In-situ Imaging of Pyrolyzing Aerospace Materials

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Sreevishnu Oruganti, Collin W. Foster, and Francesco Panerai

Center for Hypersonics and Entry Systems Studies,
University of Illinois Urbana-Champaign

Dilworth Y. Parkinson

Advanced Light Source, Lawrence Berkeley National Laboratory

Sergio Fraile Izquierdo and Nagi N. Mansour

Analytical Mechanical Associates Inc. at NASA Ames Research Center

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Grainger College of Engineering
UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

NASA

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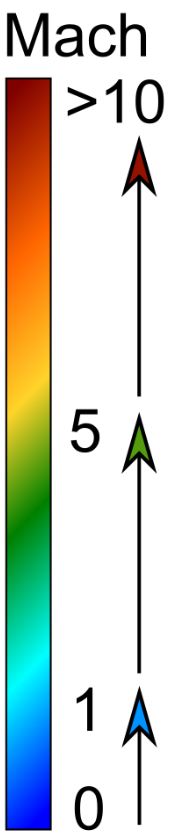
DOE ALS – [DE-AC02-05CH11231], DOE SCGSR – [DESC0014664]

Aerospace Vehicles



Inter-planetary probes

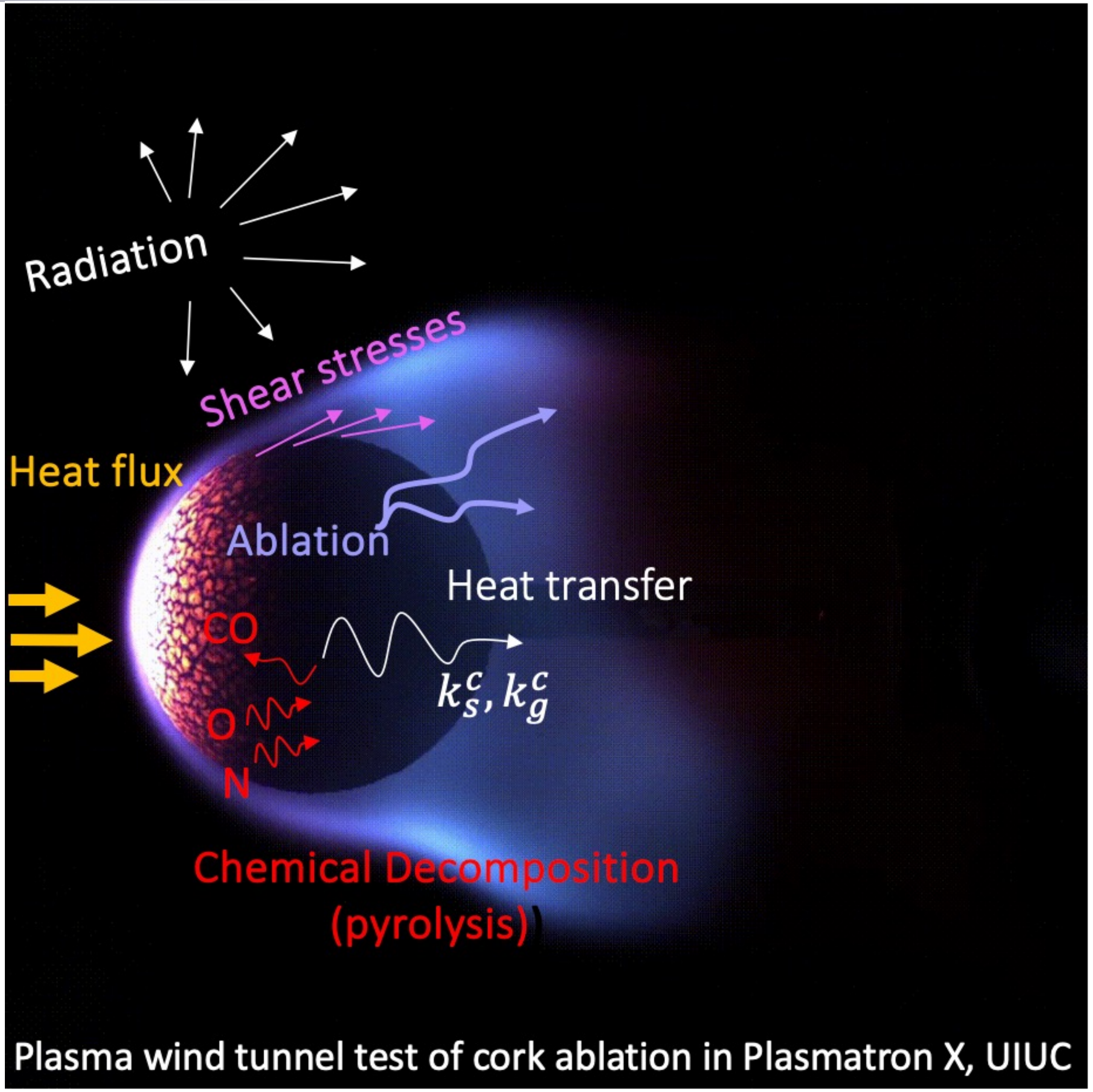
Meteorites

Defense vehicles

Military jets

Reentry parachutes

Commercial airliners



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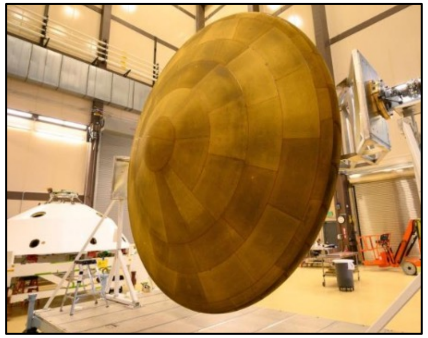
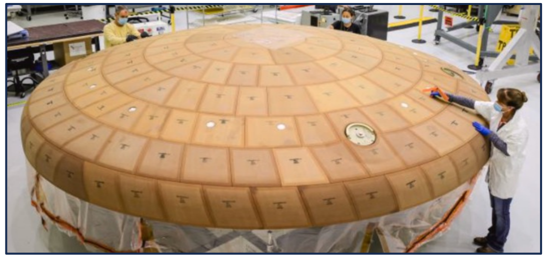
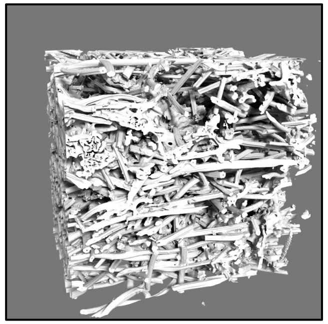
Overview of materials for hypersonic entry

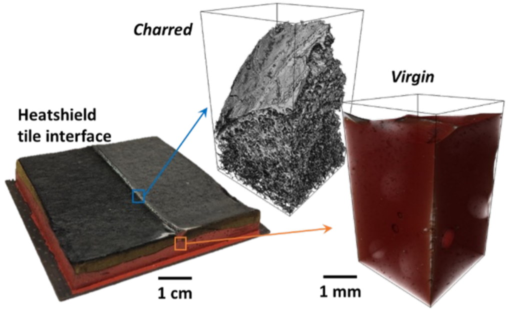
- For atmospheric entry, a sacrificial "ablative" material is used as a heatshield:
  - Carbon or silicon-based composites that endothermically pyrolyze and prevent heat from entering the inner structure.
- NASA has typically used tiled heatshields of PICA or Avcoat, bonded with Room Temperature Vulcanizing silicone (RTV560) gap-filler.
- RTV560:
  - Initially non-porous solid that pyrolyzes, becomes porous and swells as it is heated.
  - As it swells, it can cause transition of flow from laminar to turbulent and increase heating on the heatshield.


Mars Science Laboratory (MSL) heatshield with tiled PICA bonded by RTV560 [1].

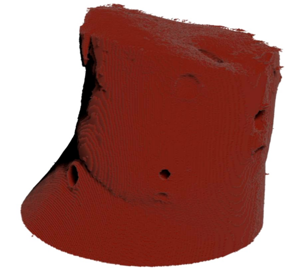

SpaceX Dragon heatshield with tiled C-PICA [Credits: SpaceX].


Artemis I heatshield made of Avcoat with RTV560 gap filler [Credits: NASA].


Tomography of carbon fibers in PICA [Credits: NASA].



Tiled heatshield showing virgin and charred RTV. [2]


In-situ tomography of RTV showing swelling and shrinking.

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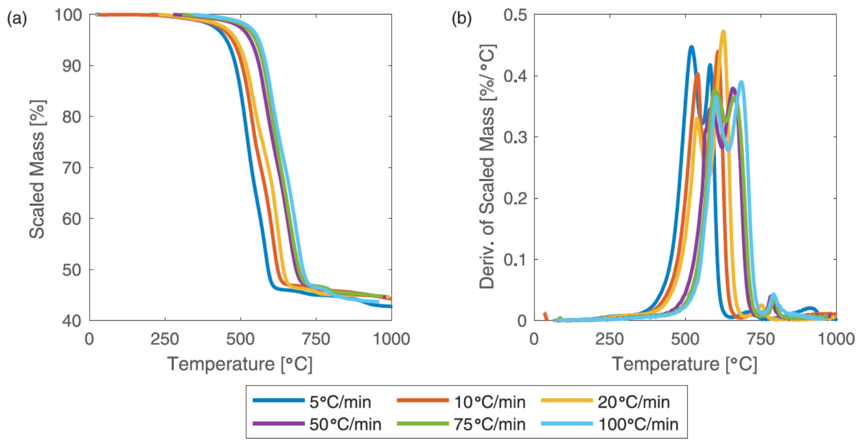
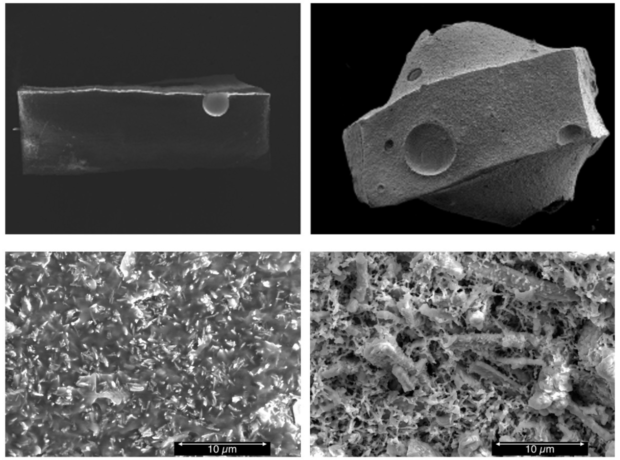
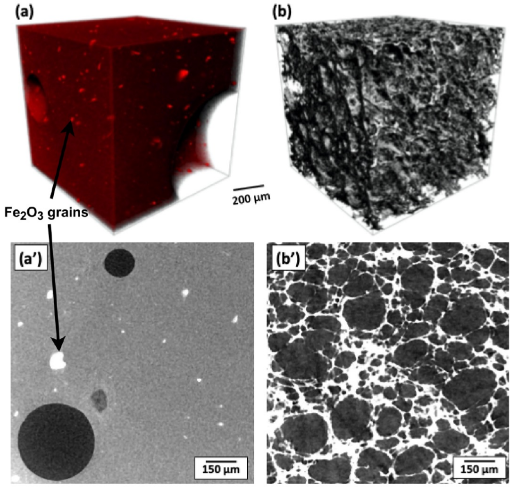
RTV 560 Pyrolysis and Intumescence

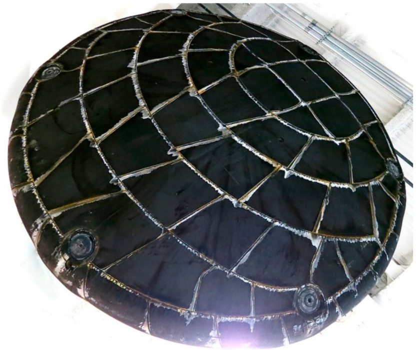
- RTV560:
  - Silicone elastomer with Fe_2O_3 grains and diatomaceous Earth,
  - Loses a significant amount of mass and changes in porosity and volume drastically [2].







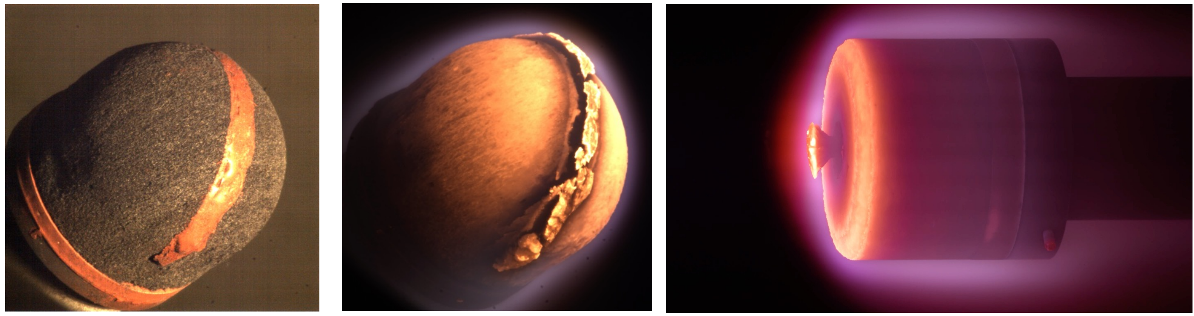
Courtesy of Space X



Post-flight Dragon

Courtesy of NASA Ames

RTV swelling in TPS [3].



RTV tested at increasing heat fluxes.

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RTV 560 Pyrolysis and Intumescence

- RTV560:
  - Silicone elastomer with Fe_2O_3 grains and diatomaceous Earth,
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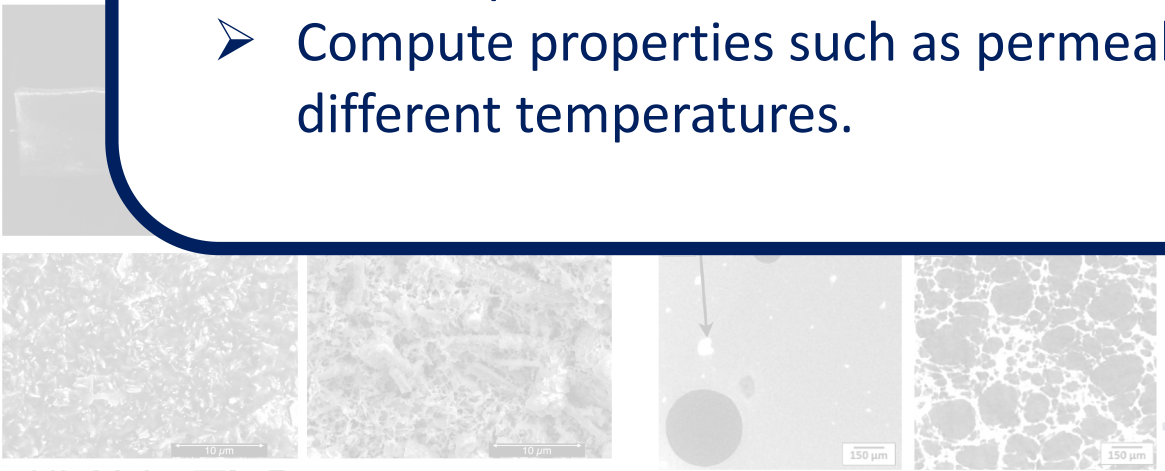
Courtesy of Space X

Courtesy of NASA Ames

Goal of current work:

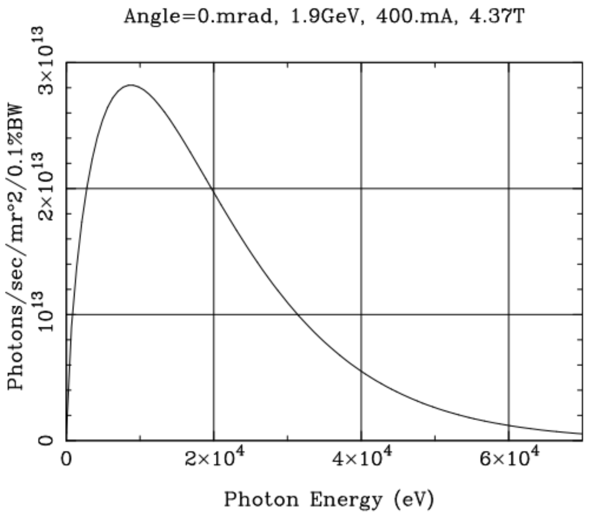
- Determine microstructure change of RTV as function of temperature using MicroCT at the Advanced Light Source,
- Obtain quantitative estimates for macrostructural change,
- Compute properties such as permeability, thermal conductivity, etc. at different temperatures.

S [3].



RTV tested at increasing heat fluxes.

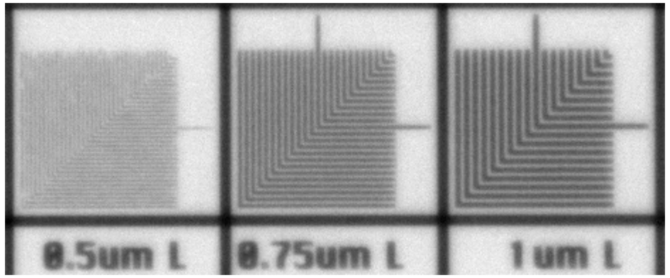
Tomography at ALS, Beamline 8.3.2



X-ray flux at different energies at Beamline 8.3.2.

	PCO Edge	**(2560x2160) [Optique Peter]**
Lens	pixels size (um)	FOV (mm)
20x	- [0.33]	- [0.8]
10x	0.65 [0.69]	1.7 [1.7]
5x [4x]	1.3 [1.72]	3.3 [4.4]
2x	3.25 [3.44]	8.3 [8.8]
1x	6.5 [-]	16.6 [-]

Different lens options available for tomography.



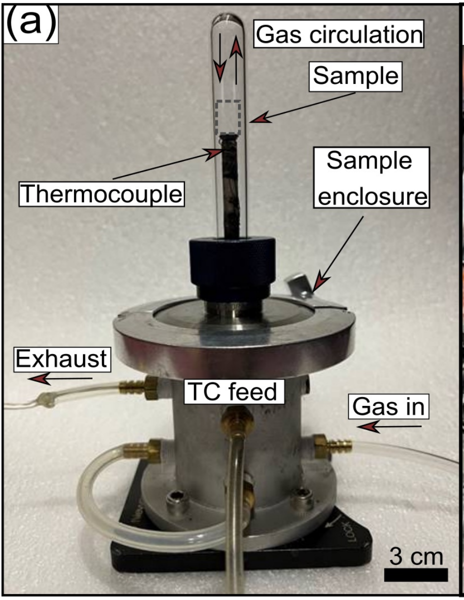
Finest resolution scan (~0.5 micron) with 20x lens.

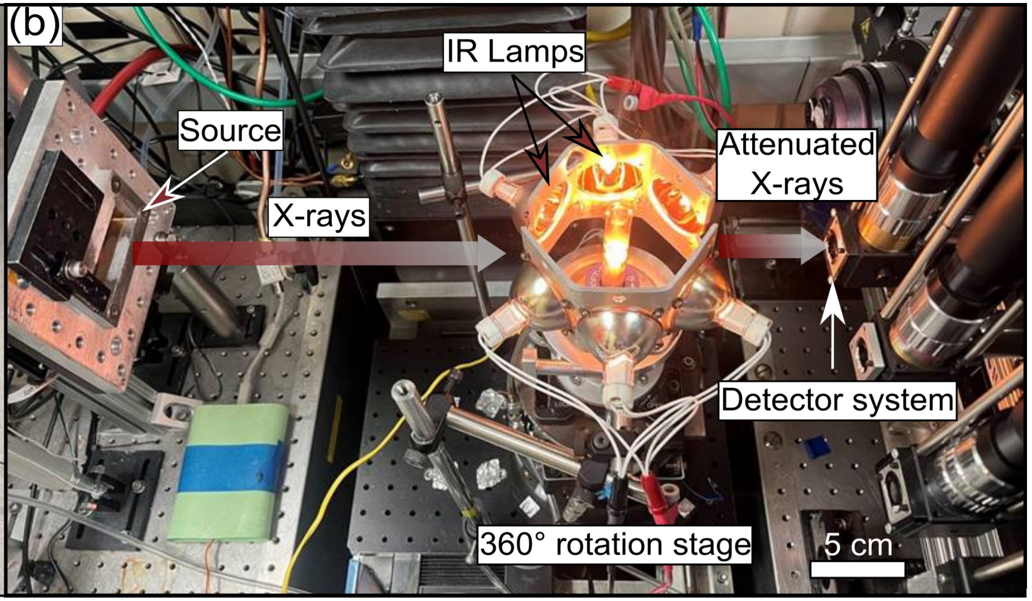
Synchrotron μ-CT of Astronaut, *Courtesy of Dula Parkinson*

X-ray system	Resolution [μm/px]	FOV, $\varnothing$[mm]	Time/tomography	Capabilities
Xradia Bio-MCT	0.9 – 10	1.5 – 10	> 2 hours	*ex-situ*
ALS 8.3.2 Beamline	0.6 – 10	1.7 – 27	30 s – 30 min	*in situ*

Pyrolysis setup for RTV [4,5]

- RTV samples are heated by IR lamps and scans are collected every 90 seconds.



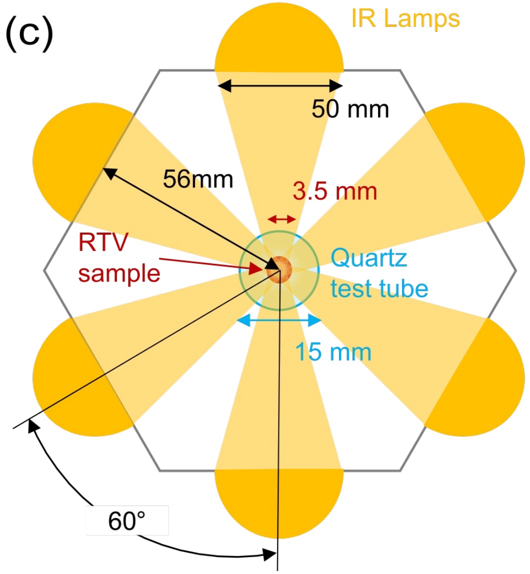


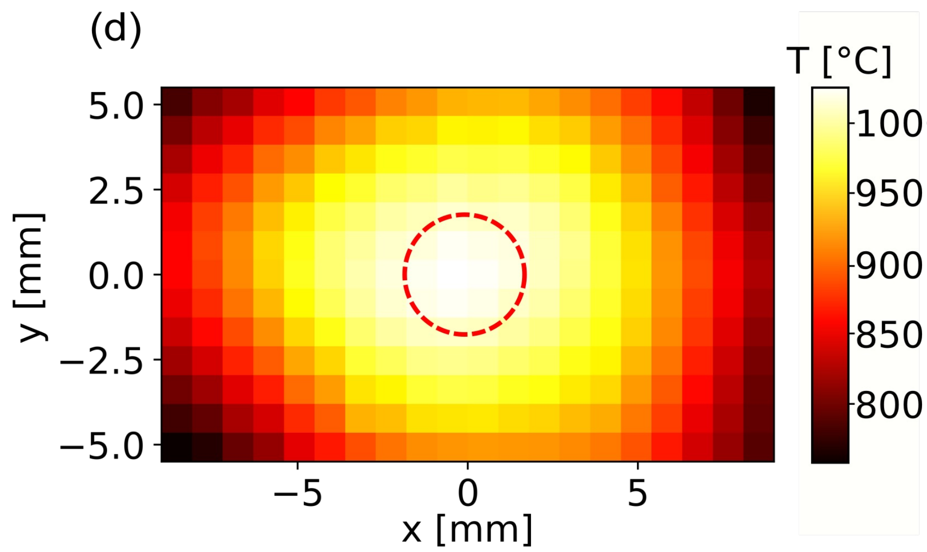
(a) Environmental chamber

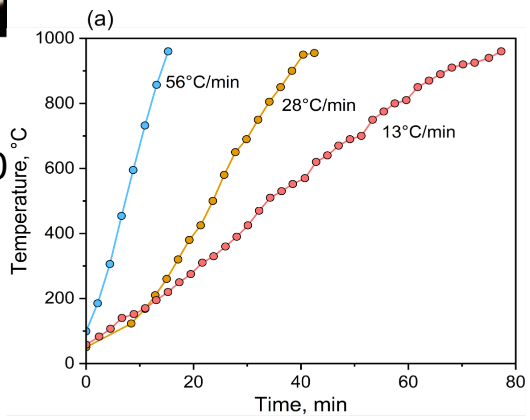
(b) Inside μ-CT hutch at ALS

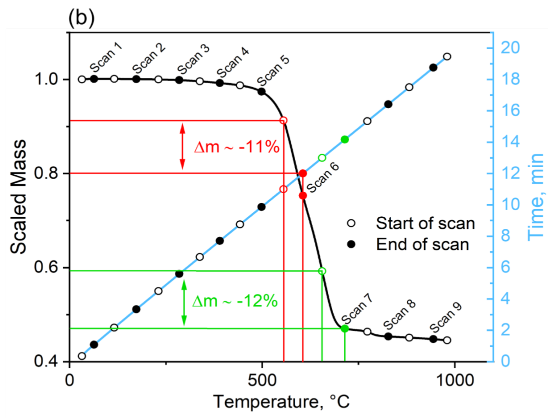
(c) Lamp schematic

(d) Spatial temperature variation

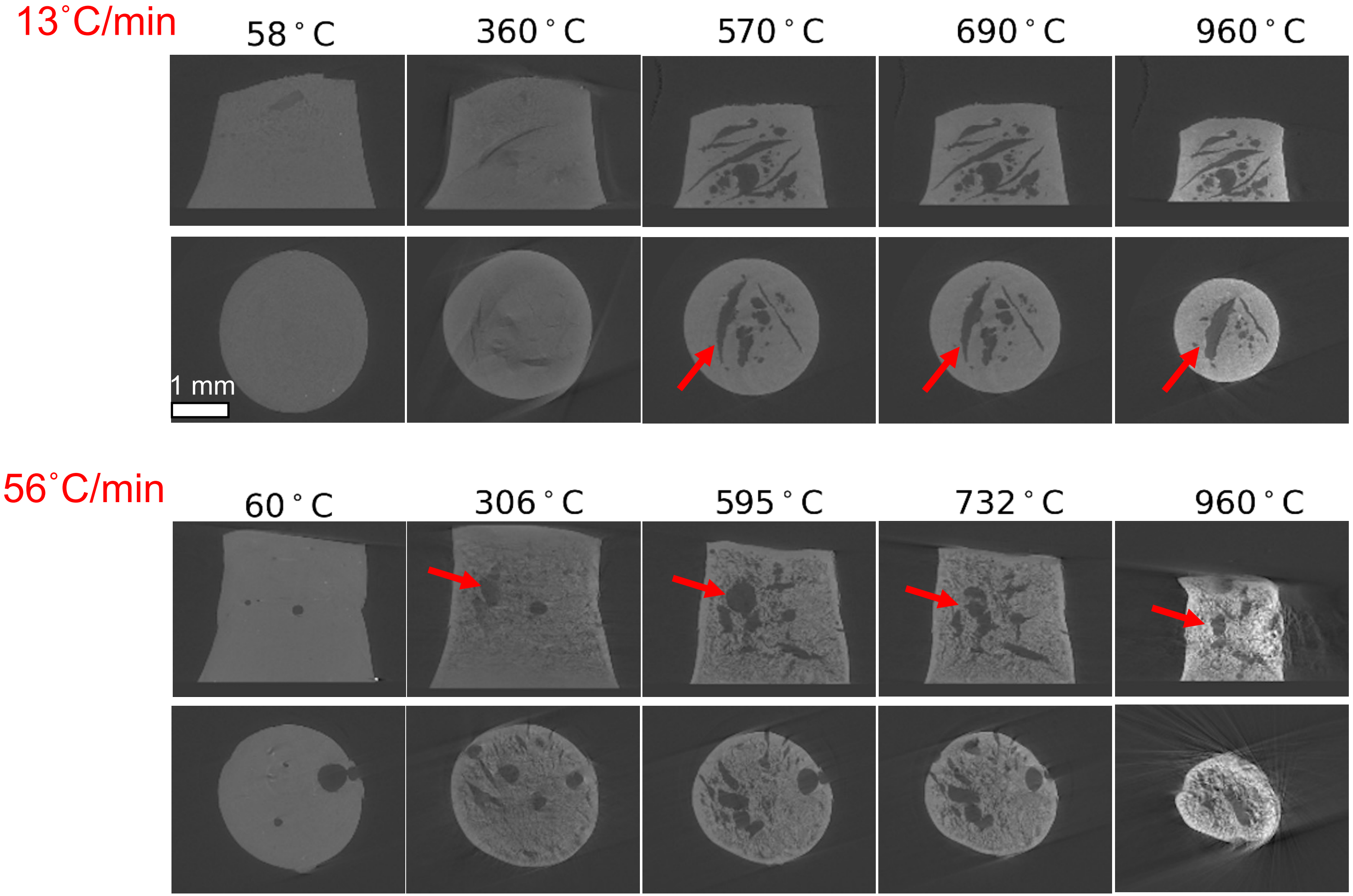


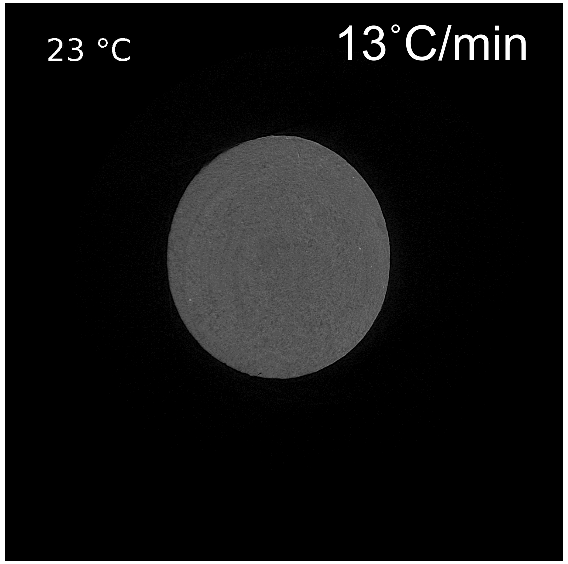


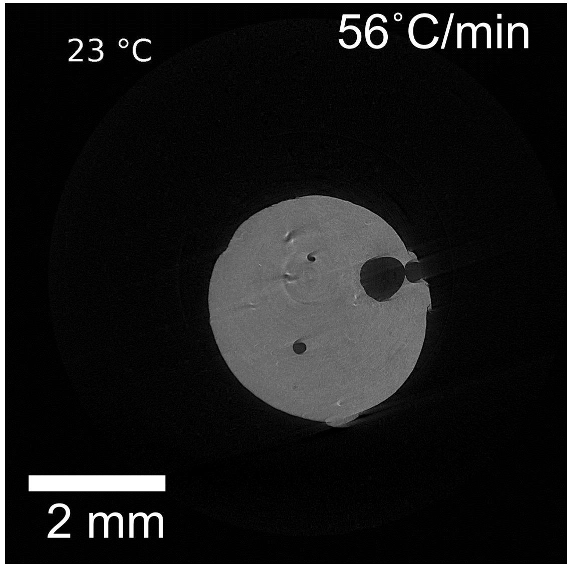




RTV Pyrolysis







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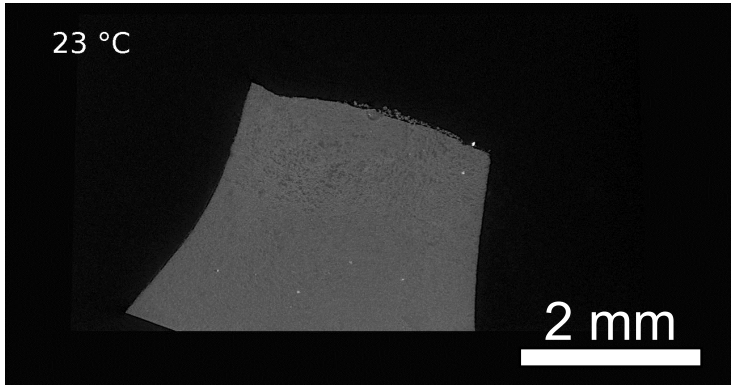
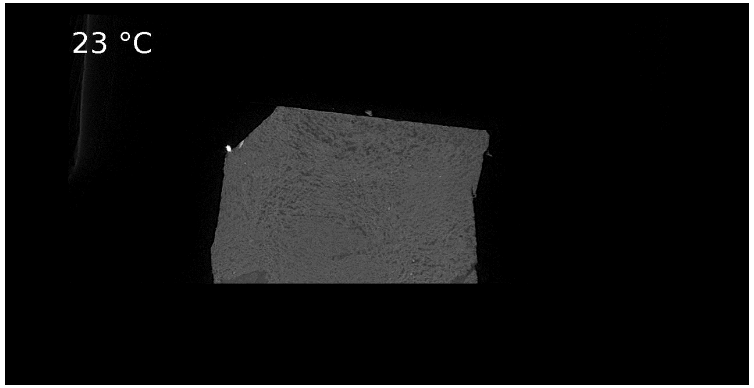
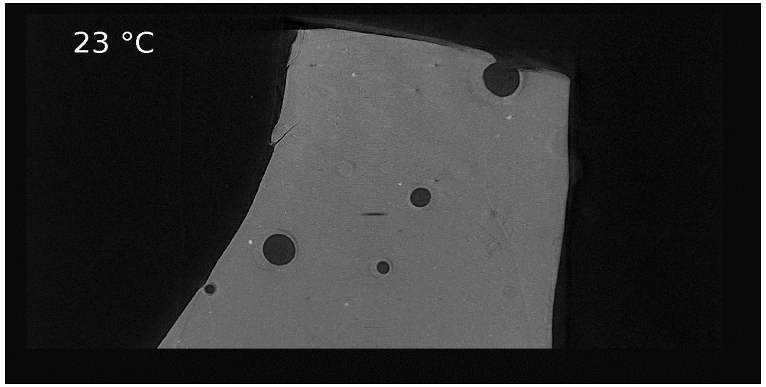
Quantitative estimates for morphological evolution



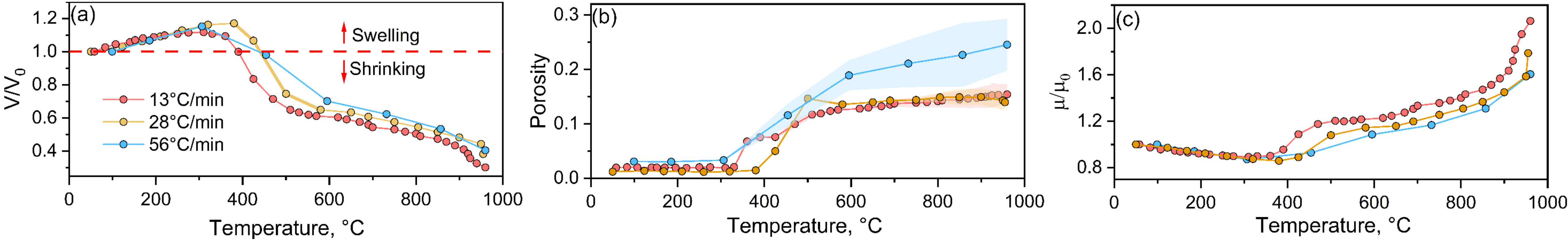
13˚C/min



28˚C/min

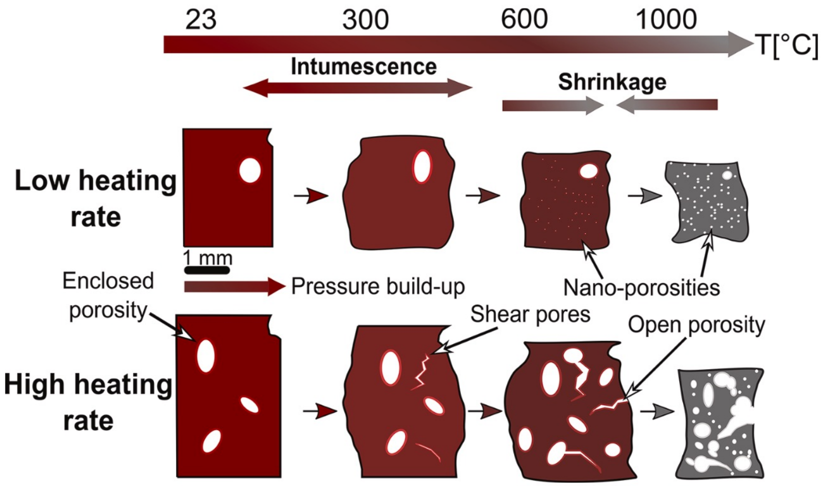


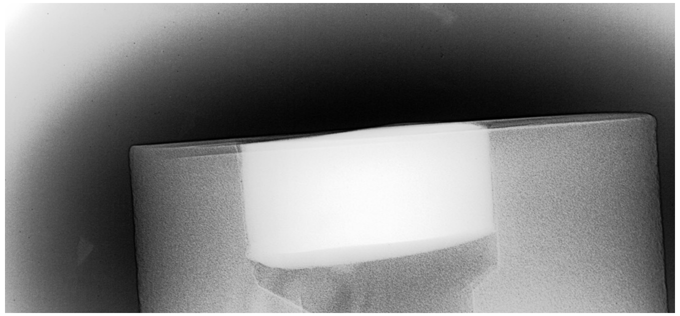
56˚C/min



Exploring higher heating rates with Radiography

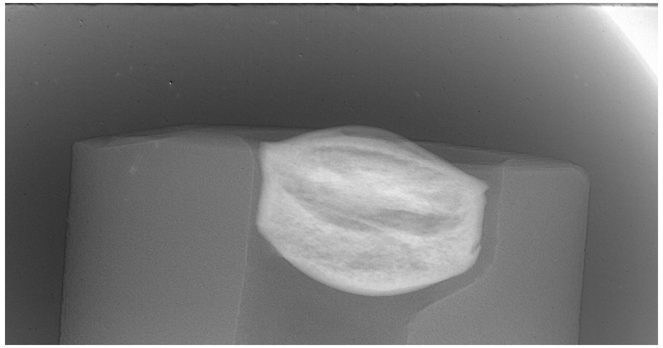
- Tomographies indicated heating-rate dependent behavior
- But do not show swelling to the extent seen in plasma wind tunnel tests
  - Heating rates for in-situ tests are significantly lower than for wind tunnel tests/flight.
  - To test at much higher heating rates, radiography was used.



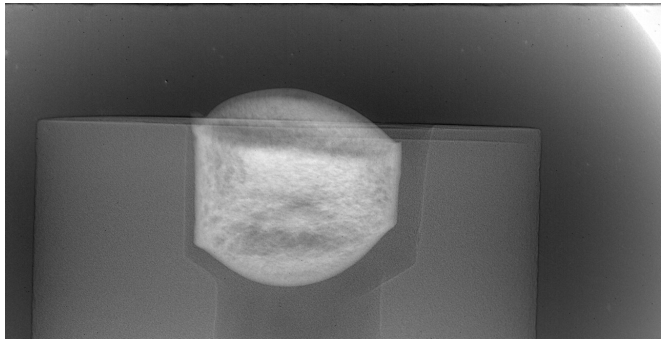


Initial radiograph of RTV sample before high-heating rate tests

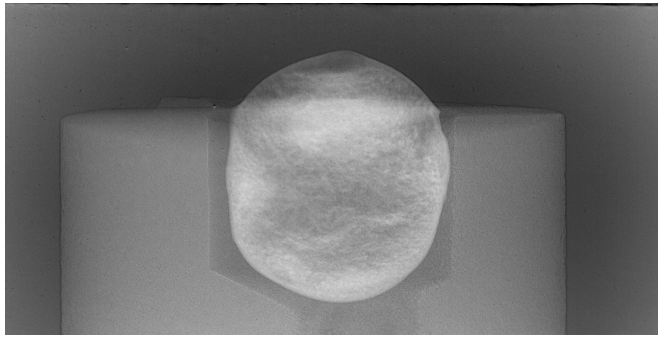
100 C/min



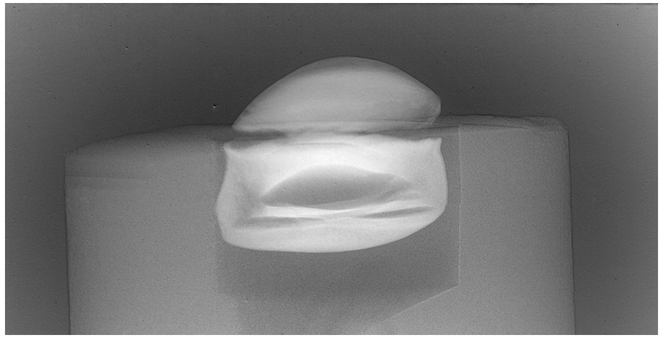
200 C/min



600 C/min



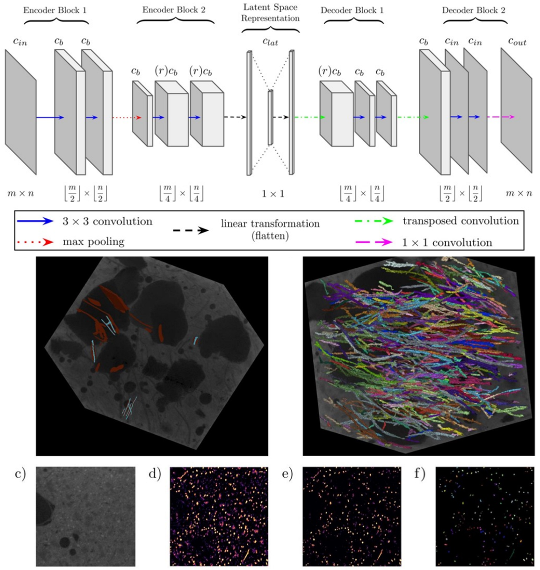
1600 C/min



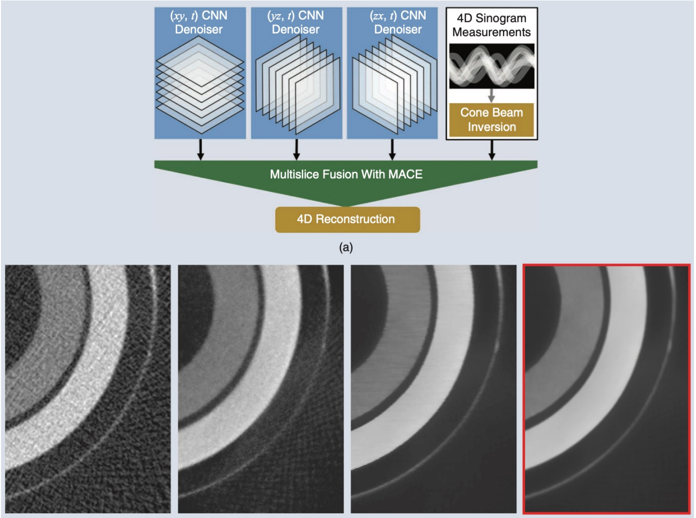
Conclusion and Future Work

- In-situ pyrolysis of RTV was performed and quantitative determination of morphologic showed:
  - A large change in RTV's porosity and permeability with increase in temperature,
  - Extent of swelling and shrinking is a function of heating rate.
- Future Work:

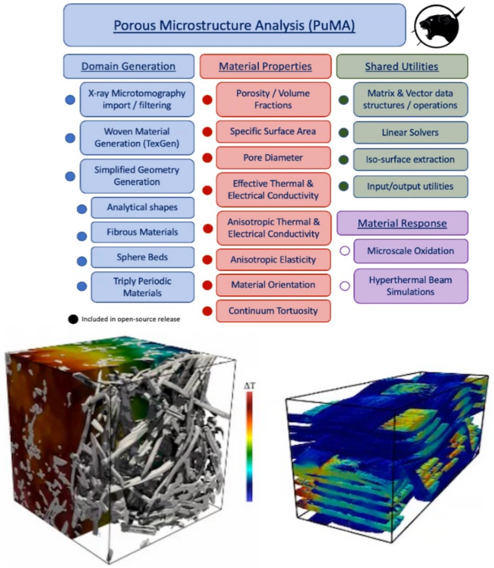
High-fidelity Segmentation with DLSIA



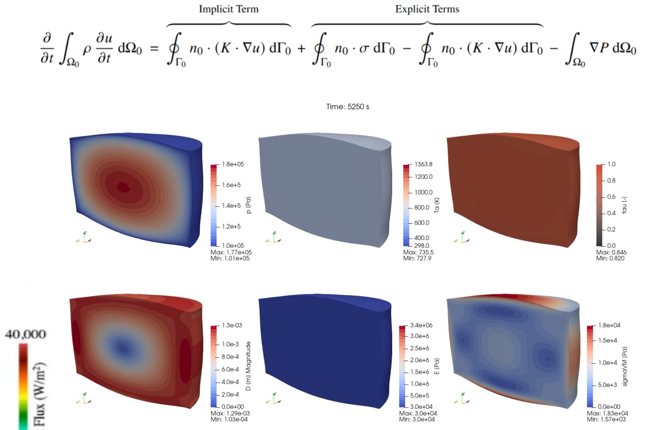
Increased temporal resolution with MBIR



Microscale properties with PuMA



Macroscale simulations with PATO



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Thank you! Any questions or suggestions?