Development of New Thermal Protection Systems Based on Polysiloxane/Silica Composites*

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Today's Presentation

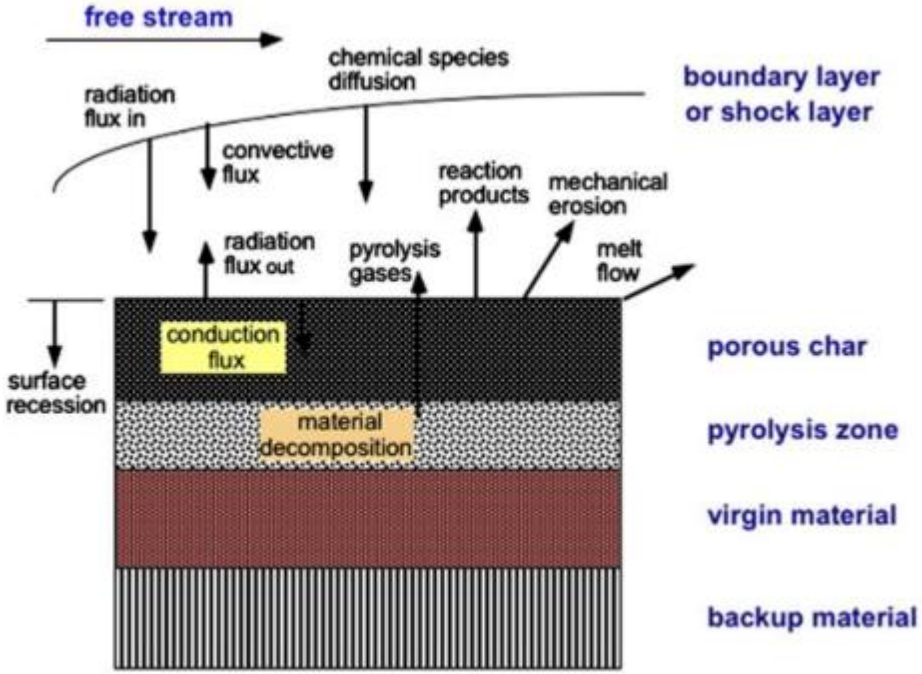
- Background and Motivation
- Research Objective
- Characterization of the Neat Resins
- Sample Manufacturing
- Ablation Testing
- Microstructure Analysis
- Conclusion and Future work

Background

- Want to investigate new ablative material systems
- Need for materials which can withstand harsher environments
  - Rocket Motors
  - TPS Materials for Re-entry Vehicle
  - Nose Cones – Atmospheric Probes
  - Vertical Launching Systems
  - Fire Prevention – Trains, Submarines, etc.

Ablative Materials

- Resist thermochemical erosion caused by harsh environment
- Formation of the protective char layer
- Different mechanisms for different materials, no one size fits all ablative
- Current SOTA resins: SC-1008, PT-15



Research Objective

- Using legacy material as control, S/Ph
- MX2600 TDS lists 30-35% resin content
- Goal of creating testing various resin content S/DG-UHTR samples
- Find optimal resin:fiber ratio

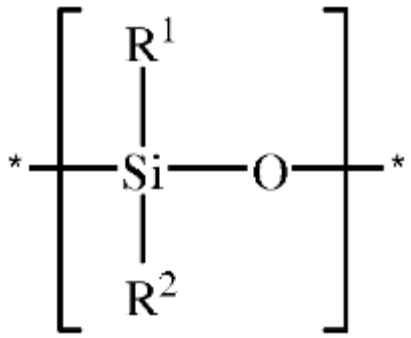
SC-1008 Phenolic

- MIL-standard phenolic resole resin manufactured by Hexion
- Typically carbon or silica fiber reinforcement
- Foamed versions of phenolic used for low density ablators
- Lots of data collected
- Diverse applications, from TPS materials to rocket motor materials
- Relatively cheap

Silica/Phenolic composite

DG Polysiloxane

- Inorganic matrix, utilizing a mixture of polysiloxane chemistries manufactured Dyna-Glas Technologies LLC
- Pre-ceramic material
- High char yield
- Low heat release rate and heat release capacity
- Good compatibility with silica fabric



Siloxane

Silica-/DG-UHTR composite

Material Characterization

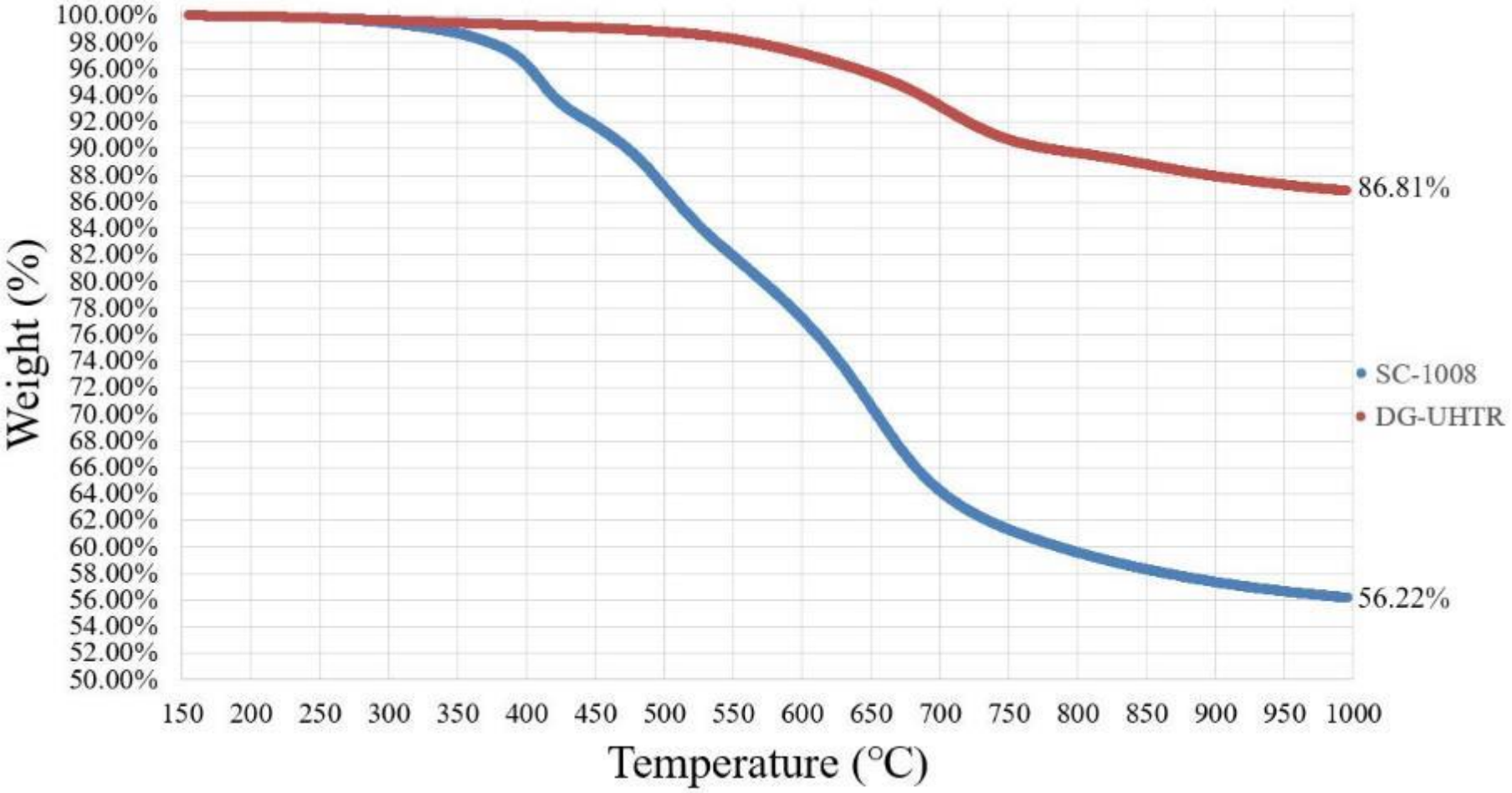
- Thermogravimetric Analysis
  - Thermal Stability & Char Yield
- Microscale Combustion Calorimeter
  - Heat Release Rate and Capacity
- Density
  - Water Displacement
- Oxygen-Acetylene Test Bed
  - Ablative Performance

Char Yield Study

1. Dry the TGA sample 150°C for 30min
2. Consistent sample size – 20mg
3. TGA heating rate of 20°C/min in nitrogen
4. Char yield is defined as the %mass remaining at 1,000°C

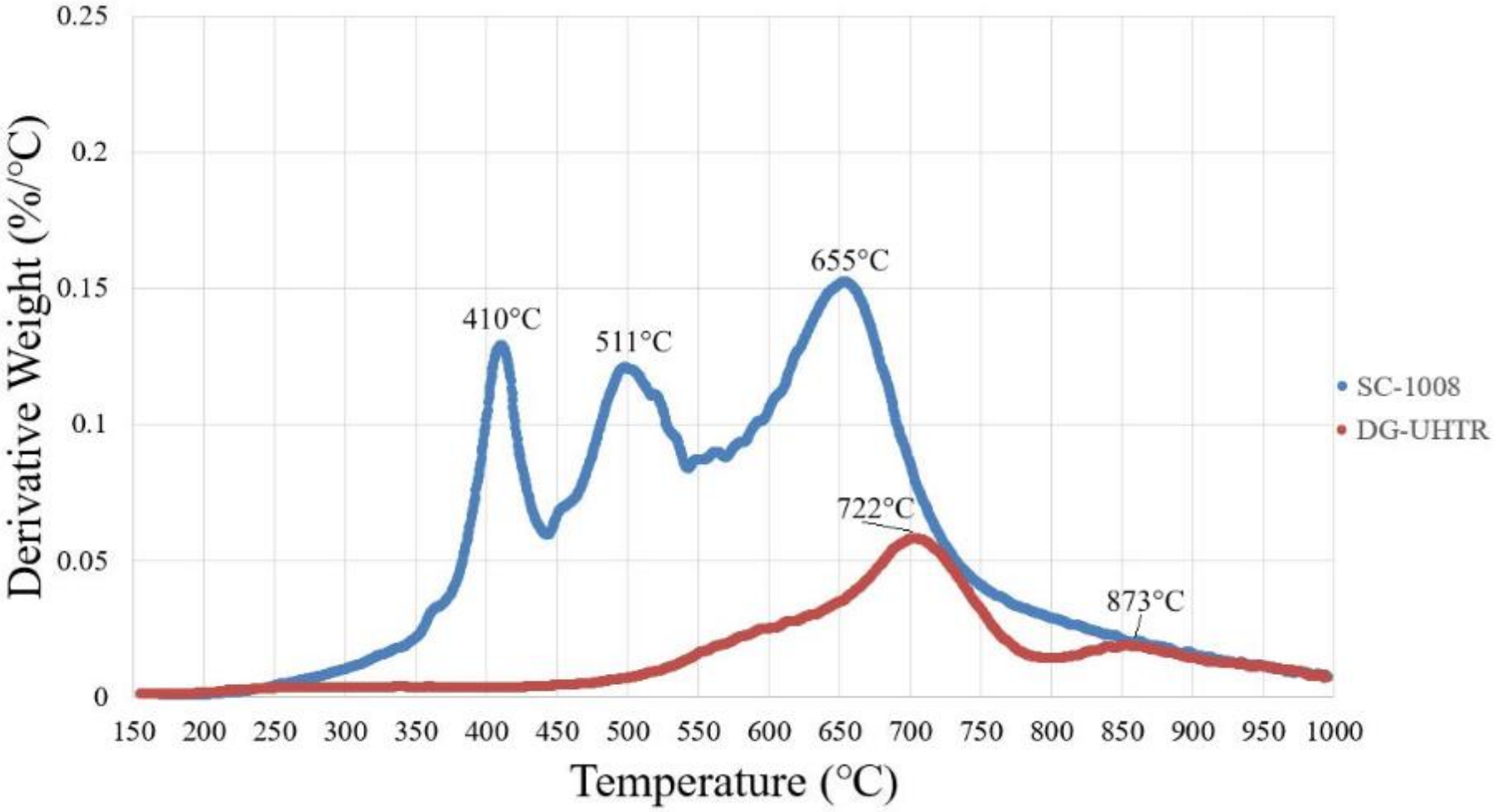
Developed based on a NASA report on PICA

Char Yield Study



Char yield results for the neat SC-1008 and DG-UHTR resins

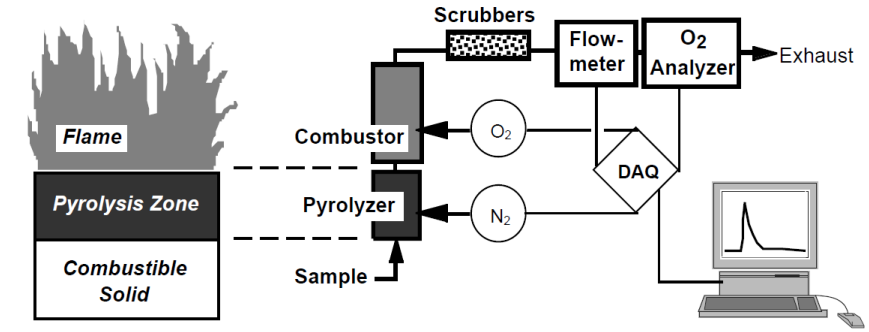
Char Yield Study



dTGA for the neat SC-1008 and DG-UHTR resins

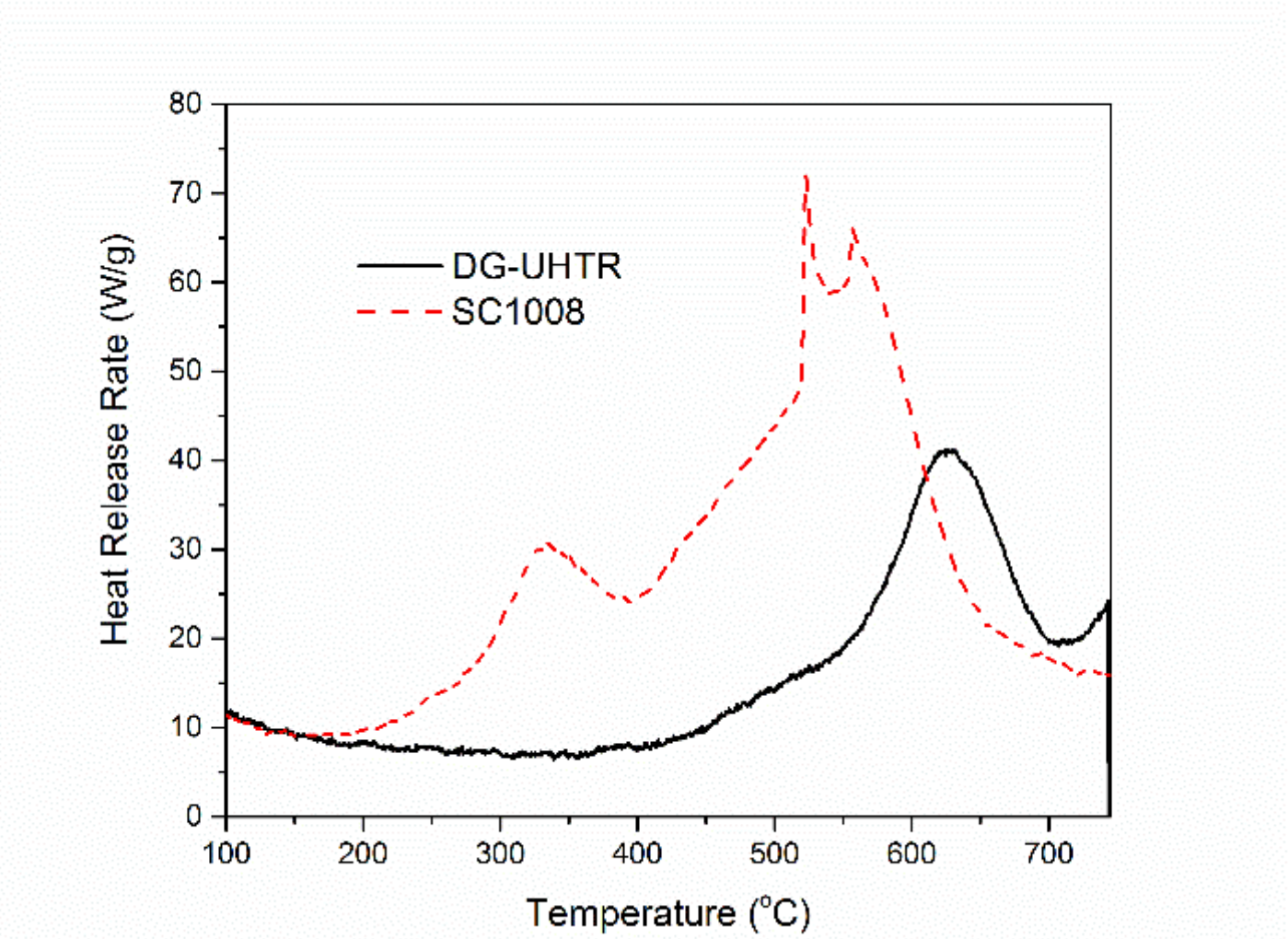
Flammability Properties

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



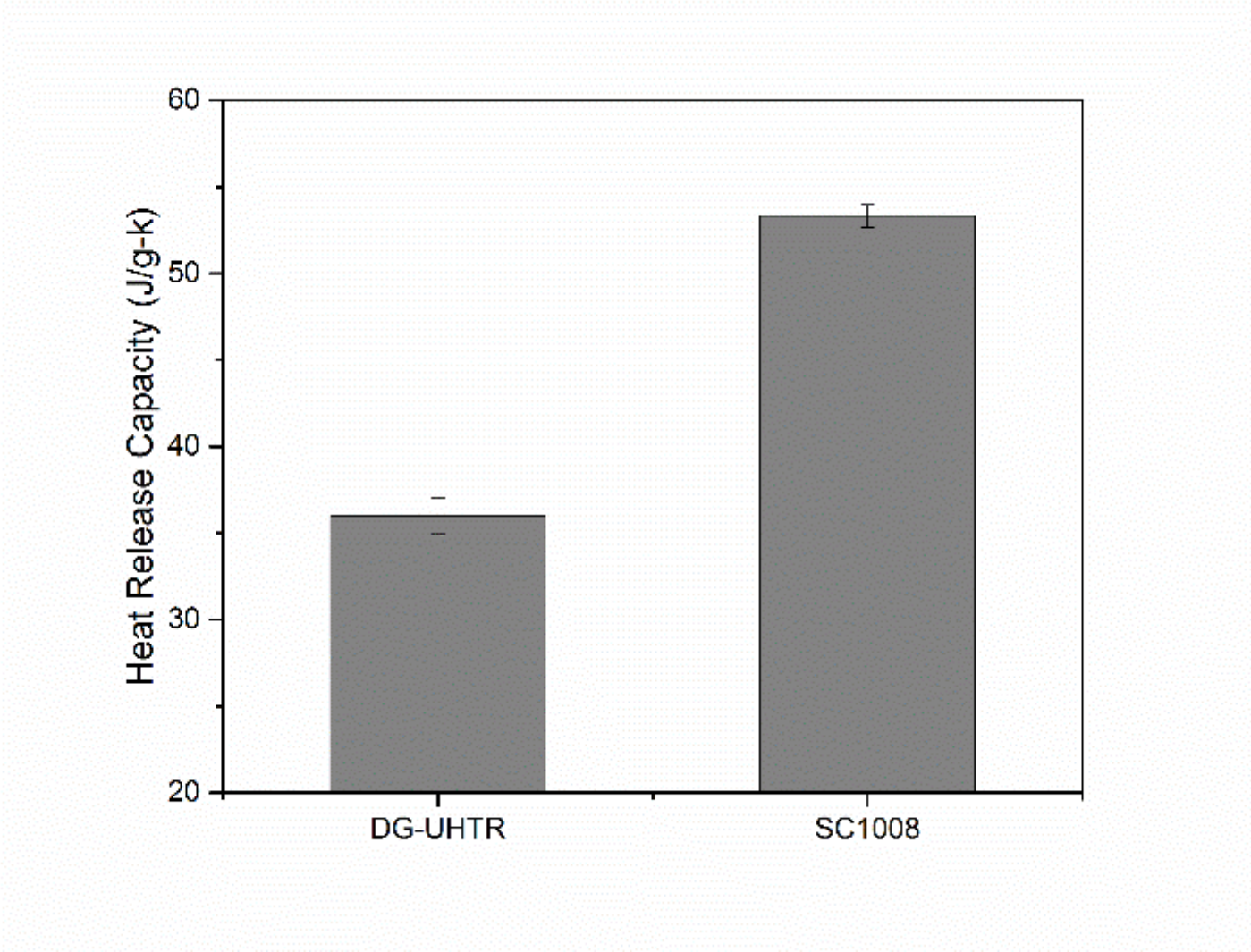
Flaming Combustion *Pyrolysis-Combustion Flow Calorimeter*

Flammability Properties



Typical heat release curves for the two resin systems

Flammability Properties



Comparison of the Heat Release Capacities for the four resin systems

Sample Preparation

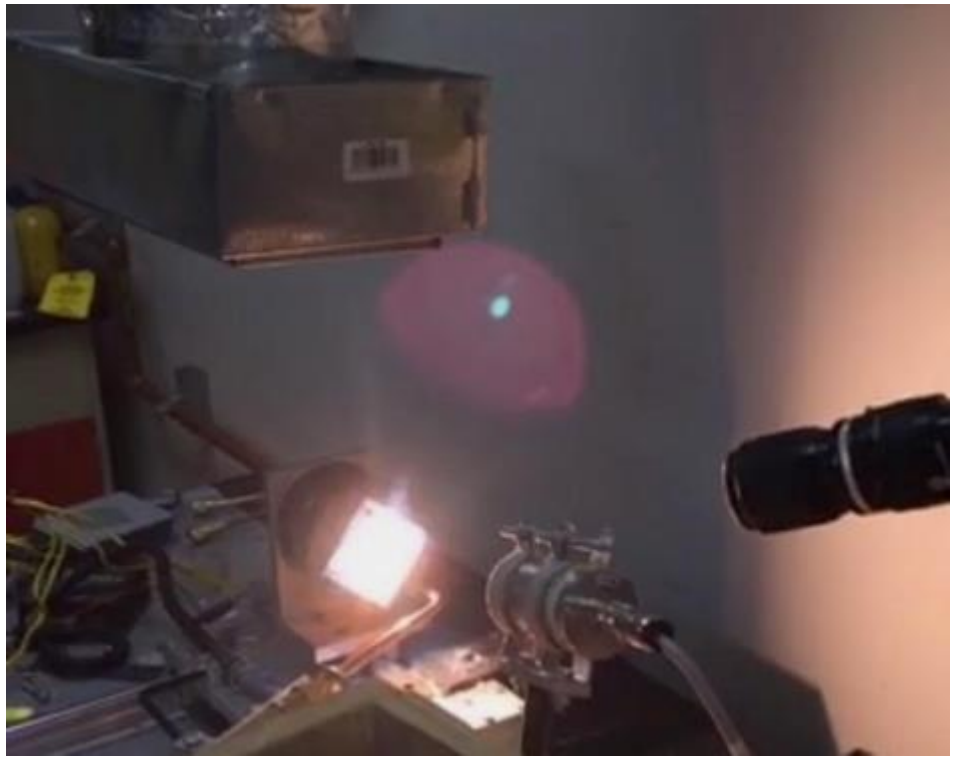
- Wet lay-up
  - Hand lay-up of fabric with resin
  - Make partially cured pre-preg
- Compression mold
  - 170°C 1hr
  - 250°C 2hr
- Water jet samples
  - 15mm diameter
  - 15-17mm thick
- Drilled holes
  - 10mm from the surface

Test Samples

Sample ID	Resin %	Fiber %	Silica filler %	Nanosilica wt% (in resin)
S/Ph MX2600	30-35% (burn-off)	64%	4.5%	0%
S/DG-UHTR F1	35%	65%	0%	0%
S/DG-UHTR F2	40%	60%	0%	0%
S/DG-UHTR F3	48%	52%	0%	0%
S/DG-UHTR F4	35%	75%	0%	3%

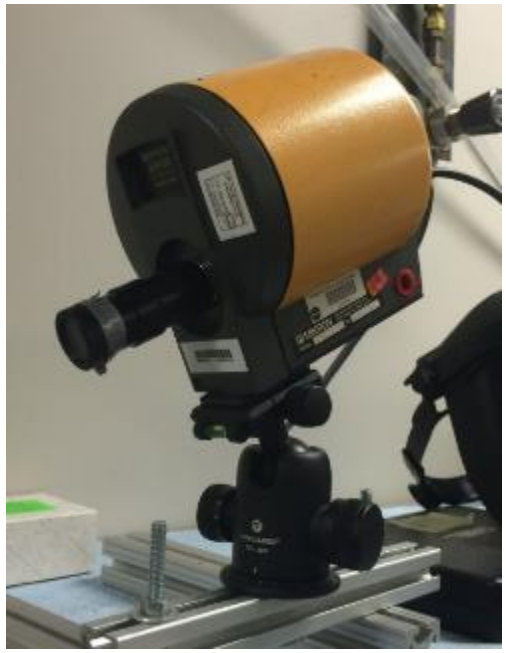
Oxygen-Acetylene Test Bed

- Welding torch setup utilizing high flow rates
- Utilized a 1:1 Oxygen to Fuel ratio
- Tested at a heat flux of 1000W/m^2, verified using gardon gauge
- 40s exposure time
- Carbon-carbon shield



Experimental Setup

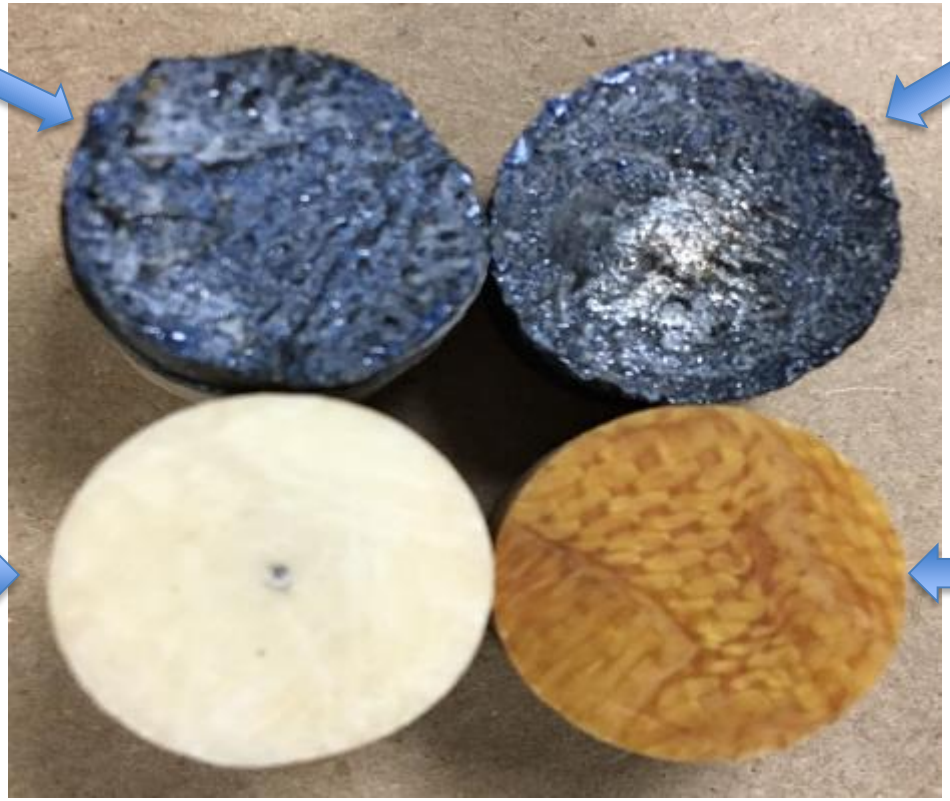
- Test Equipment
  - Two color Pyrometer
  - IR camera
  - HD camera
  - K-type thermocouple



Ablation Testing

S/DG-UHTR
Post-test

S/Ph
Post-test

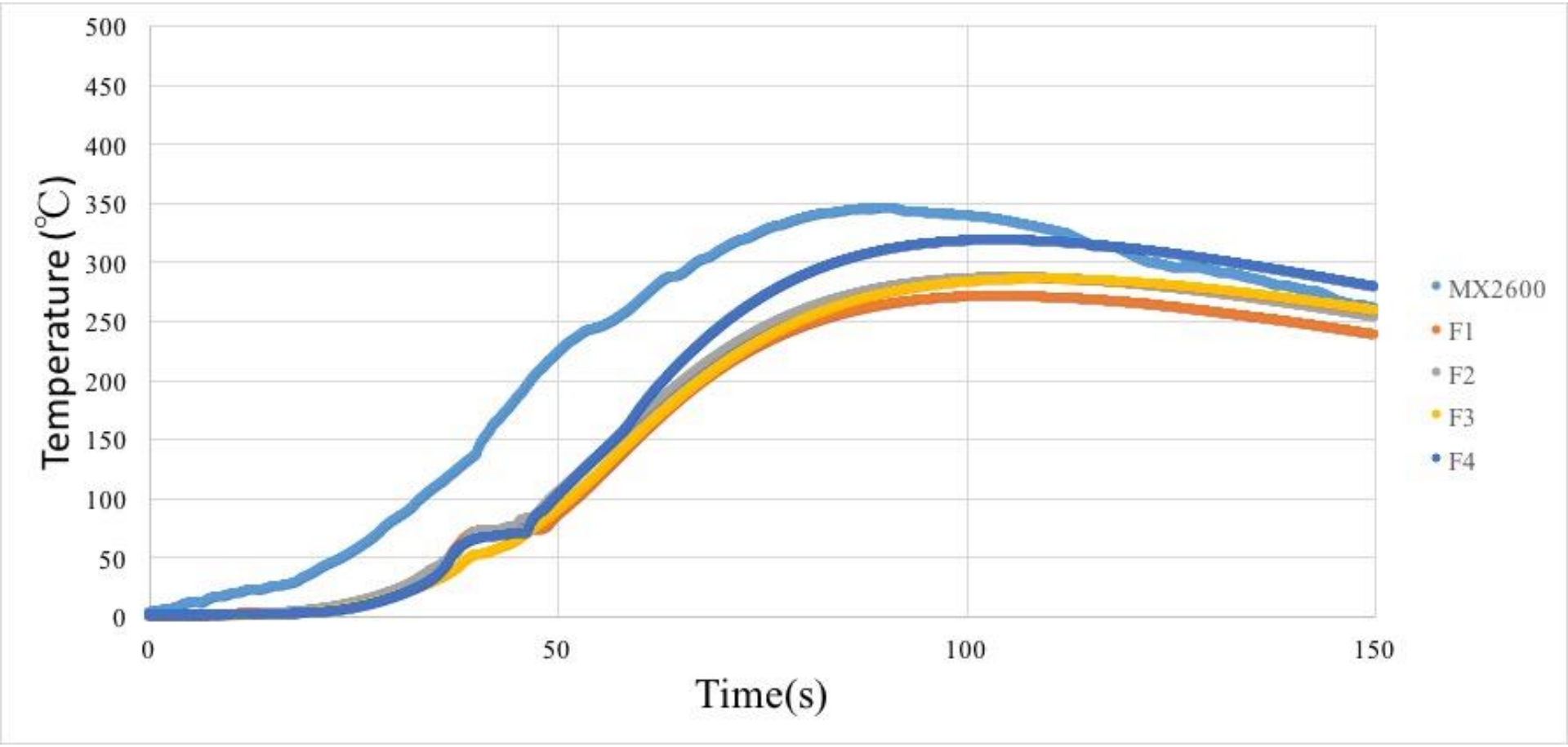


S/DG-UHTR
Pre-test

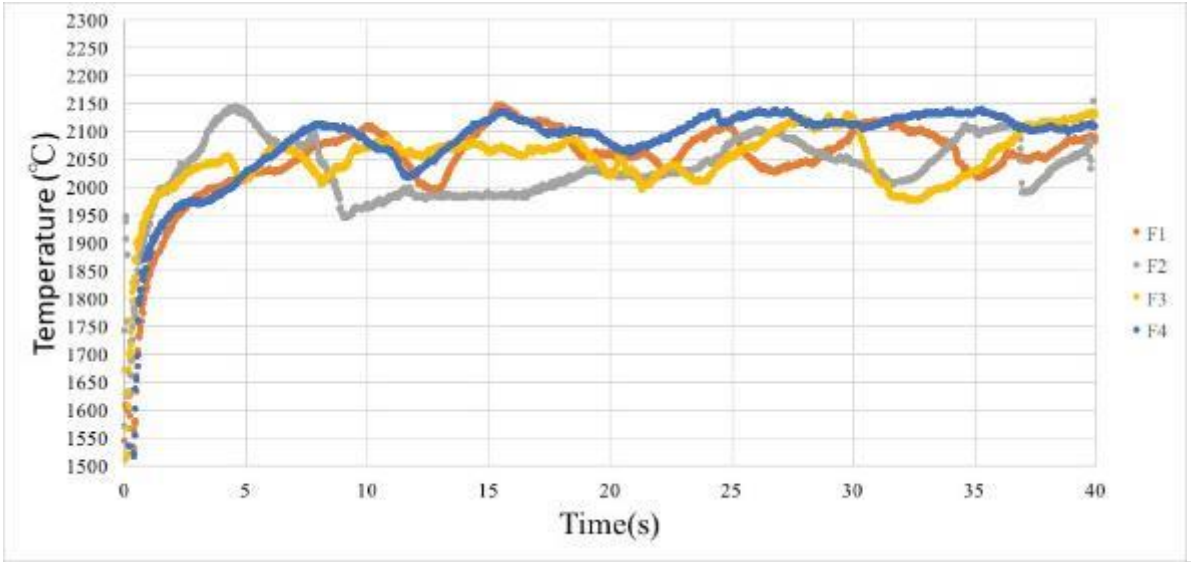
S/Ph
pre-test

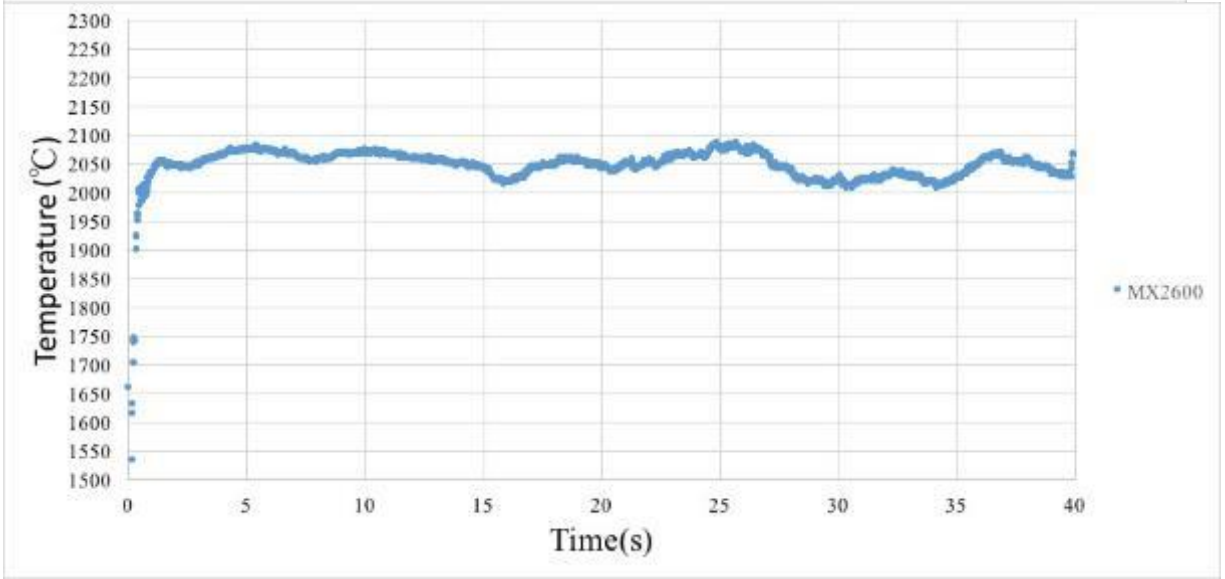
15mm diameter x 15mm thick samples

Thermocouple Data

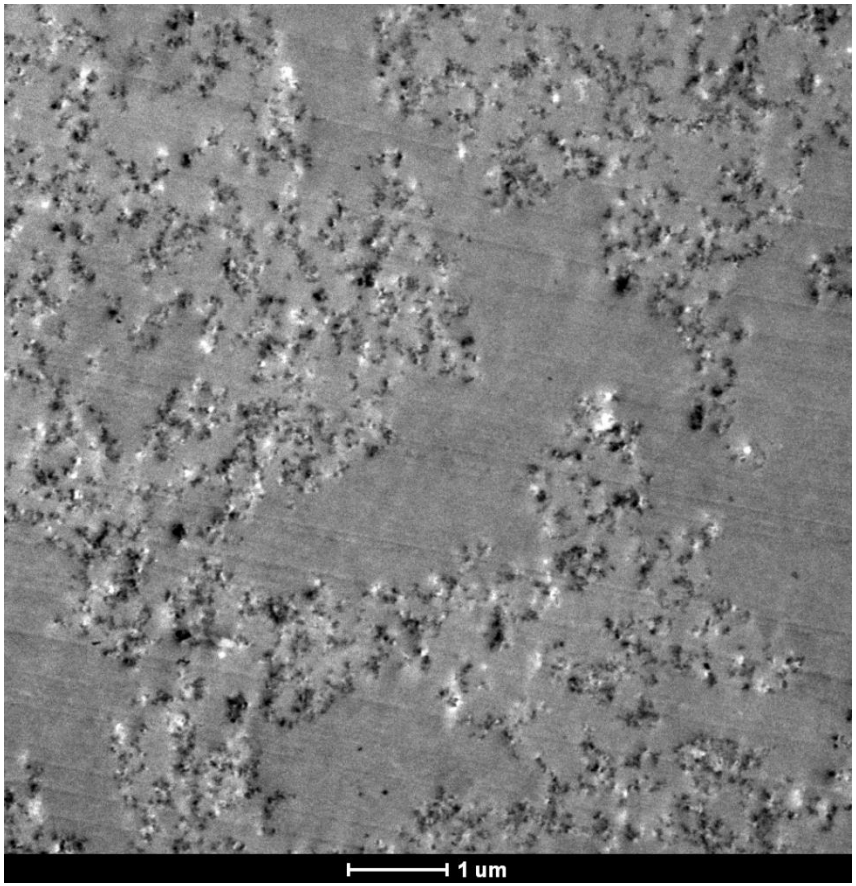


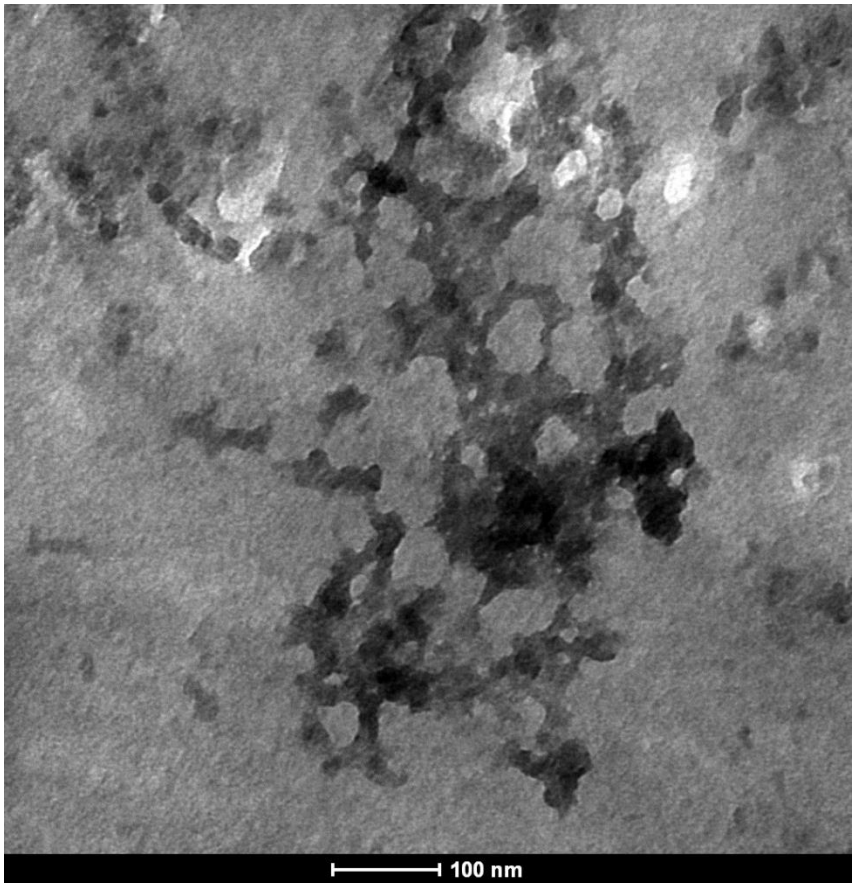
Surface Temperature Data



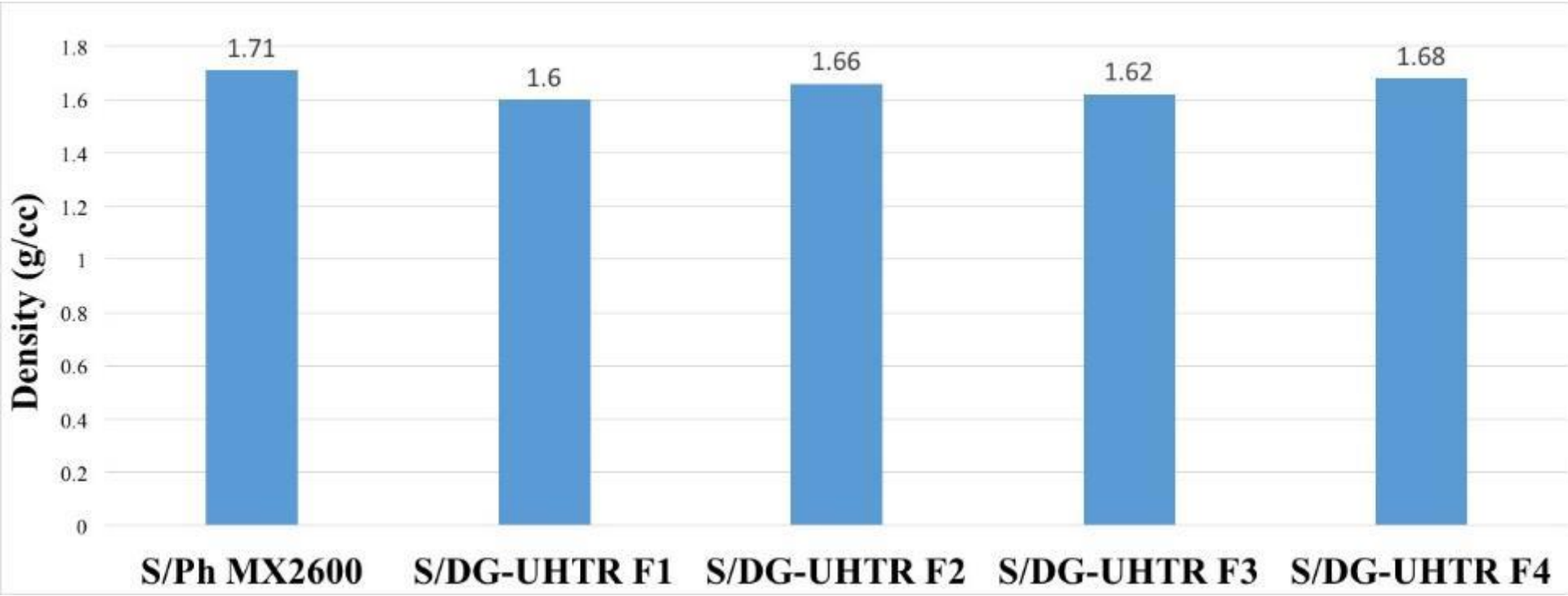


TEM



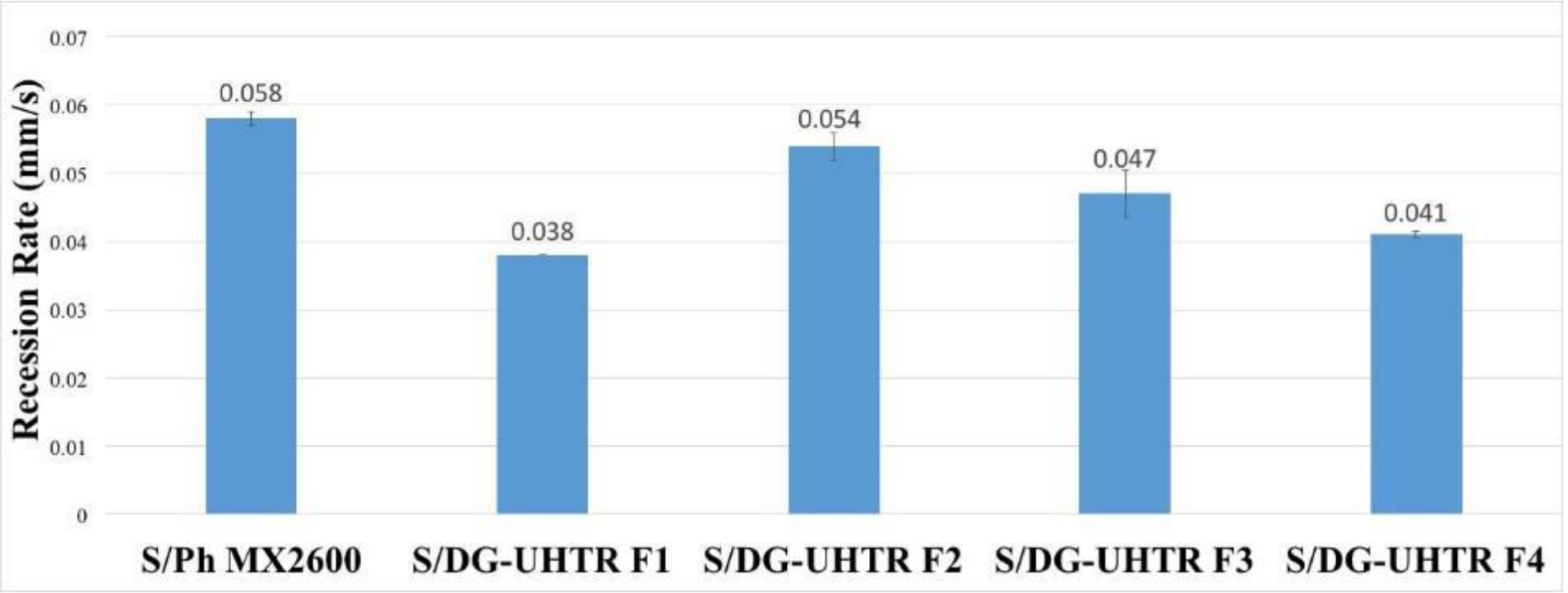


Density



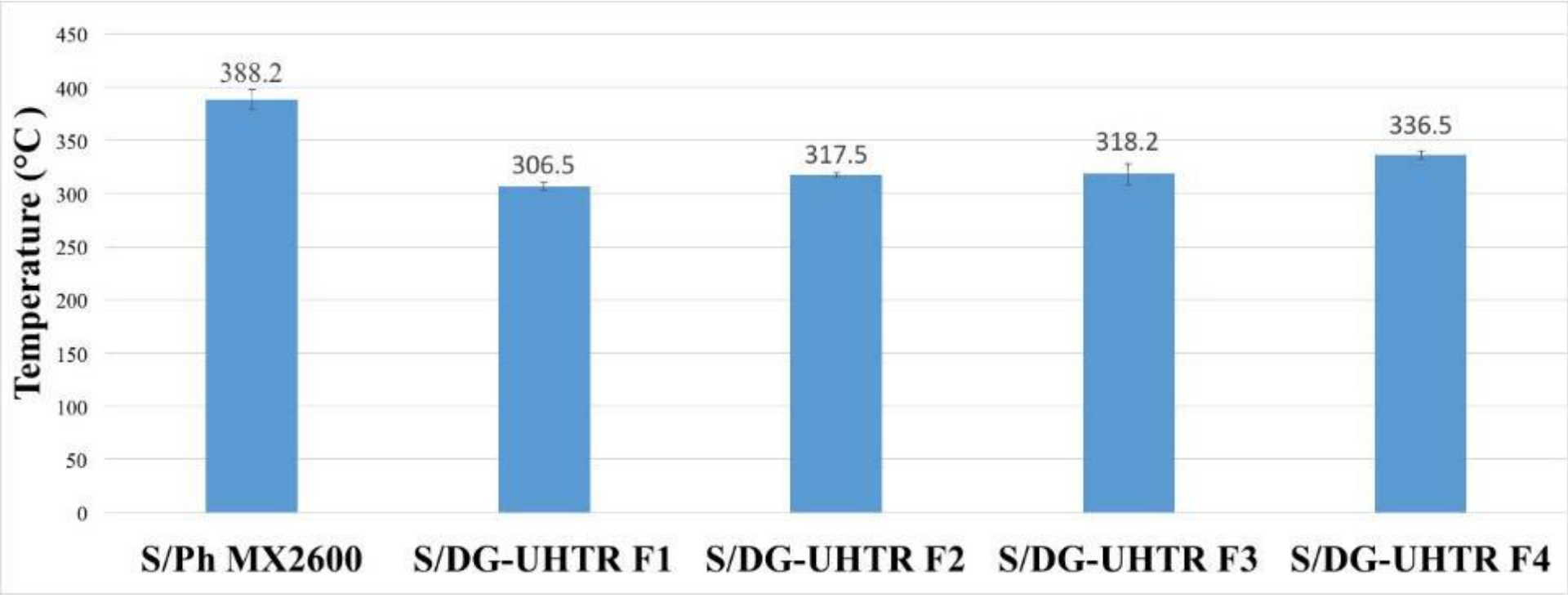
Resin:	**30-35wt%**	**35wt%**	**40wt%**	**48wt%**	**35wt% (3wt% n-silica)**

Recession Rate



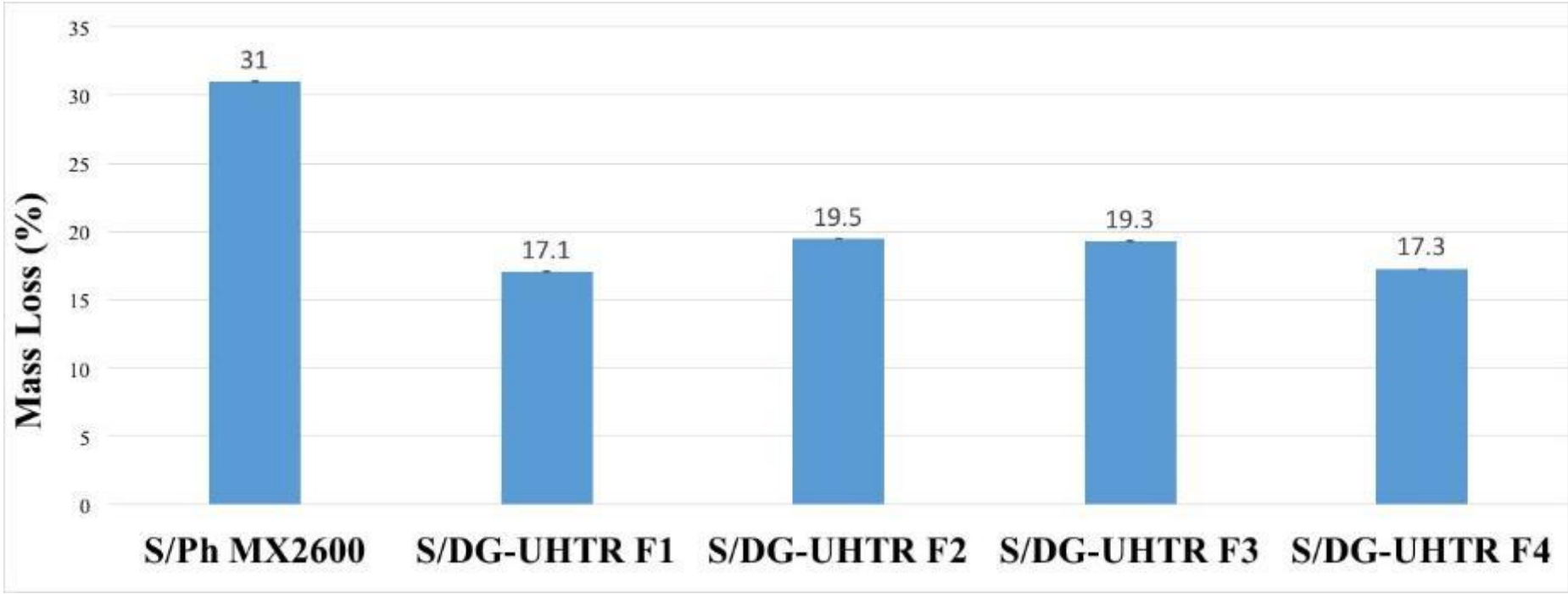
Resin:	**30-35wt%**	**35wt%**	**40wt%**	**48wt%**	**35wt% (3wt% n-silica)**

Heat Soak Temperature



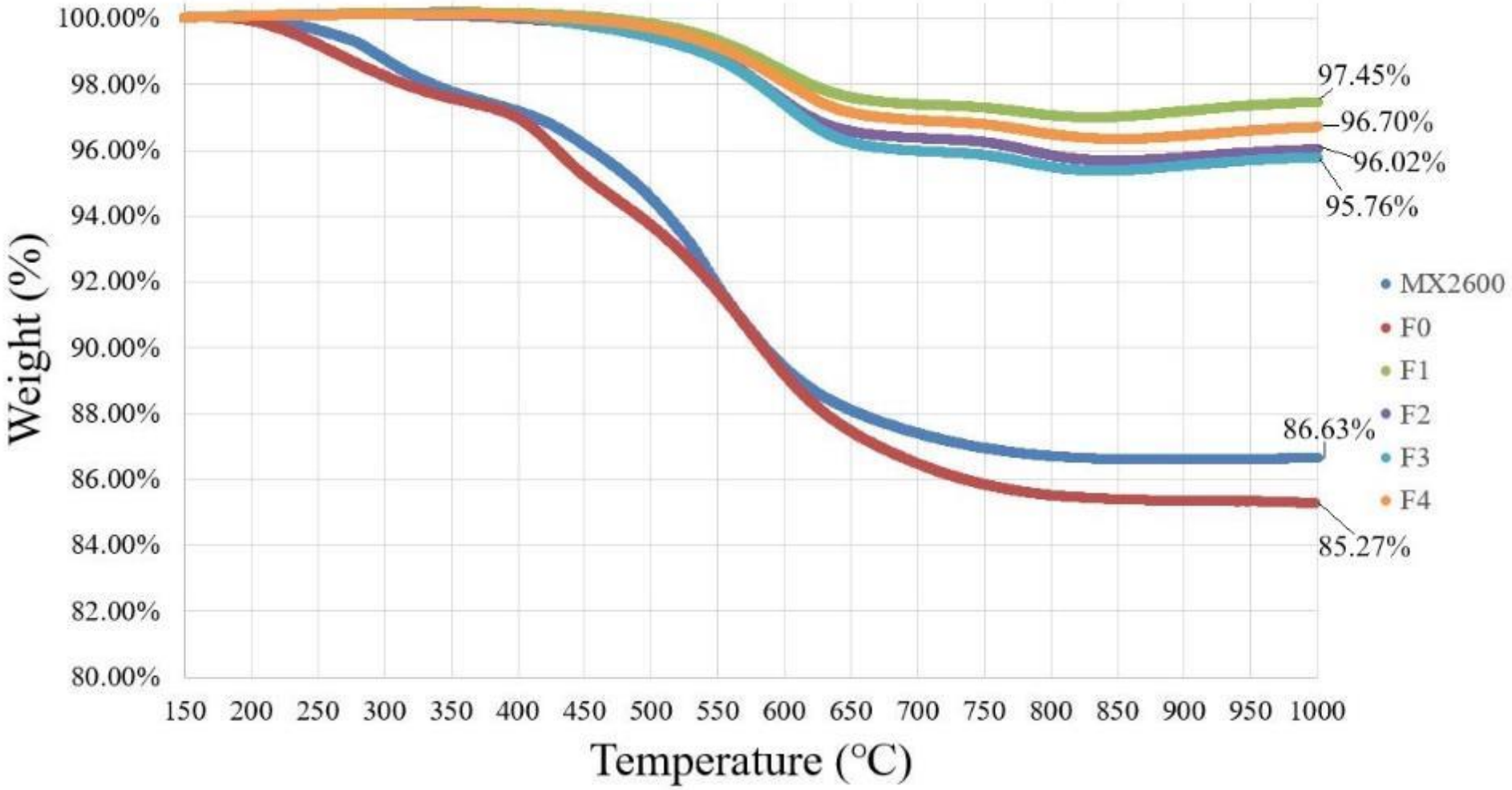
Resin:	**30-35wt%**	**35wt%**	**40wt%**	**48wt%**	**35wt% (3wt% n-silica)**

Mass Loss Percent

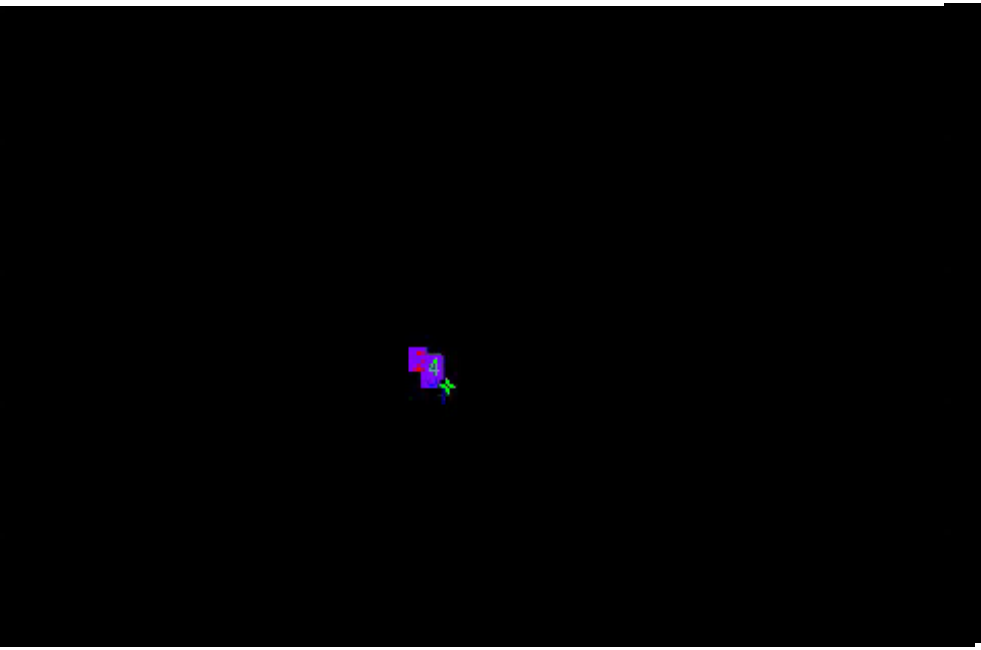


Resin:	**30-35wt%**	**35wt%**	**40wt%**	**48wt%**	**35wt% (3wt% n-silica)**

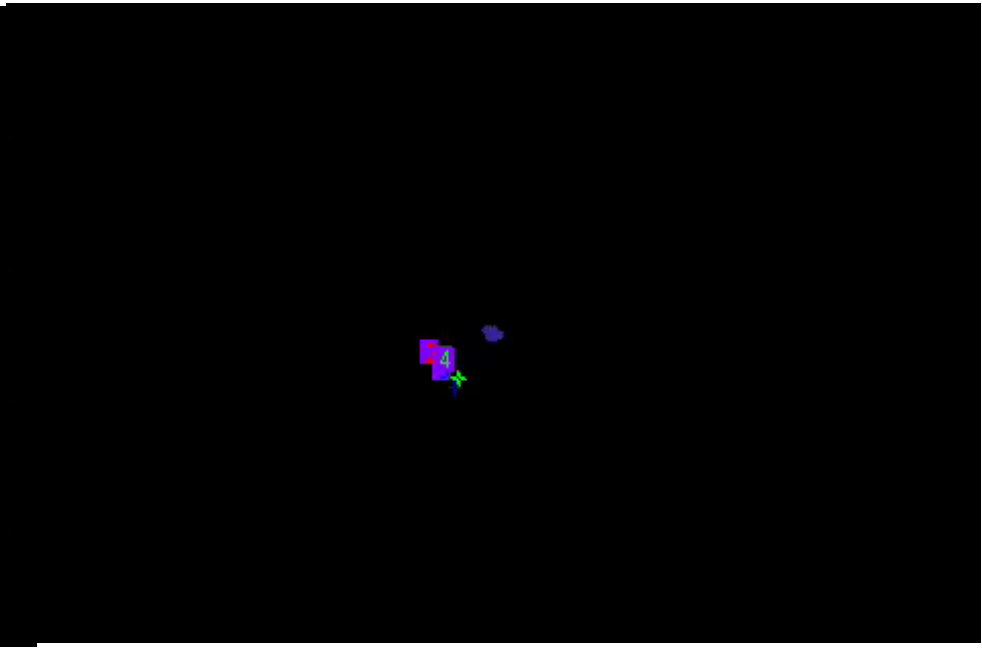
TGA


100.00%
98.00%
96.00%
94.00%
92.00%
90.00%
88.00%
86.00%
84.00%
82.00%
80.00%
Weight (%)
150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000
Temperature (°C)
97.45%
96.70%
96.02%
95.76%
86.63%
85.27%
MX2600
F0
F1
F2
F3
F4

IR Video



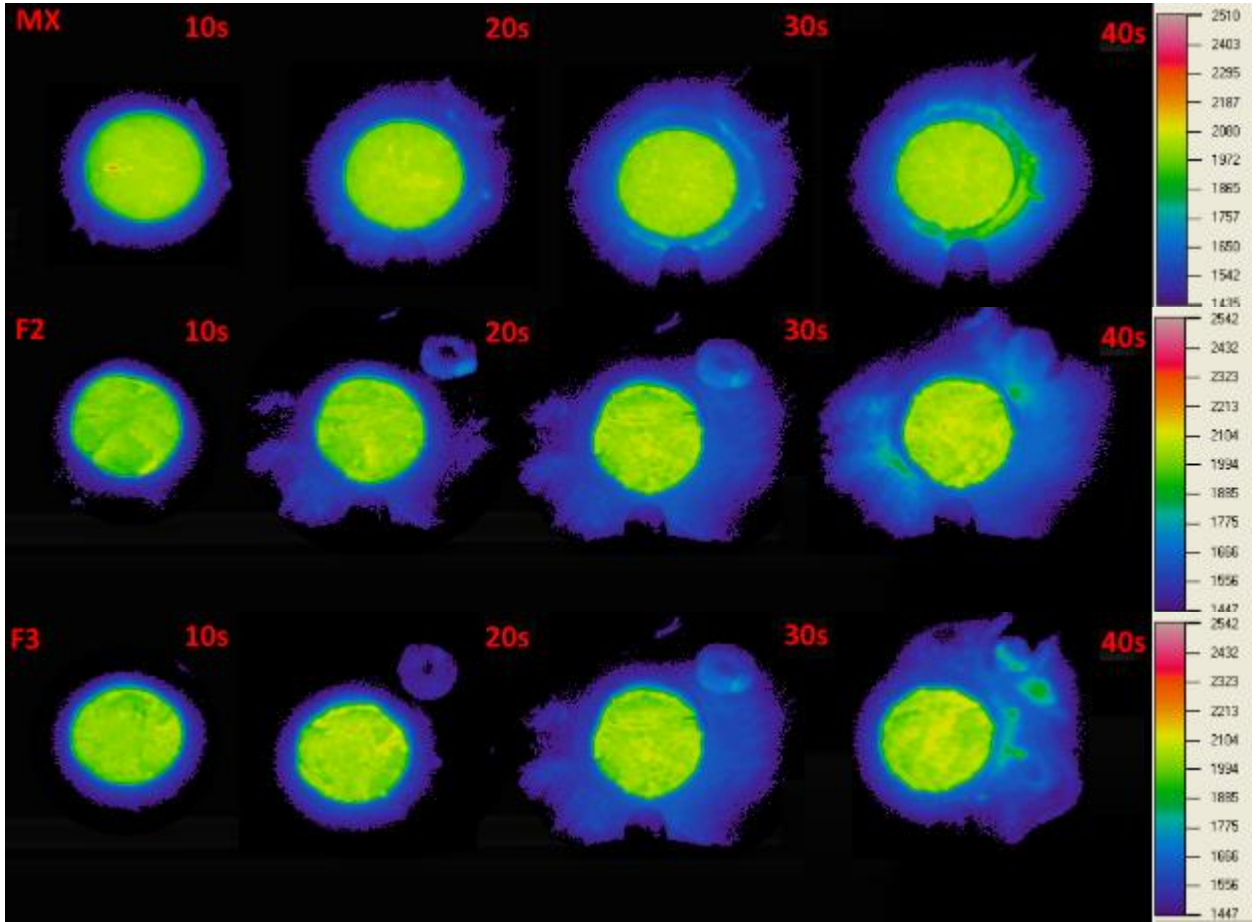
S/Ph MX2600



S/DG-UHTR F2

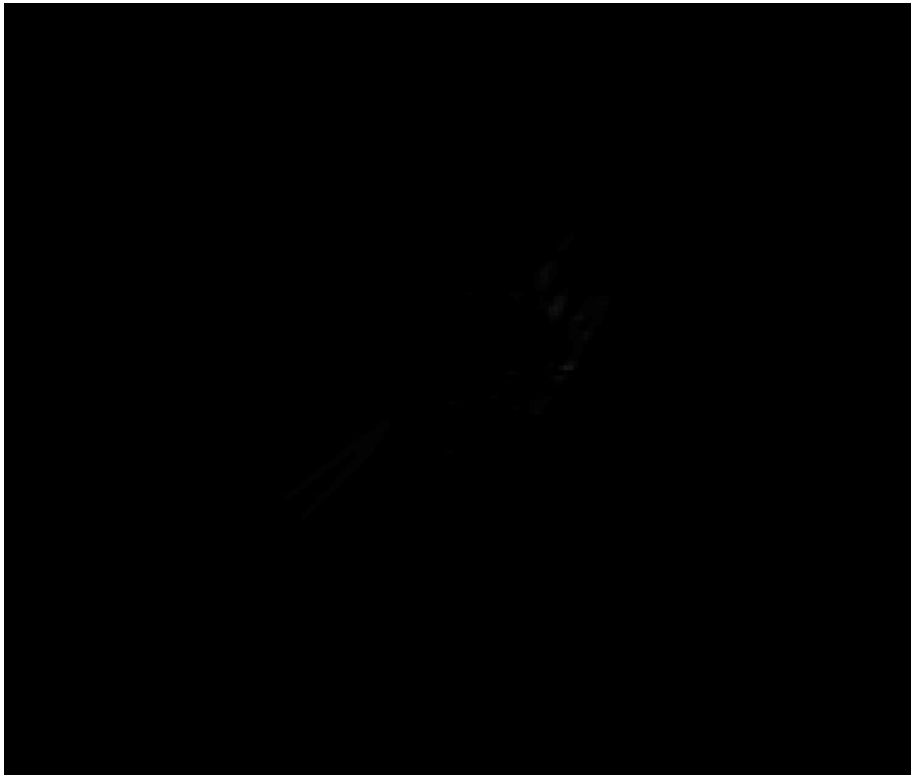
IR Video

- Growing blue ring around the sample is the radiative heat
- Yellow/Green circle is the physical sample



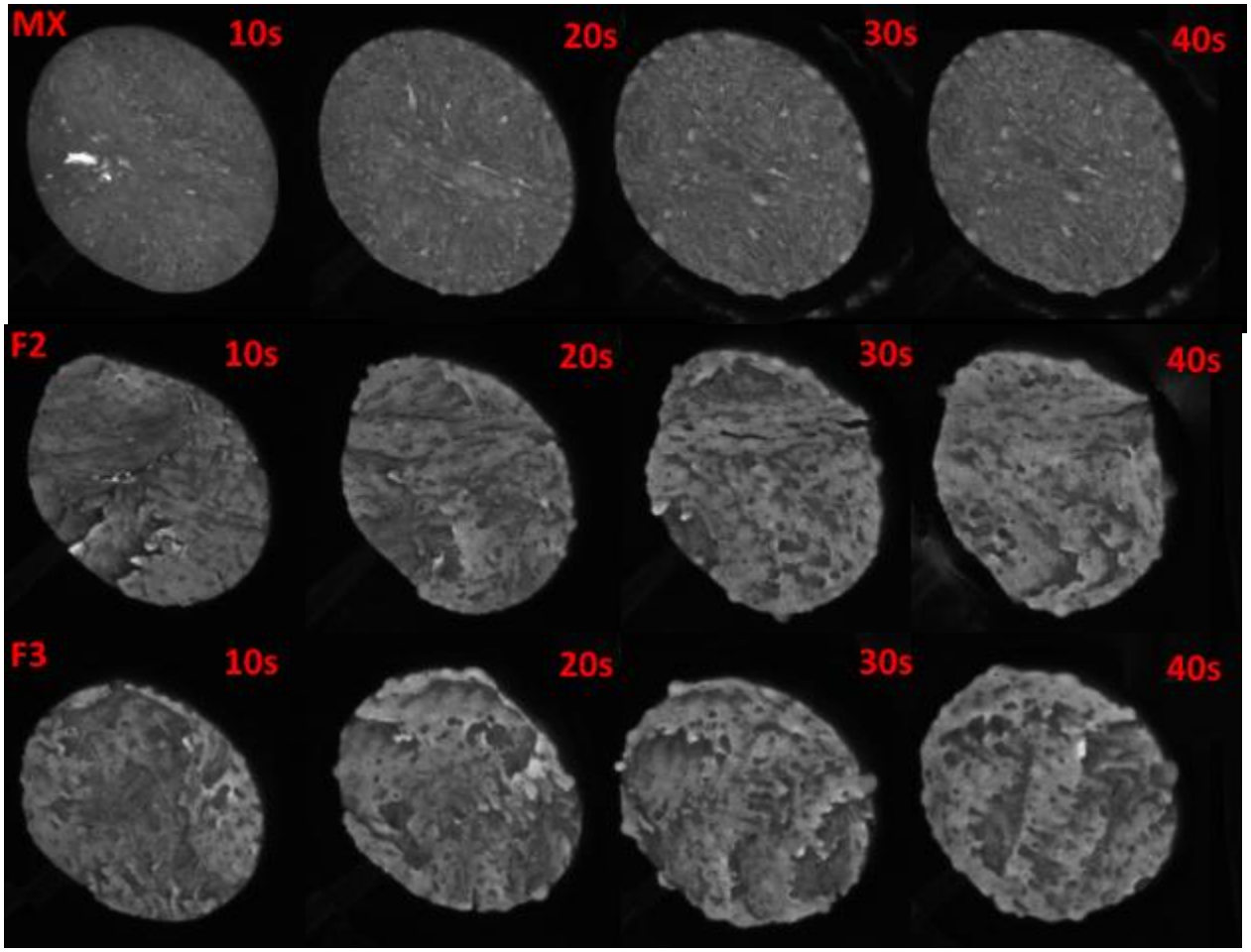
HD Video

S/Ph MX2600

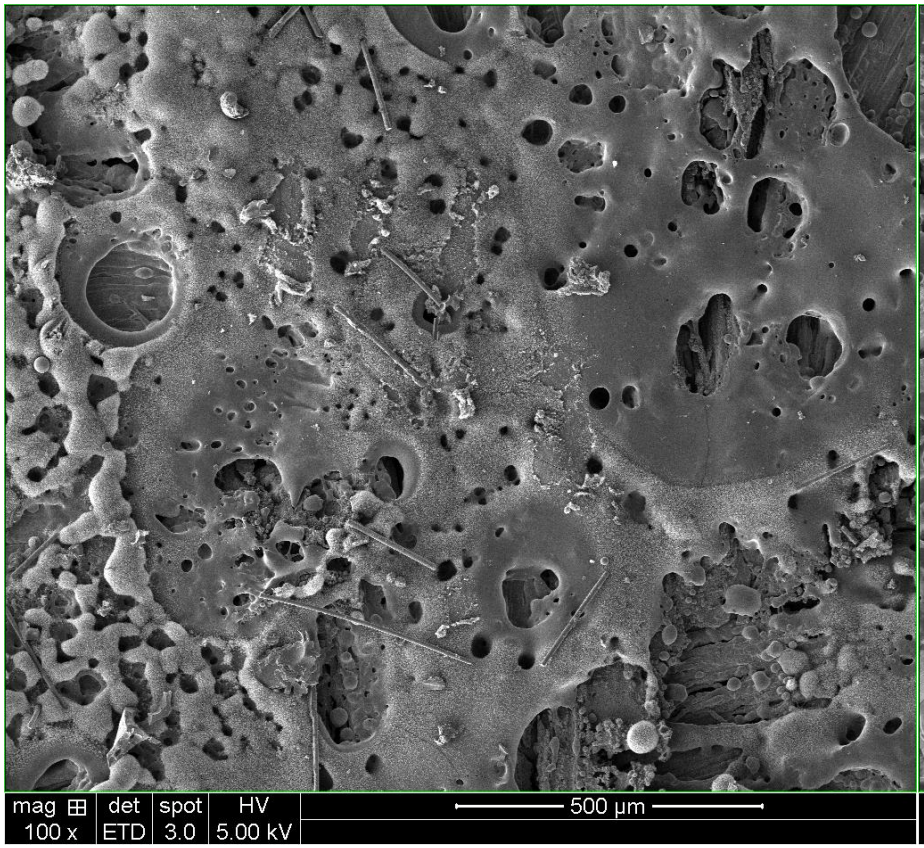


S/DG-UHTR F2

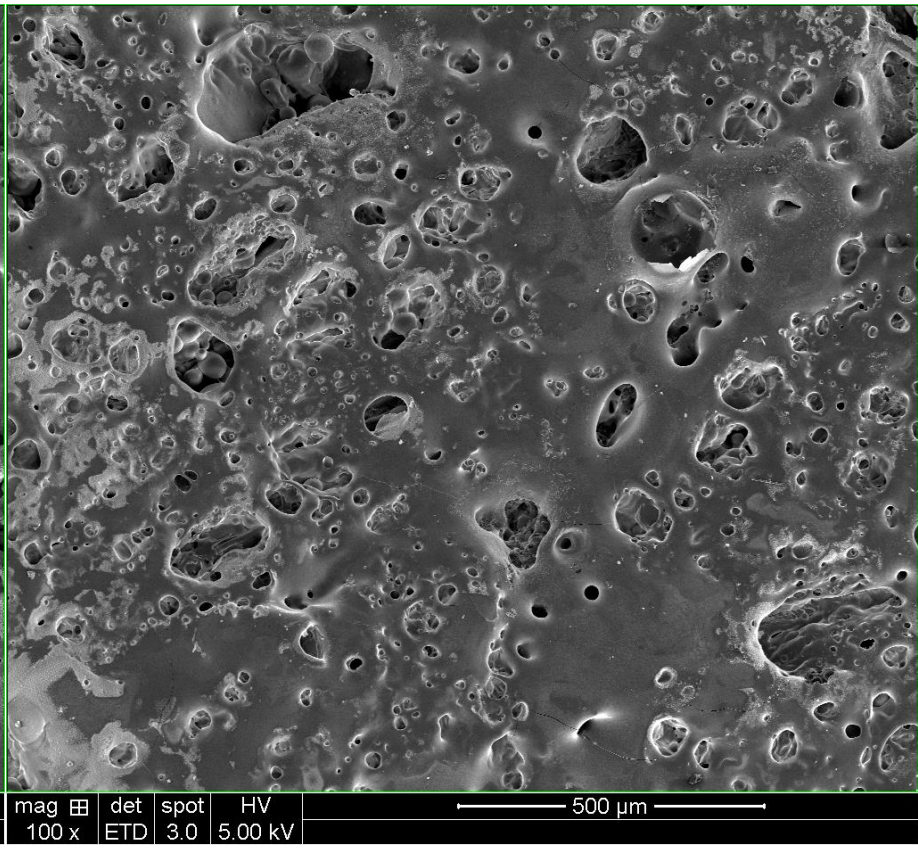
HD Video



100x Top View - Char layer

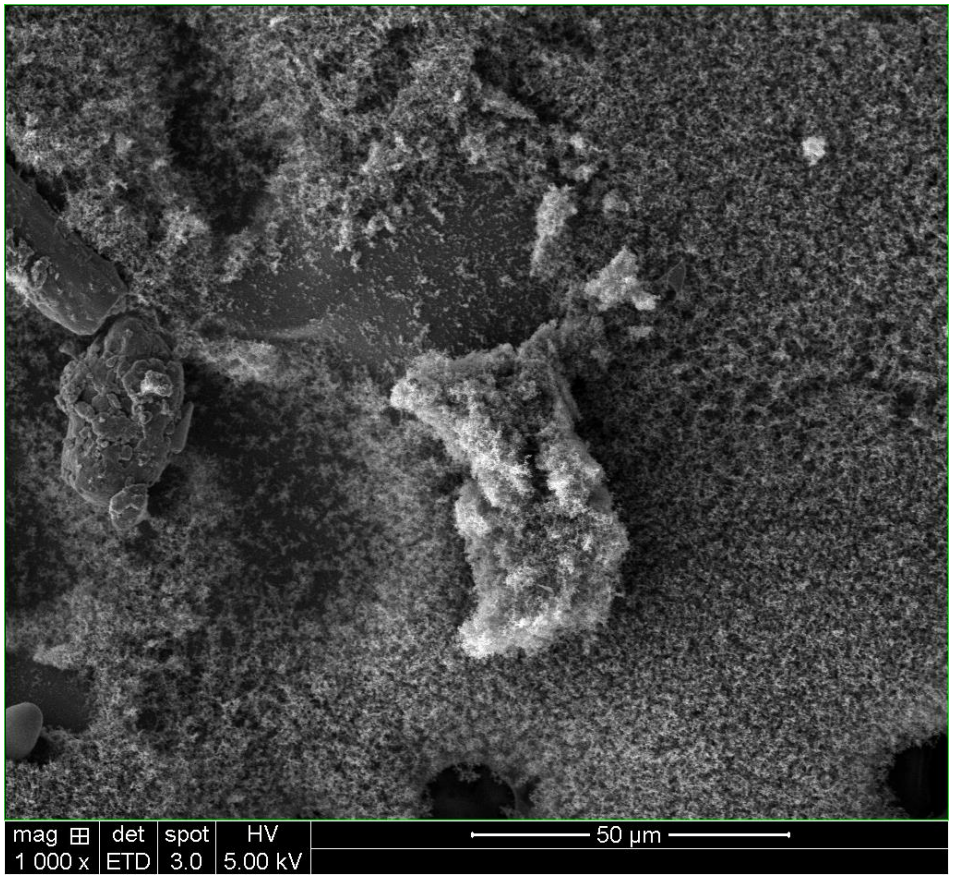


S/Ph MX2600

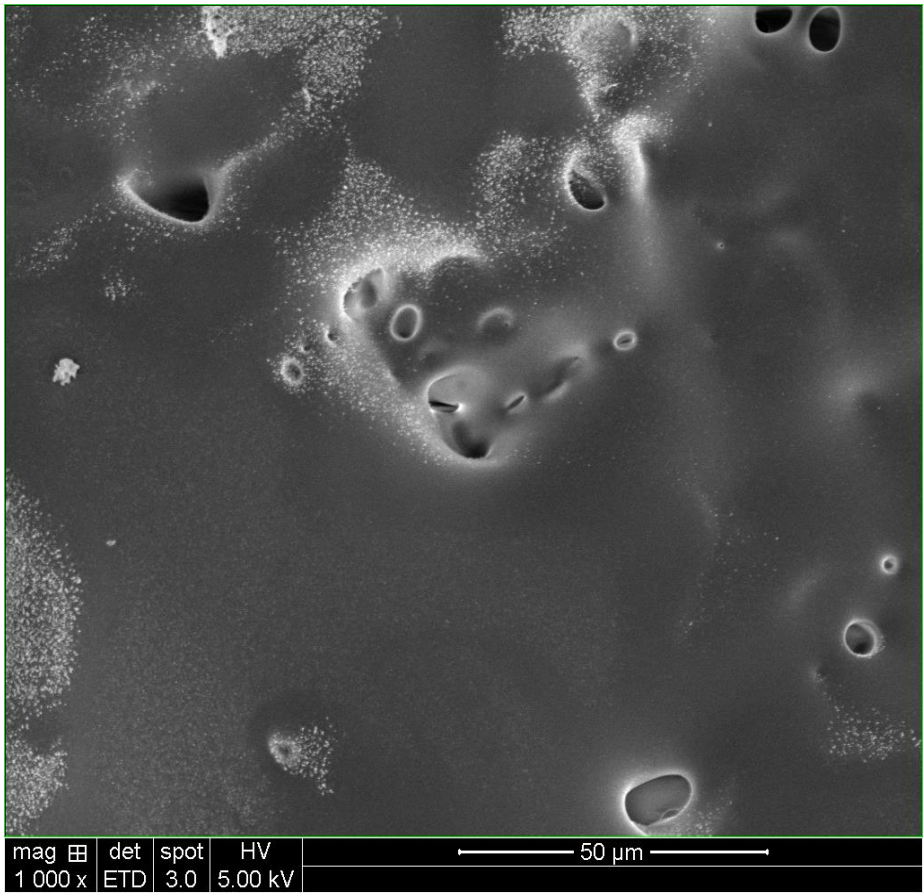


S/DG-UHTR F1

1000x Top View - Char Layer

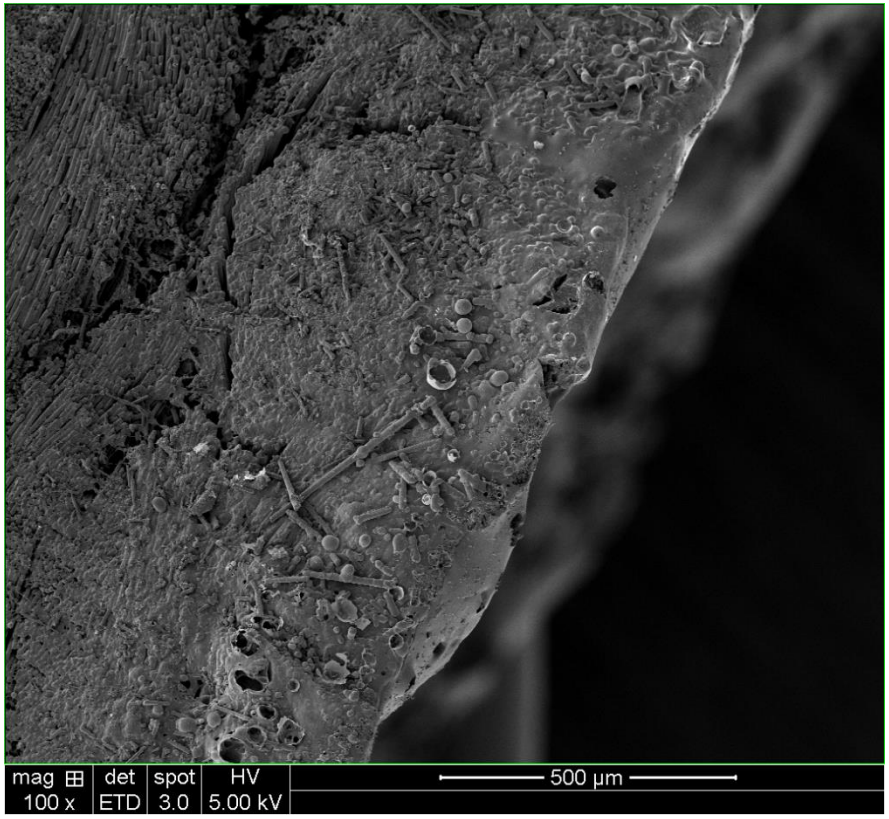


S/Ph MX2600



S/DG-UHTR F1

100x Cross Section– Char Layer



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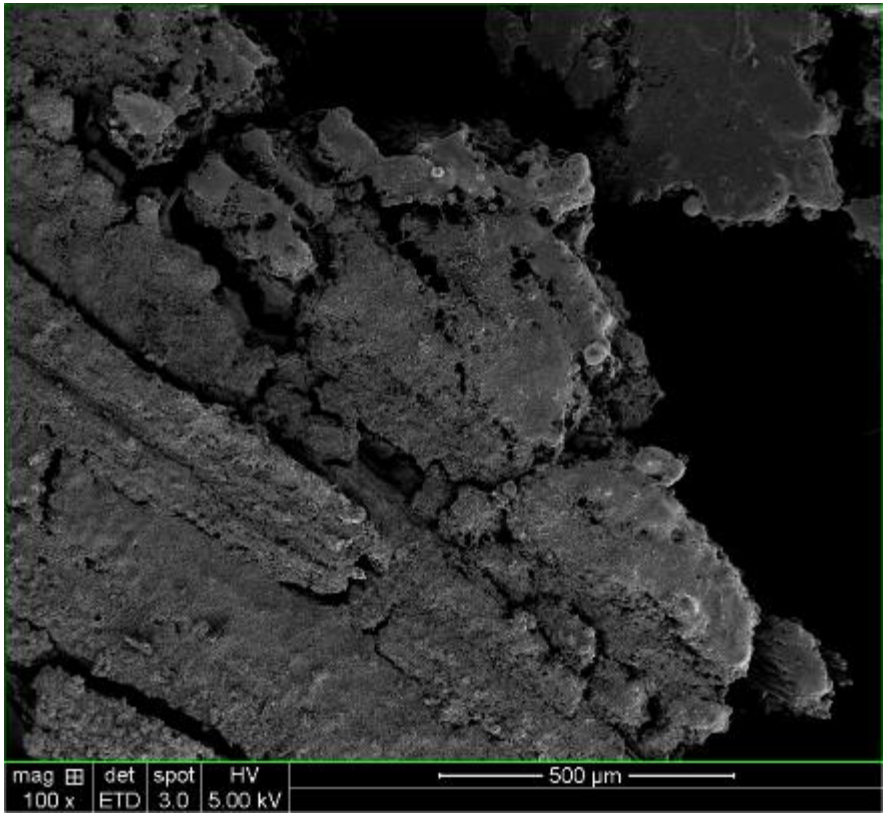
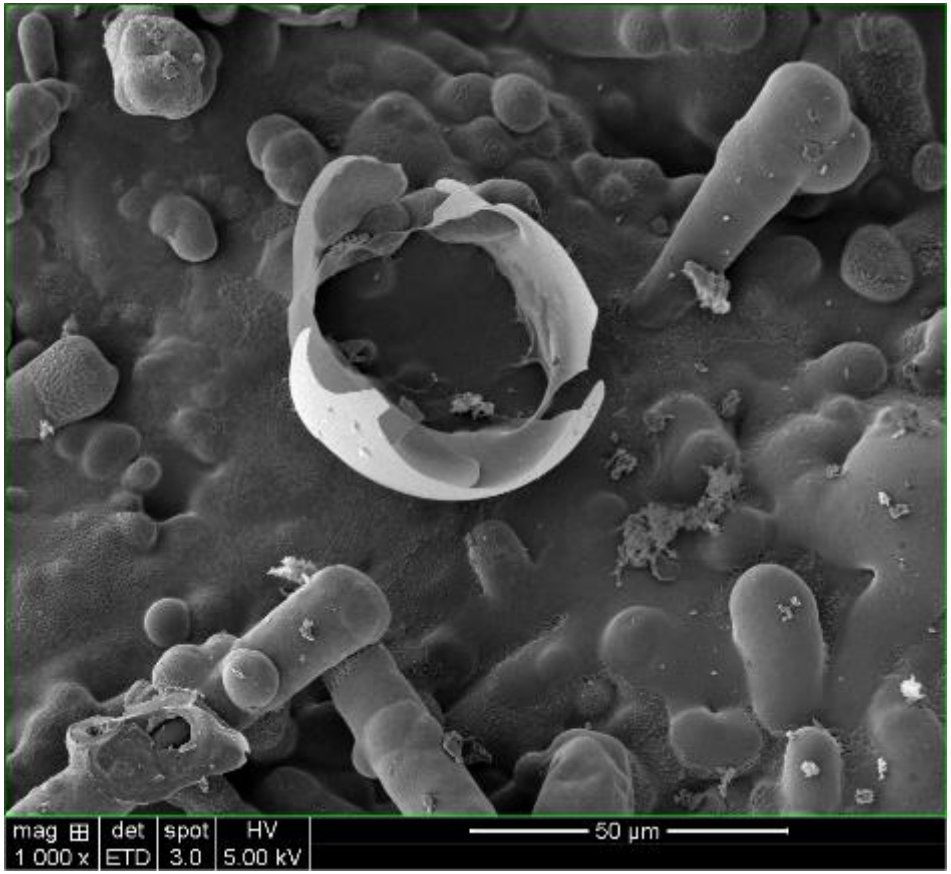
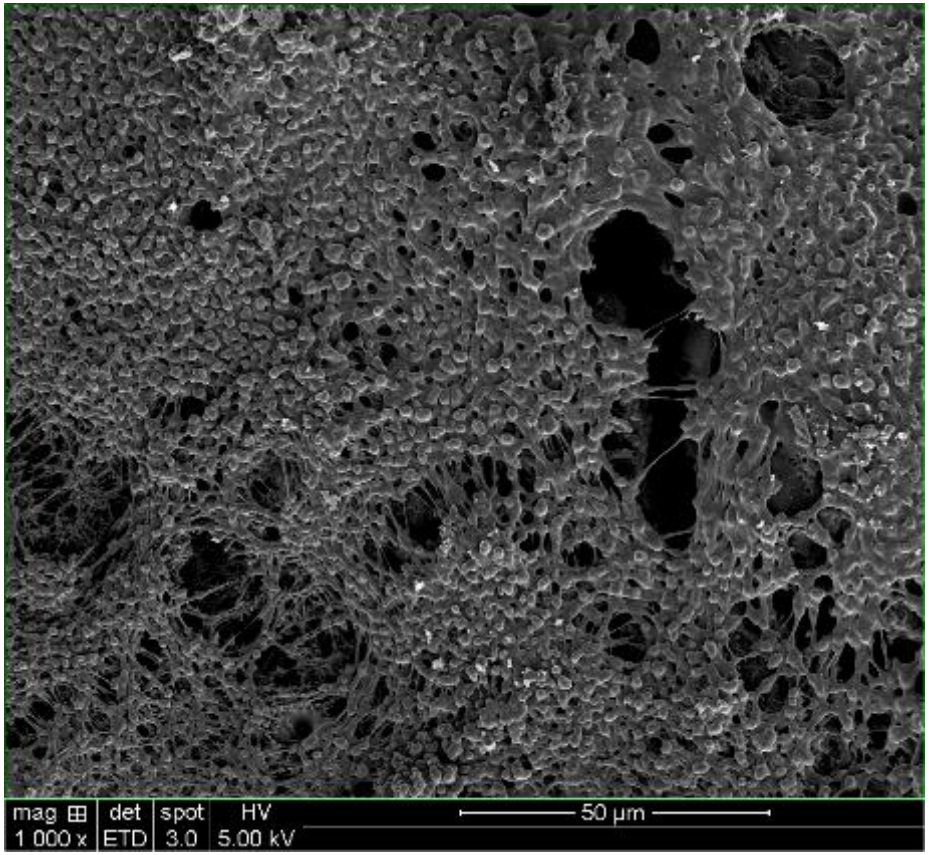


S/DG-UHTR F1

1000x Cross Section– Char Layer

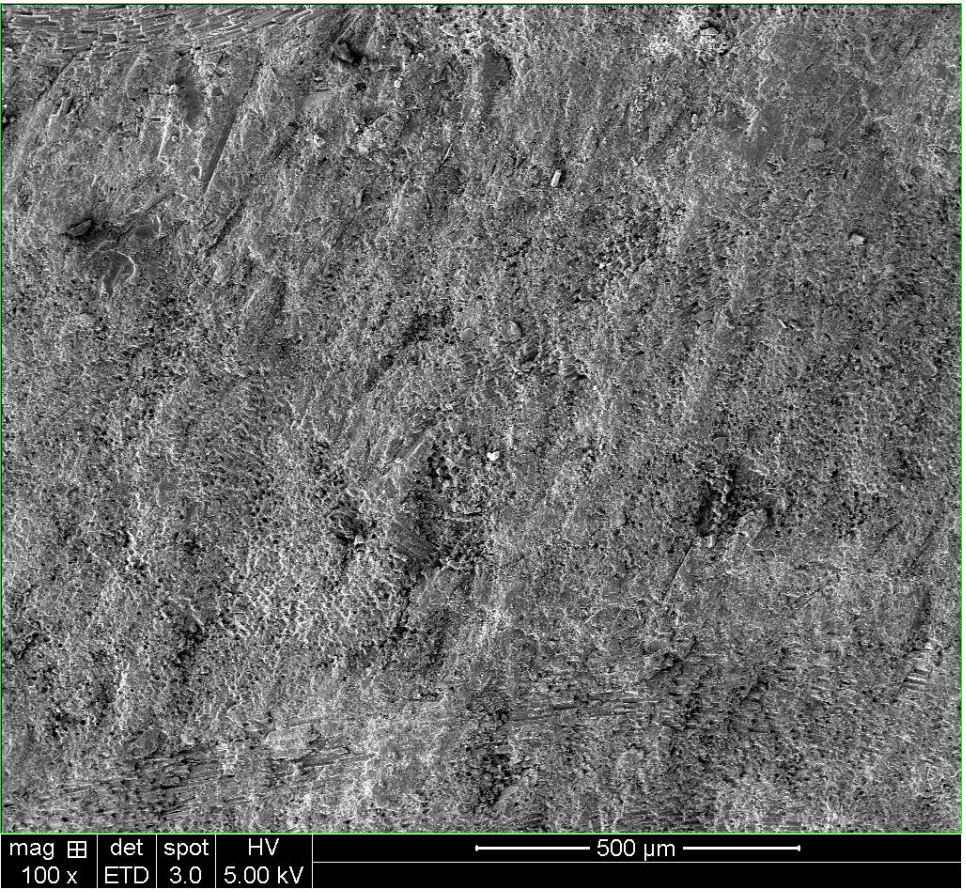


S/Ph MX2600

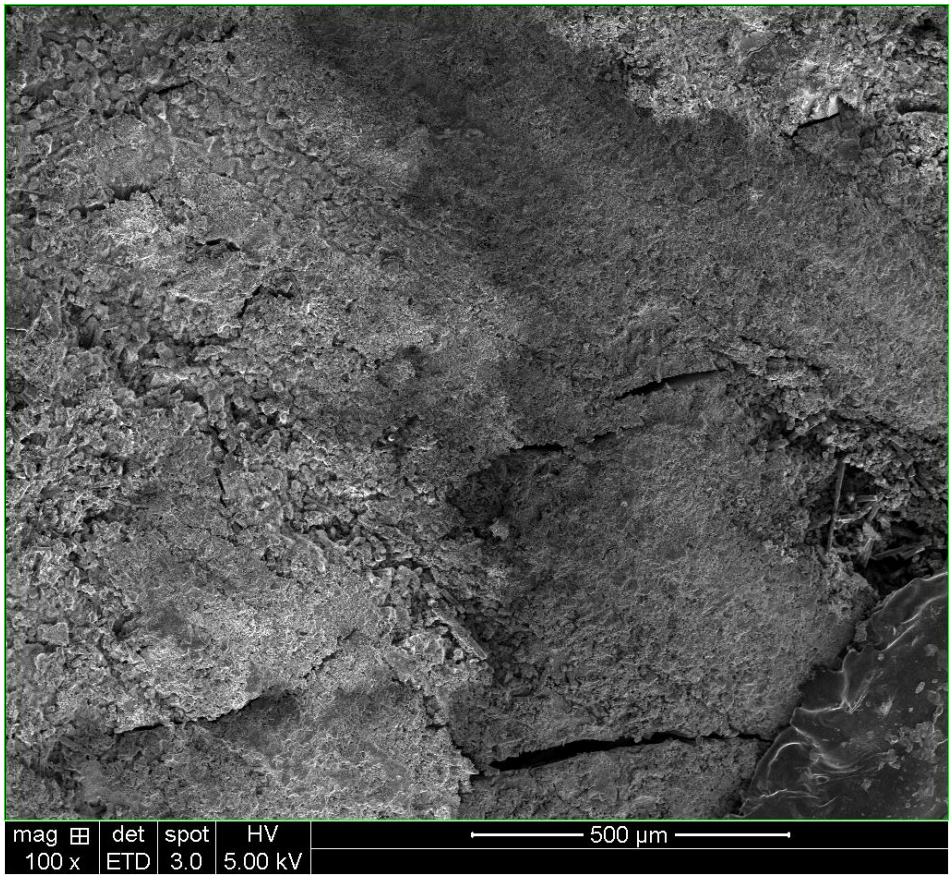


S/DG-UHTR F1

100x Cross Section– Virgin

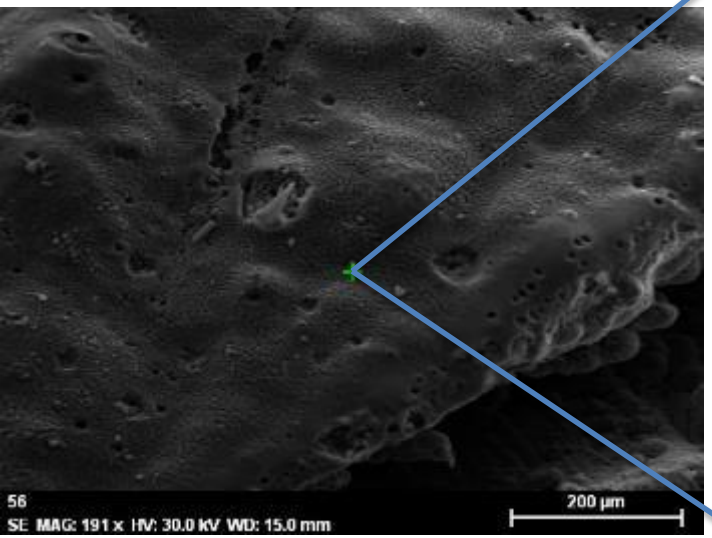


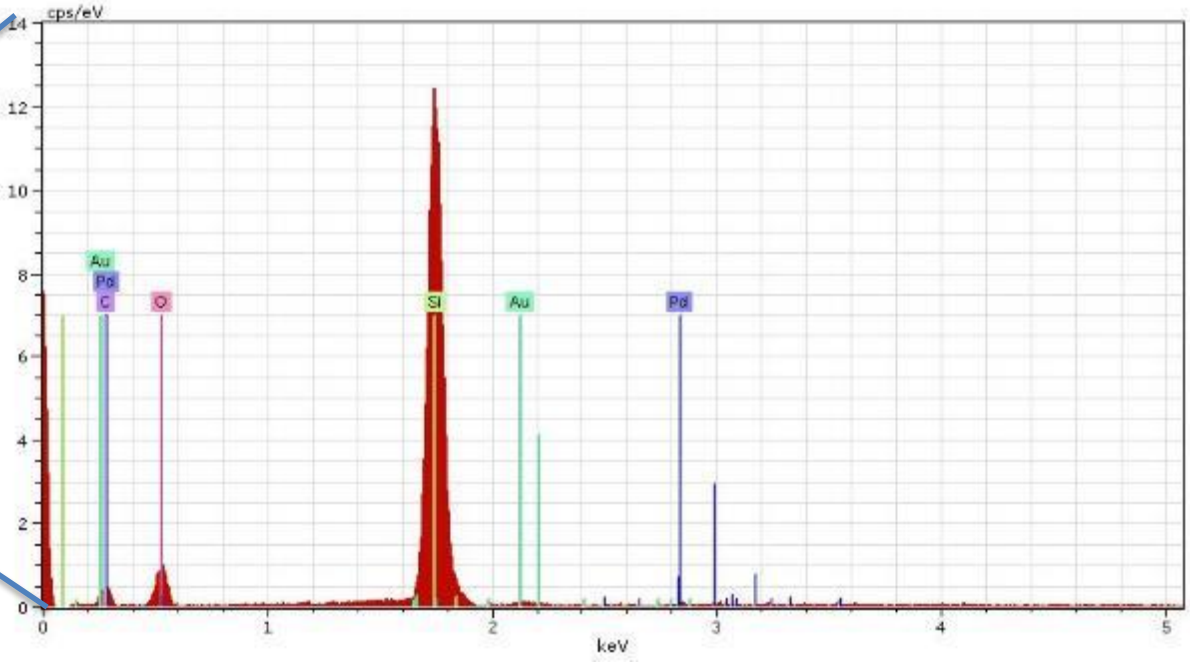
S/Ph MX2600



S/DG-UHTR F1

EDX Analysis – DG-UHTR F1 Char





Conclusion

- All Silica/DG-UHTR formulations showed the best thermophysical and ablation properties
- The 35wt% Silica/DG-UHTR F1 composite showed the highest char yield, lowest recession rate, mass loss, and heat soak temperatures
- Better processing method needed to obtain best quality samples

Future Work

- Characterization of mechanical properties
  - Tensile strength and modulus
  - Compression strength and modulus
  - Flexural strength and modulus
- Test lower S/DG Resin:Fiber ratios: 25wt%, 30wt%
- Ablation testing using Inductively Coupled Plasma (ICP) torch
- Better dispersion of nanosilica into the resin
- Test shear and compression strength of char
- Explorer incorporation different additives and nanomaterials

Thanks for attending!
Questions?

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