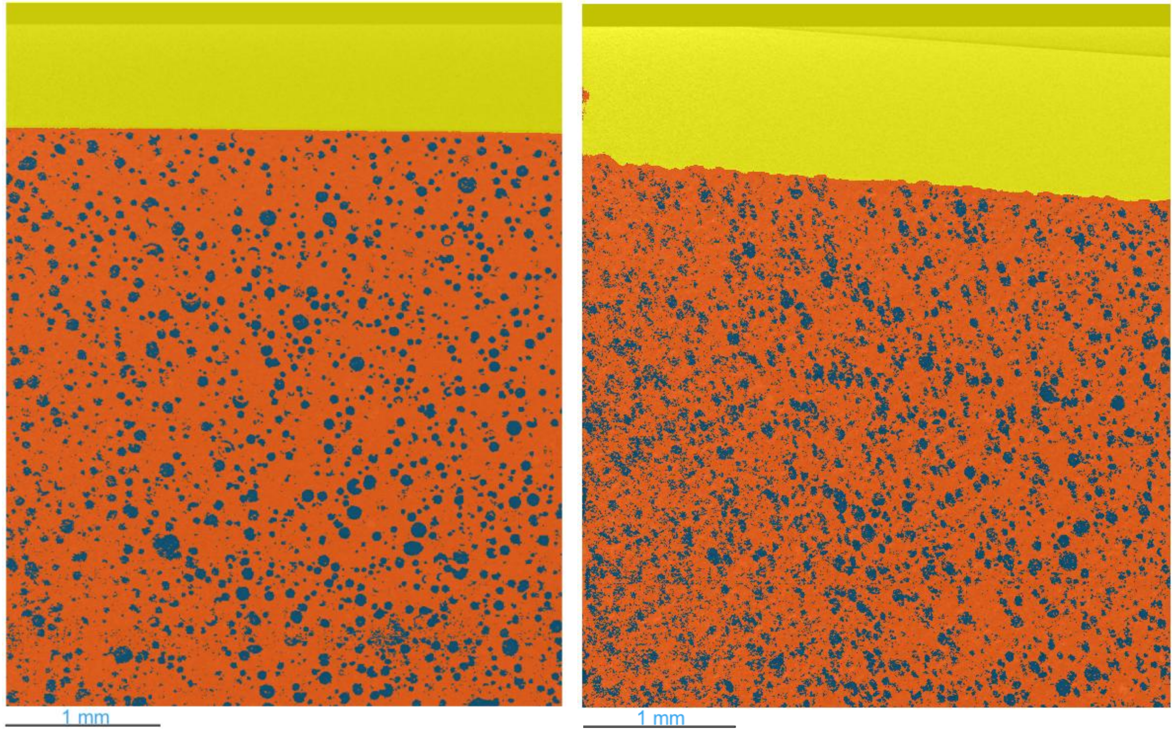


# U-Net Predictions on Different Datasets

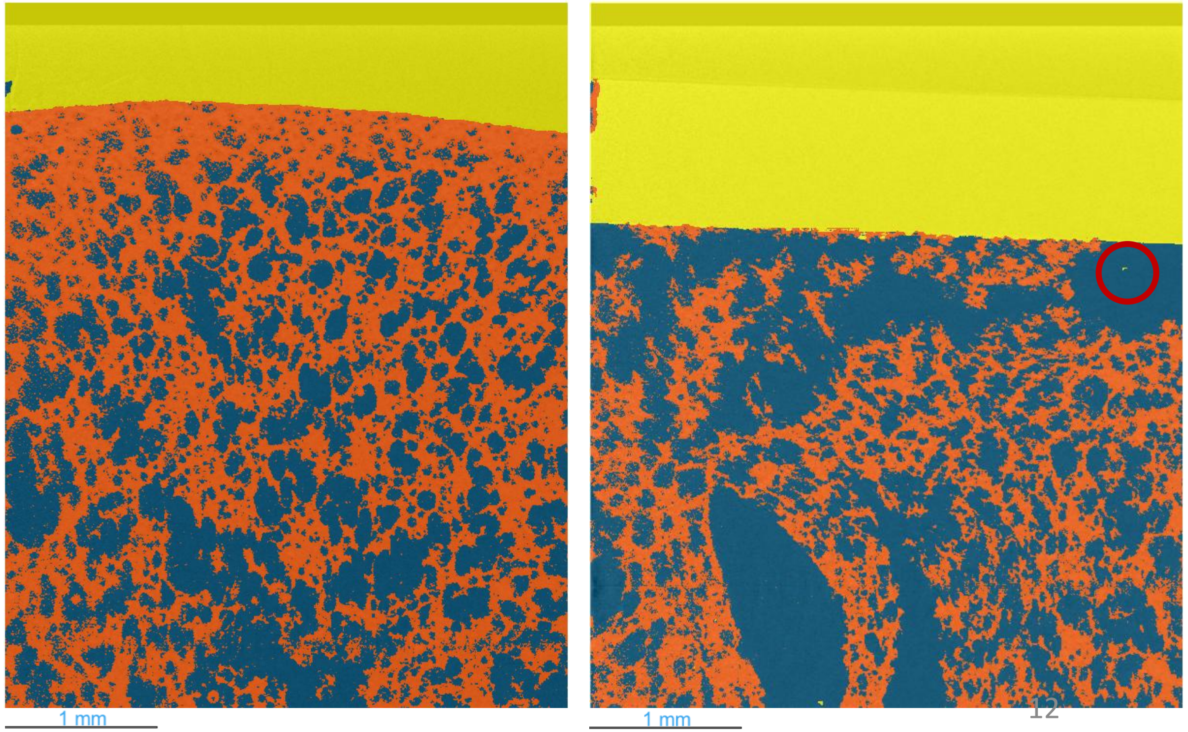
- **Computational Cost**: Prediction requires ~3 seconds per slice: ~2 hours to segment a full tile.
- **Accuracy**: Model score is over 99% compared to ground truth.

Void
Material
Background

## Prediction: Virgin / Pre-Pyrolysis



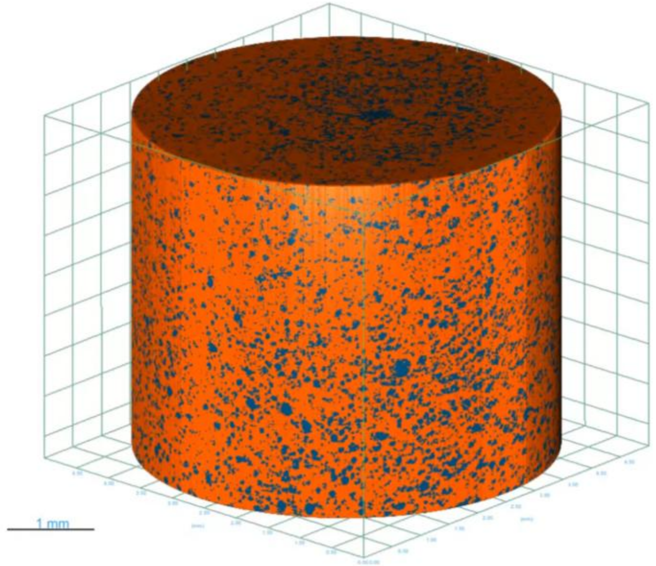

## Prediction: Charred / Post-Pyrolysis



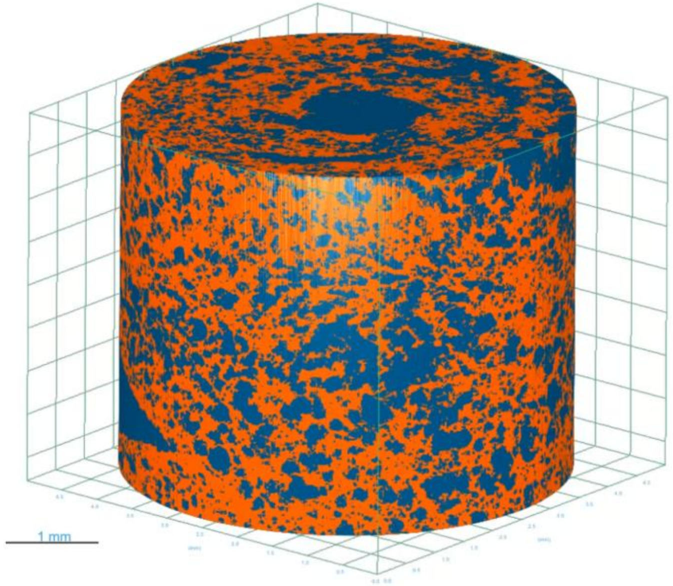
# Quantifying Porosity in Cropped Volumes

Void

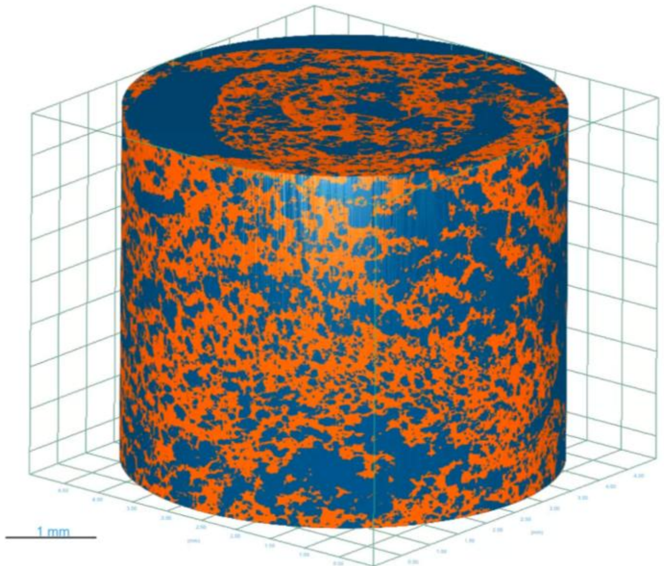
Material



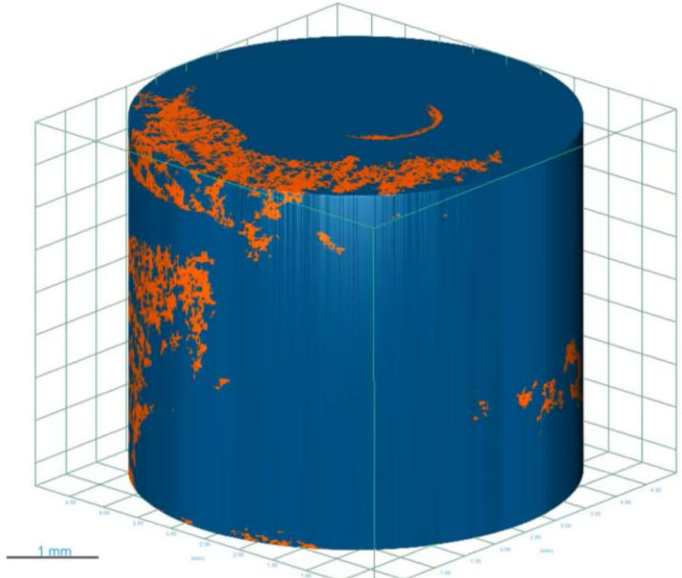

**Virgin**
Porosity: 21.5%




**Pre-pyrolysis**
Porosity: 54.0%

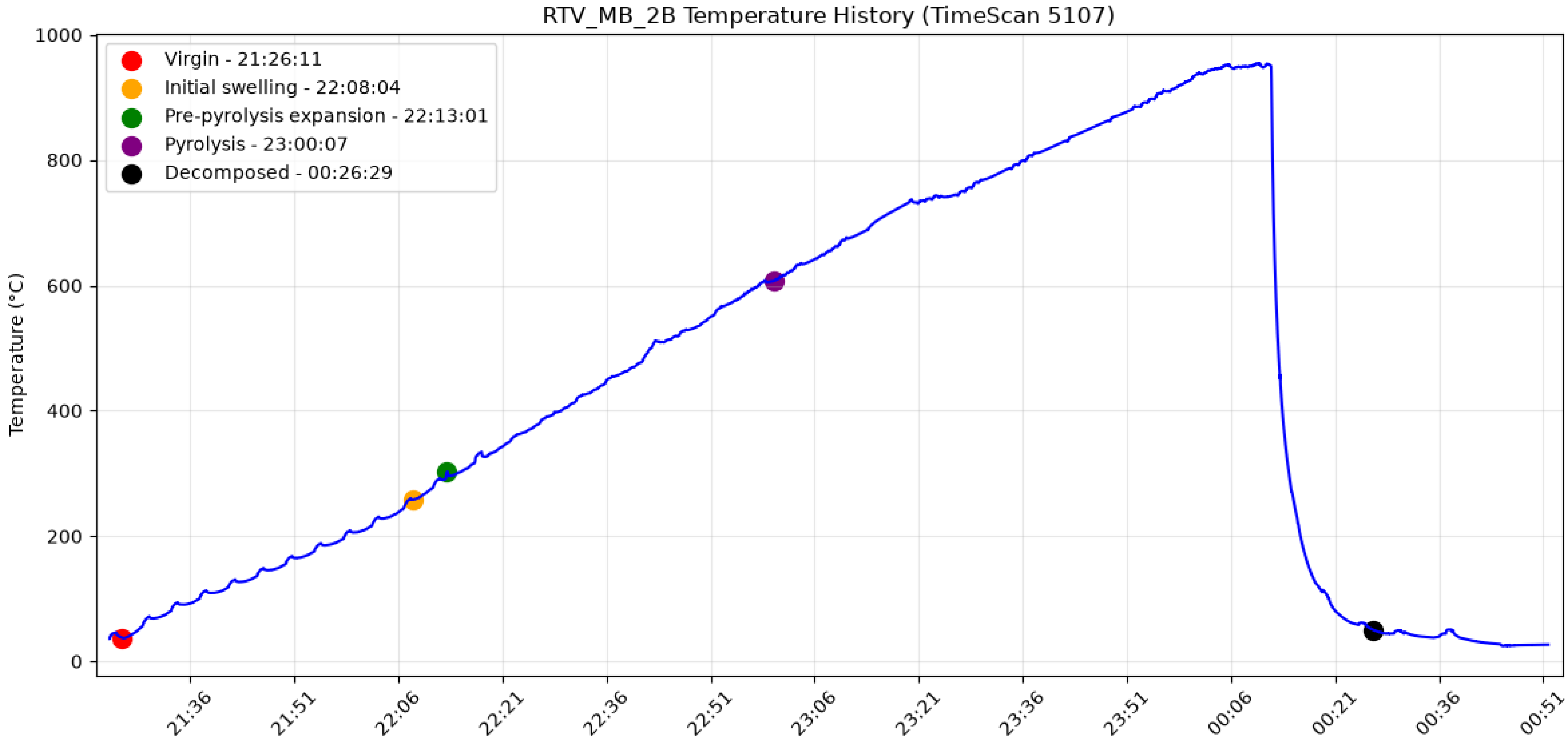



**Pyrolysis**
Porosity: 57.3%



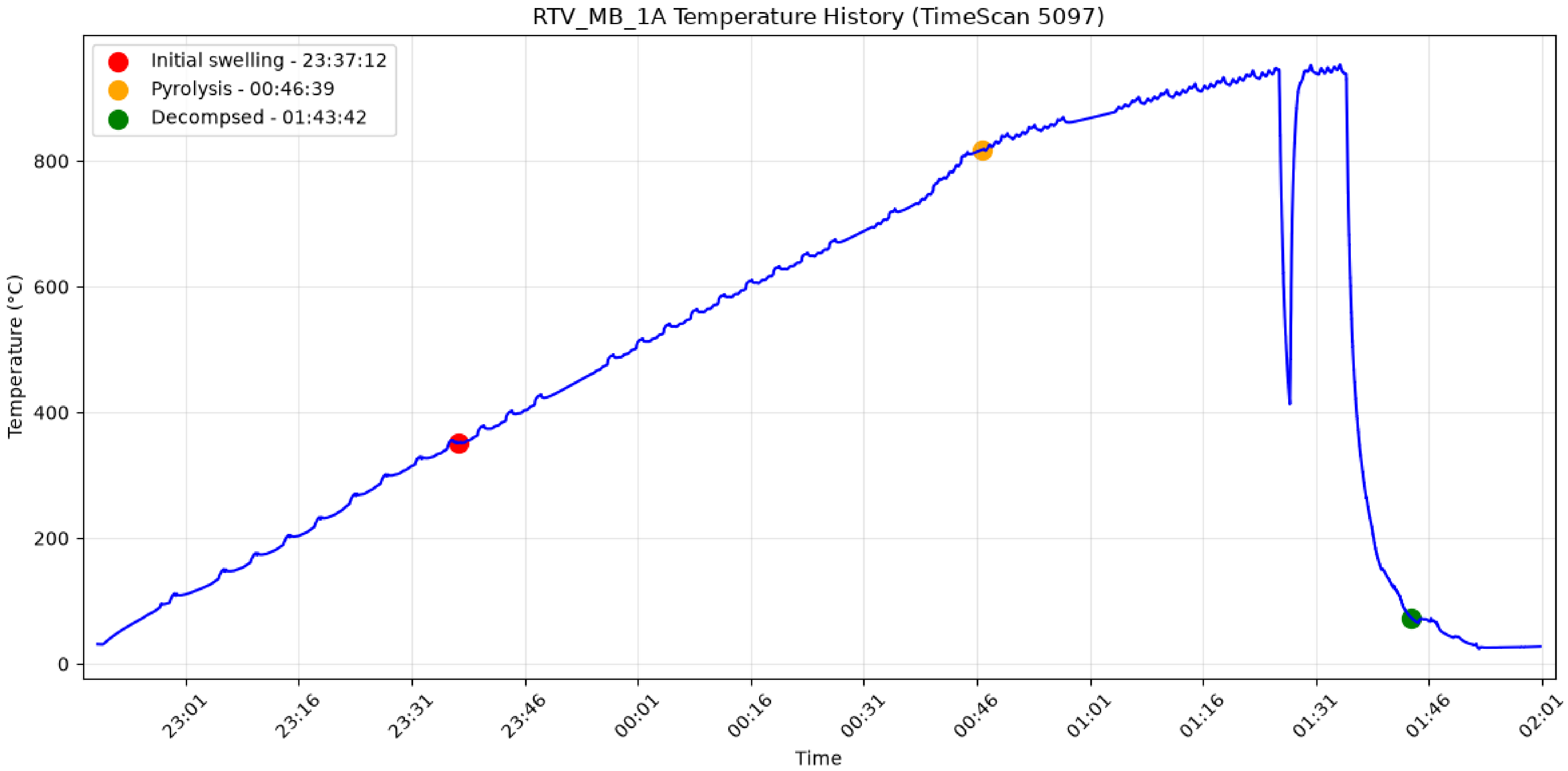

**Charred**
Porosity: 70.8%

# RTV Temperature Profile, PMB, Constrained




| File | State | Start time | End time | Start Temp (°C) | End Temp (°C) |
|---|---|---|---|---|---|
| 20251029_212611_RTV_MB_2B | Virgin | 21:26:11 | 21:30:18 | 37.55 | 68.86 |
| 20251029_220804_RTV_MB_2B | Pre-swelling | 22:08:04 | 22:12:11 | 258.31 | 290.32 |
| 20251029_221301_RTV_MB_2B | pre-pyrolysis expansion | 22:13:01 | 22:17:08 | 302.02 | 325.65 |
| 20251029_230007_RTV_MB_2B | pyrolysis | 23:00:07 | 23:04:14 | 607.34 | 633.85 |
| 20251030_002629_RTV_MB_2B | Decomposed | 0:26:29 | 0:30:36 | 50.36 | 47.25 |

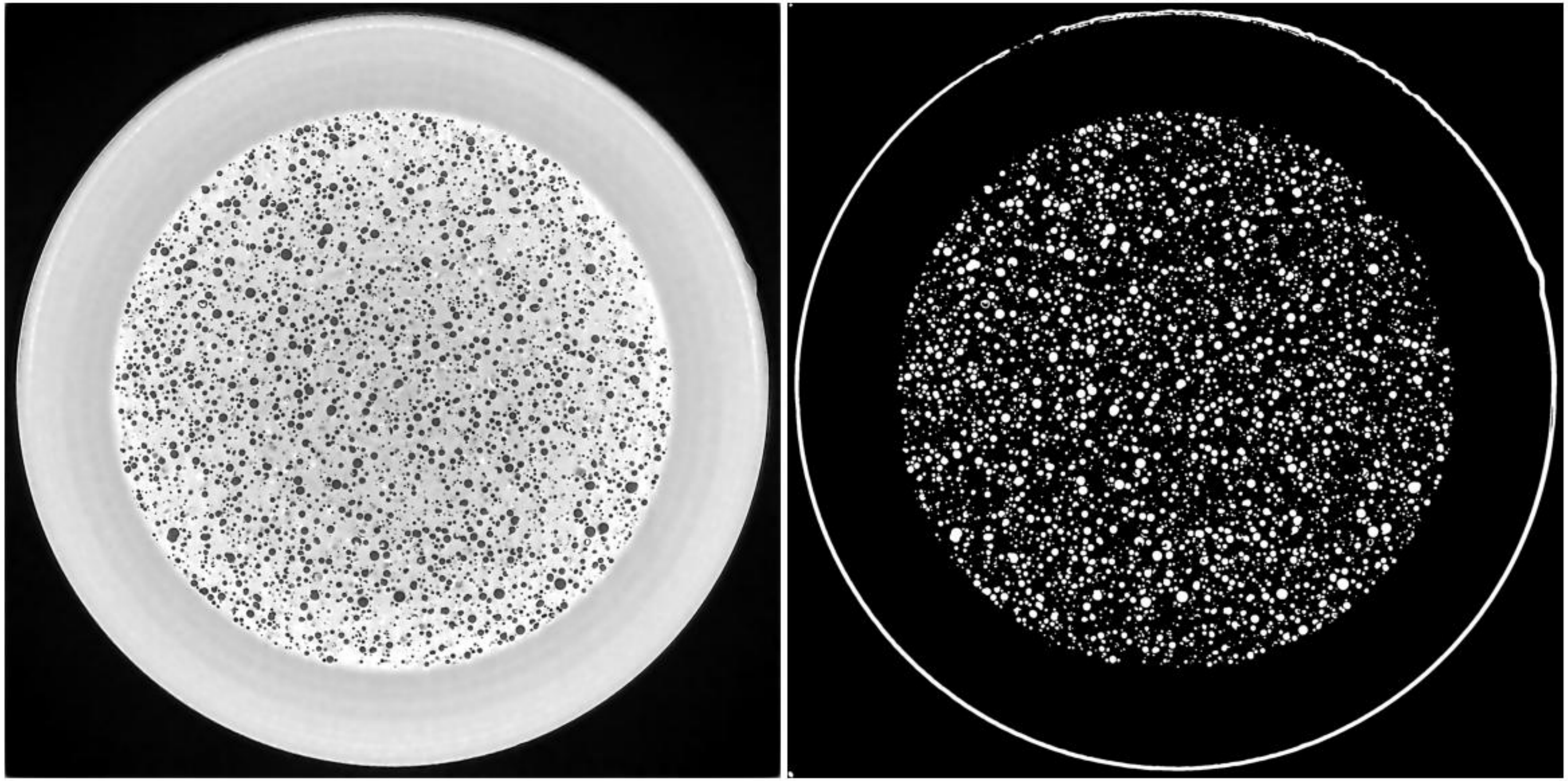
# RTV Temperature Profile, PMB, Unconstrained




| File | State | Start time | End time | Start Temp (°C) | End Temp (°C) |
|---|---|---|---|---|---|
| 20251027_214517_RTV_MB_1Af | Virgin | 21:45:17 | 21:49:17 | No temp data | |
| 20251027_233712_RTV_MB_1Af | Initial swelling | 23:37:12 | 23:41:12 | 352.02 | 377.18 |
| 20251028_004639_RTV_MB_1Af | Pyrolysis | 0:46:39 | 0:50:39 | 817.86 | 836.14 |
| 20251028_014342_RTV_MB_1Af | Decomposed | 1:43:42 | 1:47:42 | 72.82 | 50.97 |

# CT data processing: Denoising via Noise2Inverse

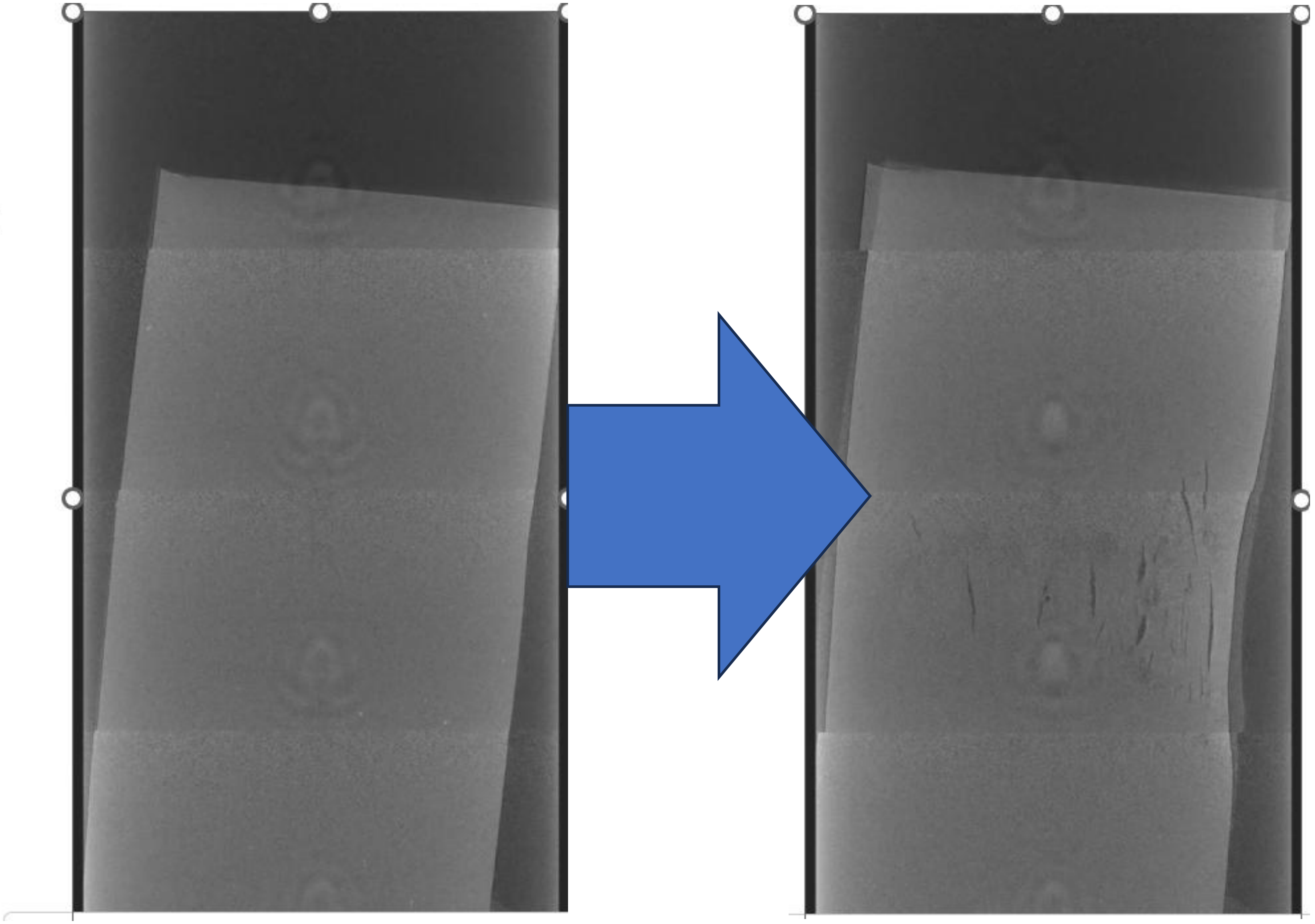
ML processing in collaboration with Argon national lab with an in-house de-noising code (still underway for the whole data set)



# RTV Failure Progression Analysis

## RTV UN A

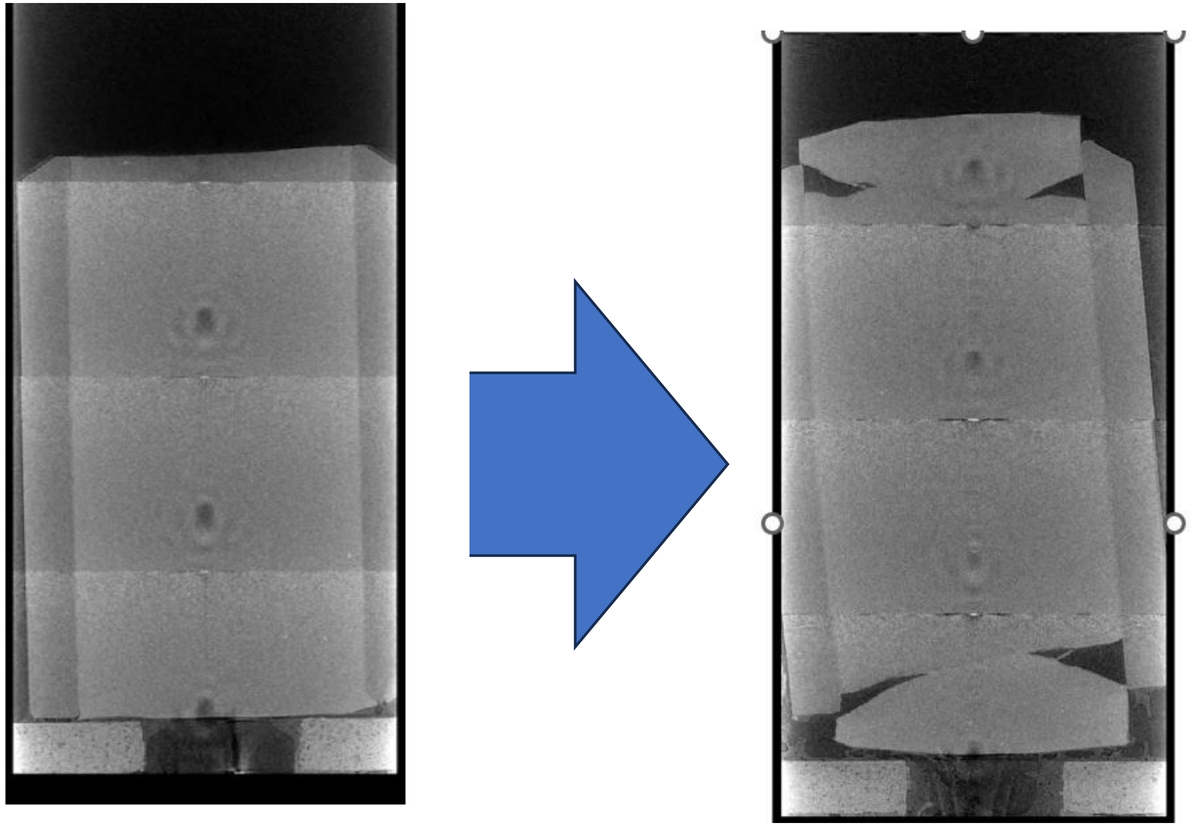
- **Description**: Unconstrained Solid RTV
- **Dryness**: N/A
- **Gas**: Argon



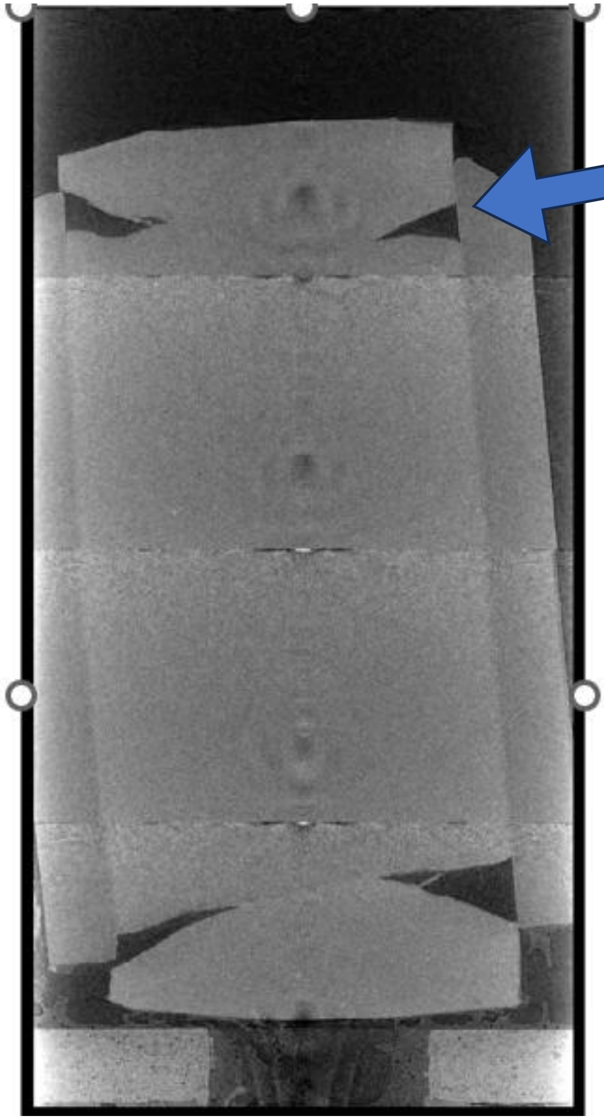
# RTV Failure Progression Analysis

## RTV CON A

- **Description**: Constrained Solid RTV
- **Dryness**: N/A
- **Gas**: Argon

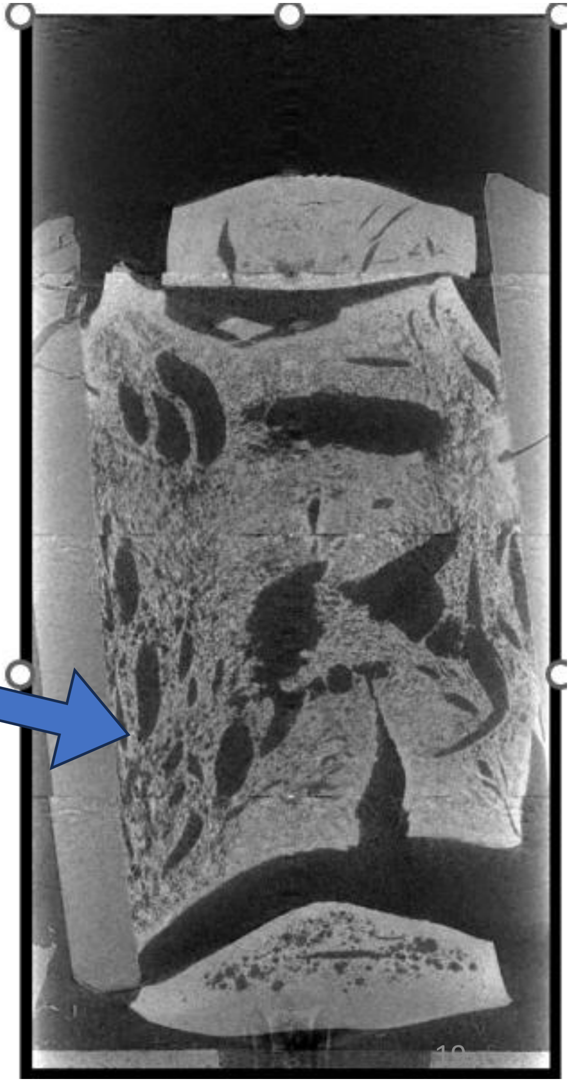


# RTV Failure Progression Analysis: Qualitative Visuals of base RTV degradation



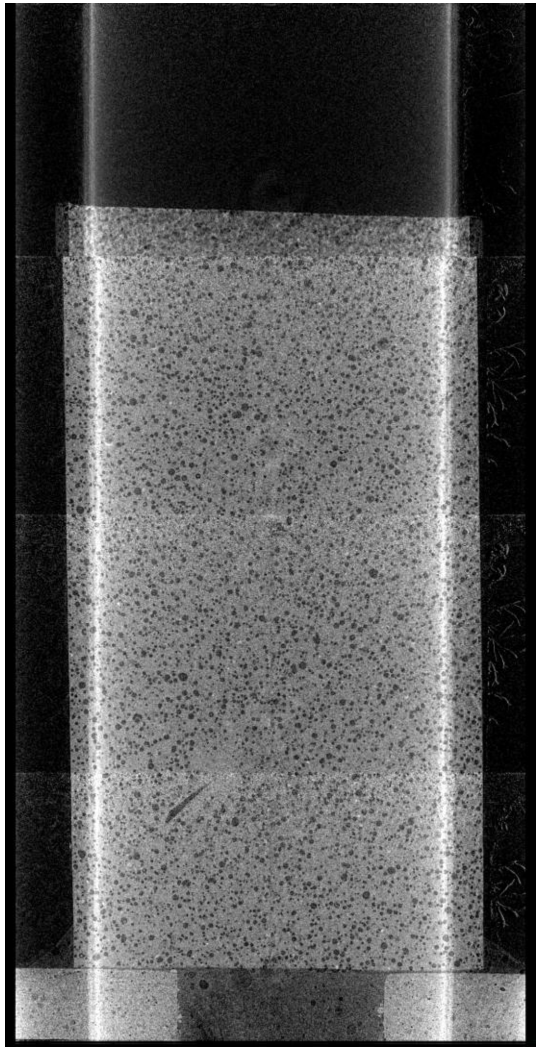
‘Capping’
Expansion-driven tearing of the constrained sample

Pyrolysis shrinkage and voids driven by polymeric decomposing and outgassing, densification and degradation.



# RTV Failure Progression Analysis: Timestep video of Uncon sample

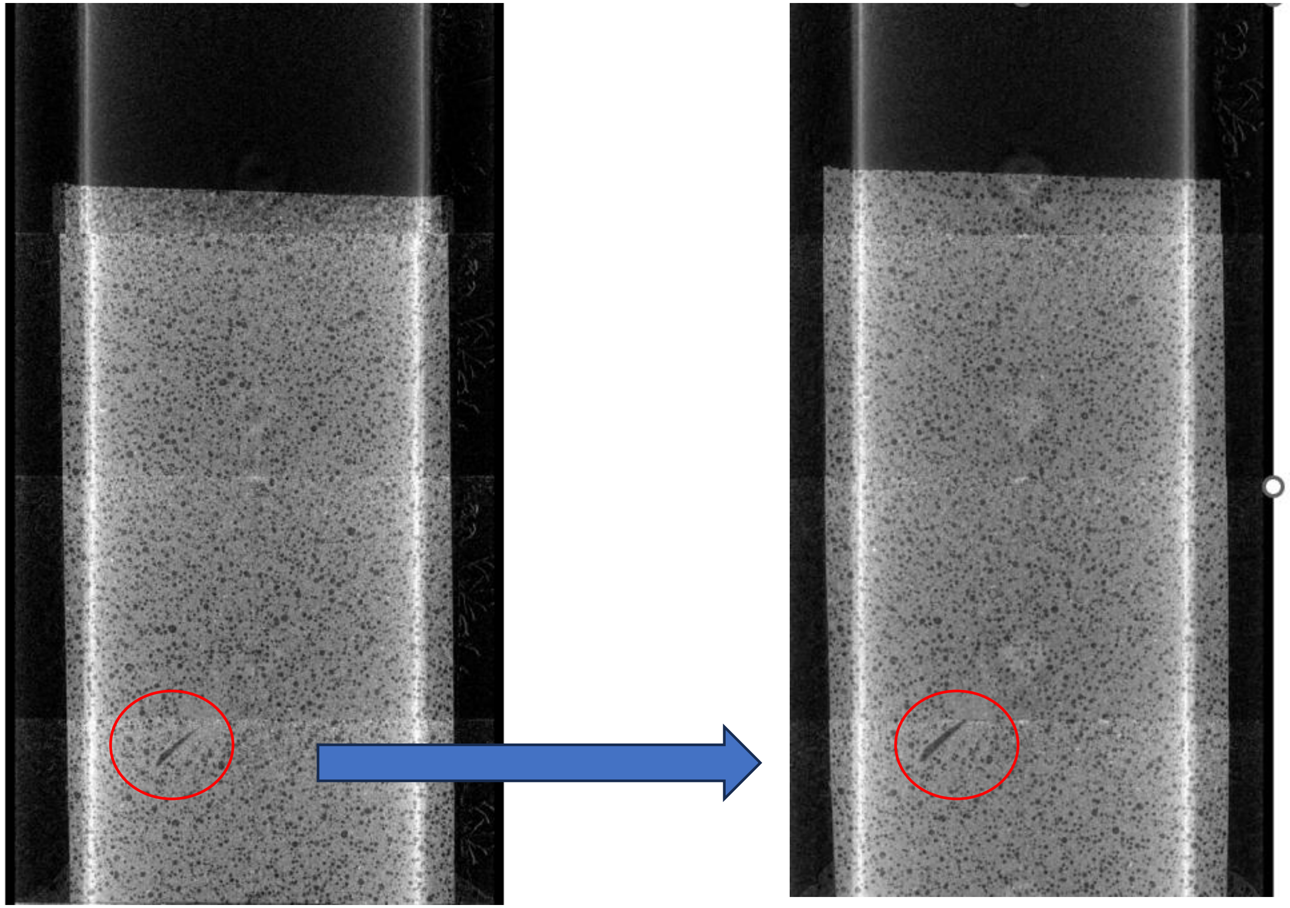
- **Description**: Default, unconstrained
- **Dryness**: Dried
- **Gas**: Argon



# RTV Failure Progression Analysis: Tracking Early Expansion of Uncon

RTV MB 3A

- **Description**: Default, unconstrained
- **Dryness**: Dried
- **Gas**: Argon

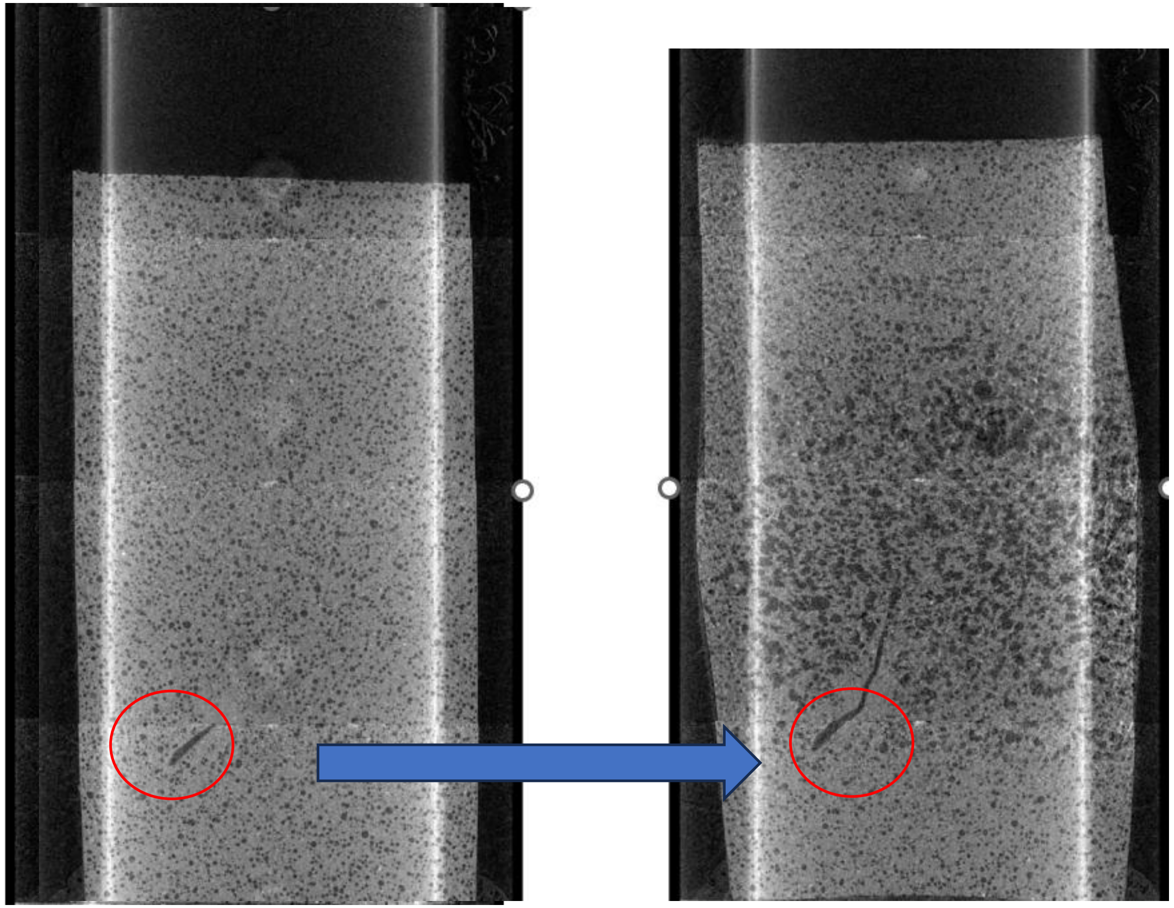


RTV expansion is coupled with gaseous expansion of microballoons and larger voids, individual balloons can initially be tracked and growth measured. Strain easy to track

# RTV Failure Progression Analysis: Pre-pyrolysis Degradation

**RTV MB 3A**

- **Description**: Default, unconstrained
- **Dryness**: Dried
- **Gas**: Argon

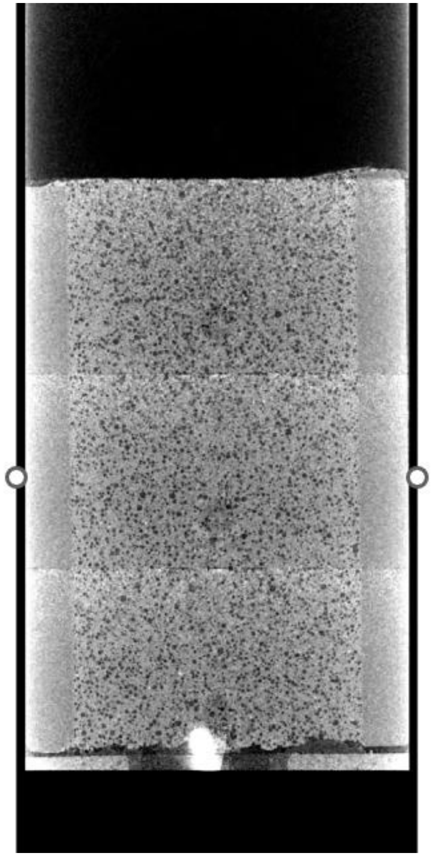


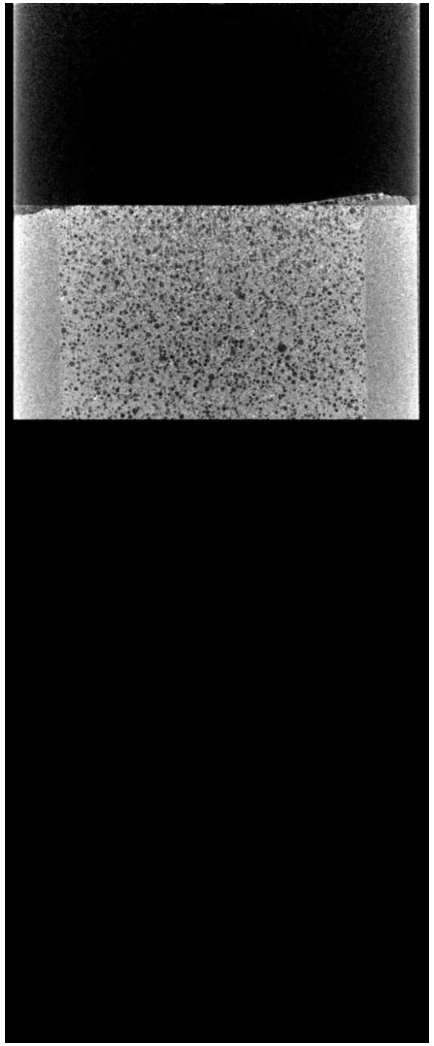
Densification and pyrolysis starts, facilitated by PMB. Starts in center of sample.

# RTV Failure Progression Analysis: Constrained Sample

## RTV MB 2A

- **Description**: Default, constrained
- **Dryness**: As-received
- **Gas**: Argon

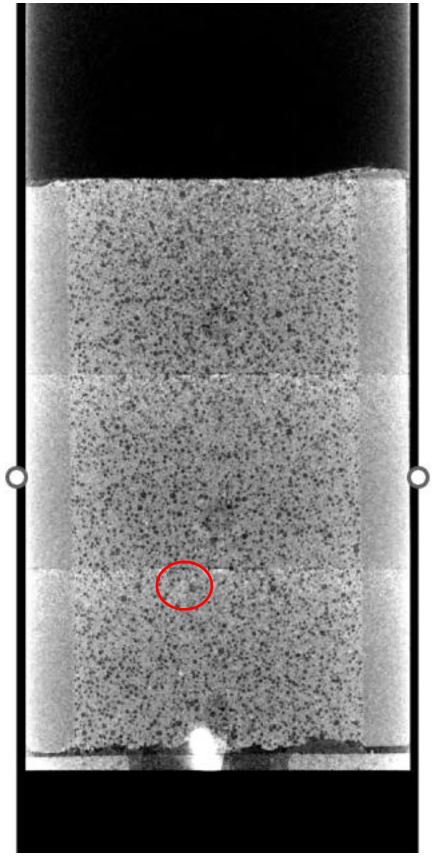




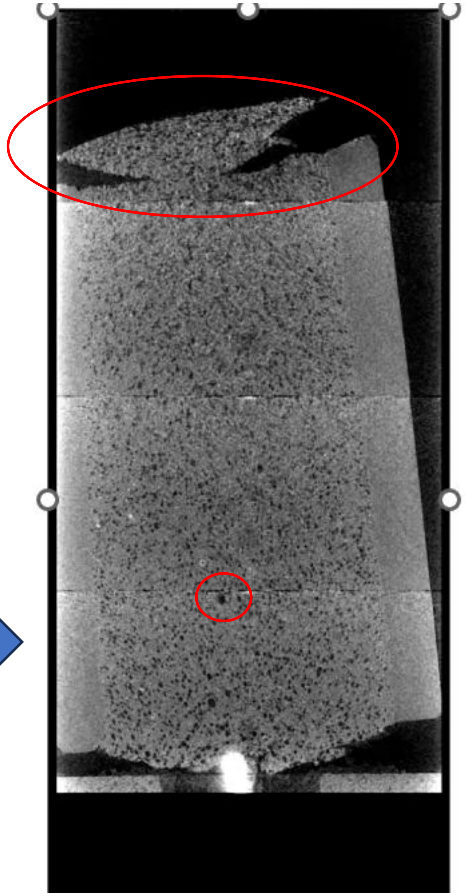
# RTV Failure Progression Analysis: Constrained Early Expansion

RTV MB 2A

- **Description**: Default, constrained
- **Dryness**: As-received
- **Gas**: Argon



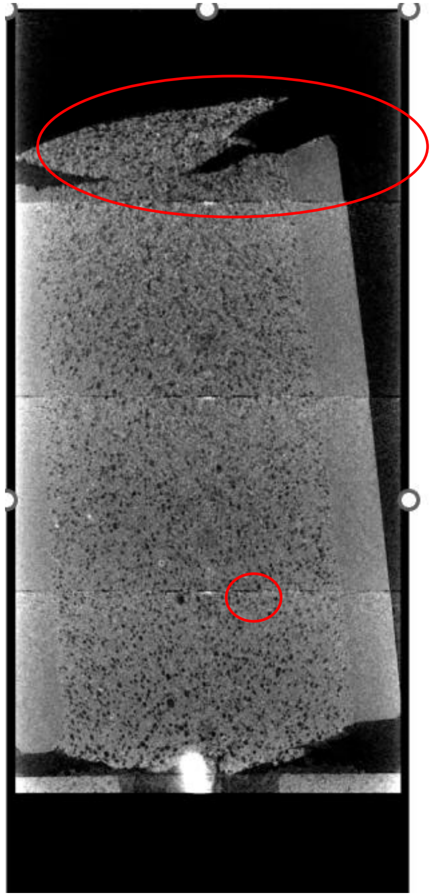
Expansion of balloons is trackable, capping/tearing still occurs (compared to solid RTV)



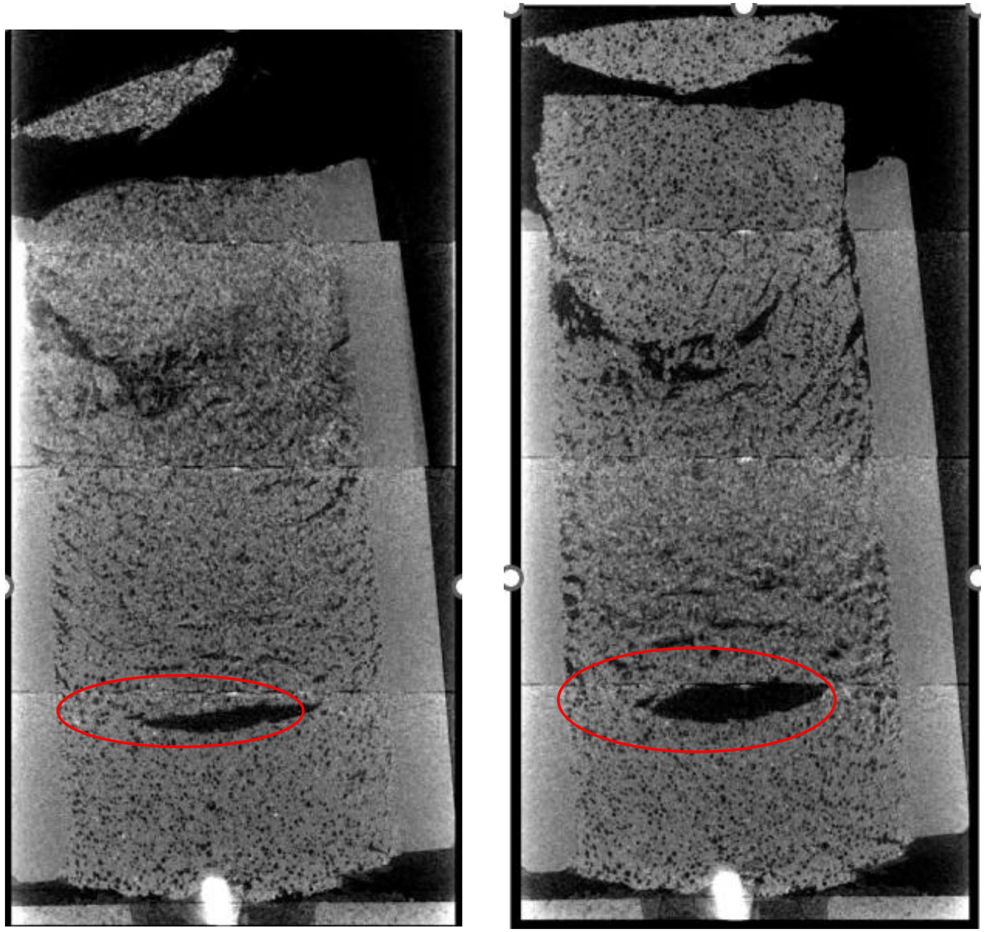
# RTV Failure Progression Analysis

## RTV MB 2A

- **Description**: Default, constrained
- **Dryness**: As-received
- **Gas**: Argon



Pyrolysis outgassing starts, coupled with tearing. Dominate features (tears) relax stress in the surrounding RTV and dominate expansion growth when no outgassing pathway is available



# RTV Failure Progression Analysis: Next Steps

- Limitations of the time-resolved data set
- Analysis directions
- Validation of hypotheses