Silica/Polysiloxane Ablative

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Outline

- Background and motivation
- Characterization methods
- Neat resin
- Prepreg manufacturing
- Creation of Si/DG composites
- Ablation testing
- Future work

Background

- To investigate next generation TPS materials
- Alternative to phenolic
- Polysiloxane resins
  - Very popular in electronics industry
  - Typically poorer mechanical properties
  - Researchers have shown success in reinforcing polysiloxane with different fillers/additives, thermal properties usually suffer
  - To find a good balance between the two

Control Sample

- Using legacy material as control, S/Ph
- Working with lower grade silica fabric, 96% SiO_2, to perfect processing
- Eventually will use aerospace grade silica fabric, 99% SiO_2
- Aiming to make 1.72g/cc control samples

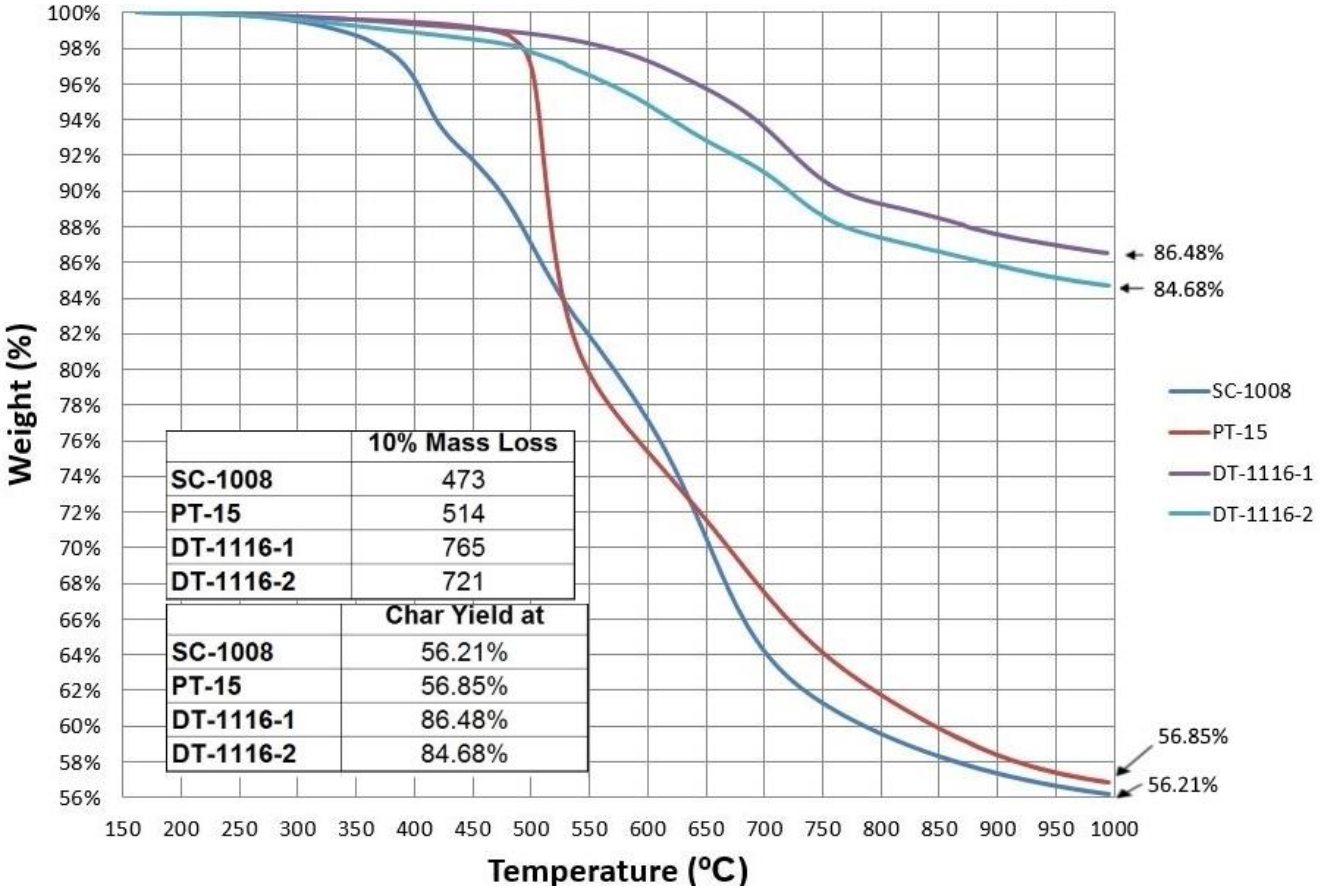
DG Polysiloxane Resin

- Inorganic matrix, utilizing a mixture of polysiloxane chemistries manufactured Dyna-Glas Technologies LLC
- Will be examined two proprietary formulations
  - DG-1 : Original neat resin
  - DG-2 : Neat resin with fillers to help curing cycle

Material Characterization

- Thermogravimetric Analysis
  - Thermal Stability & Char Yield
- Microscale Combustion Calorimeter
  - Heat Release Rate and Capacity
- Kinetic Parameters Modeling
  - Activation Energy
- Oxygen-Acetylene Test Bed
  - Ablative Performance

Char Yield Study-1

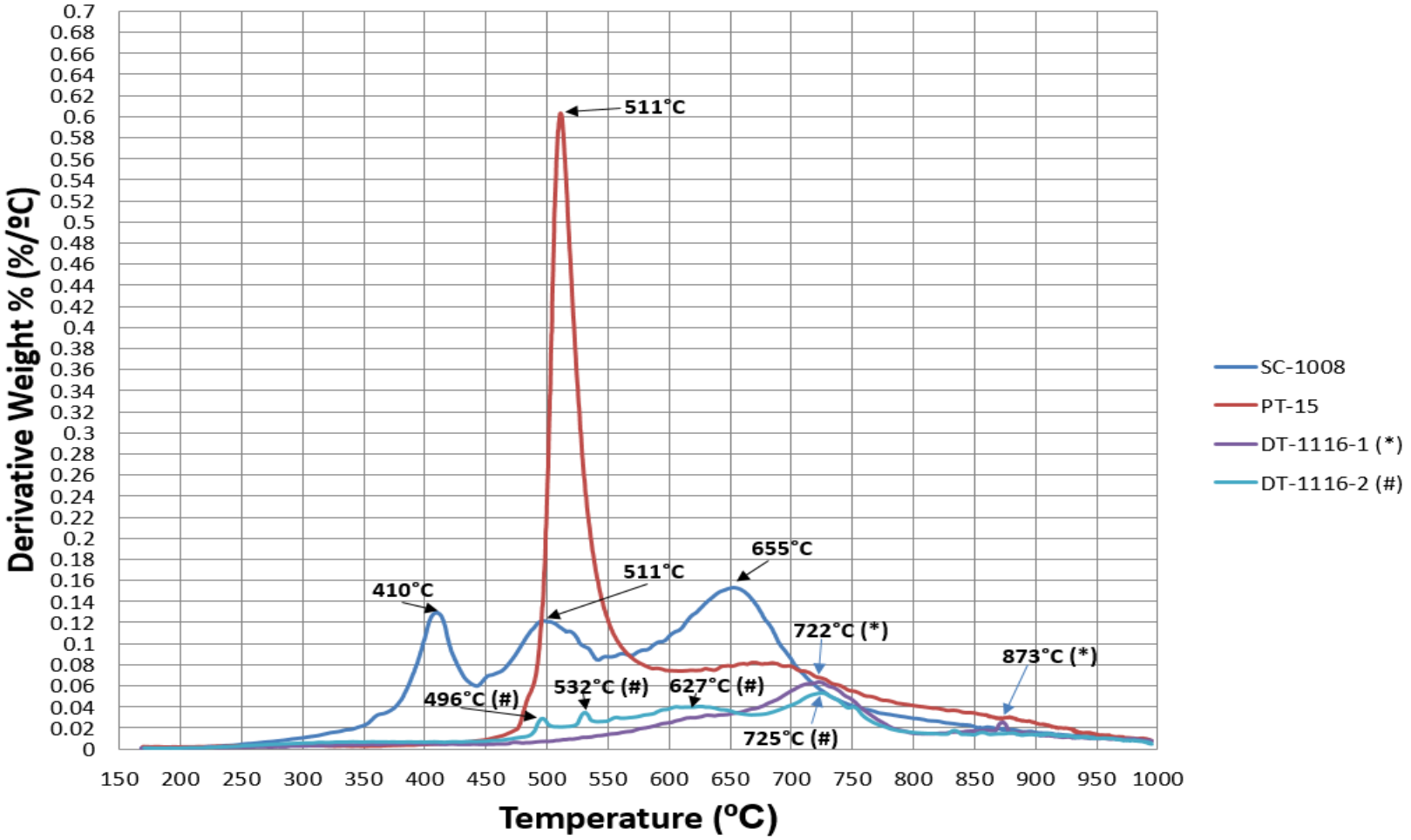


	10% Mass Loss
SC-1008	473
PT-15	514
DT-1116-1	765
DT-1116-2	721

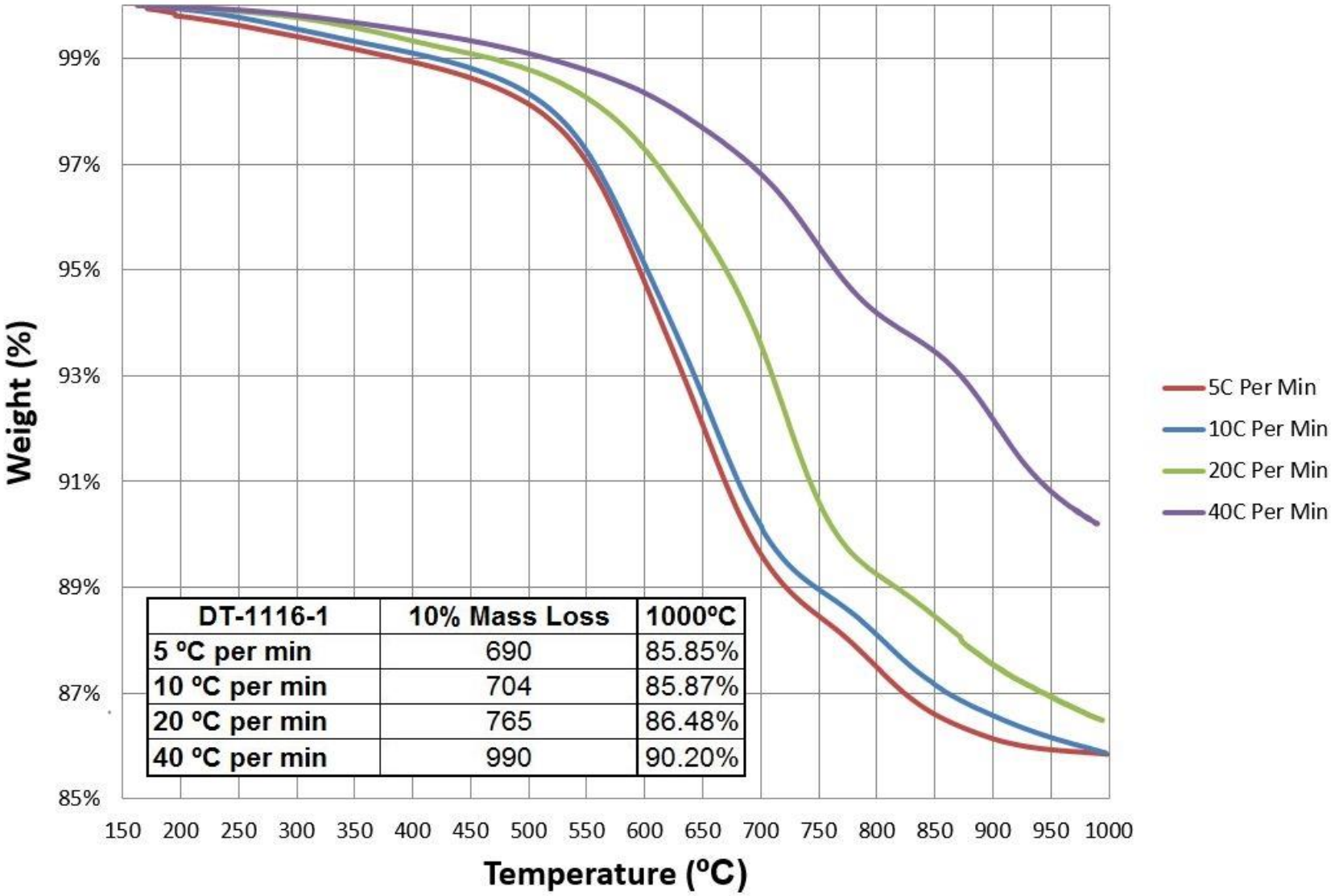
	Char Yield at
SC-1008	56.21%
PT-15	56.85%
DT-1116-1	86.48%
DT-1116-2	84.68%

Char yield results for SC-1008, PT-15, DG-1, and DG-2.

Char Yield Study-2



dTGA for SC-1008, PT-15, DG-1, and DG-2.



DT-1116-1	10% Mass Loss	1000°C
5 °C per min	690	85.85%
10 °C per min	704	85.87%
20 °C per min	765	86.48%
40 °C per min	990	90.20%

DG-1 polysiloxane at heating rates of 5, 10, 20, and 40ºC/min

	10% Mass Loss Temperature (°C)			
	5 °C/min	10 °C/min	20 °C/min	40 °C/min
SC-1008	416	439	473	557
PT-15	474	491	514	550
DT-1116-1	690	704	765	990
DT-1116-2	688	686	721	781

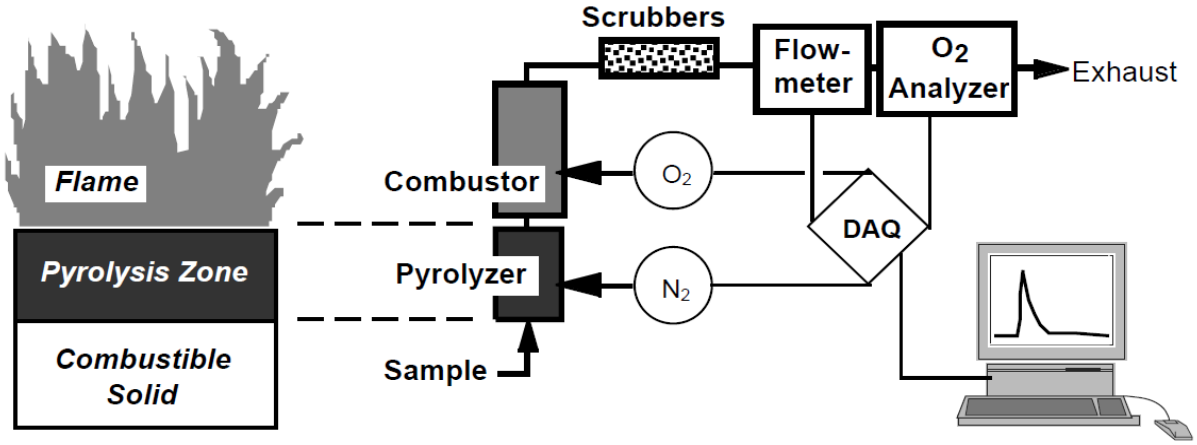
Decomposition temperature (T_d) of 10% mass loss temperature

	Weight % at 1000°C			
	5 °C/min	10 °C/min	20 °C/min	40 °C/min
SC-1008	61.38%	57.54%	56.21%	56.99%
PT-15	60.61%	57.69%	56.85%	55.44%
DT-1116-1	85.85%	85.87%	86.48%	90.20%
DT-1116-2	86.16%	85.16%	84.68%	85.14%

Weight % at 1,000ºC

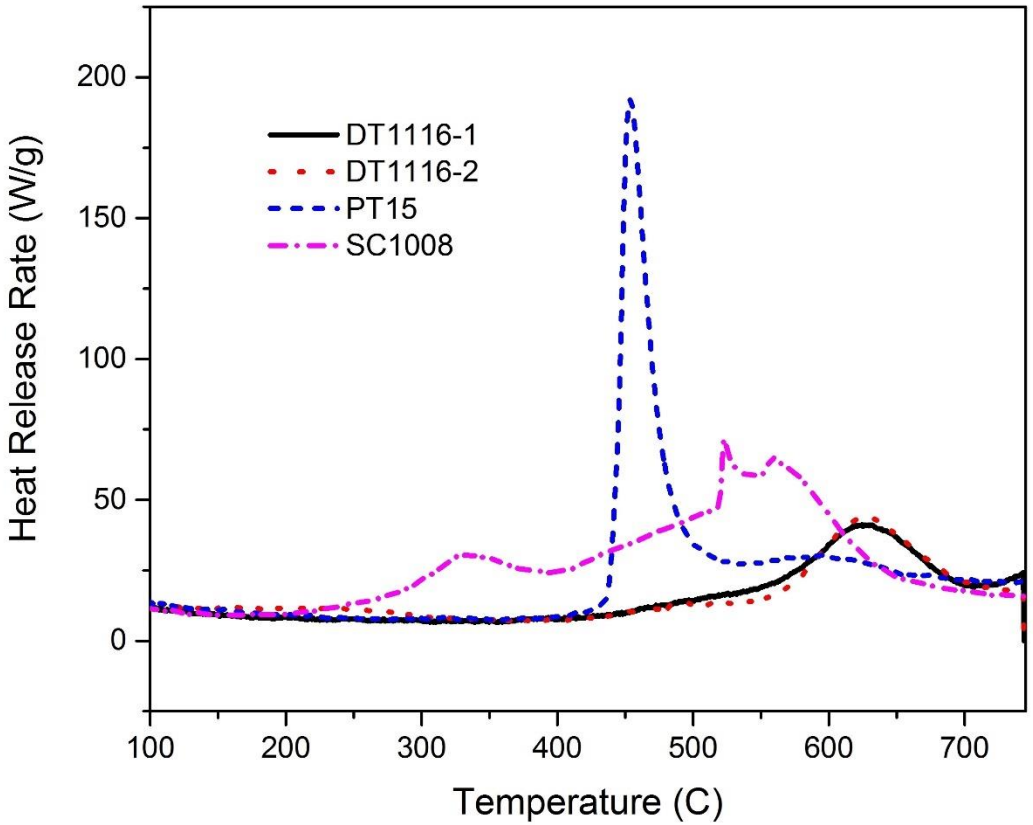
Flammability Properties-1

- Microscale Combustion Calorimeter
- Lab scale for small sizes
- Screening tool
- Good alternative to a cone calorimeter



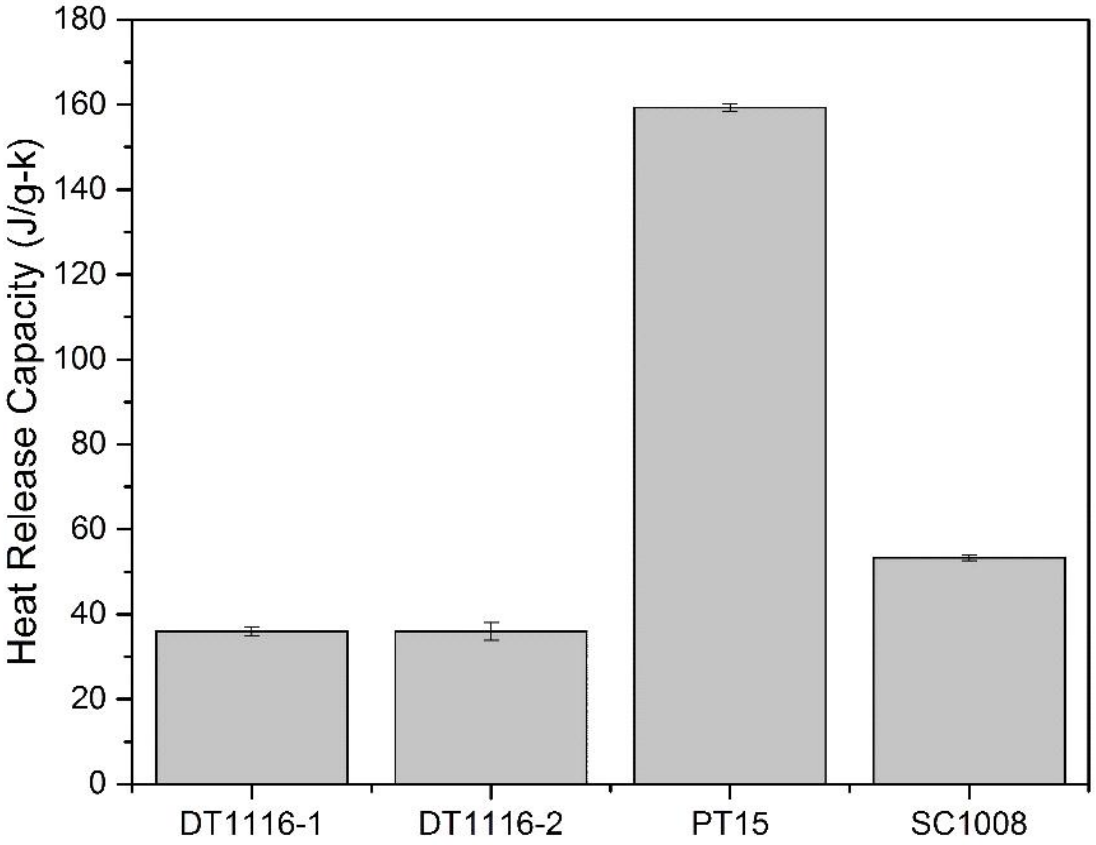
Flaming Combustion *Pyrolysis-Combustion Flow Calorimeter*

Flammability Properties-2



Typical heat release curves for the four resin systems

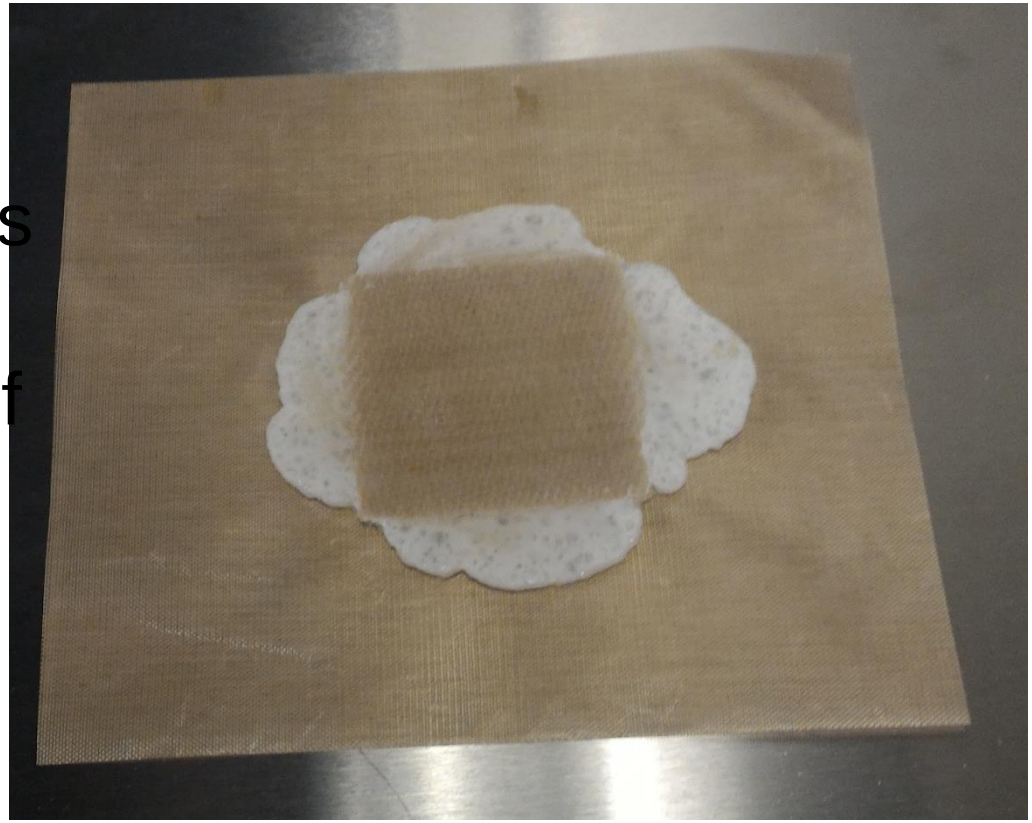
Flammability Properties-3



Comparison of the Heat Release Capacities for the four resin systems

2D Laminates

- High resin loss from flow
- Gave decent densities
- Allows for refining of prepreg and testing of different gelling procedures



Silica/DG 2D laminate coupons, 2”x2”x9plies

Solvent Selection

t-butyl acetate

IPA

xylene/toluene

50wt% Resin

40wt% Resin

Silica/DG pre-pregs made using different solvents and amounts

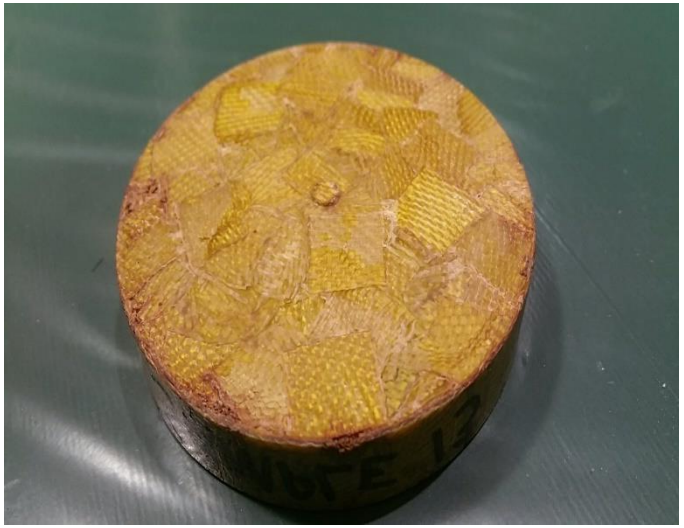
BMC Processing

- Prepreg chopped into ½”x ½” squares
- Attempt reduce resin loss
  - More gel time in oven
  - Apply gentle pressures
- Obtain density comparable to 1.71 g/cc

BMC being compressed by the weight of the mold

Test Coupons

S/DG
1.56g/cc



S/Ph
1.65g/cc

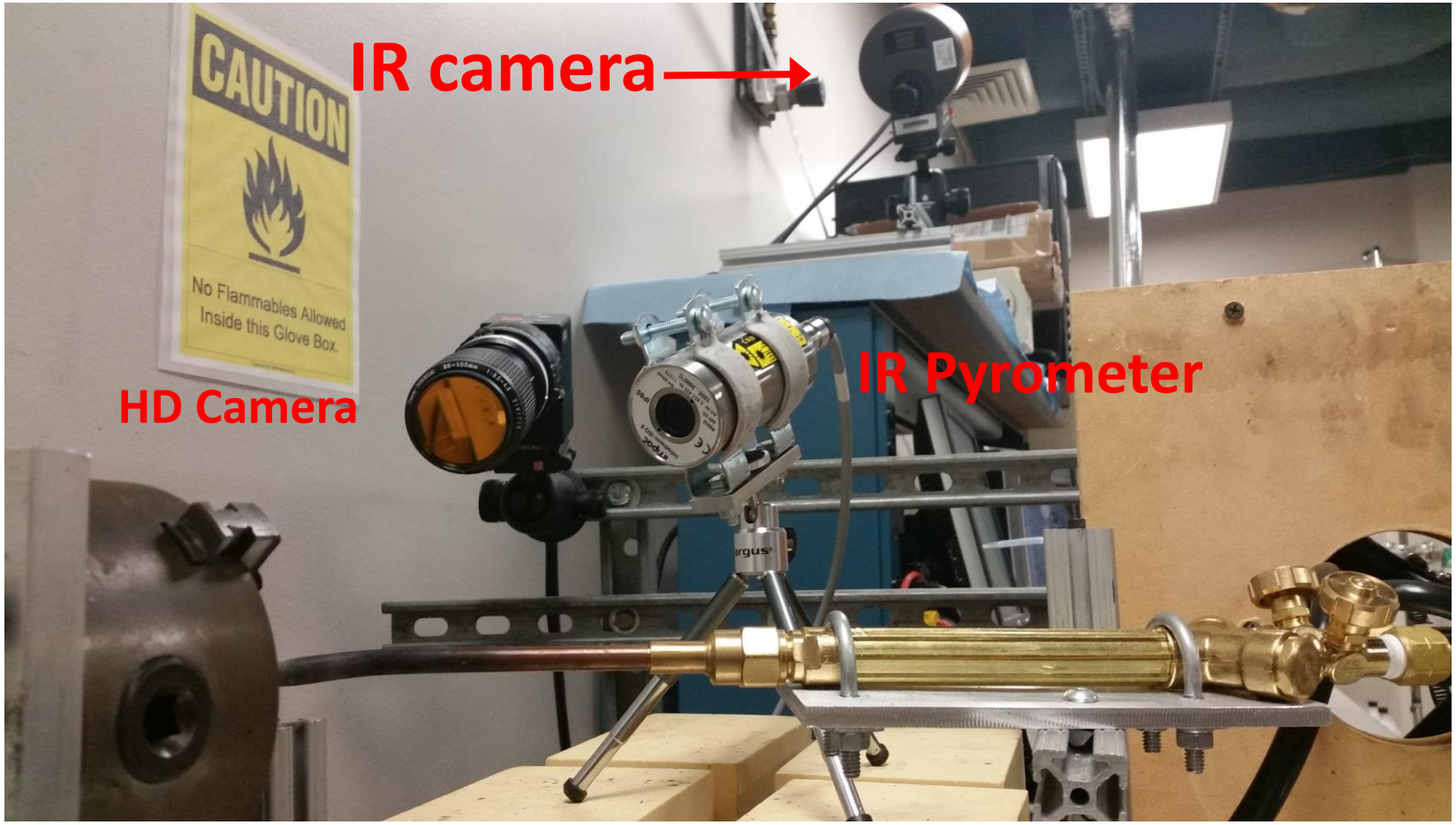
Ablation Testing

Pre-test samples

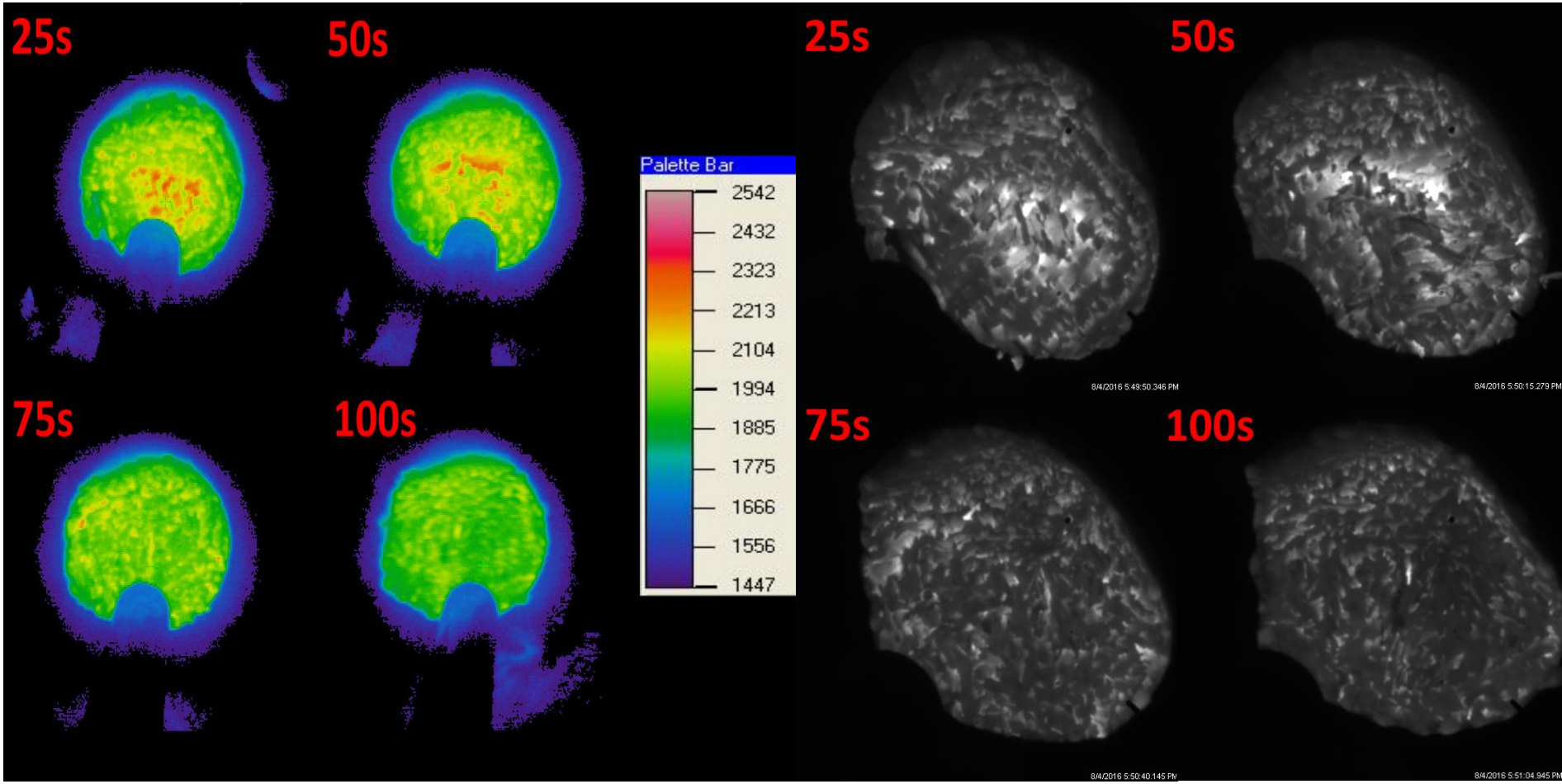
Post-test samples

Instrument Placement

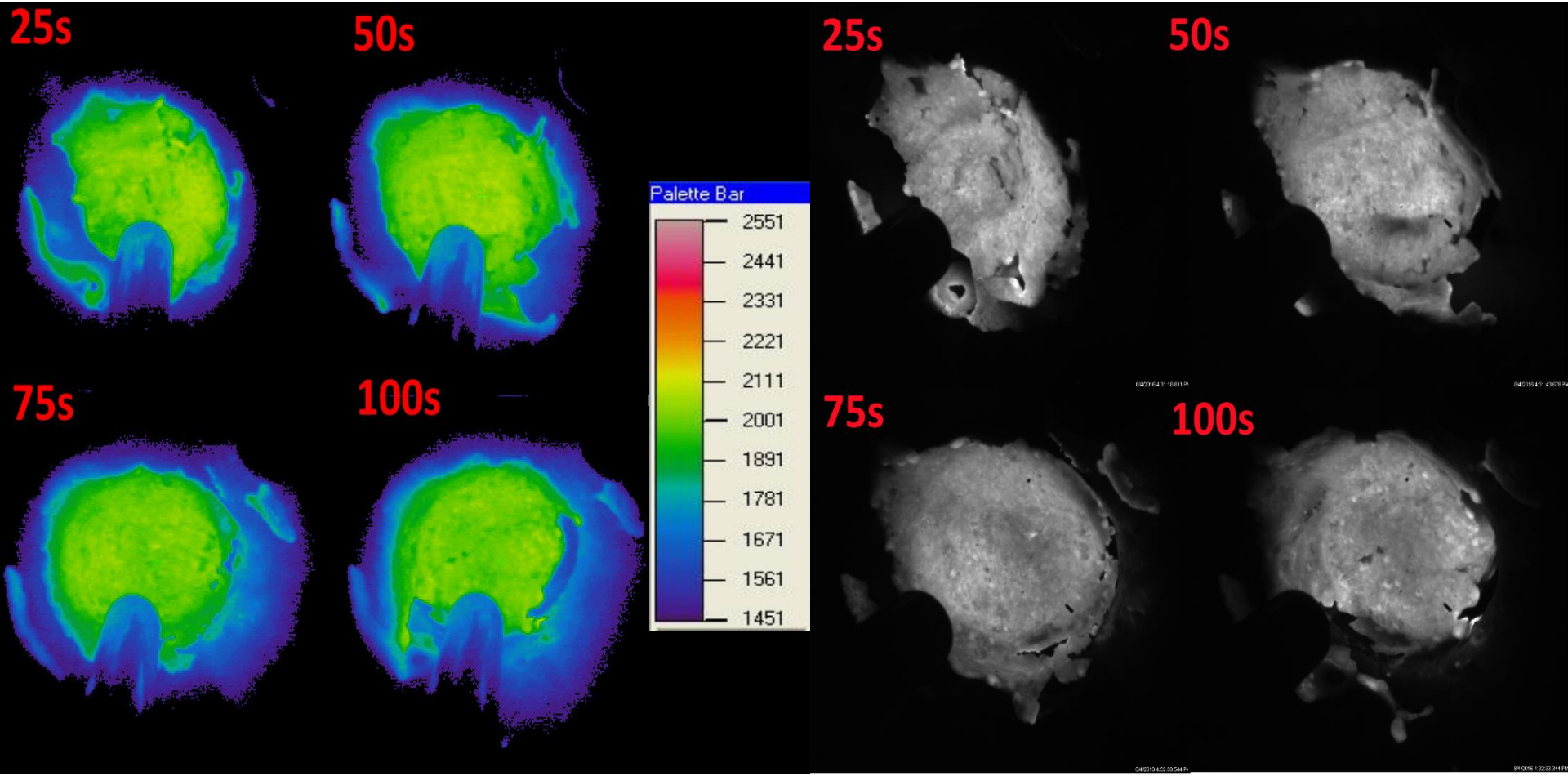
Ablation Testing



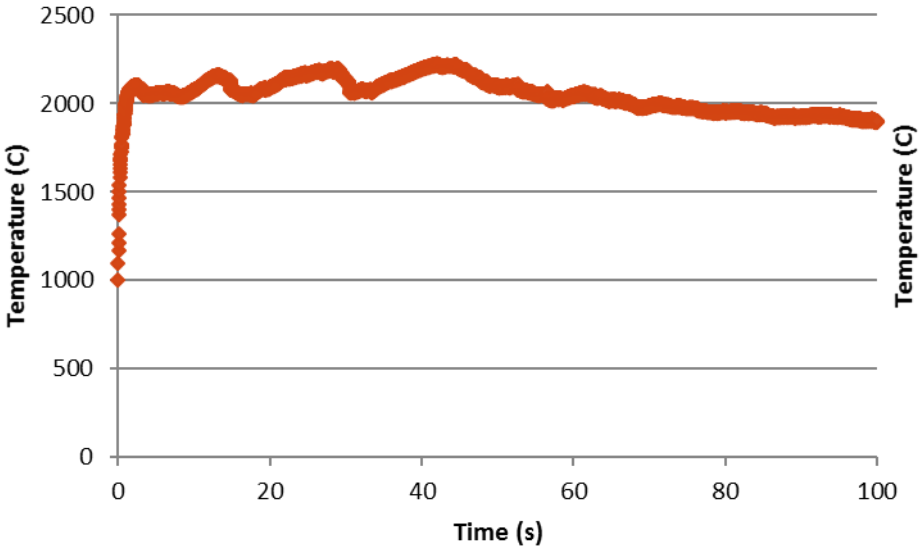
Ablation Testing S/Ph



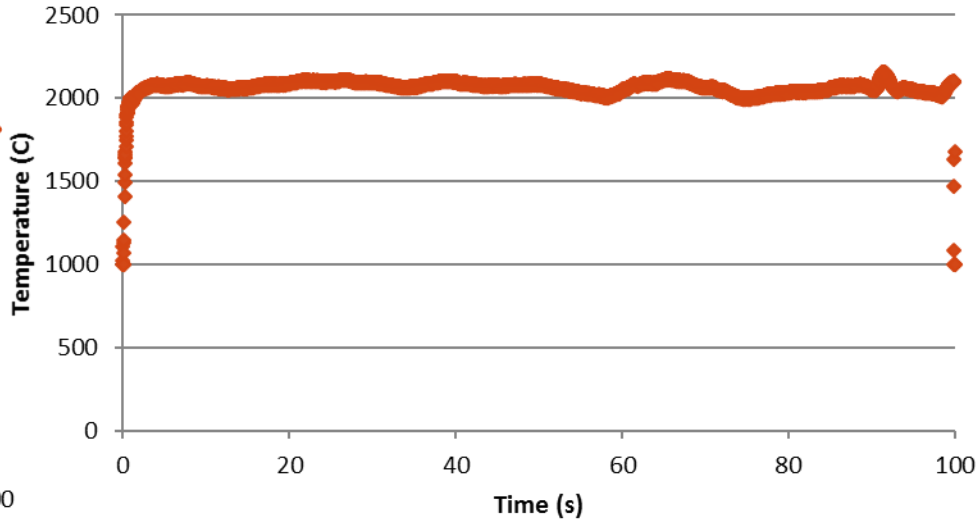
Ablation Testing S/DG



Surface Temperature



IR pyrometer data for S/Ph sample



IR pyrometer data for S/DG sample

Summary of Test 1

Material:	Silica Phenolic	Density is 1.62 g/cc							
Sample Number	Heatflux (W/cm^2)	OFR	Test Time (s)	Mass loss (%)	Recession (mm)	Recession Rate (mm/s)	Max IR Pyro Temp (°C)	Peak Heat-Soak time (s)	Peak Heat-Soak Temp (°C)
4	867	4.8 : 4	100*	47.90	5.46	0.05	2159		**
6	867	4.8 : 4	180	61.18	10.25	0.06	2146		**
8	867	4.8 : 4	173*	61.18	8.66	0.05	2128		**
5	867	4.8: 4	100	31.70	3.17	0.03	2226	139	110

*Testing time was reduced on some trials due to flame deflecting

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Summary of Test 2

Material:	Silica/ DT 1100	Density is 1.56 g/cc							
Sample Number	Heatflux (W/cm^2)	OFR	Test Time (s)	Mass loss (%)	Recession (mm)	Recession Rate (mm/s)	Max IR Pyro Temp (°C)	Peak Heat-Soak time (s)	Peak Heat-Soak Temp (°C)
4	867	4.8 : 4	180	52.82	7.63	0.04	2220	190	313
7	867	4.8: 4	180	44.81	8.09	0.04	2243		**
9	867	4.8: 4	139*	40.45	6.91	0.05	2264		**
6	867	4.8: 4	100	28.20	3.6	0.04	2161	100	354
8	867	4.8: 4	100	29.36	3.9	0.04	2158	100	304

** TC failure during test.

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S/DG-1 (t-butyl)

S/DG-1, 1.56g/cc

S/DG (IPA)

S/DG-2, 1.62g/cc

S/DG-3, 1.55g/cc

Conclusion

- DG-1 exhibited the best results with 87% char yield. An increase of ~54% compared to phenolic and cyanate ester resins.
- DG-1 had a HRC of 36 J/g-K. SC-1008 phenolic's HRC was 48% higher at 53.31 J/g-K and PT-15 cyanate ester's HRC was 443% higher at 159.33 J/g-K
- Preliminary ablation data from non-ideal sample looks promising
- Better processing method needed to obtain best quality samples

Future Work

- Test various S/DG Resin: Fiber ratios
- Ablation testing using Oxygen-Acetylene Test Bed (OTB) and Inductively Coupled Plasma (ICP) torch
- Dispersion of nanosilica into the resin
- Incorporation of aerospace grade silica fabric into the polysiloxane resin
- Mechanical properties
- Incorporation of Nafen alumina nano-fiber