SAND2012-2216 C

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The Behavior of Carbon Fiber-Epoxy Based Aircraft Composite Materials in Unmitigated Fires

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The 2012 Meeting of the Western States Section of the Combustion Institute
March 19-20, 2012, Tempe, AZ, USA

Presentation 12S-23

Fire Science & Technology

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Broad Objectives

1. **Perform testing to determine composite decomposition characteristics to aid in defining fire tests for transportation safety analysis.**
2. **Create data that can be used to develop and validate computational models capable of simulating composite material fires.**

Methods:

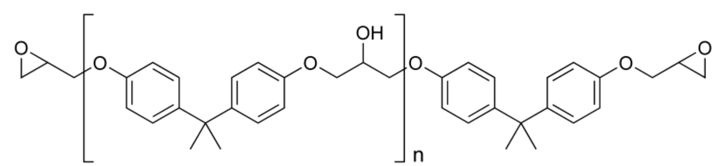
- **Intermediate scale fire testing provides decomposition data approaching practical scales**
- **This test series is designed to evaluate the intensity of the fire environment under thermally severe conditions**
- **An insulated enclosure was constructed to test materials**

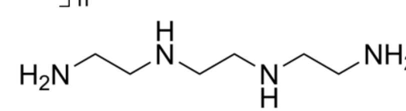
About Carbon Fiber Epoxy Aircraft Composites

- **Around ~35% epoxy, ~65% carbon fiber**
- **Fabric (woven) or uni-tape sheets, usually multiple layers thick**
- **Possibly sandwich material with high void fraction material between two composite sheets**
- **Pressed and cured in an autoclave**
- **Fibers around 5 μm diameter, 95% carbon**

Epoxy and TETA hardener (From wikipedia):

$[C_{12}H_{20}O_3]_n$

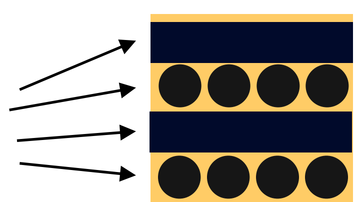




$C_6H_{18}N_4$

A four layer cross-section illustration:

Fibers in varying orientation



Key:

Carbon Fibers

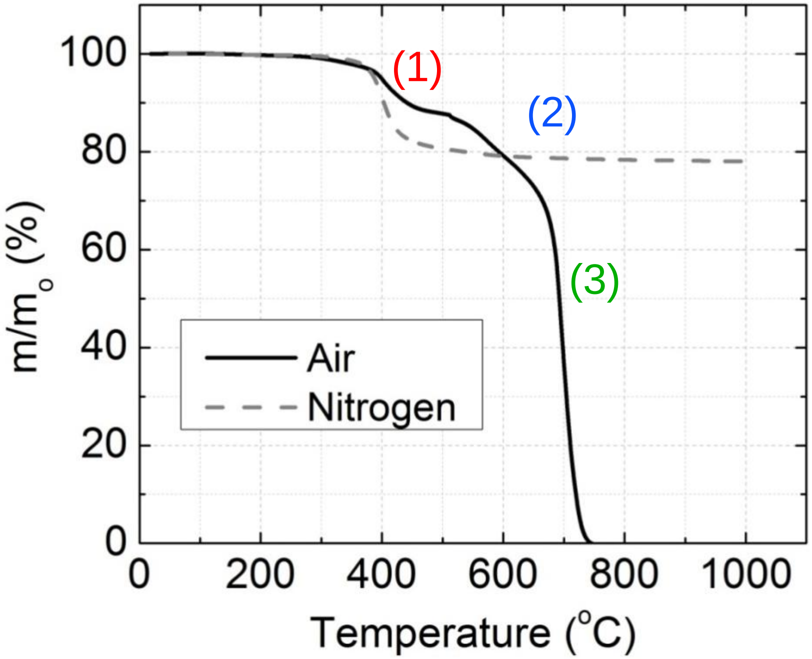
Epoxy Resin

Full Testing Progress/Plan

	Characteristic Length Scales	*Characteristic Mass*	*Experiments*	*Purpose of Testing*
Very small	0.1 mm to 1 mm	Milligrams (initial mass)	TGA, DSC	Fundamental kinetic, chemistry, decomposition behavior, and property measurements
Small	mm to 10 cm	Hundreds of grams	Cone calorimetry, radiant heat	Burn rate and scaled dynamics determination, simple validation testing
Intermediate	10-100 cm	0.1-100 kg	Radiant heat and environmental chamber tests	Bridge the gap between small and very small scale and large scale testing to discover dynamics not exposed at the smaller scales that will be present at larger scales
Large	Meters and above	Hundreds of kg and above	Full-scale fire testing	Full-scale with all physics represented in appropriate scale range

This presentation is focusing on a small portion of the full test plan.

TGA Results, 3 Regimes



TGA Details:

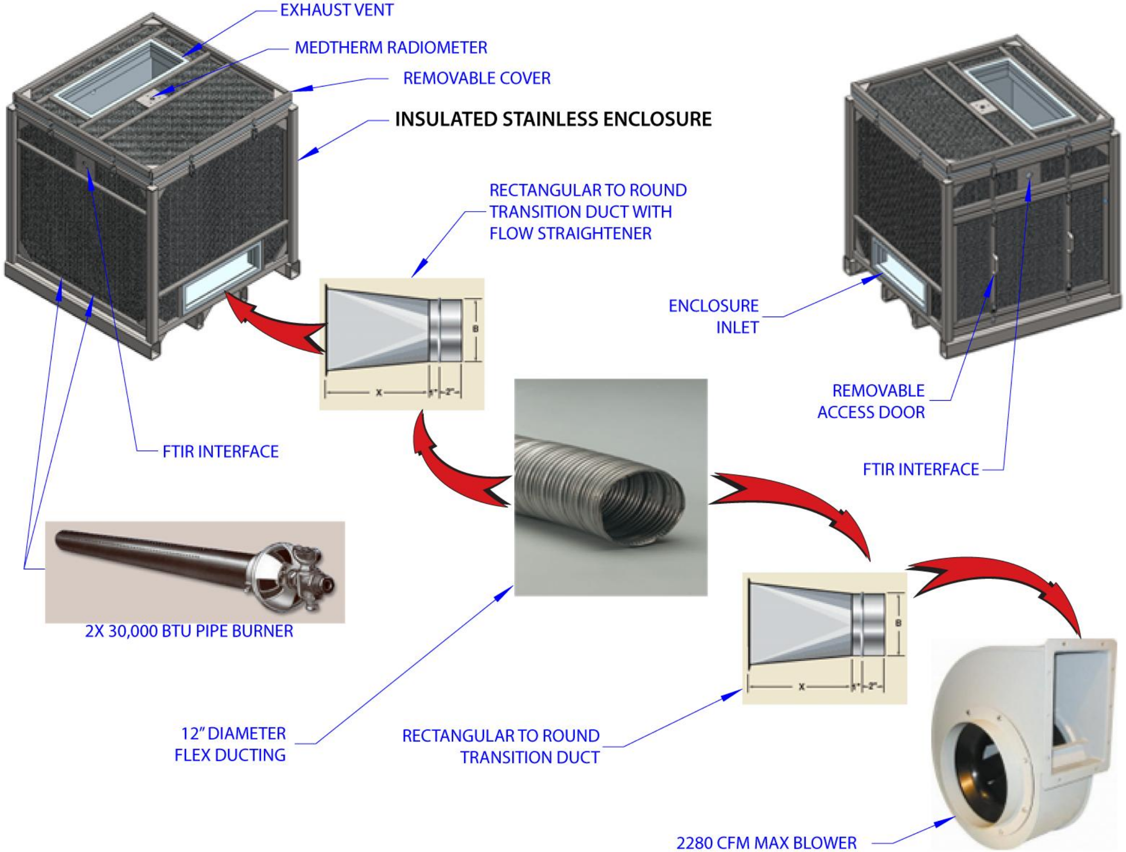
- 1-2 mg samples
- 20ºC/min
- Cytec 977-3 resin
- IM7 Fibers
- Single sheet cured in 1 atm oven

(1) Epoxy Decomposition (both Thermal and Oxidative Pyrolysis) and Char Formation
(2) Slow Char Oxidation
(3) Carbon Fiber Oxidation

- In N_2, pyrolysis reaction generate organic vapors/fuel and char
- In air, O_2 interacts with the epoxy and changes rate at which organic vapors are generated
- Char formation inhibits combustion of carbon fibers
- Char oxidation occurs BEFORE carbon fiber oxidation

Test Enclosure

- **91.0 cm aspirated internal cube designed to create an idealized semi-adiabatic environment**



Instrumentation

- FTIR
- RGA/Mass Spec.
- Radiometers
- Calorimeter
- Thermocouples
- Pitot Velocity Probe
- Video

Test Matrix

Wood Pre-test Fires

Test #	*Description*	*Quantity*
1	Wood panels, roughly ~8" x 13", ½ to 1" thick,	100-300 lbs
2	30" long, 1.5" wide, roughly 7/16" thick strips	Around 100 lbs

Carbon Fiber Composite Fires

Test #	*Description*	*Quantity*	*Perceived Quality*
3-4	IM7 epoxy woven body armor pieces, roughly oval (~8" x 13"), 3/8" thick, varying in shape, and some with heavy excess epoxy along the edges	100-300 lbs	Low
5	Same as test 3-4, more uniform materials		Moderate
6	28" long, 1" wide, 3/32" thick strips of Hexcel woven fiber materials (from the SNL shop). Some end pieces with variations in the sizing.	Between 550 and 600 strips	Moderate to High
7	32" long, 3" x 3" I-beams made at the composite training center. Apparently all loaded to failure, most with cracking and delamination.	31 I-beams	Moderate to High

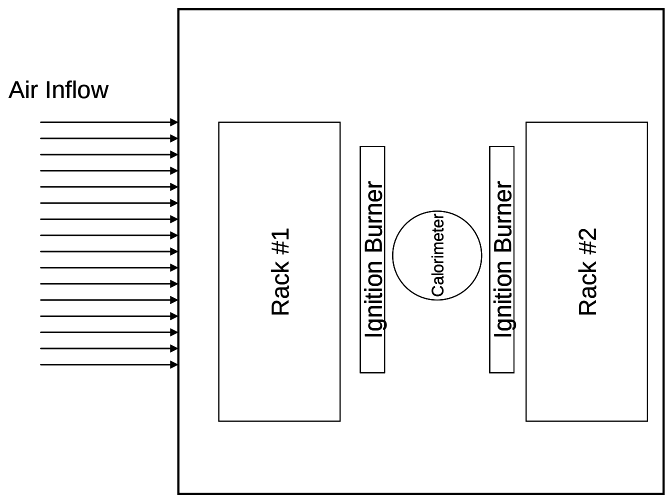
Test Details

Parameter	*Test 5*	*Test 6*
Material Description	Body Armor	Strips
Manufacturer	Hercules	Hexcel
Epoxy	3501-6 resin	3501-6 resin
Fibers	AW370 woven carbon fibers	Woven carbon fibers
Mass	36.6 kg	39.3 kg
Est. SA/Vol ratio	2.0 cm^{-1}	9.2 cm^{-1}
Arrangement	Two racks	Crib

Test 5 & 6 Layout

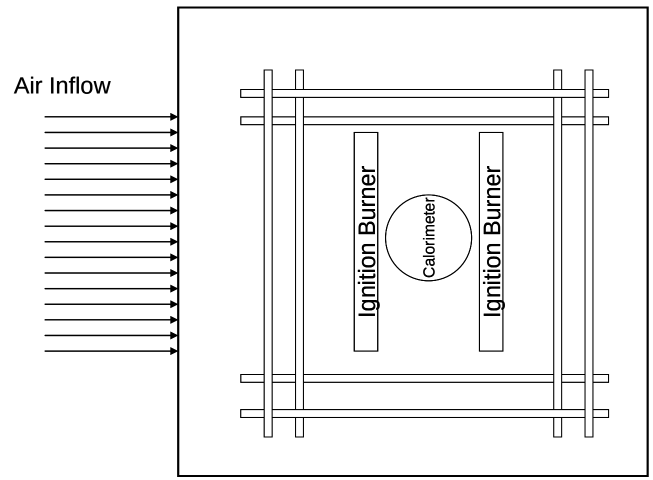
Test 5

Top View

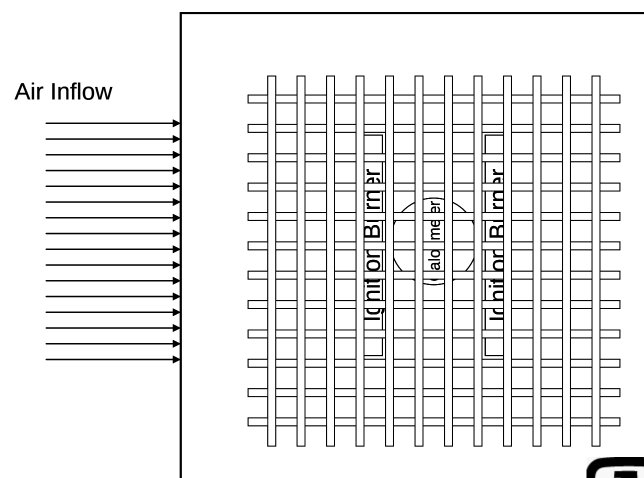


Test 6

Top View

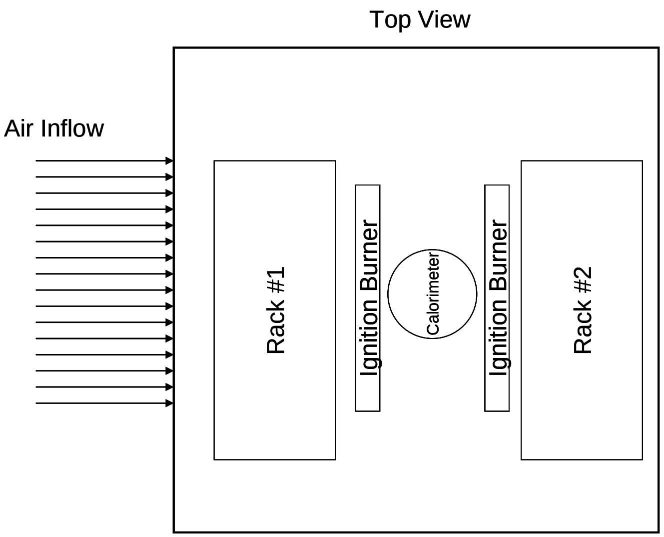


Top View

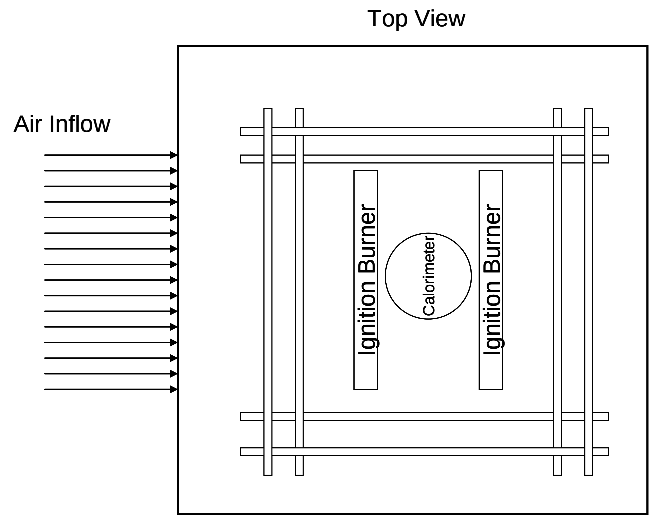


Test 5 & 6 Layout

Test 5



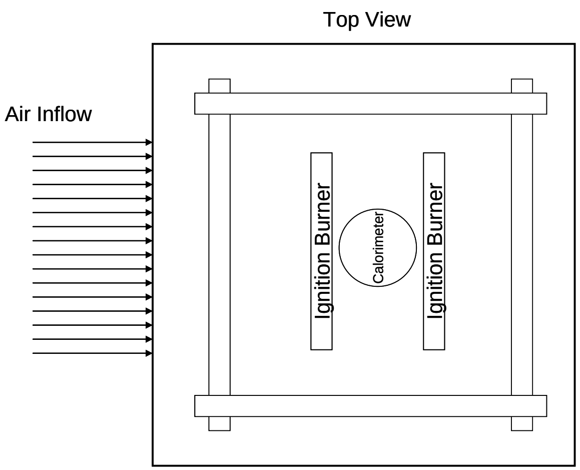
Test 6



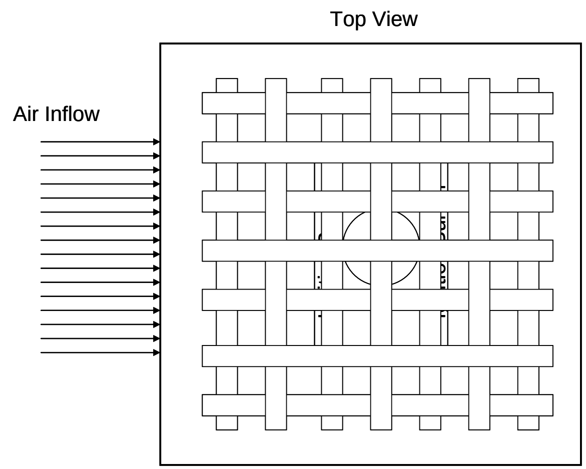
Top View

Test 7 Layout

Lower Layer



Upper Layer

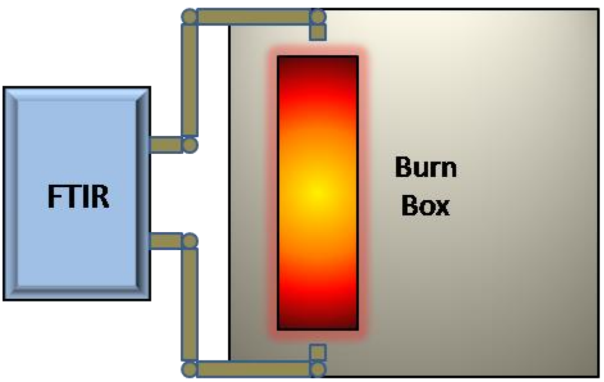


→ Air inflow was significantly varied for this test

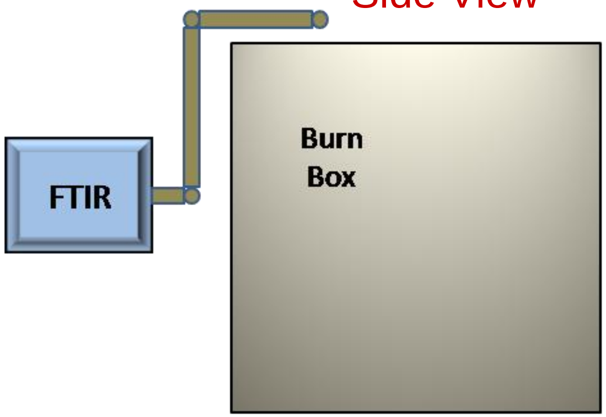
Instrumentation

FTIR

Top View



Side View



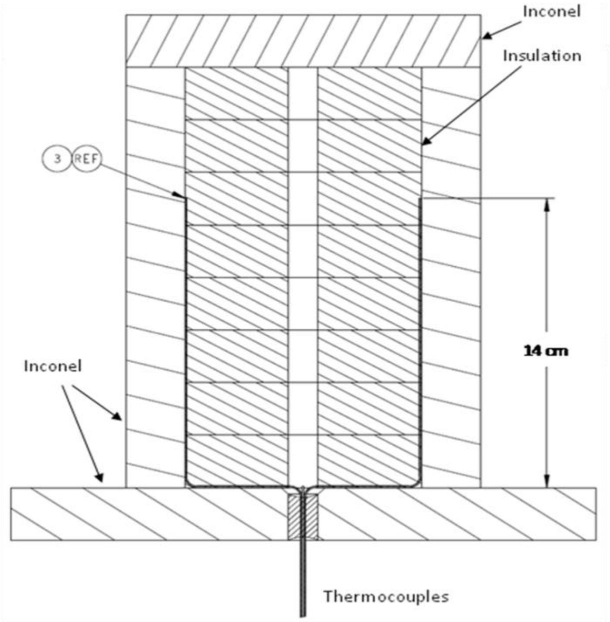
Signals obtained except during highest intensity burning times

Medtherm Radiometers

Pre-test / Post-test

Despite aspiration, some deposition occurred
Post-test calibrations were used to correct results

Inconel Calorimeter



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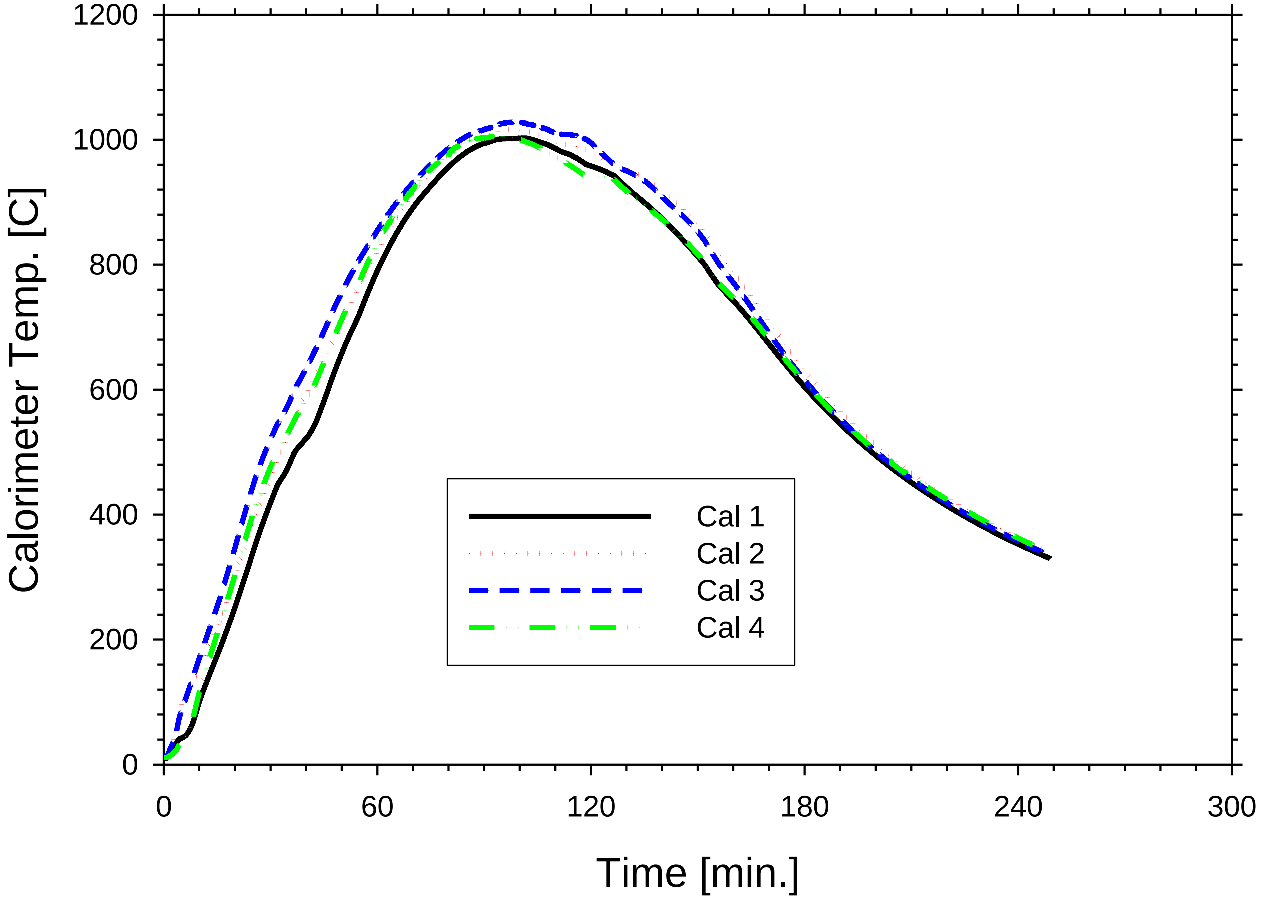
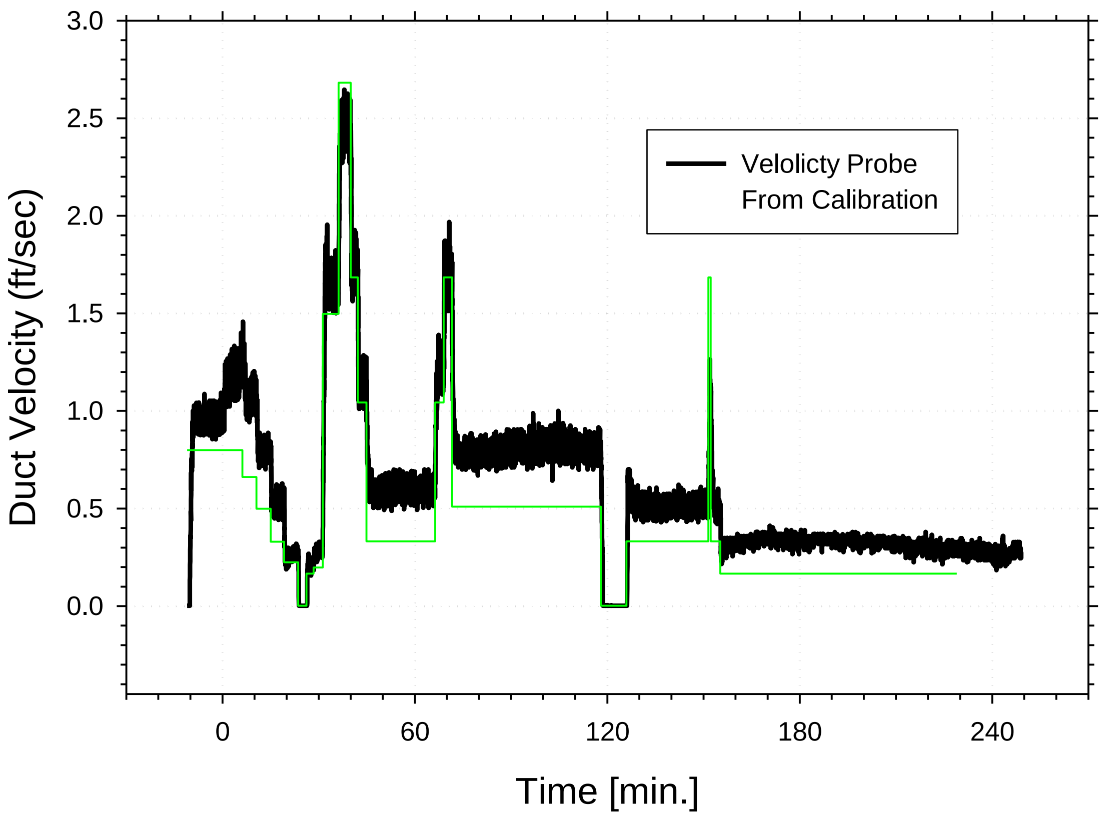
Test 7 (I-beams) Calorimeter Temperature Results



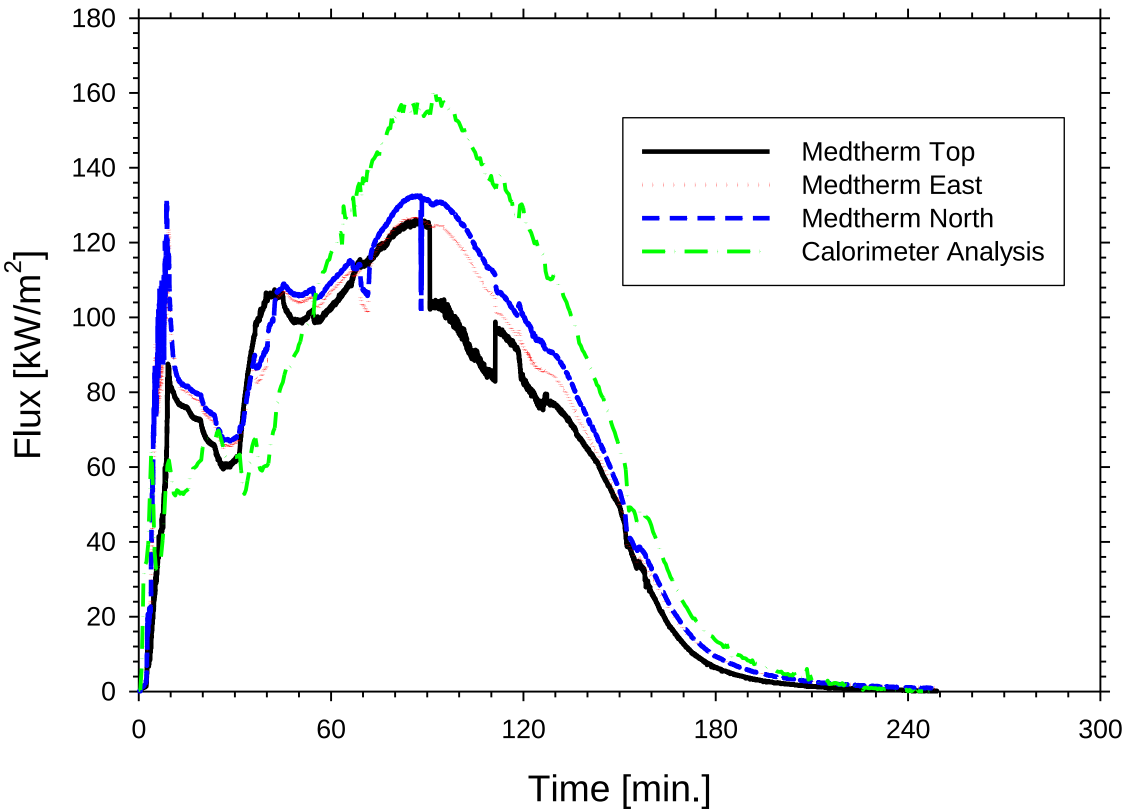
Almost no discernable variations to air flow
Peak Temperatures were just over 1000 ºC

Test 7 (I-beams) Inflow Velocity



Substantial inflow changes result in only moderate changes in flux, temperature

Test 7 (I-beams) Heat Flux and Calorimeter Heat Flux Results

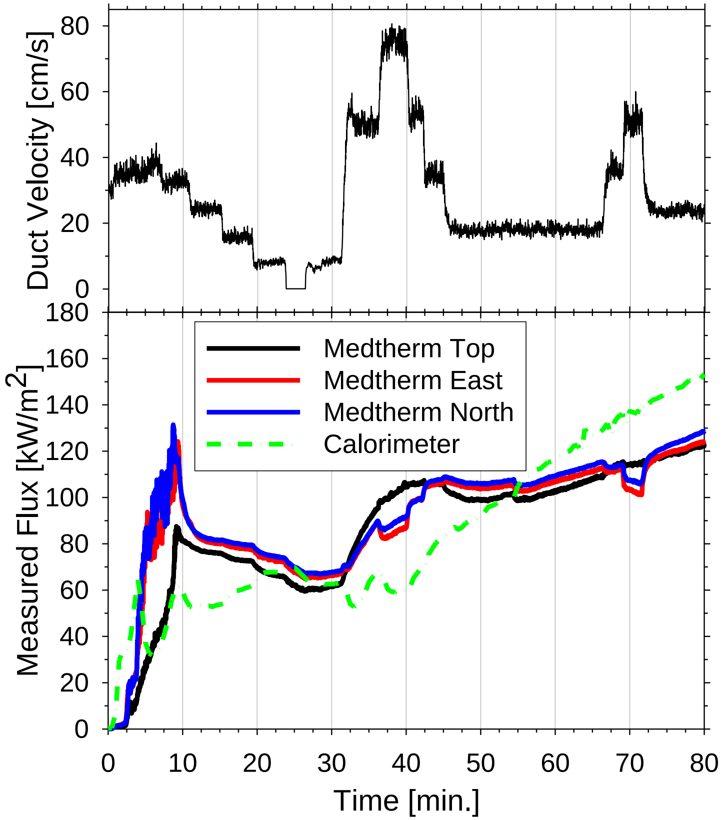


Calorimeter shows lower flux during flaming, higher during glowing combustion times

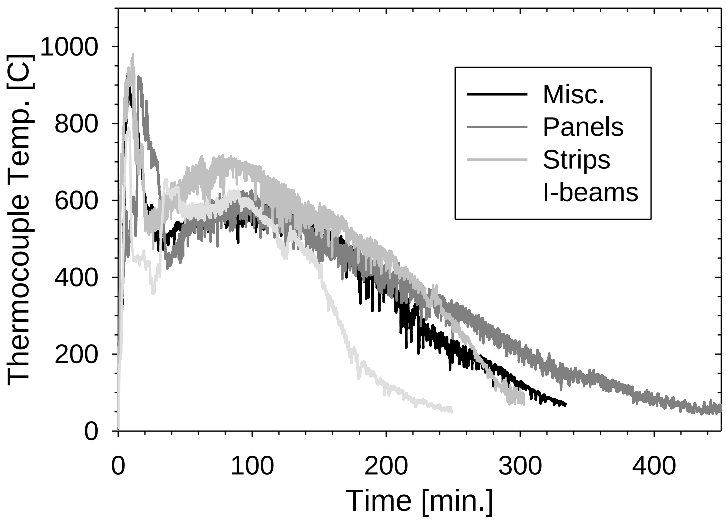
Fluxes as high as 160 kW/m^2 are found during glowing combustion in the bed

Test 7 (I-beams) Results

Flux/Velocity Plotted Together



Thermocouple in the Exhaust for Four Tests



Minor effect of velocity on measured flux. Reversing trends suggestive of a transitional region between diffusion and kinetic reaction control.

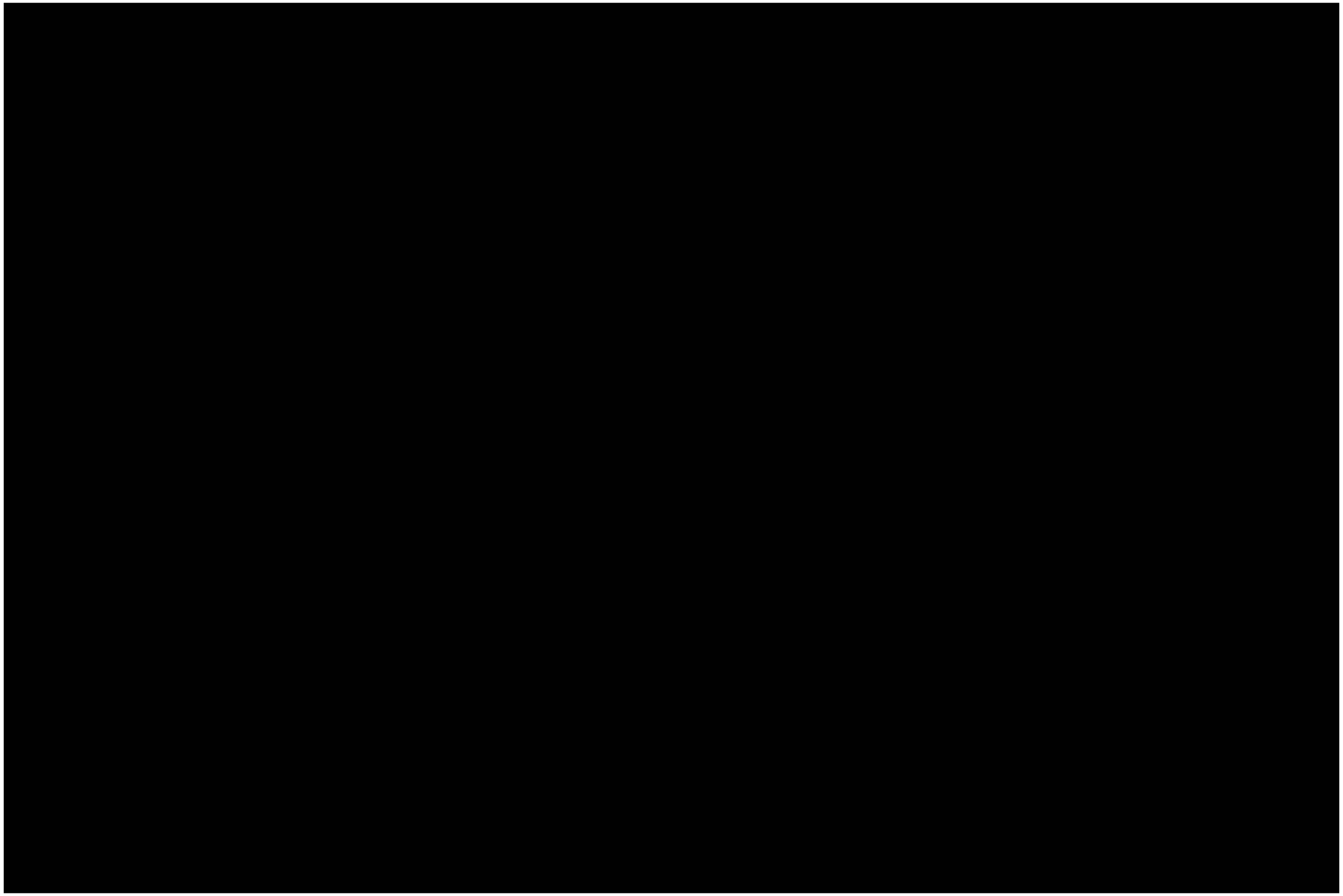
Test 7 (I-beams) Video Frames

Frames show the progression of the reactions through the glowing combustion phase

Test 7 (I-beams) Stills

Not certain what the white residue is
Air inlet is from the right in these photographs
Glowing furthest away from the air inlet at late burn-out
Final mass: 10.3% of the original mass

Test 7 (I-beams) Video



Testing Summary

	Test	Initial Mass	Residual Mass	Peak Flux	Flaming Duration	Total Duration	SA/V	Mean Consumption Rate
	#	*kg*	*%*	*kW/m^2*	*min*	*min*	*cm^{-1}*	*g/s*
Wood	1	40.8	-	220	-	90	2.4	7.56
Wood	2	31.8	-	220	-	60	1.3	8.82
Composites	4	36.5	9.56	180	25	330	-	1.84
Composites	5	38.5	2.59	175	30	420	2.0	1.53
Composites	6	39.3	6.74	220	20	300	9.2	2.18
Composites	7	26.5	10.34	160	10	240	6.9	1.84

Compared to wood, peak fluxes tend lower, consumption rates are much lower, thermal release duration is much longer.

Surface Area to Volume appears to relate to consumption rate.

Very low residual mass.

Summary

- A flaming region of 10-30 minutes followed by 4-8 hours of glowing surface oxidation reactions.
- Flaming combustion heat fluxes from the radiometers range from 80 kW/m^2 to 220 kW/m^2.
- Glowing combustion heat fluxes range from 60 kW/m^2 to 160 kW/m^2 from the radiometers, and calorimeter data suggests peak fluxes in the 100 kW/m^2 to 150 kW/m^2 range.
- Under idealized fire conditions it is possible to get high burnout of the composite material (90%+).
- Thermal and oxidative pyrolysis of epoxy generates flaming combustion early in time. Later in time, char and carbon fiber oxidation ensues.
- Evidence of both diffusion and kinetic reaction control with low air in-flow.

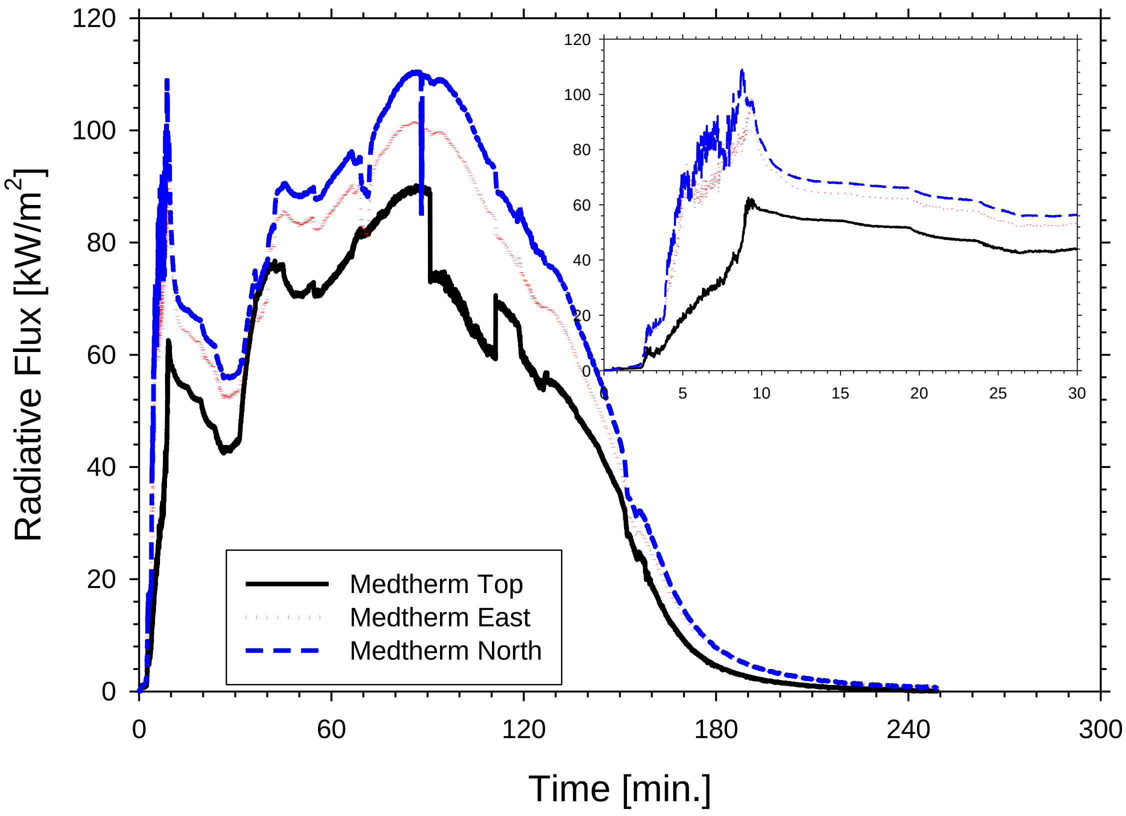
Acknowledgements

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

This work was made possible by support from the Sandia WSEAT, and B61 programs.

The authors would like to acknowledge the contributions of the technologists who conducted the tests, including Sylvia Gomez as the test director, Ciro Ramirez, Randy Foster, and Bennie Belone. Richard Simpson provided videography. The design and construction of the enclosure was overseen by Richard Streit.

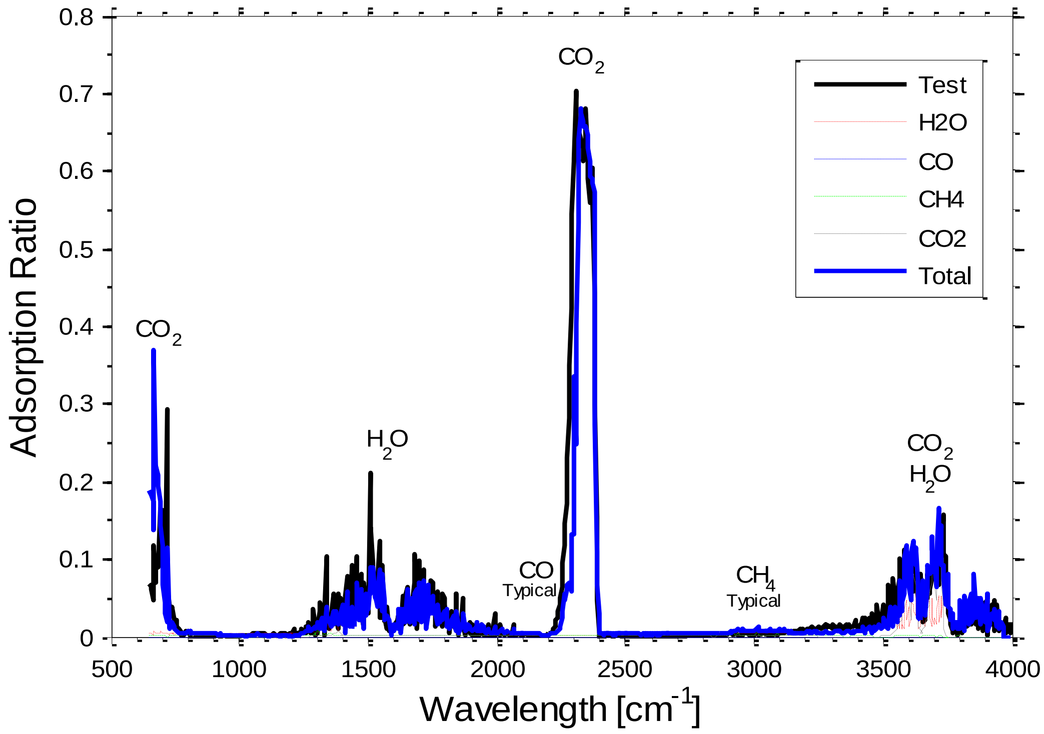
Test 7 (I-beams) Uncorrected Heat Flux Results



Uncorrected radiometers measured peak intensities during flaming around 110 kW/m^2
Flaming combustion lasted less than 10 minutes
Glowing combustion lasted 4 hours, with peak fluxes around 110 kW/m^2

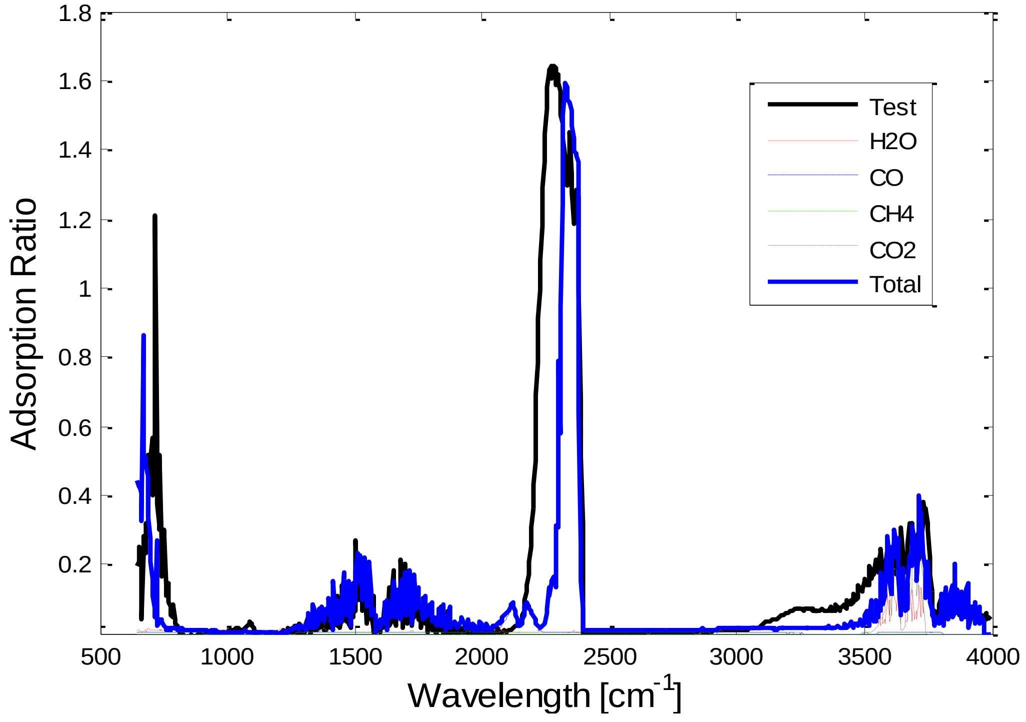
Extra Viewgraphs

Typical extraction



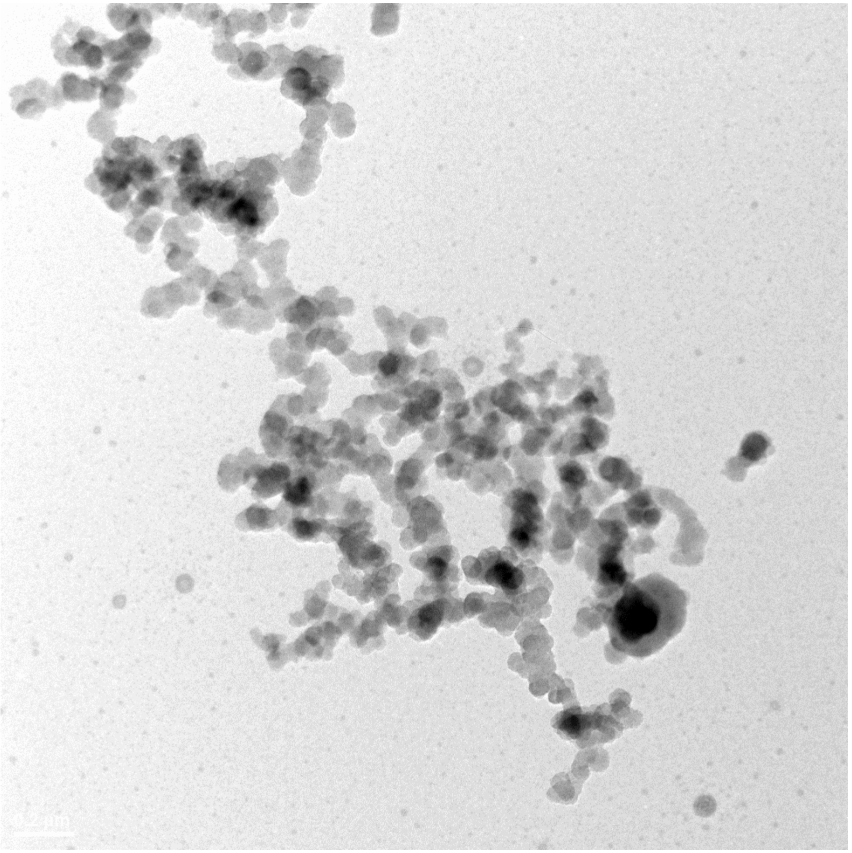
Extra Viewgraphs

Stretching during turbulent regime

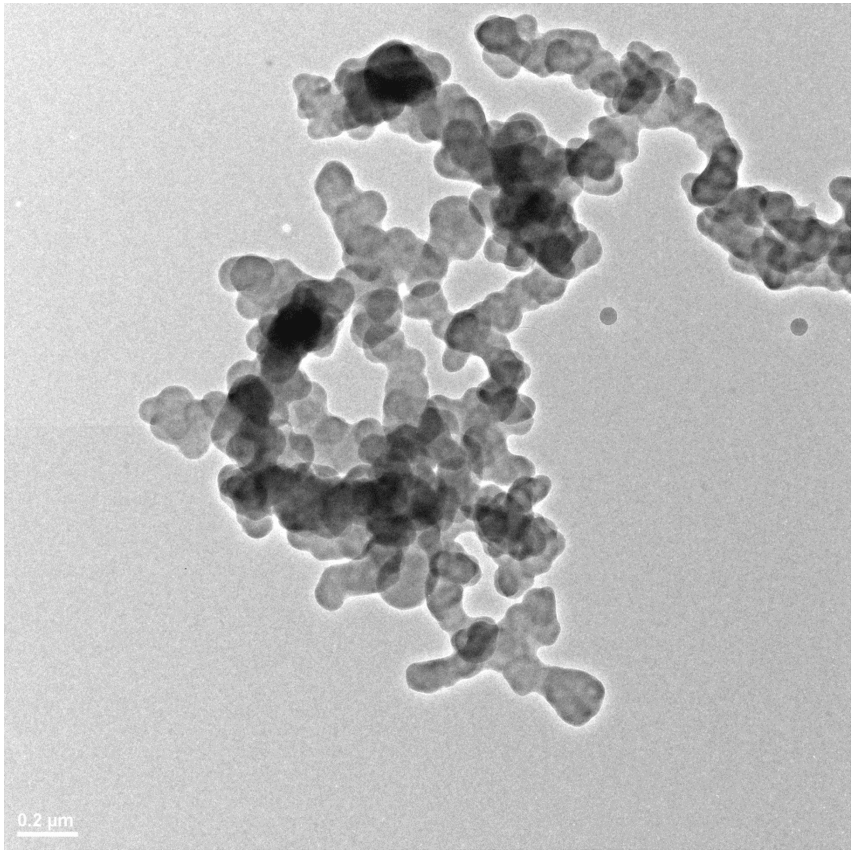


TEM Images of Soot

Test 5



Test 6



Larger soot spheres for Test 6 (same scale)

Both show large agglomerates

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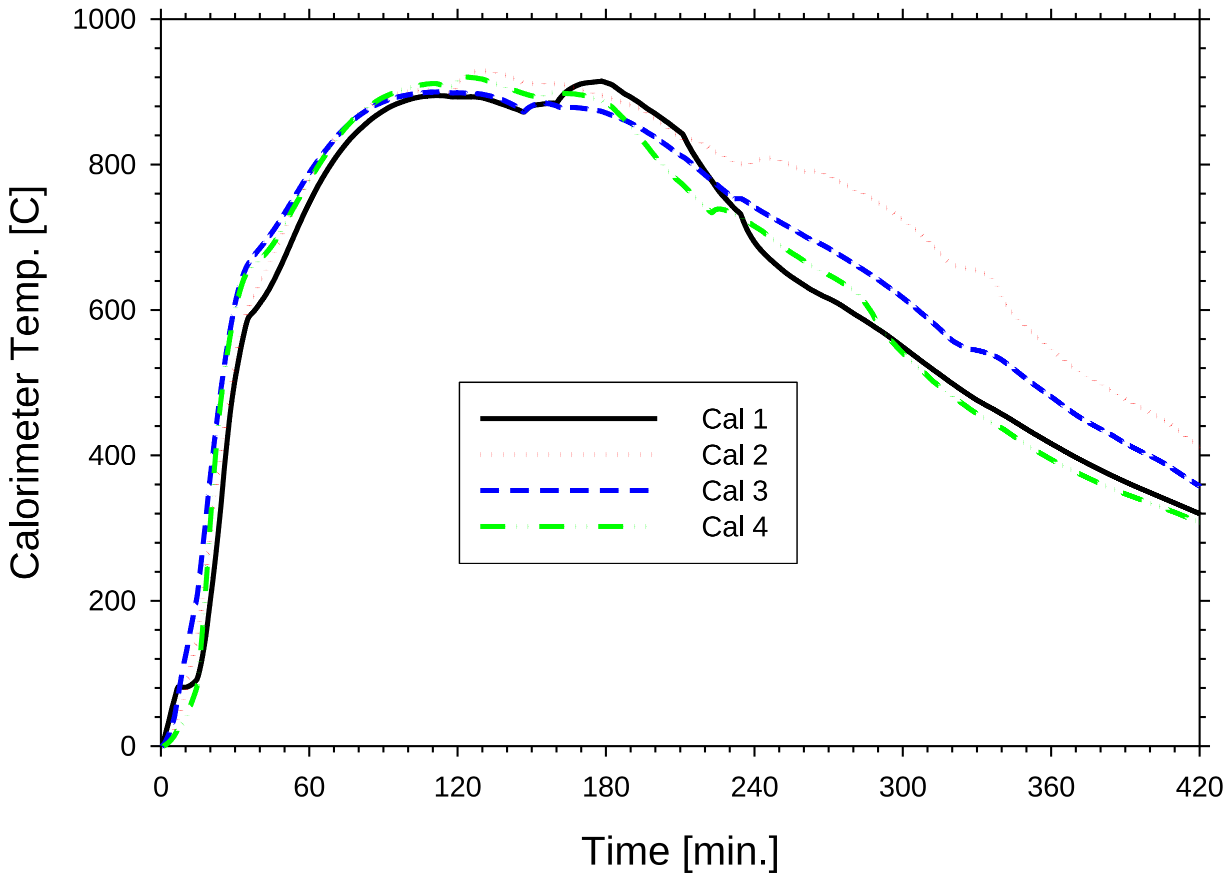
Test 5 (Body Armor) Calorimeter Temperature Results



Calorimeter temperatures showed small variation at early times
Peak Temperatures were around 900 ºC

Interpreting Calorimeter Results

- **Calorimeter temperatures can be analyzed to extract estimates of heat flux**
- **Inverse methods were employed, and comparisons suggested a simpler lumped capacitance approximation was adequate:**

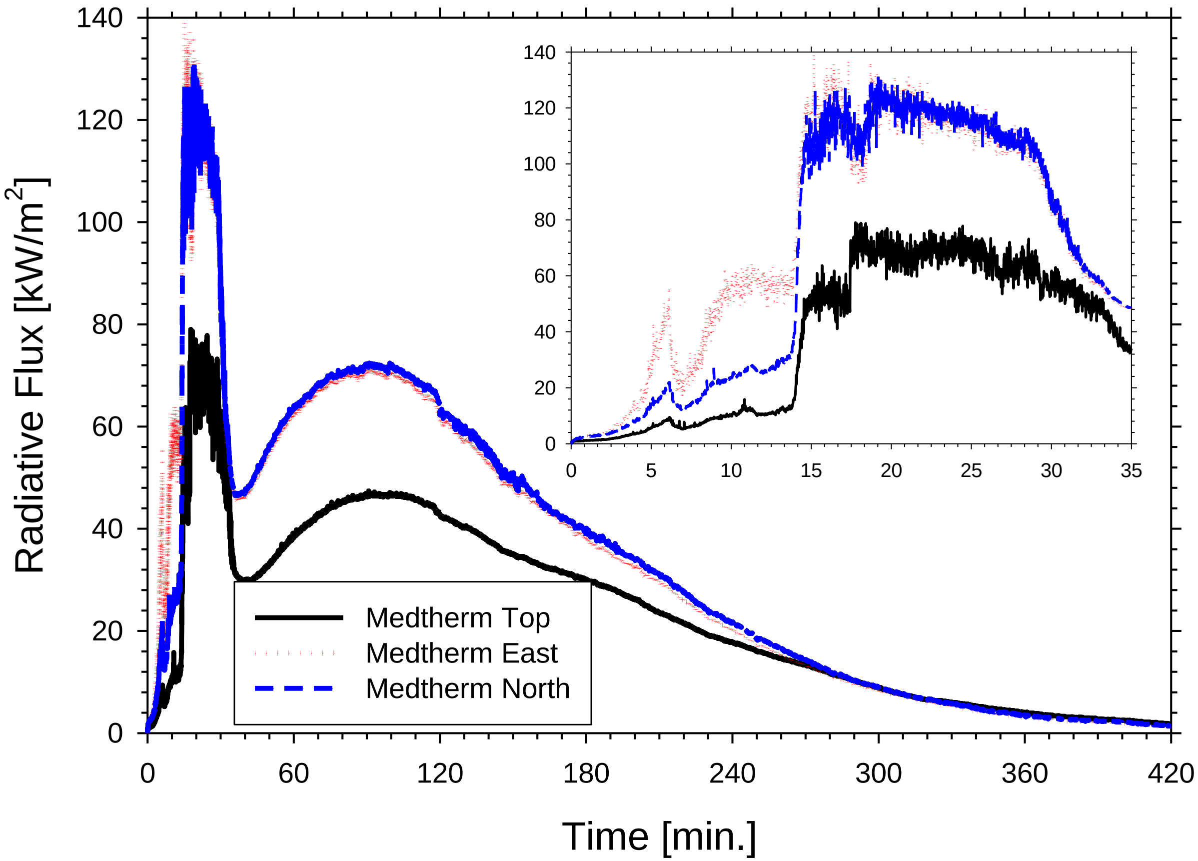
$$\varepsilon q_{itotal} = q_{abs} + \varepsilon q_{emit.}$$

$$q_{abs} = \rho C p L \frac{dT}{dt}$$

$$q_{emit.} = \sigma T_{Cal}^{4}$$

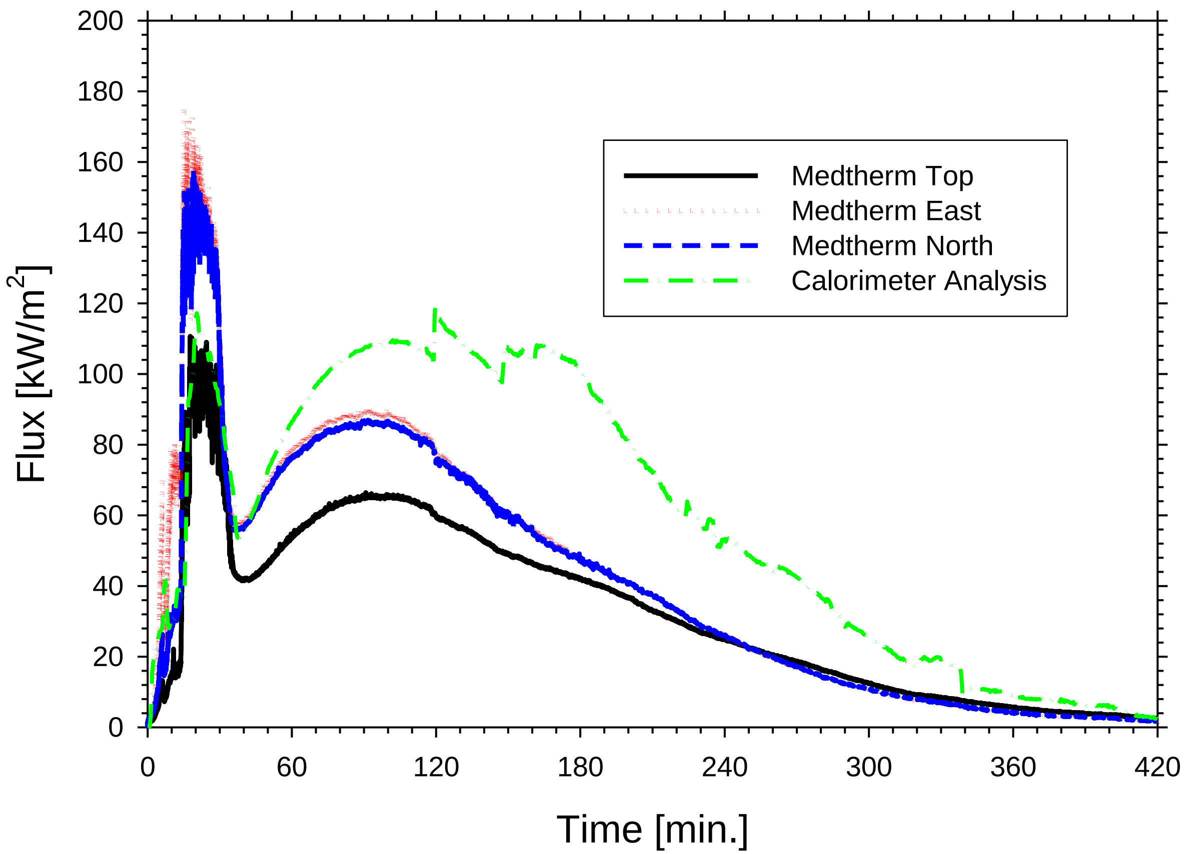
Property	Units	Value
Inconel Density	kg/m^3	8430
Inconel Specific Heat	J/kgK	540
Surface Emissivity	-	0.6

Test 5 (Body Armor) Uncorrected Heat Flux Results



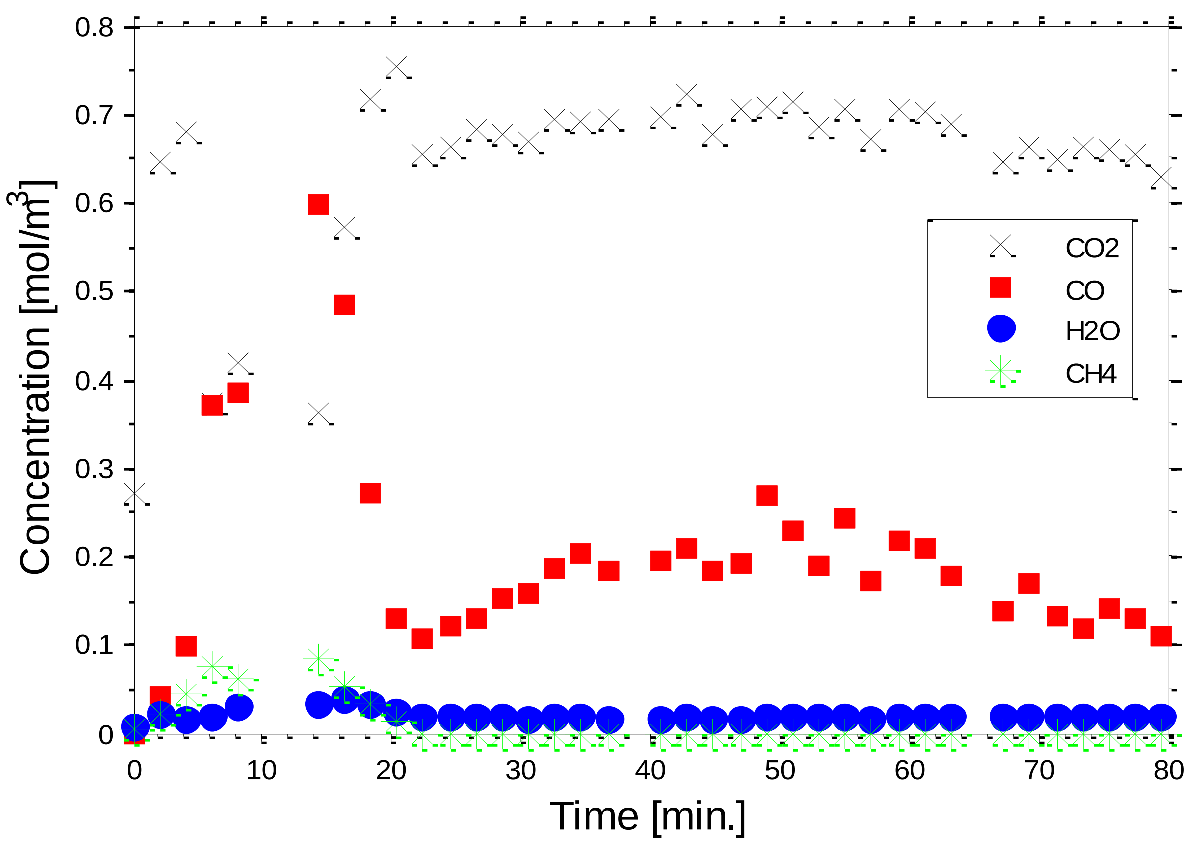
- Uncorrected radiometers measured peak intensities during flaming around 130 kW/m^2
- Flaming combustion lasted approximately 30 minutes
- Glowing combustion lasted 6+ hours, with peak fluxes around 80 kW/m^2

Test 5 (Body Armor) Corrected Heat Flux and Calorimeter Heat Flux Results



Calorimeter shows lower flux during flaming, higher during glowing combustion times
Fluxes as high as 120 kW/m^2 are found during glowing combustion in the bed

Test 5 (Body Armor) FTIR Results



Low signal during peak flaming
H2O, CO, and CH4 peak during flaming, CO persistent during glowing combustion

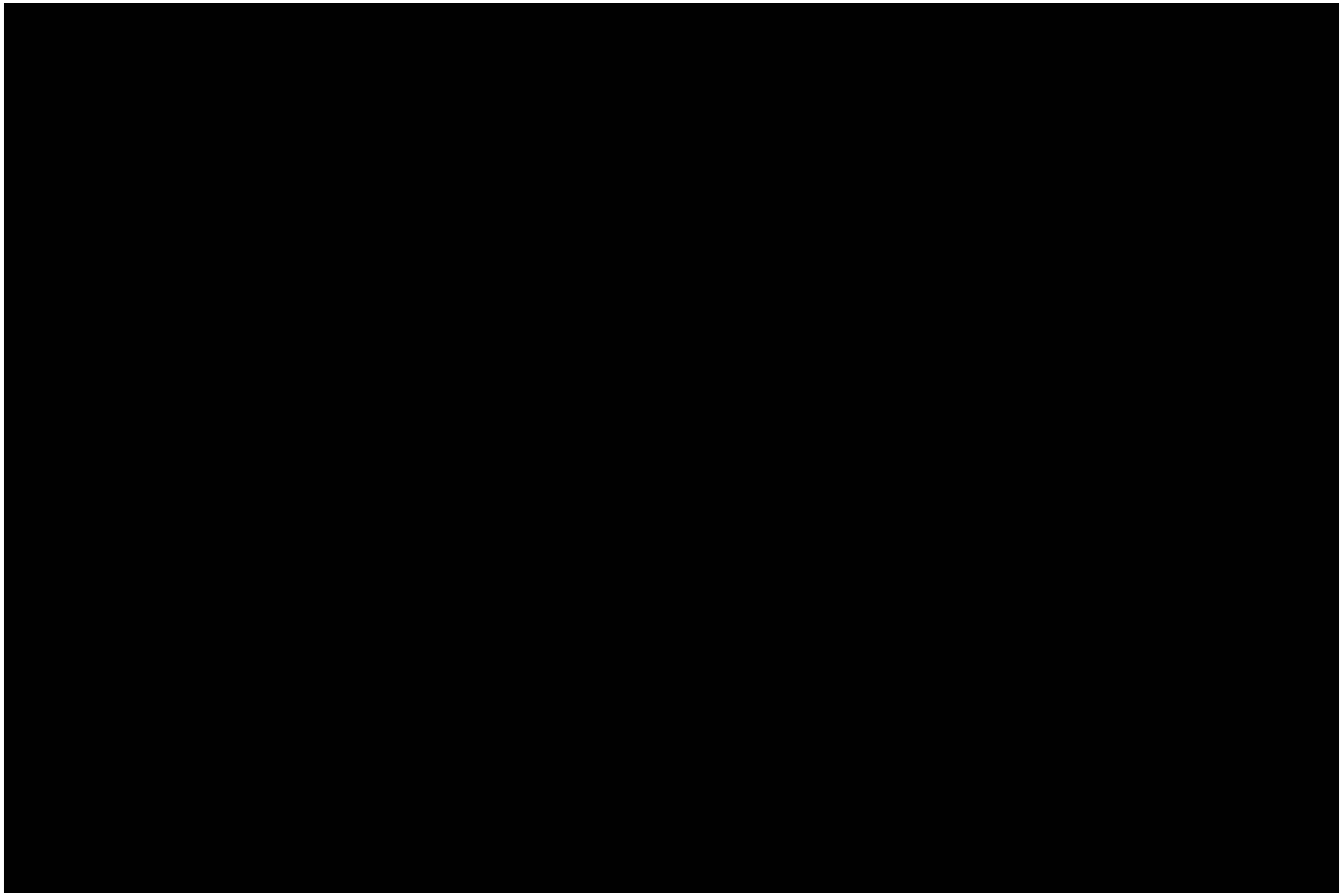
Test 5 (Body Armor) Video Frames

Frames show the progression of the reactions through the glowing combustion phase

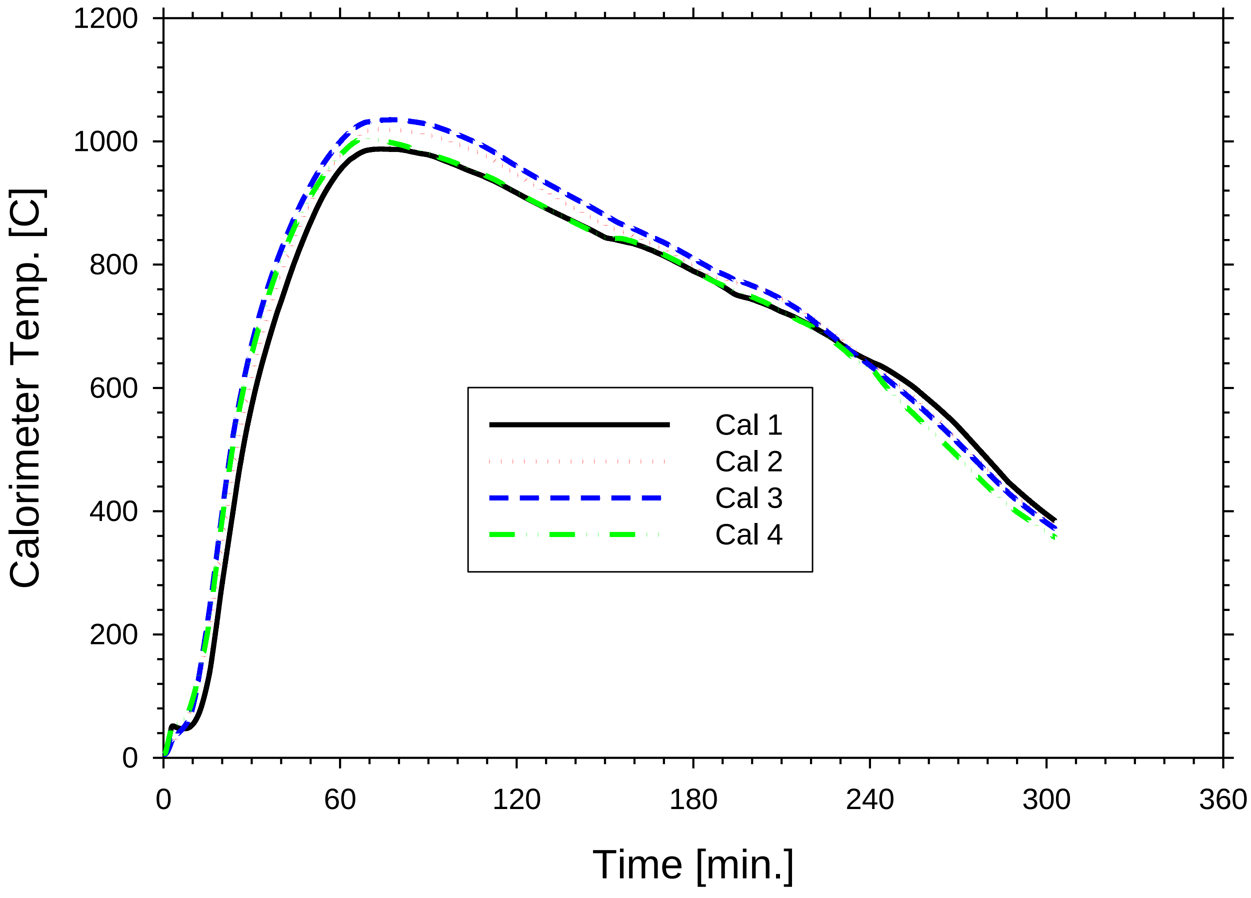
Test 5 (Body Armor) Stills

Late-term burnout pattern evident in still photography
Air inlet is from the left in these photographs
Final mass: 2.7% of the original mass

Test 5 (Body Armor) Video

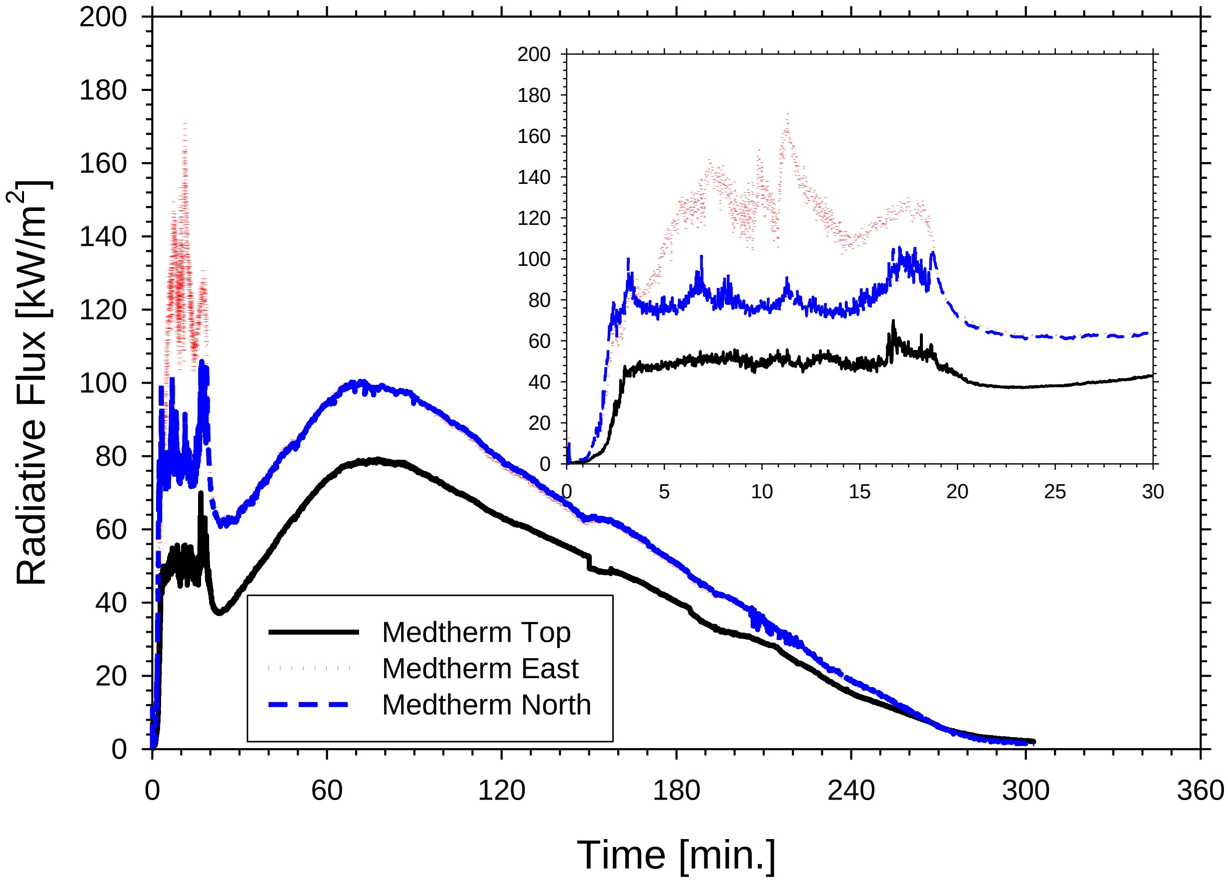


Test 6 (Strips) Calorimeter Temperature Results



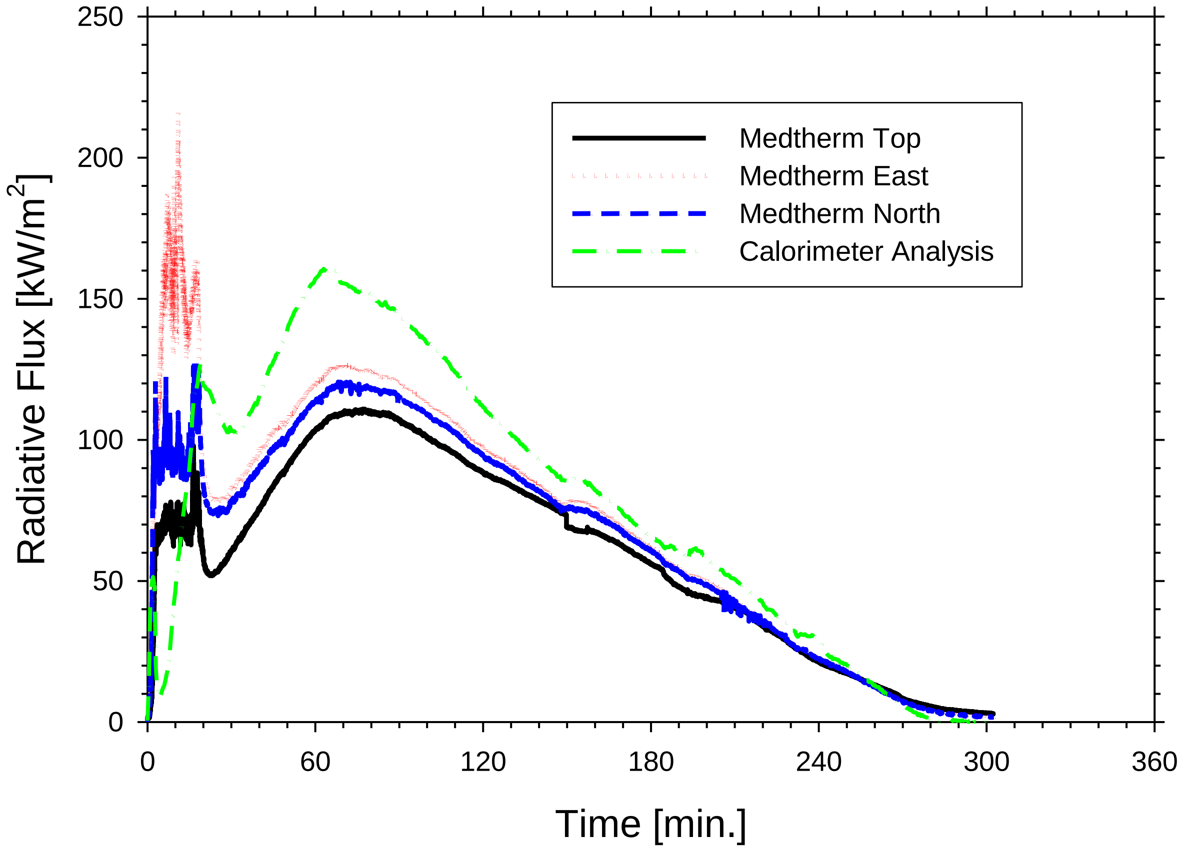
Calorimeter temperatures showed small variation at early times
Peak Temperatures were just over 1000 ^oC

Test 6 (Strips) Uncorrected Heat Flux Results



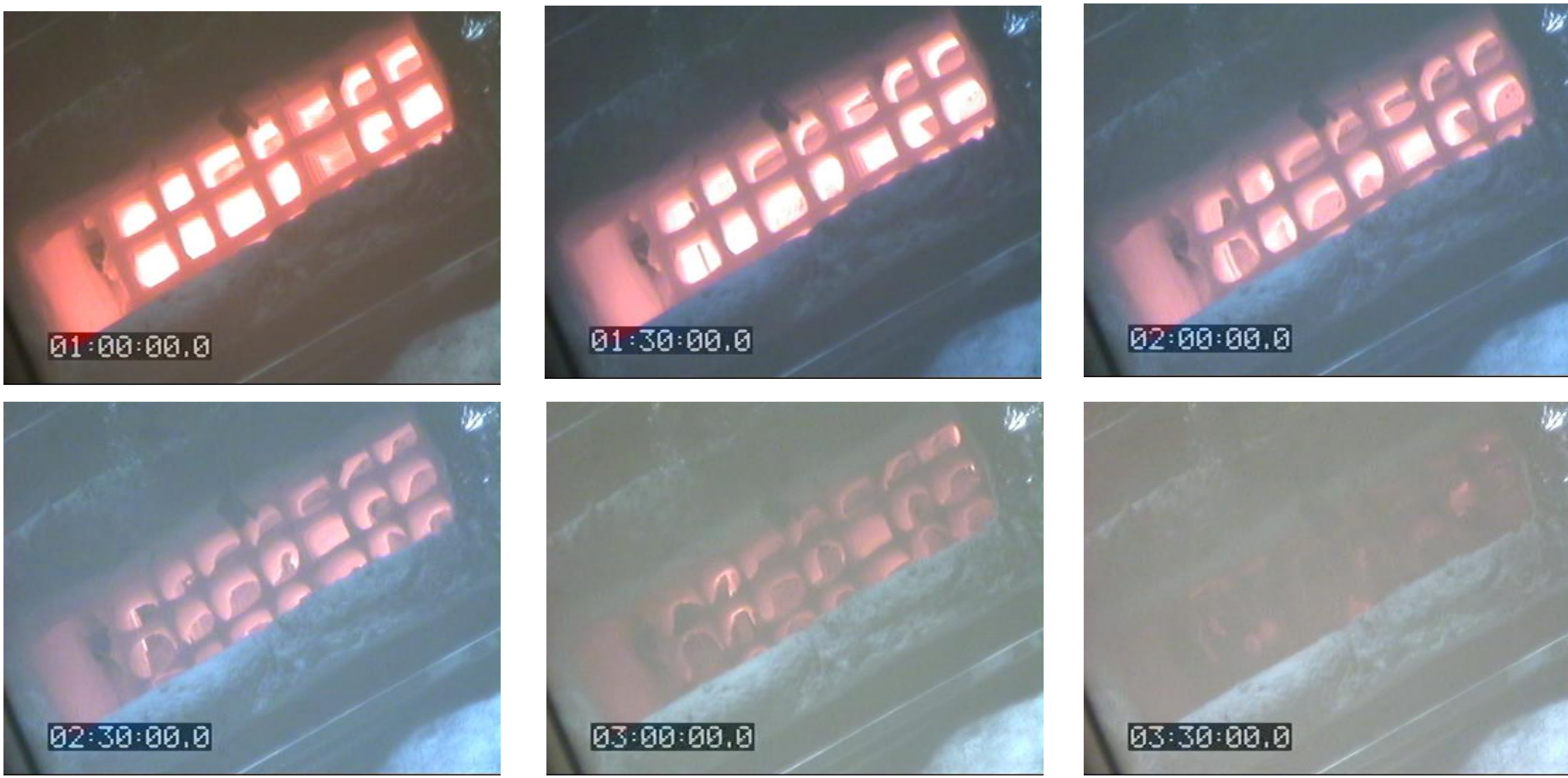
Uncorrected radiometers measured peak intensities during flaming around 150 kW/m^2
Flaming combustion lasted approximately 20 minutes
Glowing combustion lasted 4+ hours, with peak fluxes around 100 kW/m^2

Test 6 (Strips) Corrected Heat Flux and Calorimeter Heat Flux Results



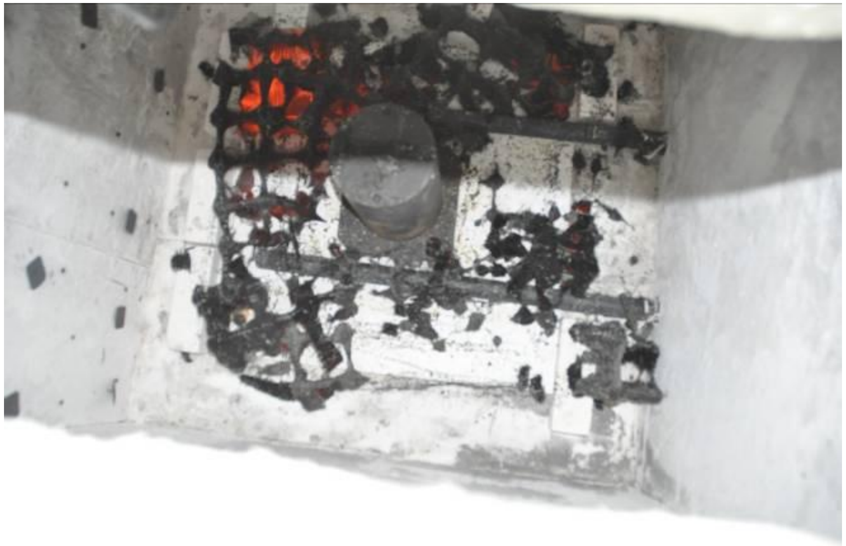
Calorimeter shows lower flux during flaming, higher during glowing combustion times
Fluxes as high as 150 kW/m^2 are found during glowing combustion in the bed

Test 6 (Strips) Video Frames



Frames show the progression of the reactions through the glowing combustion phase

Test 6 (Strips) Stills



Late-term burnout pattern evident in still photography
Air inlet is from the top in these photographs
Glowing nearest the air inlet at late burn-out.
Final mass: 6.7% of the original mass

Test 6 (Composite Strips) Video