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Pyrolysis Under Extreme Heat Flux Characterized by Mass Loss and Three-Dimensional Scans

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With **A.L. Brown**; Sandia National Laboratories

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Outline

- Introduction and motivation
- Literature Review
- Solar Furnace & Experimental Methods
- Mass loss, crater size, & 3D scans.
- Discussion
- Conclusions

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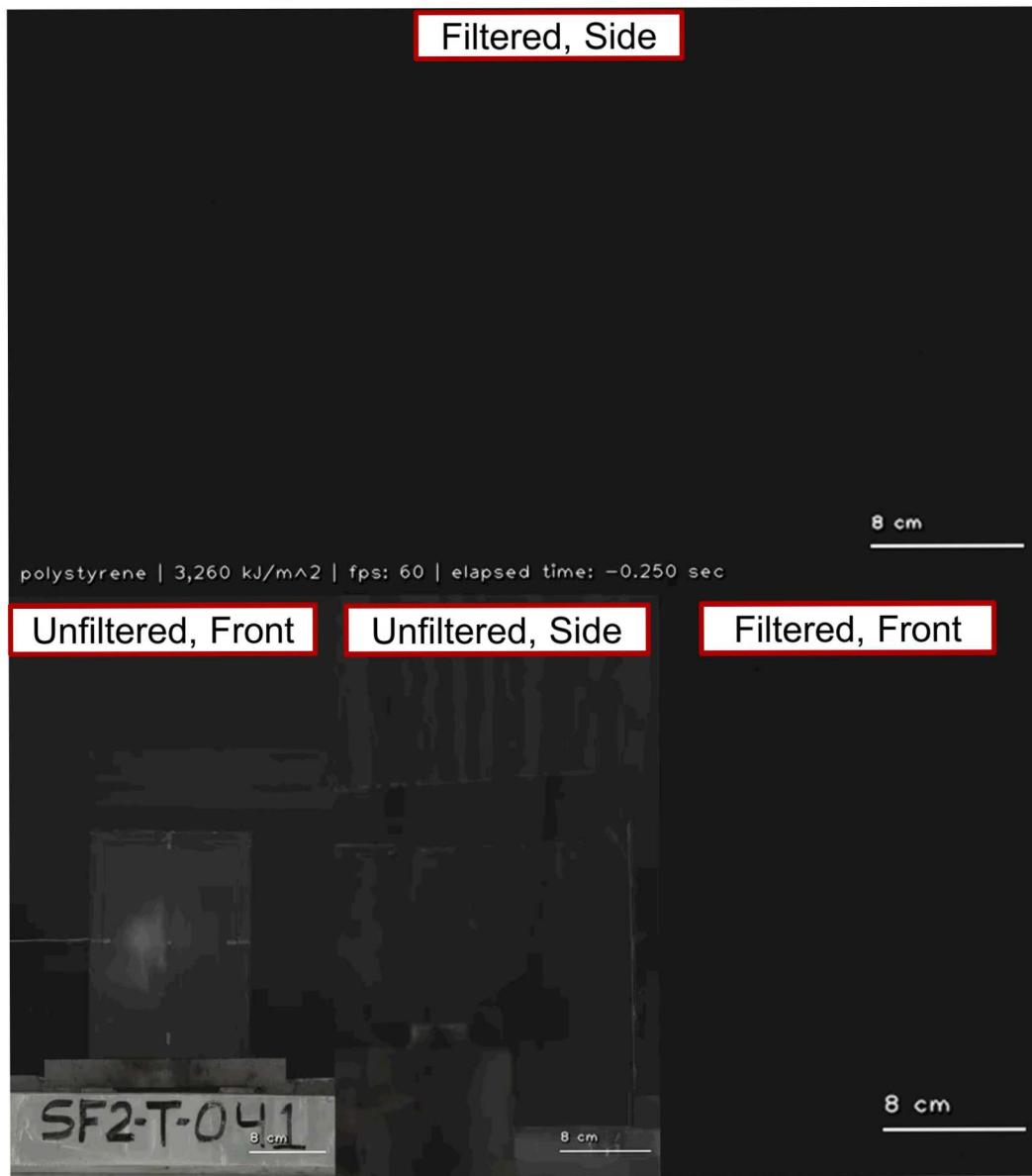
Introduction and Motivation

- A variety of sources can produce heat flux well beyond those typical of fire environments:
 - Directed Energy Weapons
 - Nuclear Weapons
 - Explosives
 - Propellants
- At extreme ($\sim 1 \text{ MW/m}^2$) heat flux, the incident energy dominates the surface energy balance
 - Radiation ($\sim 100 \text{ kW/m}^2$) and convection ($\sim 10 \text{ kW/m}^2$) are relatively small even when the surface reaches ignition temperatures ($\approx 600 \text{ }^\circ\text{C}$)
- **Objective:** Improve experimental characterization through **quantitative image analysis** and **surface topology** of samples after exposure to intense thermal irradiation.

Response to Extreme Irradiation

- Under extreme irradiation ($\sim 0.1 - 1 \text{ MW/m}^2$), a material can:
 - Pyrolyze strongly as the surface chars and/or recedes.
 - The material chemically decomposes, producing combustible gases.
 - Ignite by a variety of mechanisms:
 - Transient Flaming
 - Sustained Flaming
 - Sustained Smoldering
 - As well as other responses:
 - Melt, spall, char, exfoliate, etc.

Example: Polystyrene



Material:

High-Impact Polystyrene

3.2 mm thick

Exposure:
3.26 MJ/m²

Ignition Data:

Sustained Flaming

Ign. time: 0.85 s

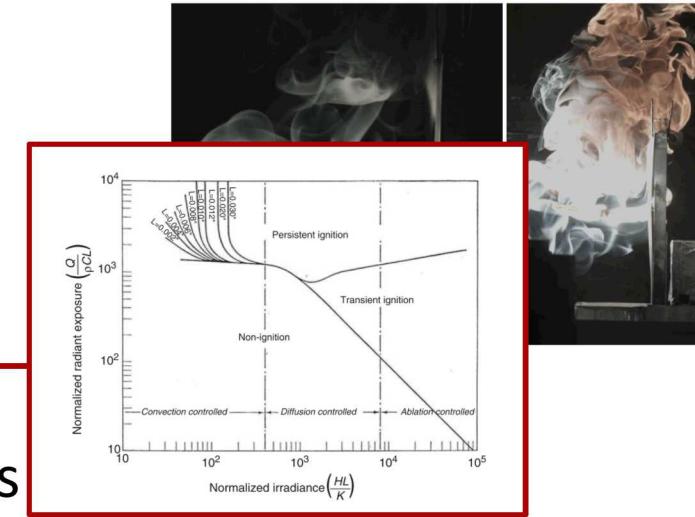
Pyr. time: 0.38 s

Programmatic Objectives

- This study is part of a wider program, **focusing on pyrolysis and ignition** at extreme radiative heat flux.
 - **Experimental** data at small and large scale
 - Solar Furnace (\approx 10 cm spot)
 - Solar Tower (\approx 1 m spot)
 - **Simple ignition models** (e.g., empirical correlations) that can predict material response to a given environment.
 - **High-fidelity computational models** that capture the complex physics and accurately predict pyrolysis and ignition.
- These advancements will produce **ignition models** spanning a wide range of **environments** and **materials**.

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Literature Review

- Most data at extreme heat flux from three sources:
 - Nuclear Testing
 - Real environment, large scale
 - Poorly Characterized
 - Glasstone
 - Expansive reference table
 - Citations not listed, qualitative information
 - Martin
 - Rigorously characterized
 - Narrow focus (black alpha-cellulose papers)
- Previous research focuses on **categorical data (*ignition*)**.
 - Supports underlying objective: **simple empirical ignition models**.
 - **Categorical data is inferior for high-fidelity computational models!**

Sample Analysis in Prev. Literature

Glasstone & Dolan:

- Minimal sample characterization
- Broad range of materials.
- **Qualitative** response
- *Only ign. threshold is quantitative.*

Material	Weight (oz/yd ²)	Color	Effect on Material	Radiant Exposure*		
				35 kilotons	1.4 megatons	20 megatons
CLOTHING FABRICS						
Cotton	8	White	Ignites	32	48	85
		Khaki	Tears on flexing	17	27	34
		Khaki	Ignites	20	30	39
		Olive	Tears on flexing	9	14	21
		Olive	Ignites	14	19	21
		Dark blue	Tears on flexing	11	14	17
		Dark blue	Ignites	14	19	21
Cotton corduroy	8	Brown	Ignites	11	16	22
Cotton denim, new	10	Blue	Ignites	12	27	44

Martin et. al.:

- Well-characterized samples.
- Mostly black cellulose.
- **Categorical** response.
- *Only ign. threshold is quantitative.*

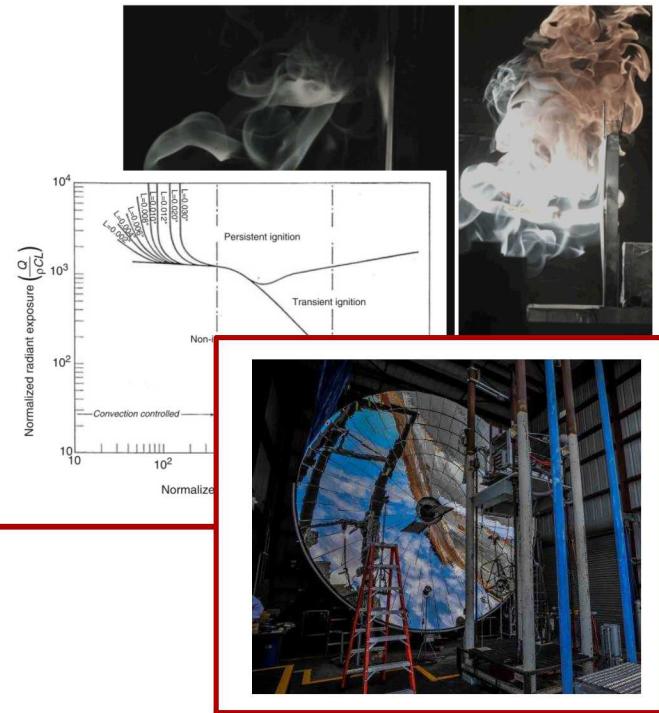
Ignition Data for 0.7 g cm⁻³ Nominal Density Material

Thickness and Physical Properties	Effect	Peak Irradiance cal cm ⁻² sec ⁻¹	Time to Peak (±s.d.) sec	Radiant Exposure cal cm ⁻²
Manufacture No. 4096 L = 31 mil = 0.0795 cm ρ = 0.69 g cm ⁻³ c = 0.35 cal deg ⁻¹ g ⁻¹ K = 2.2 × 10 ⁻⁴ cal cm ⁻² deg ⁻¹ sec ⁻¹	Flame	21.9 18.3 16.4 15.1 14.6 13.1 10.4 10.2	0.30 0.35 0.38 0.40 0.39 0.41 0.83 (±0.02) 0.85	13.5 13.4 12.9 12.4 11.4 11.2 17.9 18.0
	Transient Flame	15.1 14.6 13.7	0.29 0.26 0.40	9.1 7.9 11.4
	Glow	7.1	1.45	21.3

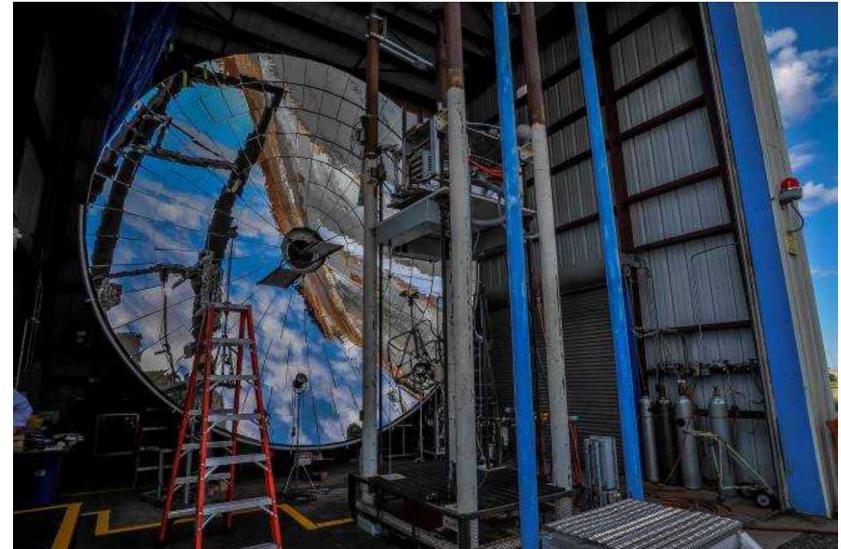
Simple data is appropriate for simple models. Need better data for high-fidelity models (gas-phase dominant ignition).

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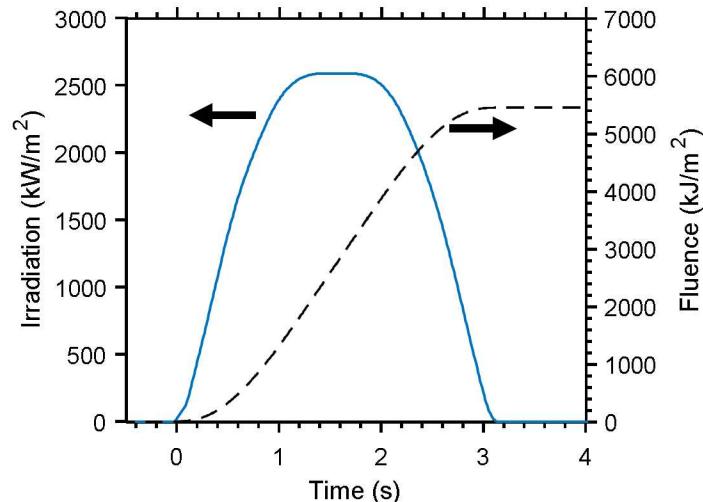
Solar Furnace



- Heliostat tracks sun
- Parabolic dish focuses light
- Attenuator controls temporal flux profile
- 3-axis table positions sample/instrumentation
- Generates heat flux of up to 6 MW/m^2 on a $\approx 8 \text{ cm}$ spot

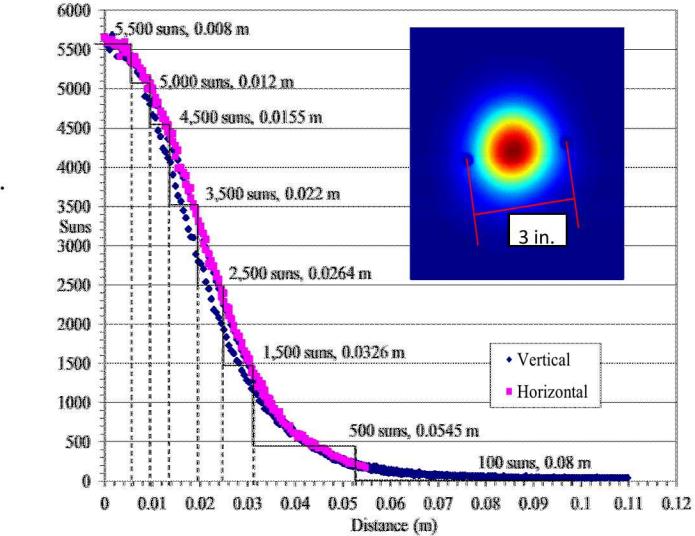
Solar Furnace Environment

- Temporal Profile
 - Ramp up/down.
 - Limited by attenuator speed.



- Spatial Profile
 - Characterized in previous paper.
 - Ho, C. K. et.al., Proc. Int. Conf. on Energy Sustain. 4 (2010) 1-9.

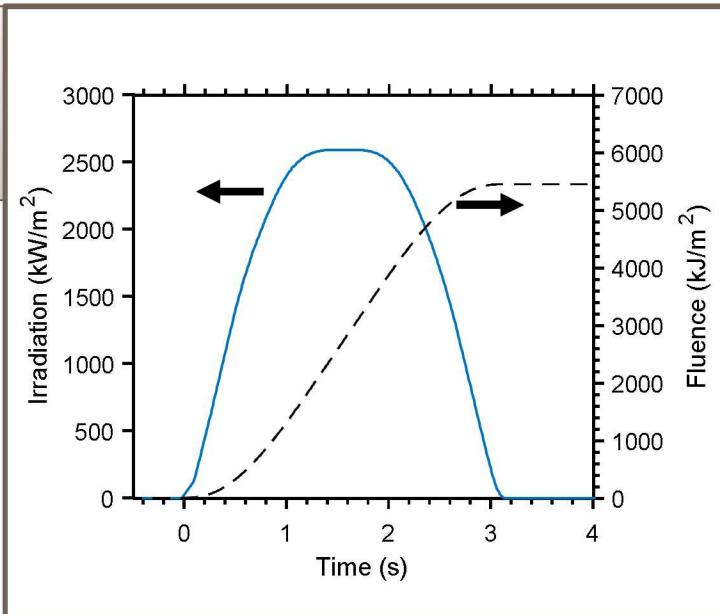
$$q''(t, r) = \frac{q''_o(t)}{1 + \exp\left(C \left(\frac{R_0}{R_1} r\right)^D - B\right)}$$



- Both distributions complicate data analysis!

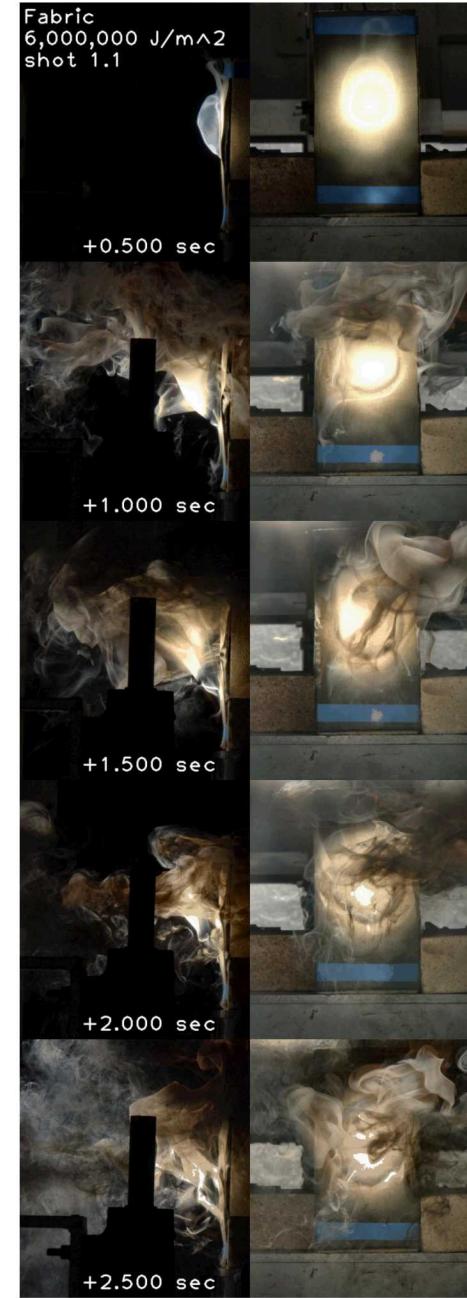
Instrumentation

- Radiometer and heat-flux gauge quantify flux/fluence
- Cameras capture material response
- Photographs taken before and after
- Mass recorded before and after
- 3D Scans after



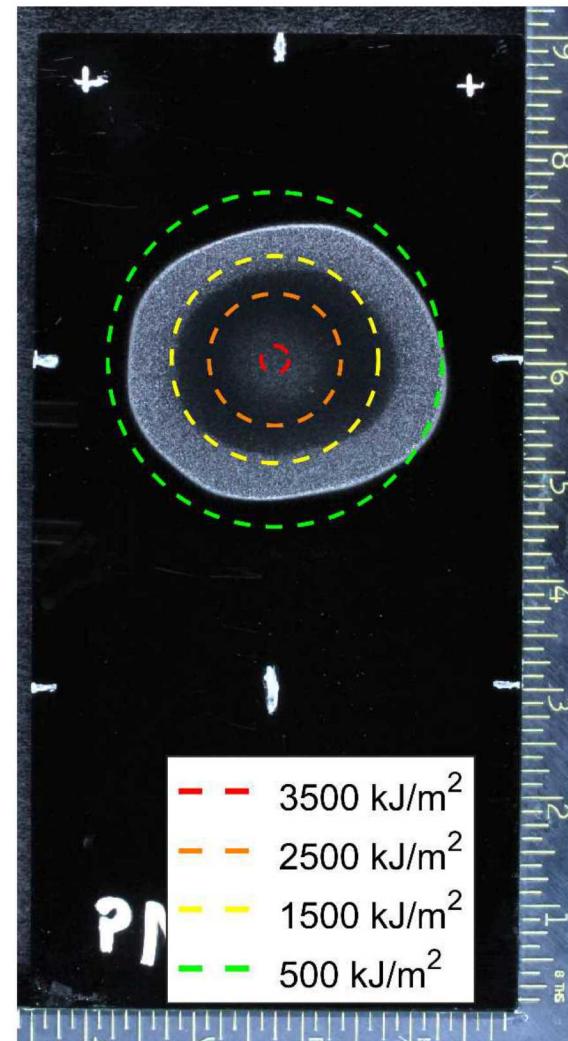
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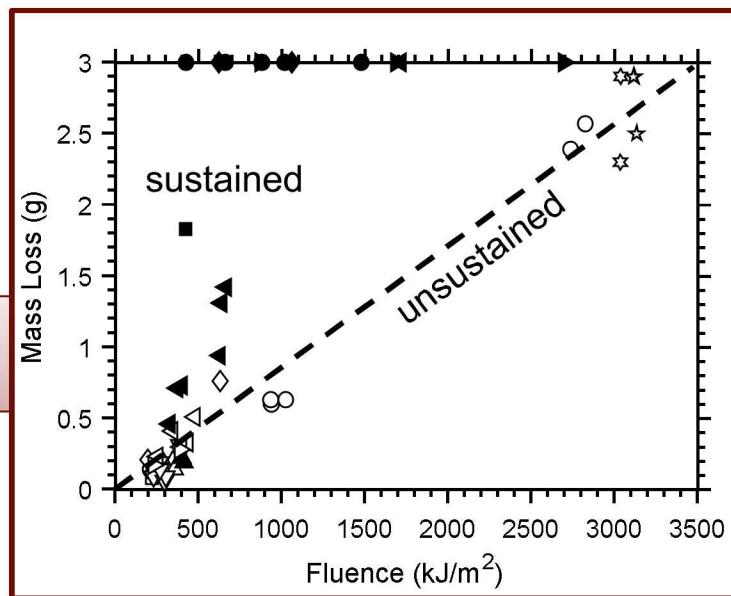
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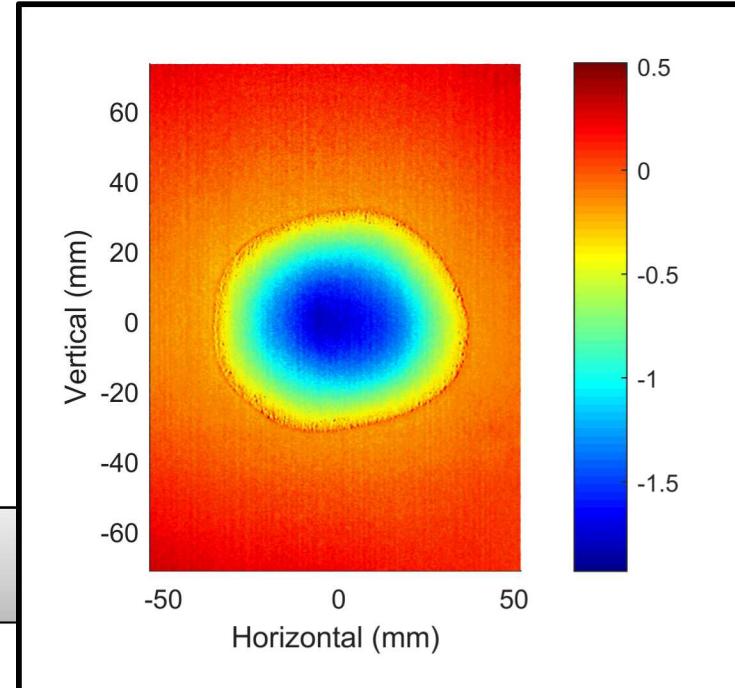
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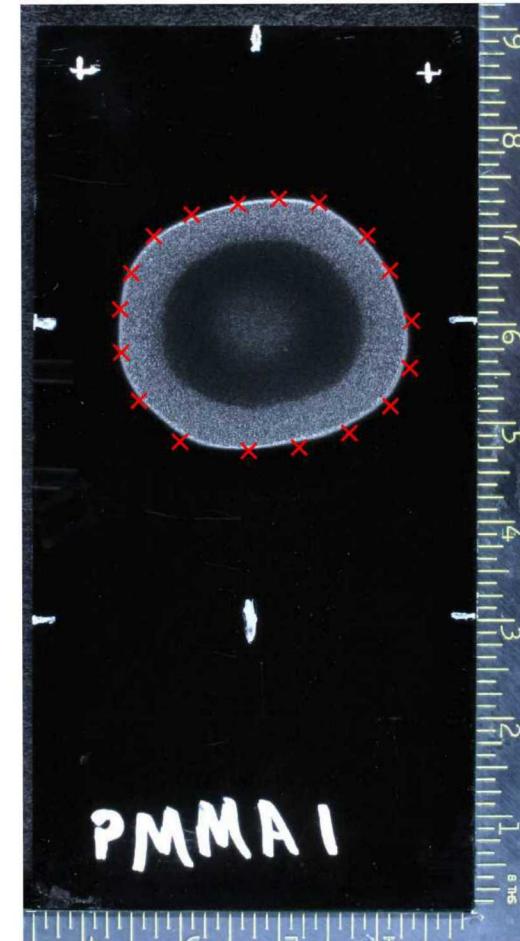


Enhanced Post-Test Analysis

- Better, quantitative empirical data through:
 - Ignition thresholds (see our previous papers)
 - Similar to past data.

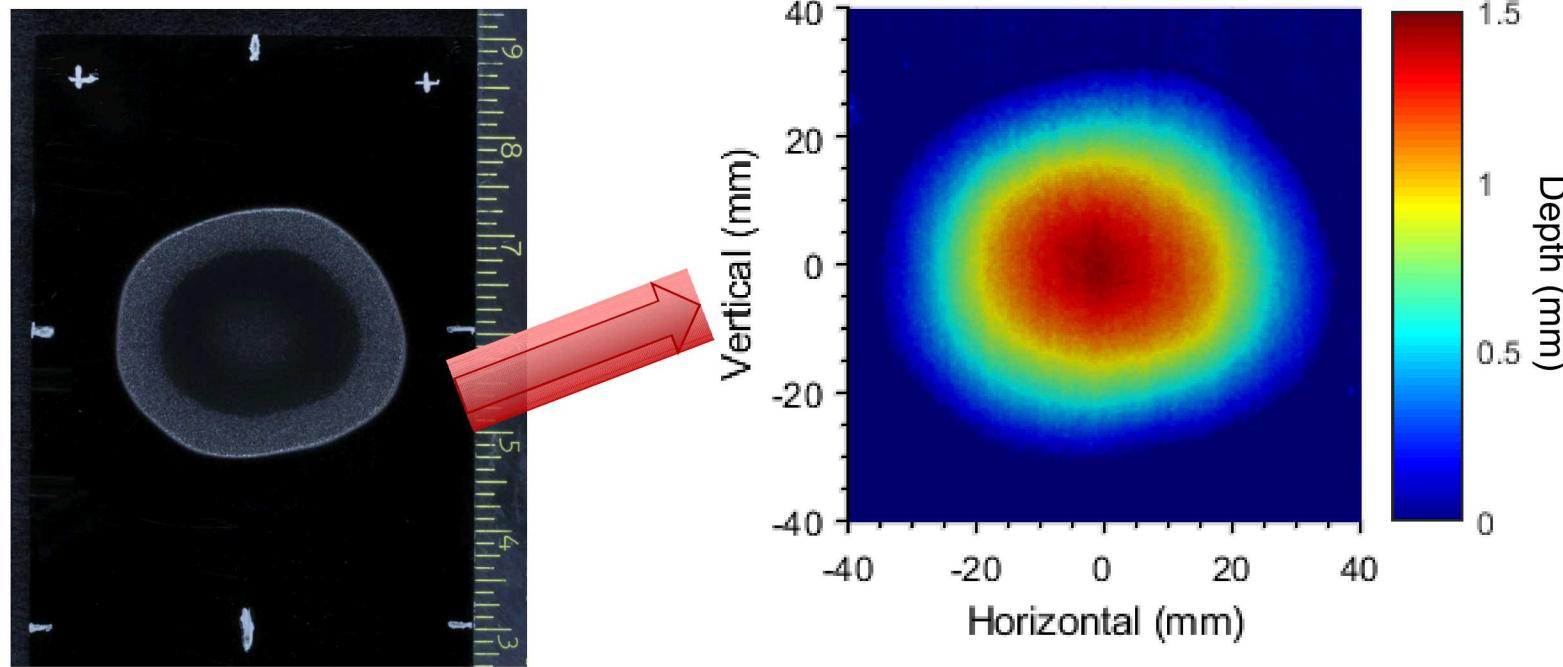
- Mass-loss data
- Photographic data
 - Quantitative analyses including:
 - Char area.
 - Heat flux at crater rim.
 - Net energy delivered to crater.
- 3D Scanner data
 - Quantitative analyses, similar to above.
 - Spatially resolves surface recession.
 - Resolves 2D mass-loss map (non-charring materials)
 - Maps mass loss to heat flux.

Newly Implemented Analysis



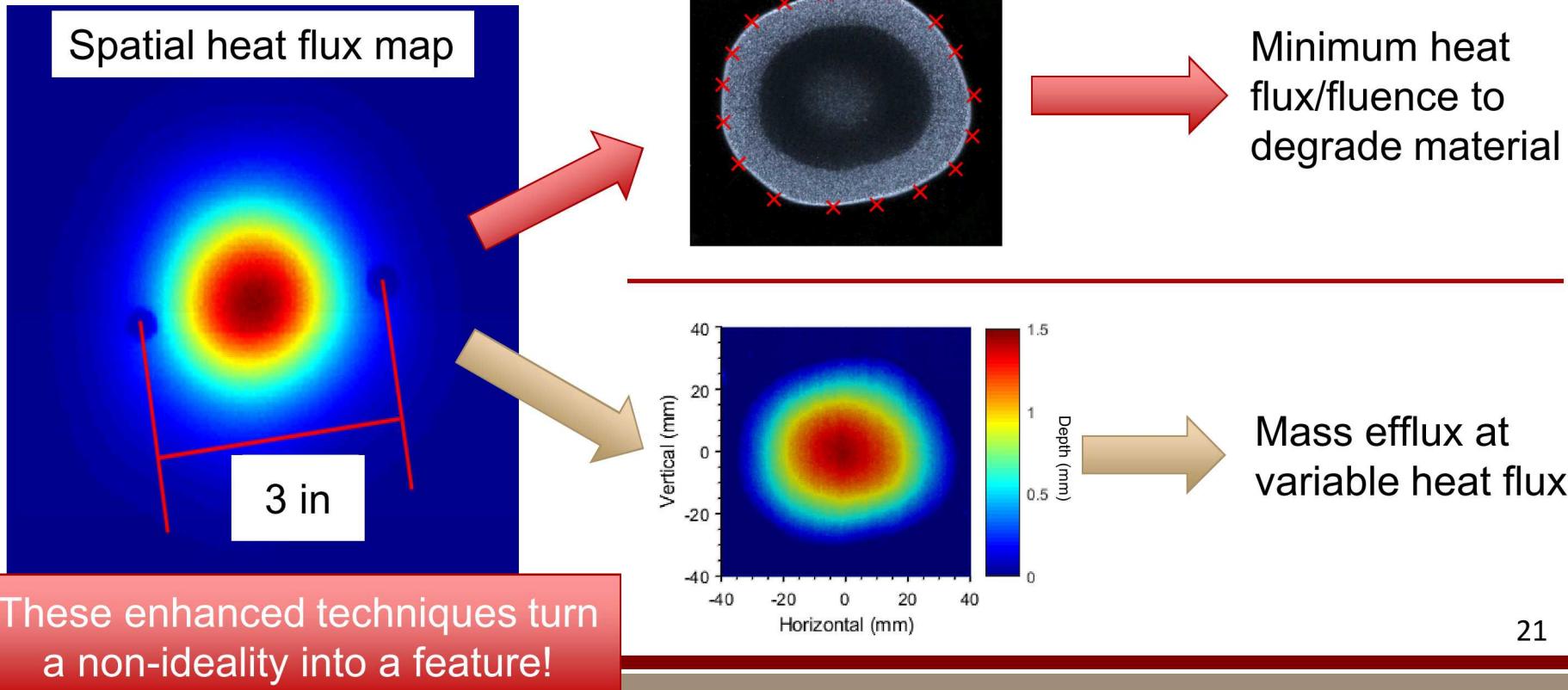
3D Scanner Technique

- 3D Scanner most valuable if surface receded (unstable char)
 - Technique presented in previous paper. (Engerer & Brown, AIAA 2018-3761)
 - Scanner spatially resolves surface recession.
 - Localized mass loss calculated from surface recession
 - Assumption: Residual material density unchanged.



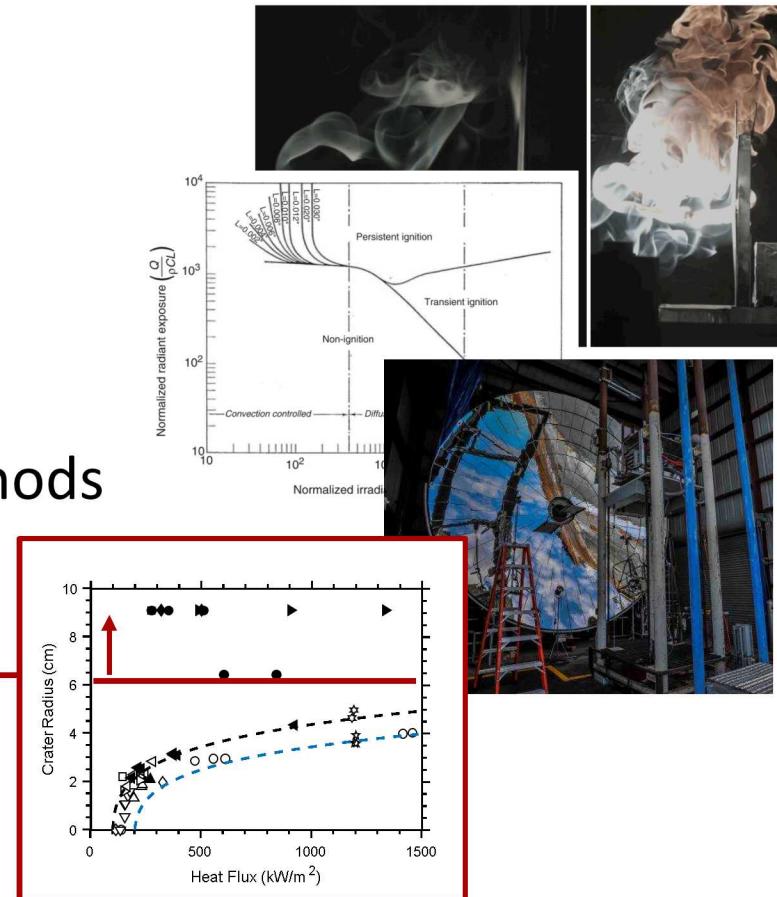
Mapping Data to Heat Flux

- Spatially distributed heat flux complicates analysis.
- However, sample exposed to a variety of heat flux conditions.
 - If deconvolve the problem, more data than in uniform heating case.



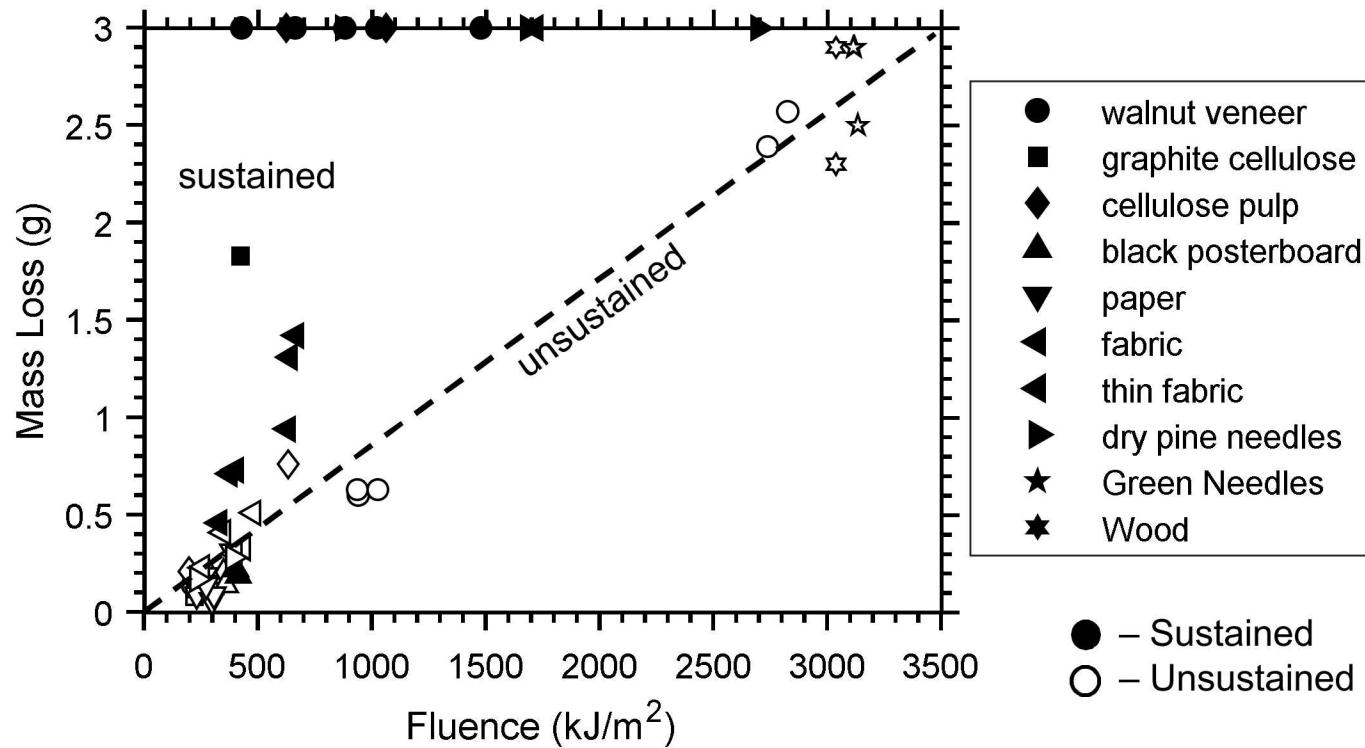
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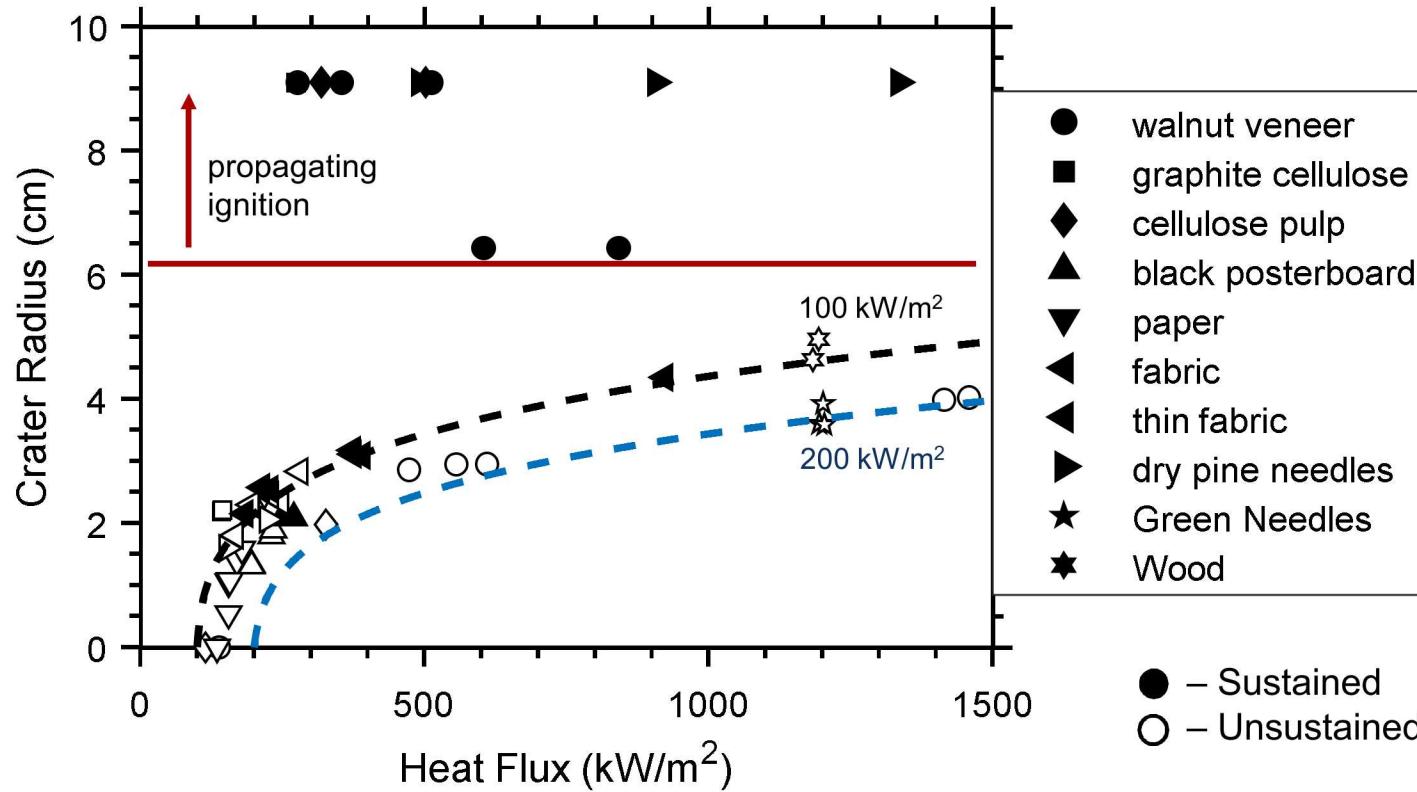
Cellulosic Materials

- Mass loss for cellulosic materials are consistent.
 - Mass loss linear with fluence for unsustained ignition.
 - Mass loss higher for sustained ignition cases.
 - Data are coupled with other effects (e.g., increasing degraded area).



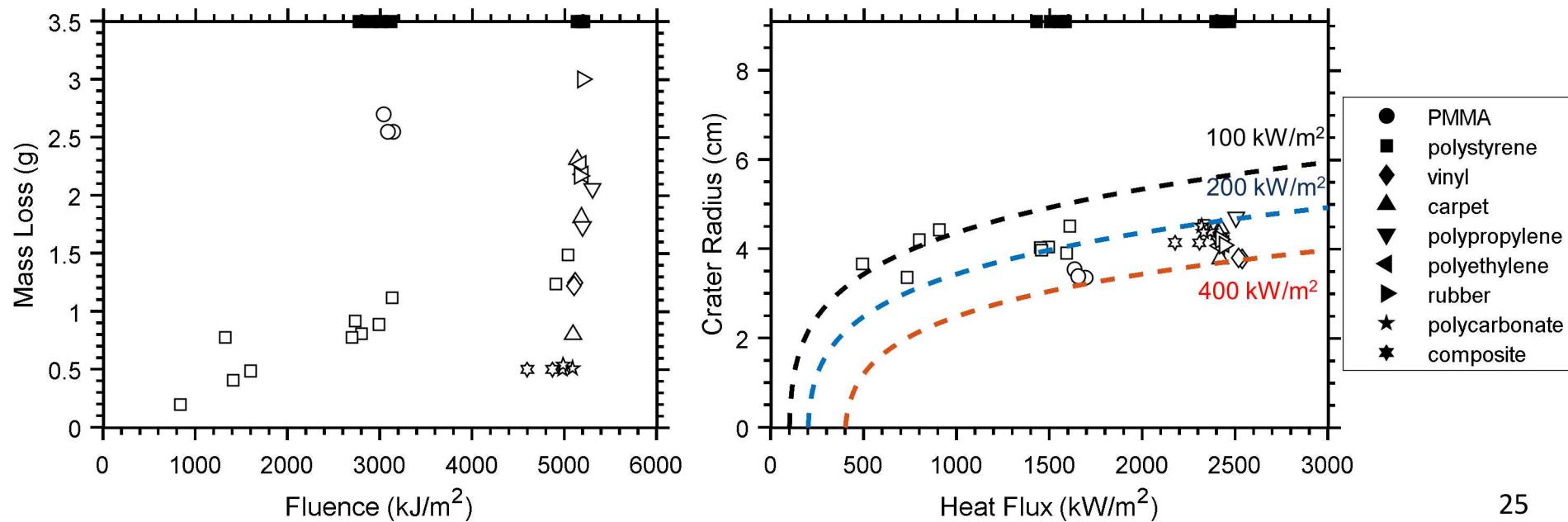
Cellulosic Materials

- Crater radius determined from post-test images.
 - Char extent predicted by heat flux at perimeter.
 - Propagating ignitions consume upper-half or all of the sample.



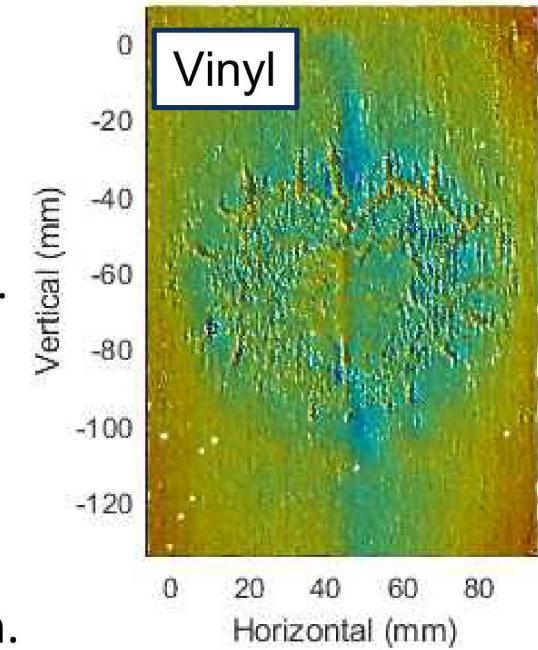
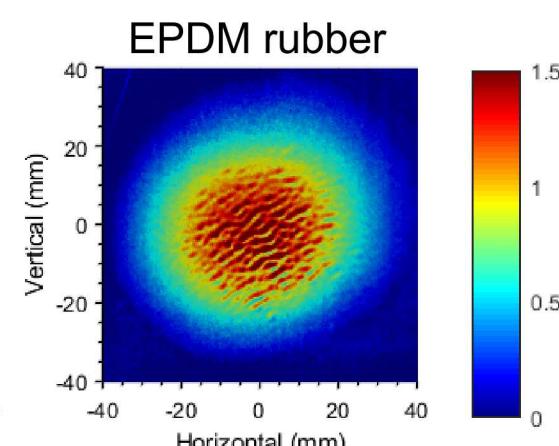
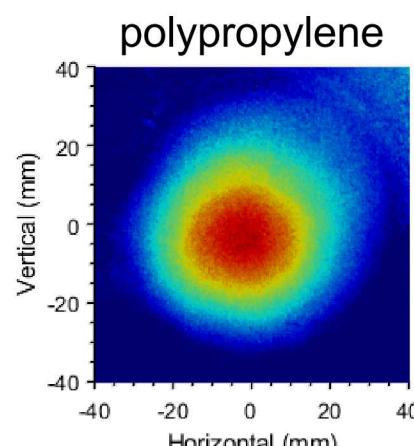
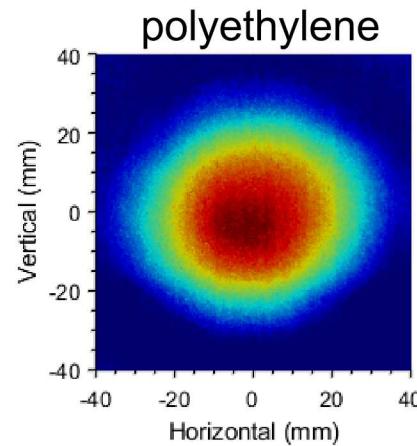
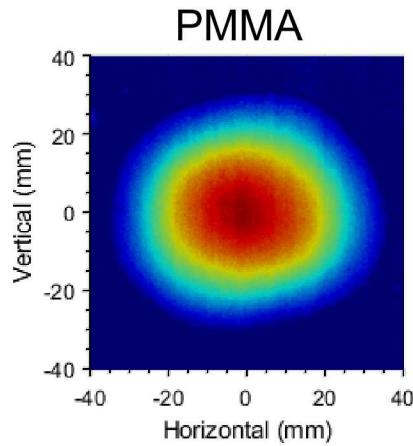
Synthetic Polymers

- Mass loss for synthetic polymers are less consistent.
 - Chemical composition varies
 - Heat flux was varied only for polystyrene experiments.
 - Mass loss fairly linear.
 - Crater radius consistent across various exposure magnitudes.
 - Physics more complex? (e.g., screening of radiant energy?)



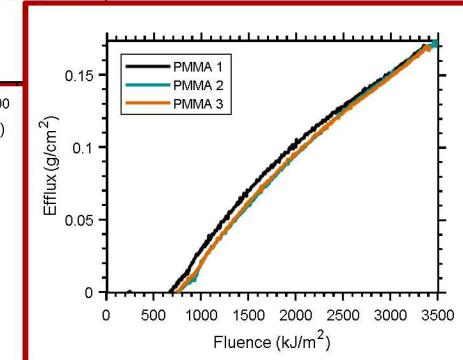
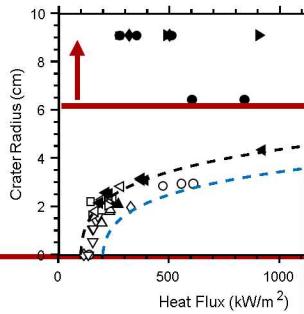
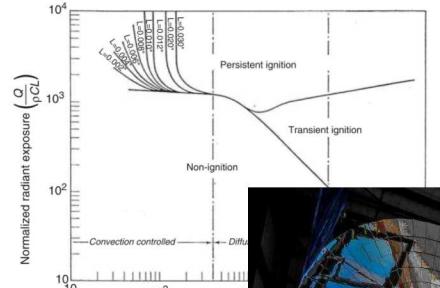
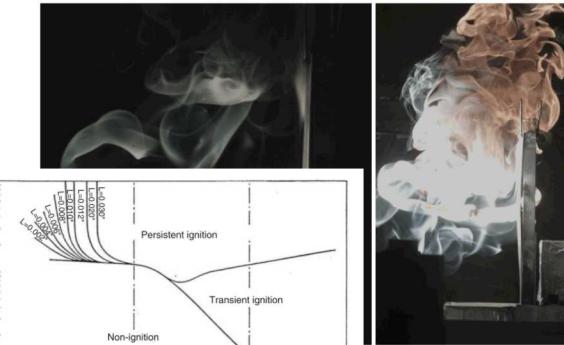
3D Scanner Results

- Among materials with significant thickness:
 - Stable Chars inhibited surface recession.
 - Data at/near resolution of scanner; lacking trends.
 - Examples:
 - walnut veneer
 - epoxy/fiber composite
 - polycarbonate
 - vinyl
 - Without stable char, significant surface recession.



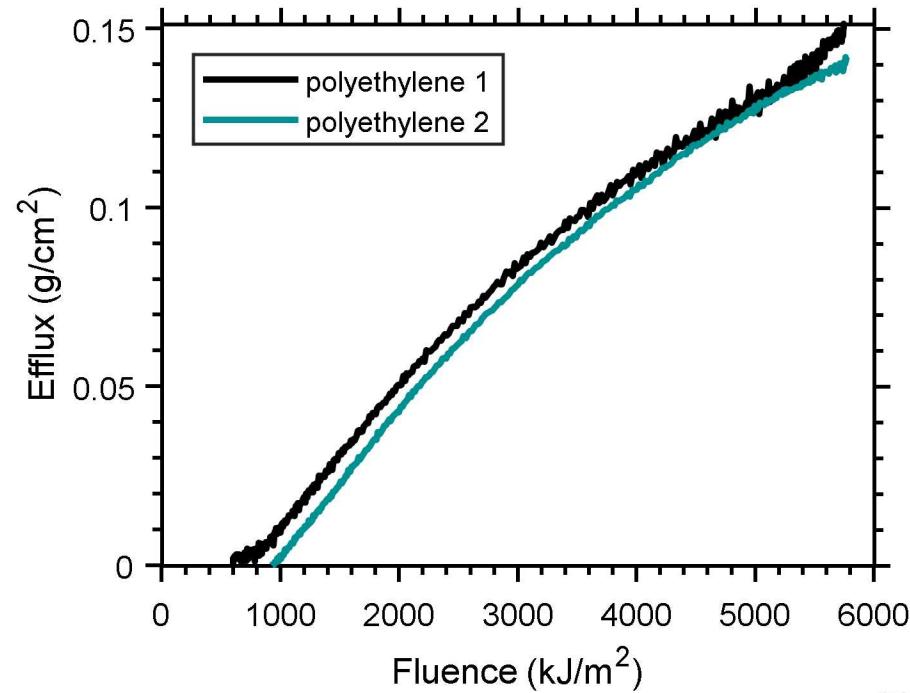
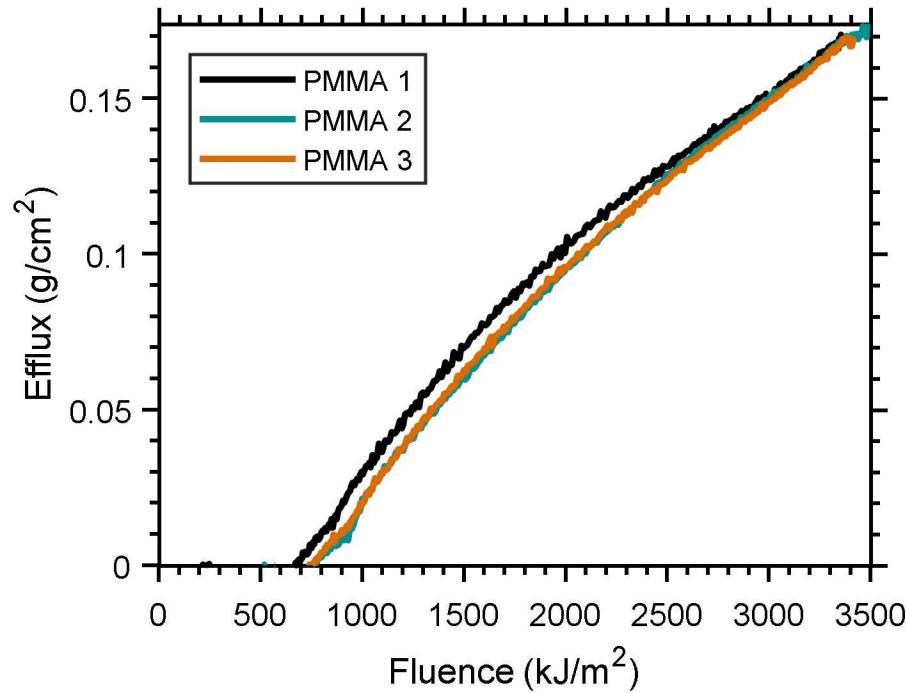
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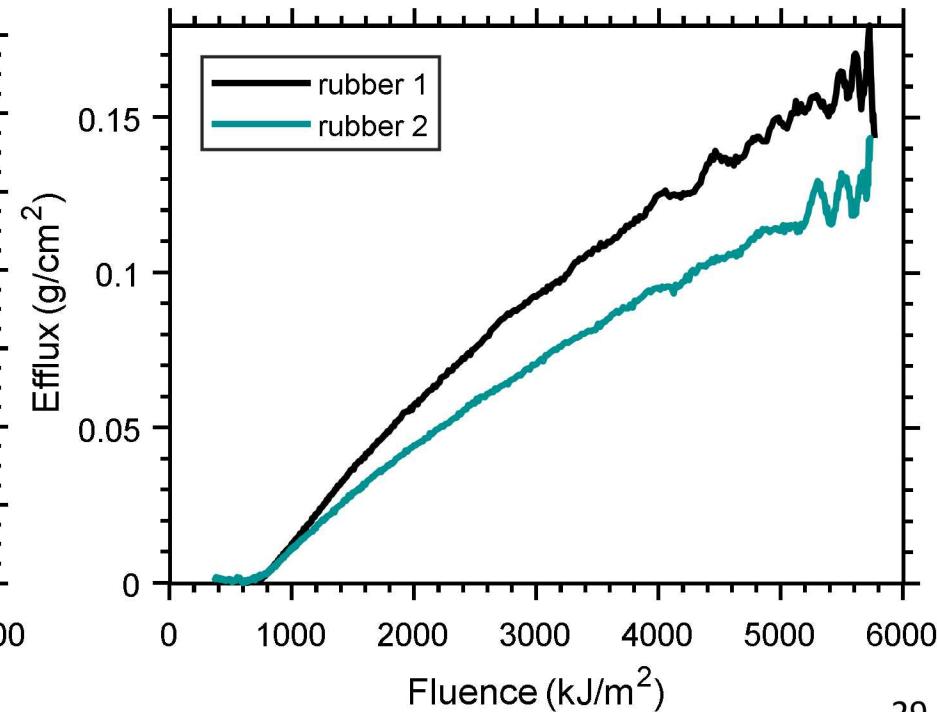
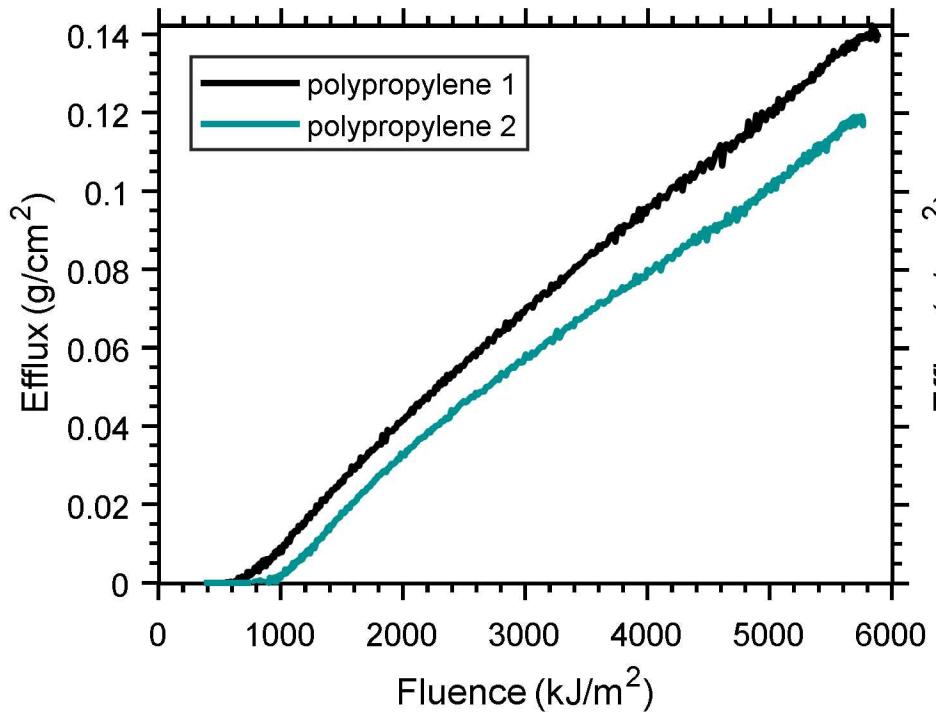
Spatially Resolved Mass Loss

- Mass loss calculated from crater depth.
 - Assumption: Density unchanged by exposure (no residual char/swelling)
- Localized efflux approximately linear with fluence.



Spatially Resolved Mass Loss

- Polypropylene and rubber samples show variations, despite having same nominal exposure level.
 - Perhaps, radiation screening from pyrolyzate.



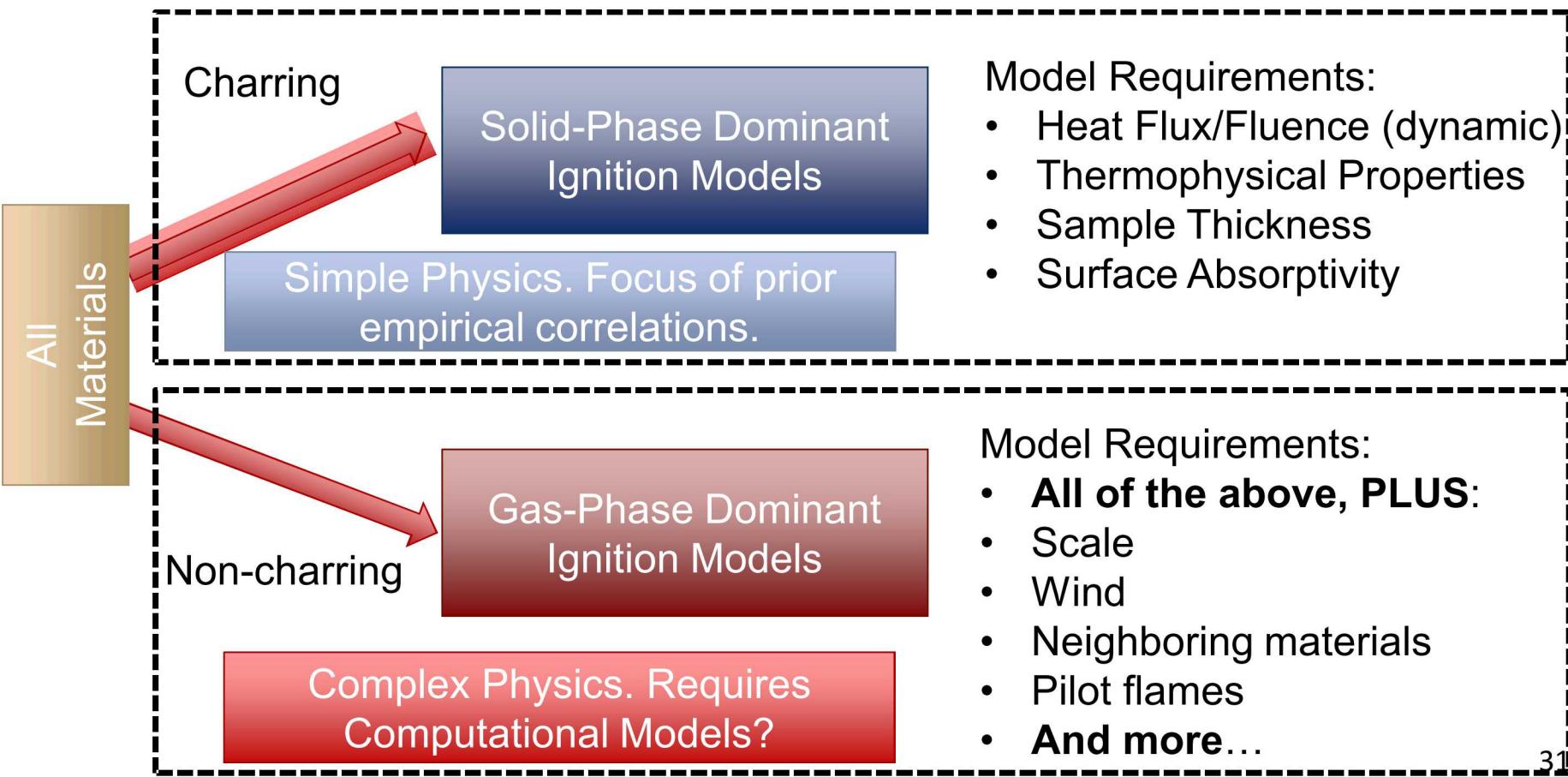
Radiation Screening

- Many polymers produced large pyrolyze clouds.
- Example:
 - Polypropylene



Extreme Heat Flux Models

- We theorize ignition models determined from the presence or absence of a char (exceptions likely exist).



Conclusions

- Radially distributed heat flux complicates data analysis, but in many cases enhances the end data.
- Quantitative image analysis provides estimates:
 - Heat flux required to degrade sample.
 - Area / total energy contributing to pyrolysis reaction.
- 3D Scans better characterize sample response:
 - Recession depths.
 - Mapping localized efflux to heat flux.
- Data will contribute to model development for gas-phase dominant ignition.
 - Solid-phase dominant ignition typically well-predicted by empirical models (Engerer, et al., AIAA 2018-3764).
 - Gas-phase dominant ignition requires further development.

Acknowledgements

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