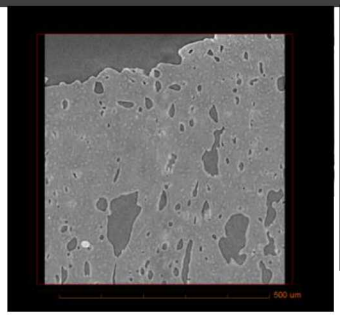
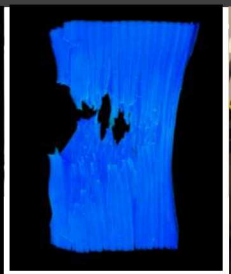
SAND2019-8664C

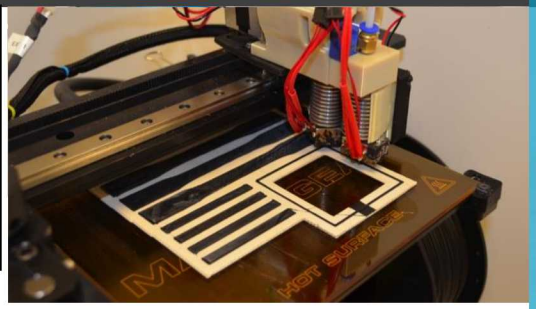
Sandia National Laboratories

Additively Manufactured Polyphenylene Sulfide

Structure and Properties of Composites and Blends







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Polyphenylene Sulfide (PPS) Overview

- Chemical resistance
  - No known solvents below 200 °C
- Mechanical properties
  - Strength of 65.0 MPa
  - Modulus of 4.00 GPa
- Thermal stability
  - Melting temperature of 280 °C
  - HDT of 115 °C at 1.8 MPa
- Dielectric strength
  - >500 V/mil
- New to additive manufacturing
  - Commercial availability began after the start of this study

Source: http://www.matweb.com/search/datasheet.aspx?matguid=44827ef8a0834126954deb6e605cff3b&ckck=1

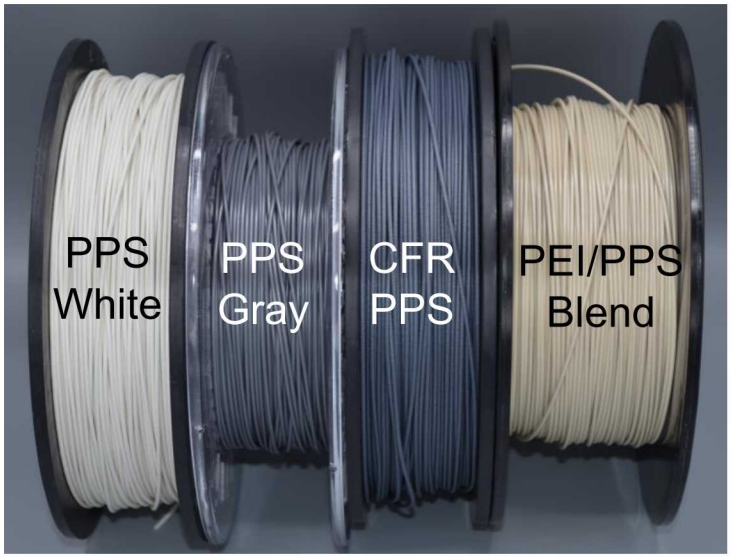
Research Motivation and Overview

- Property set of interest in aerospace applications
- Need for qualified materials and processes
- Commercial filaments subject to change without notification

Research Plan

1. Characterize commercial filaments and parts
   - Use as a baseline for properties and part quality
2. Extrude PPS filament with known PPS, additives, and fillers
3. Characterize properties and print quality, improve recipe and processing parameters

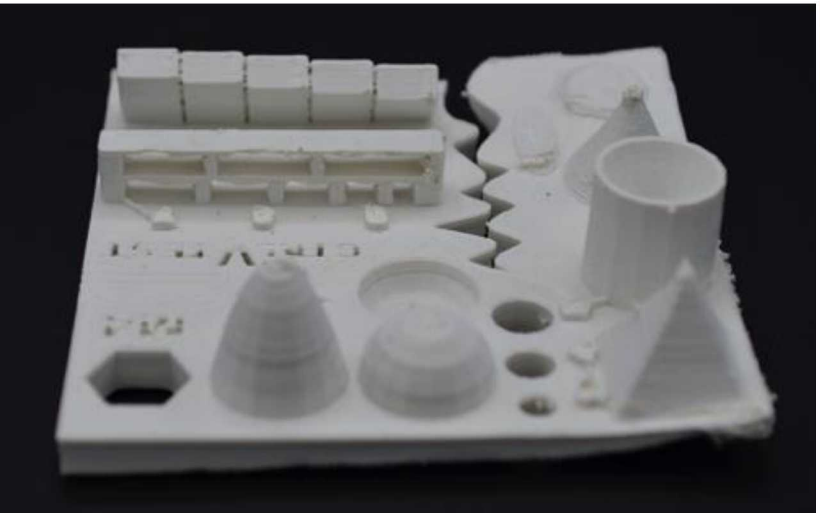
- Materials Studied
  - Commercial Filament

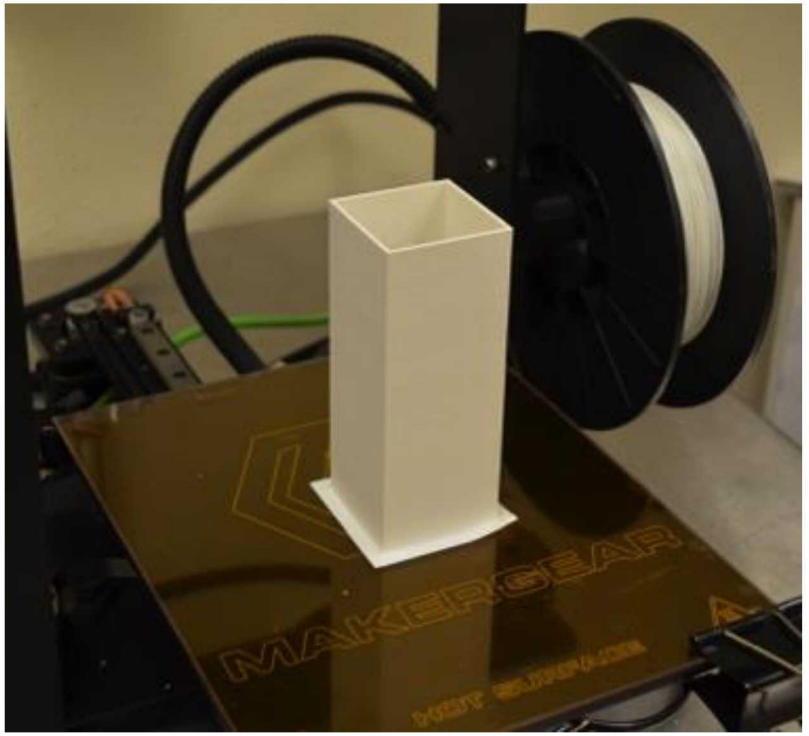


  - Extruded Fortron Filament (Sandia)

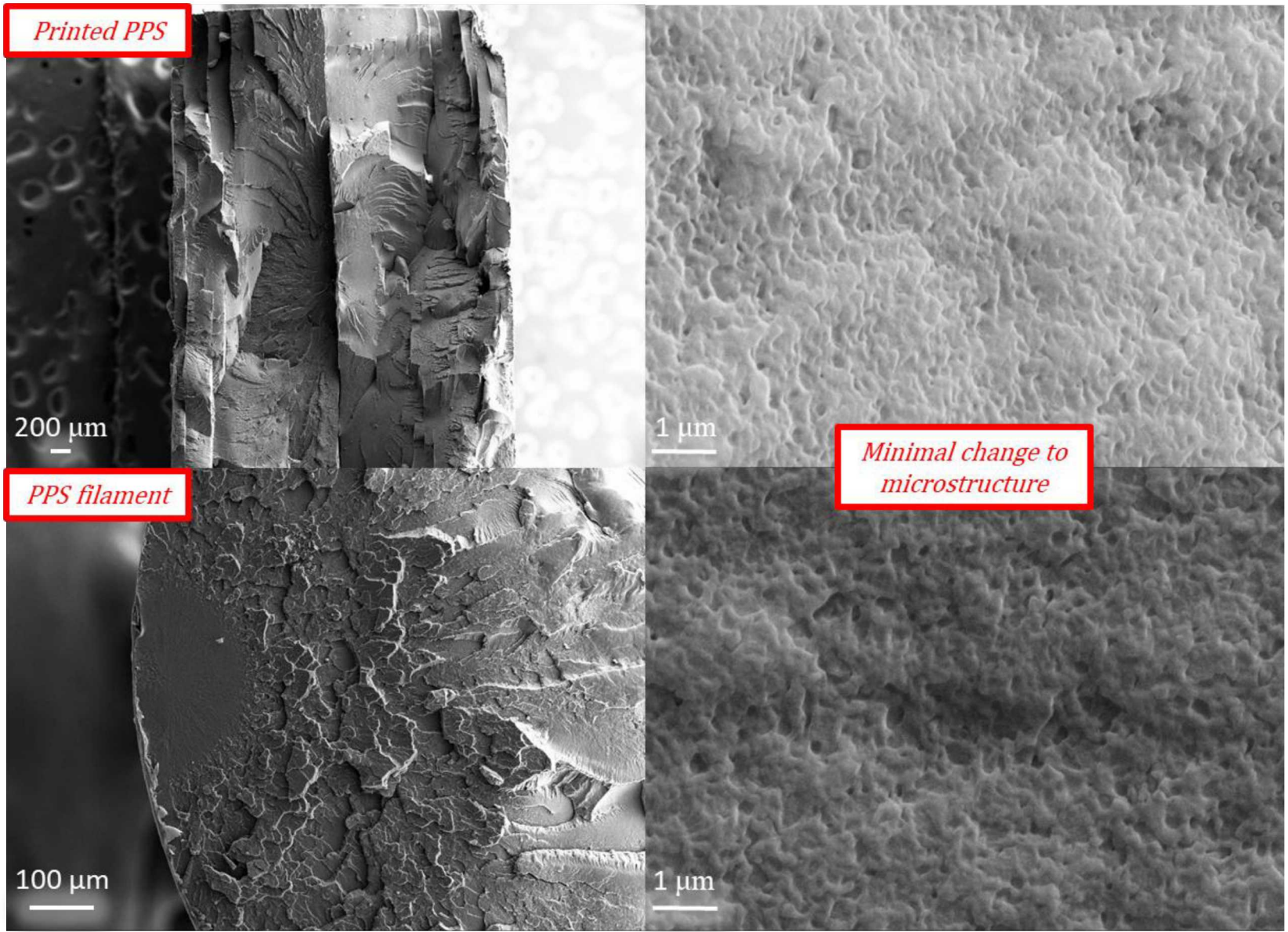
Printing definition tied to thermal strain and print bed adhesion

- Self supporting structures and overhangs are achievable with unloaded PPS
  - High dimensional accuracy
  - Thin continuous walls (0.015”-0.060”) achievable
  - Part warping and detachment from heated bed observed

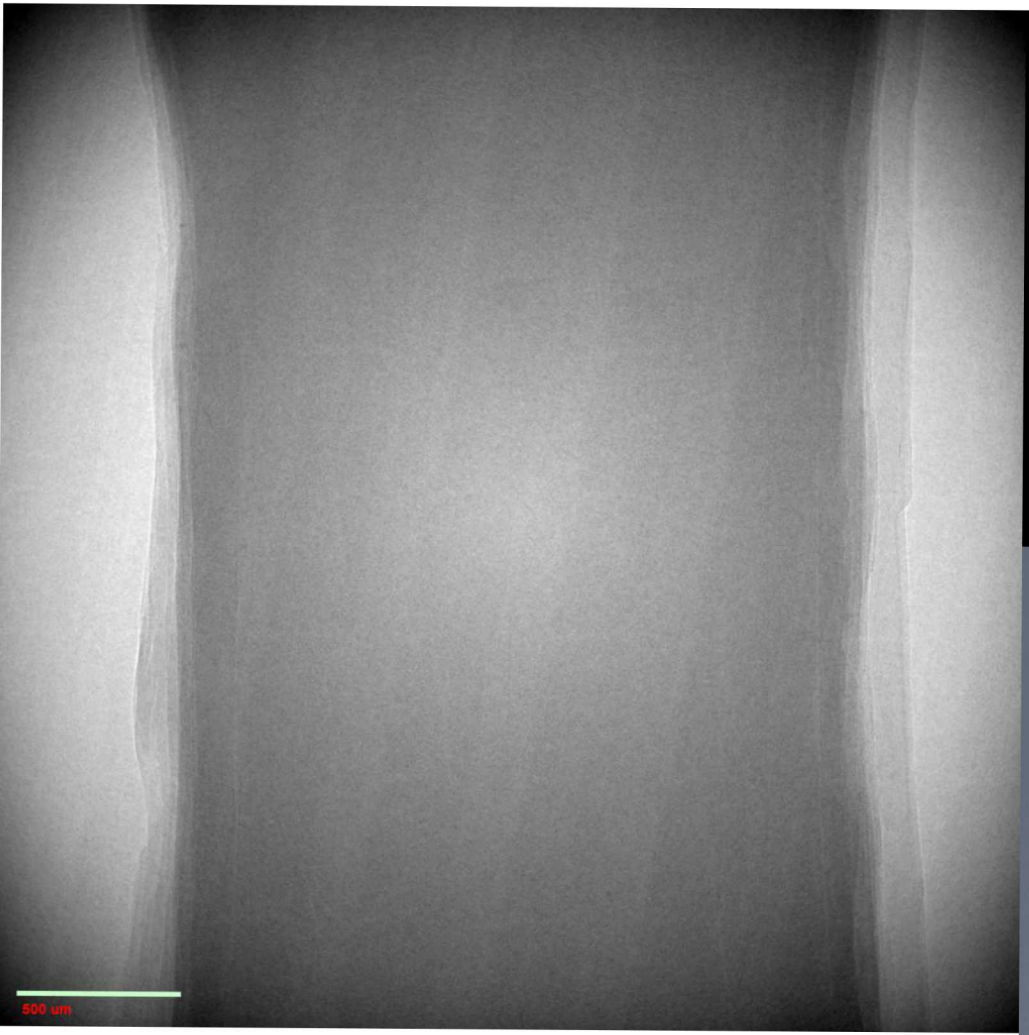


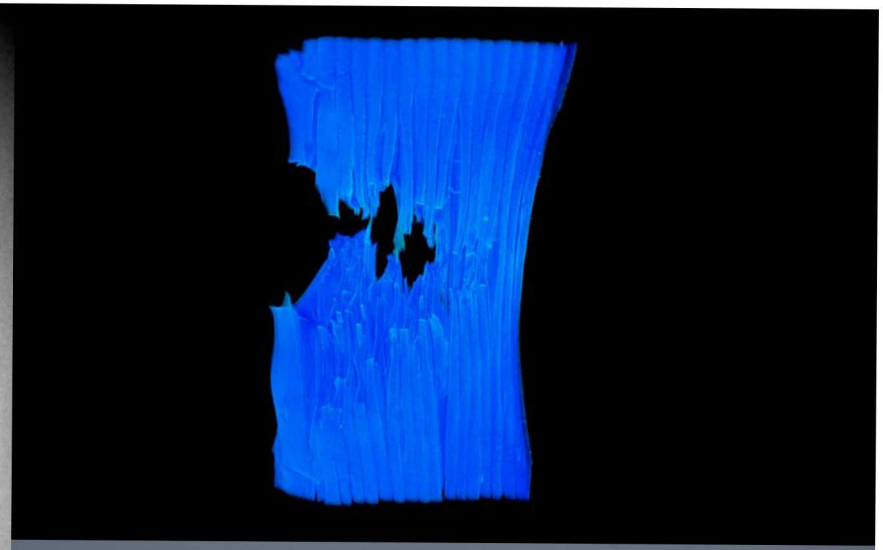


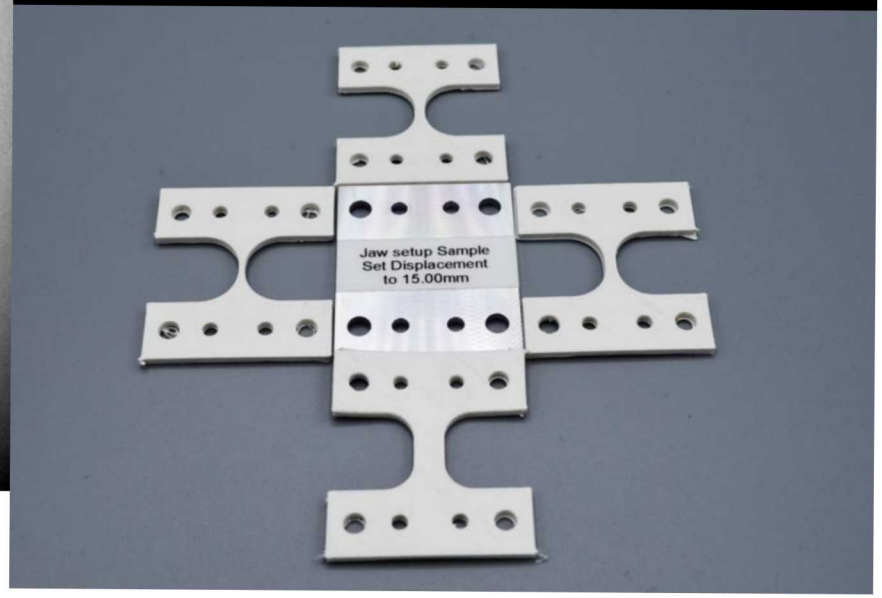
SEM Shows Minimal Changes to Microstructure after Printing Filament



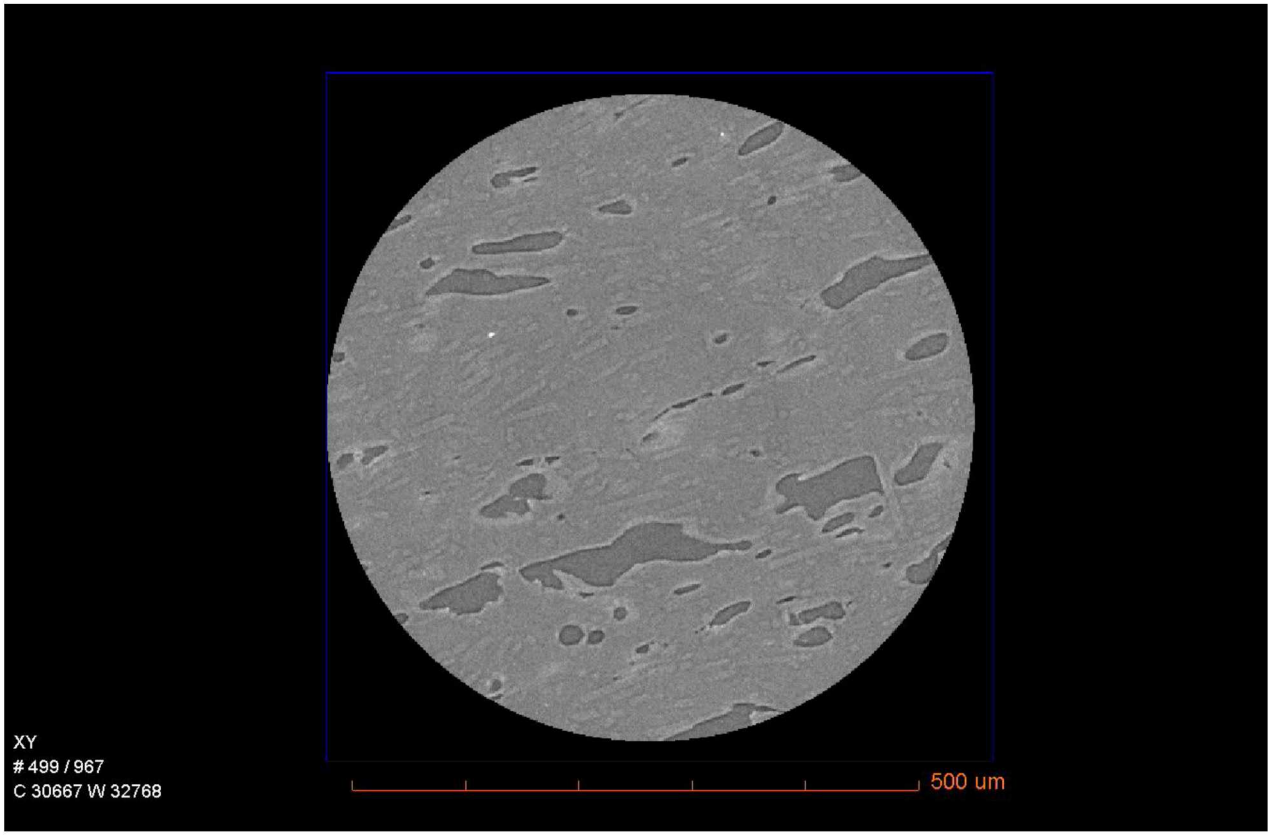
μCT reveals interlayer failure and necking in AM coupon tensile test

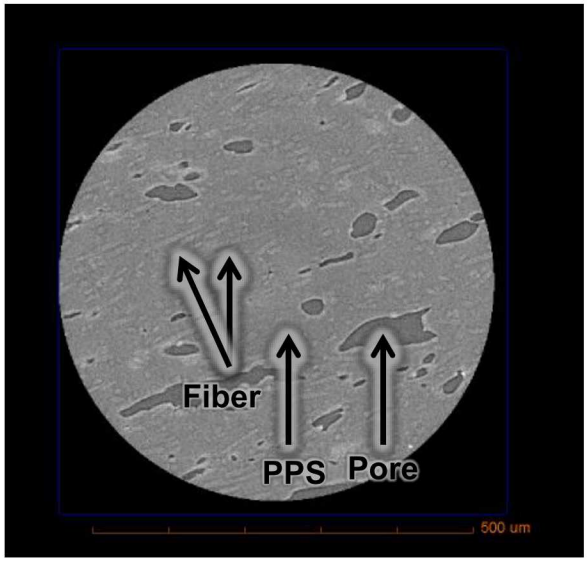


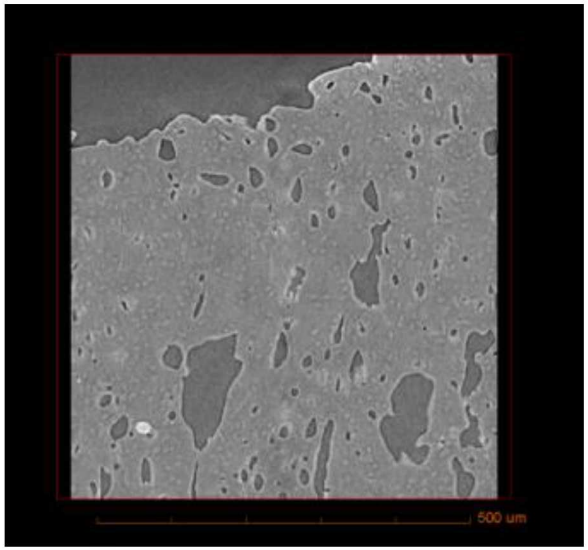




CFR PPS printed parts and filament show voids in matrix under μCT





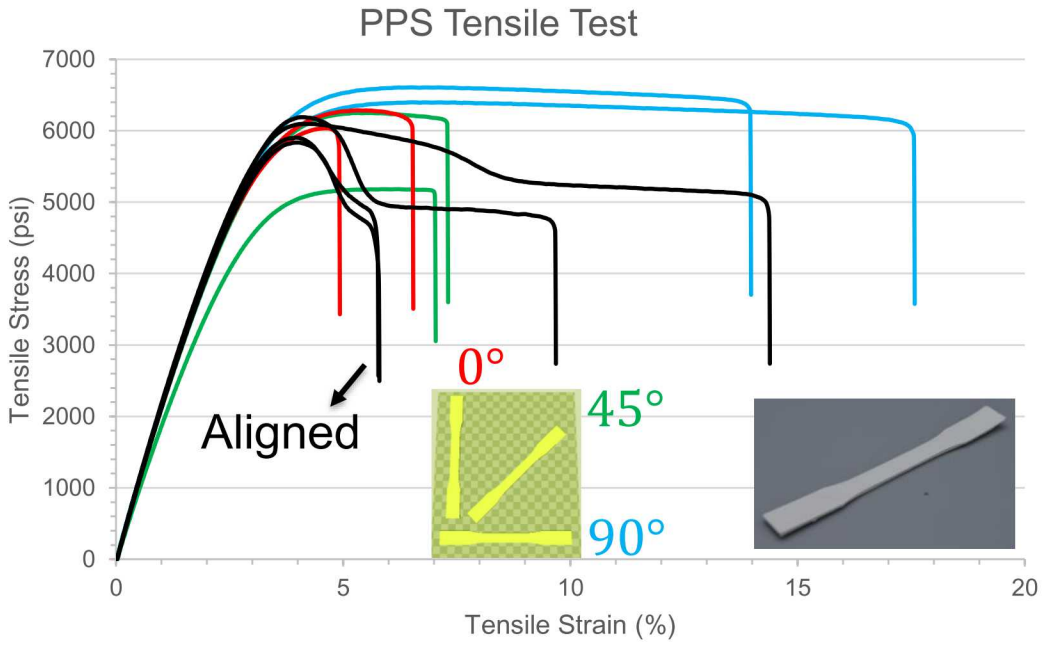


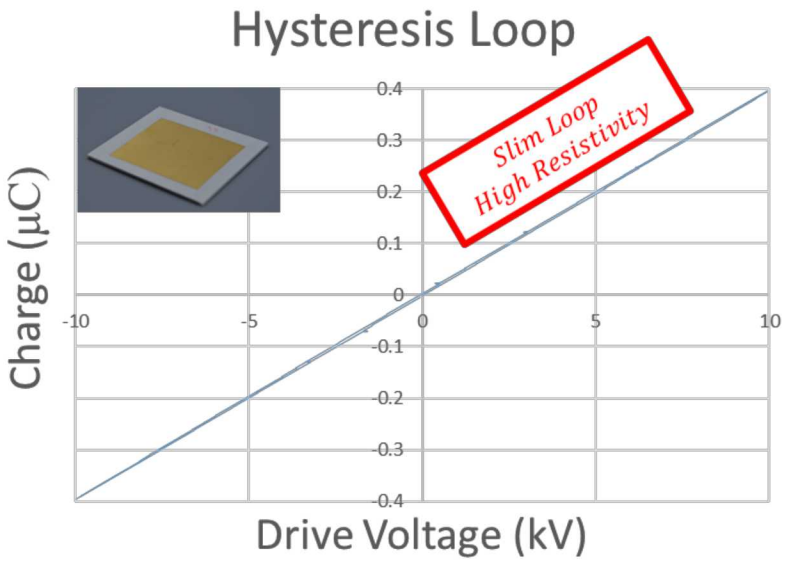
- Chopped CF filler can be used to reduce CTE
- Resulted in substantial voids in filament
- Can lead to reduced dielectric strength
- Need for alternative filler (SiO2)

Summary of commercial PPS properties

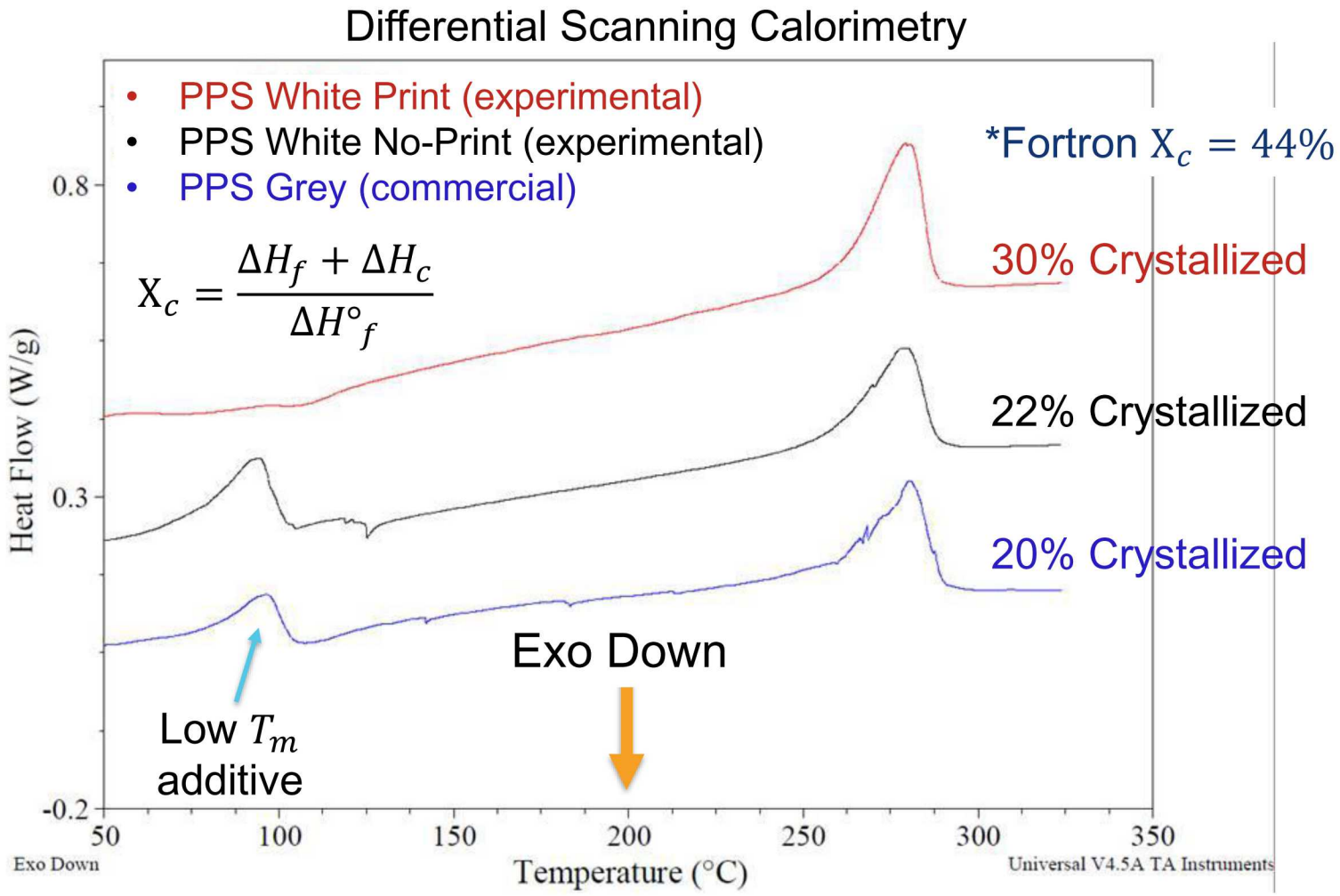
- Yield strength and modulus independent of print orientation
- Retention of electrical properties

PPS Properties	Printed	Reported [2]
Density (g/cc)	1.25	1.35
Elastic Modulus (ksi)	190-215	580
Strength (ksi)	5.8-6.5	9.4
Permittivity	3.5	3.3
Resistivity (ohm-cm)	10^{15}	10^{15}
Dielectric Breakdown (V/mil)	>850	523





Low crystallinity in commercial PPS filaments compared with Fortron



$\Delta H^\circ_f = 80\ J/g$ (Spruiell, J. E. (2005) (No. ORNL/TM-2004/304))

PPS and PPS/SiO2 composite filament extruded at Sandia

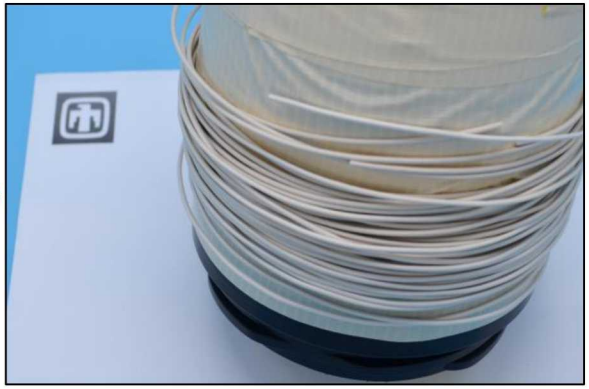
- Collin Teachline Extruder
- 5-zone system with high and low shear flights
- 1.75mm die orifice
- 270-280°C die temperature

- Melt viscosity, zone temperature, winding rate, extrusion rate, and cooling rate allow tuning of the spooled fiber diameter.

PPS Powder

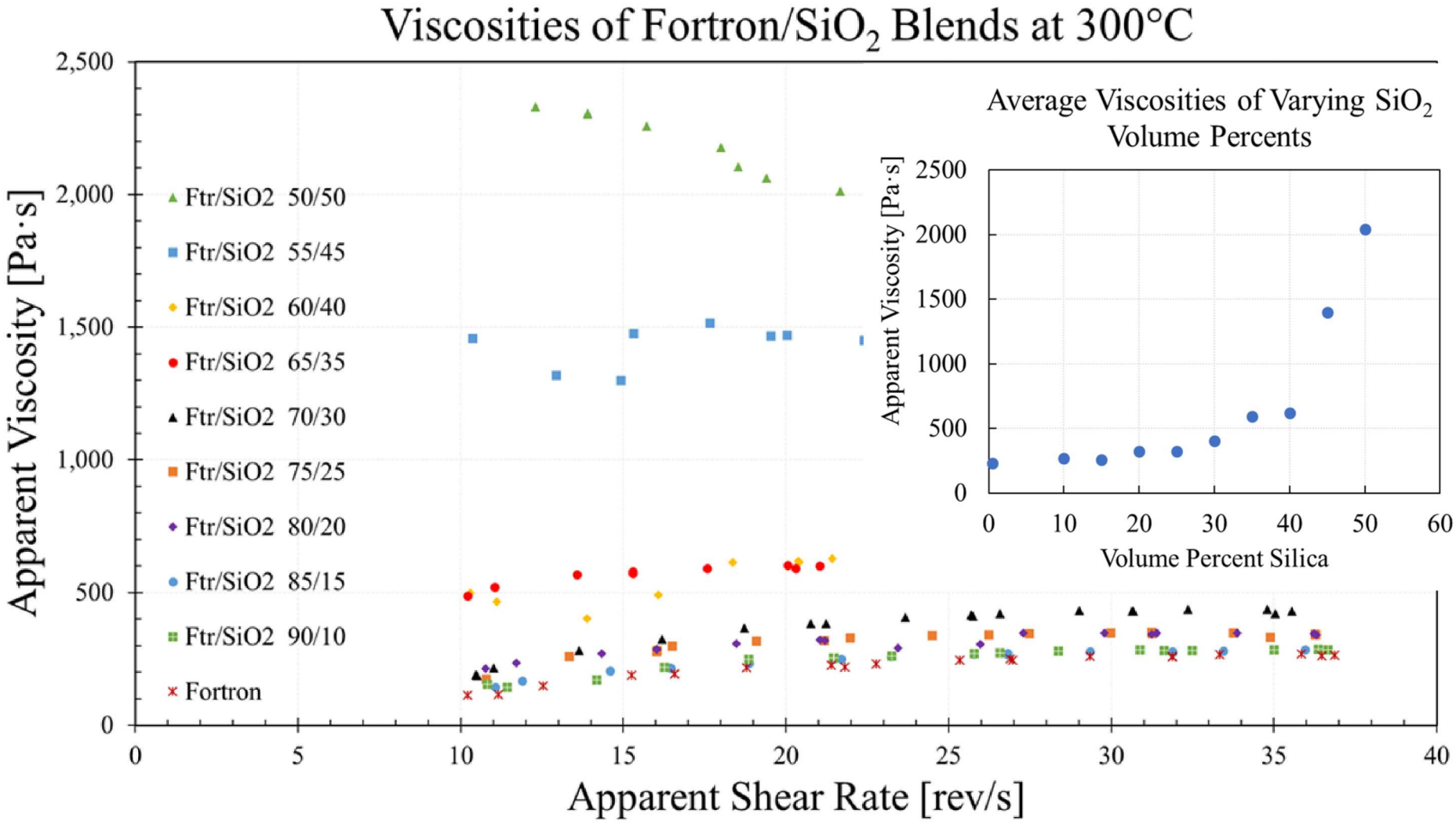
SiO2 Powder

Collin Teachline Extruder

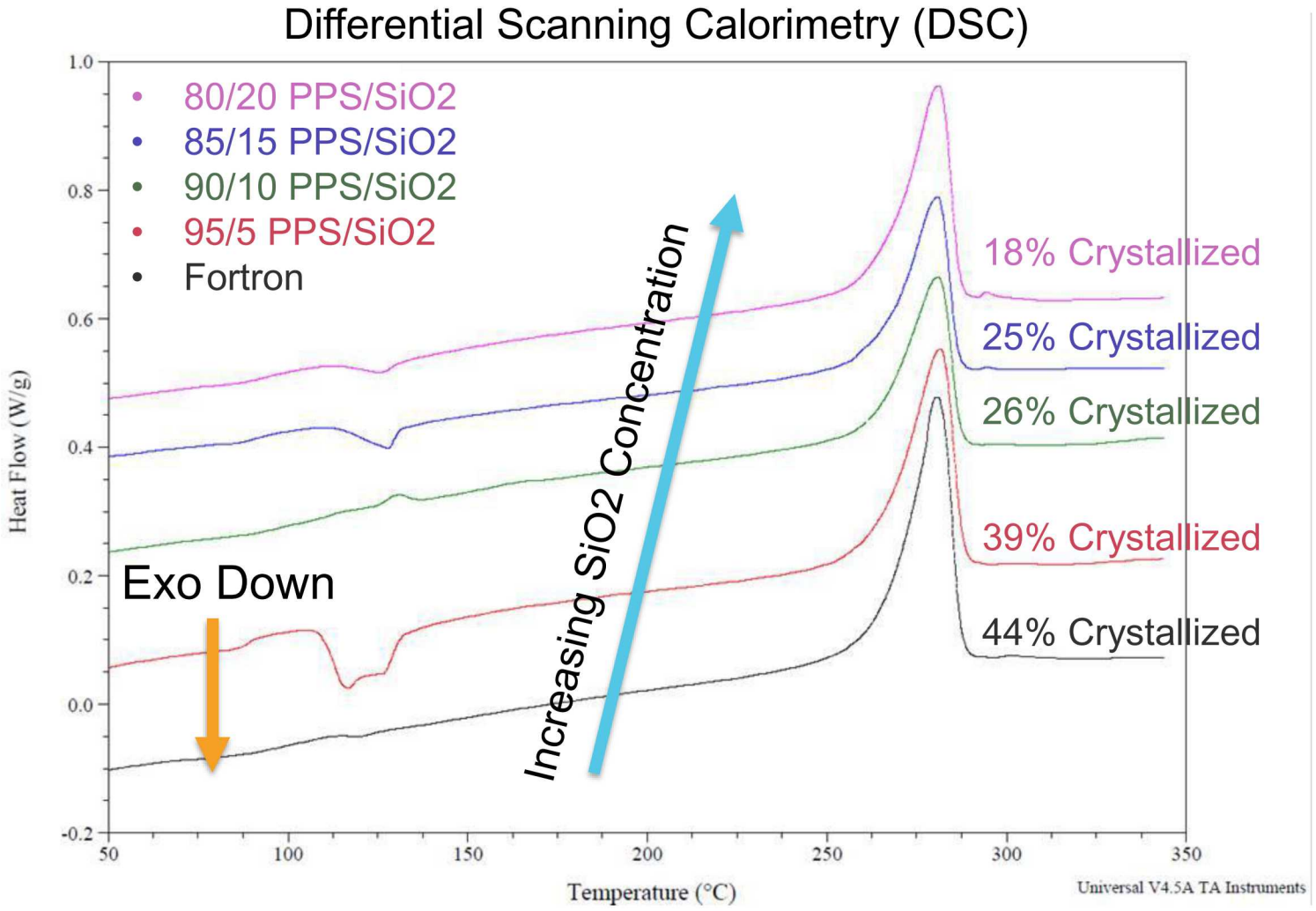


Composite FDM Filament

Viscosity increases with increasing SiO2 content

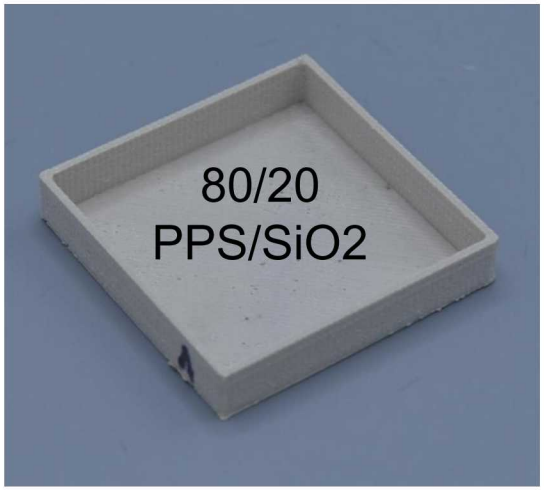


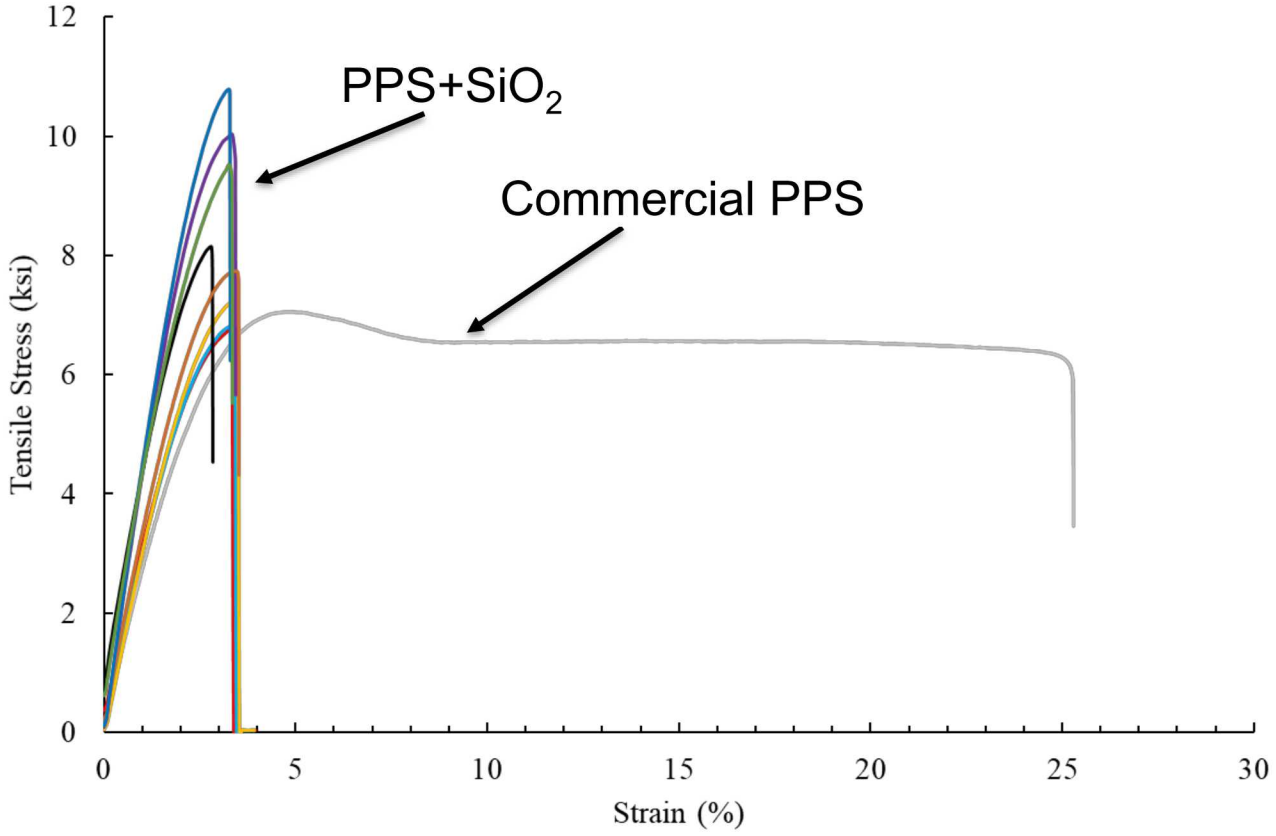
Increasing SiO2 content results in a decrease in crystallinity



Fortron and composite filament displays increased strength and modulus, reduced ductility over commercial filament

- Differences in crystallinity
- Additives/plasticisers in commercial material
- SiO2/PPS interactions



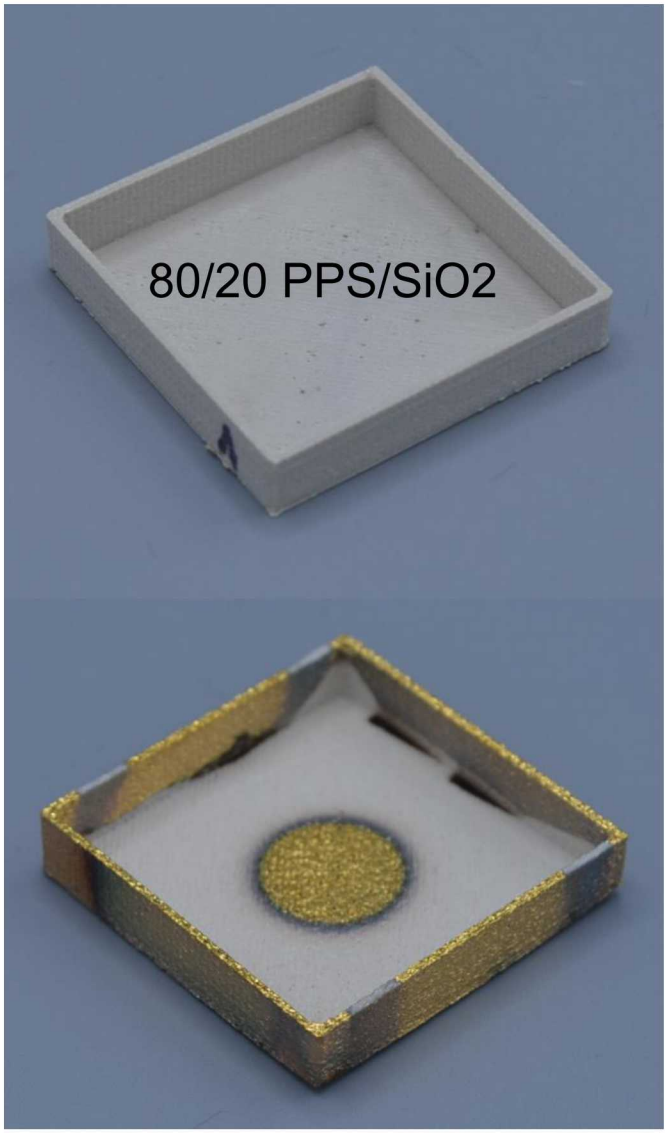


Questions?

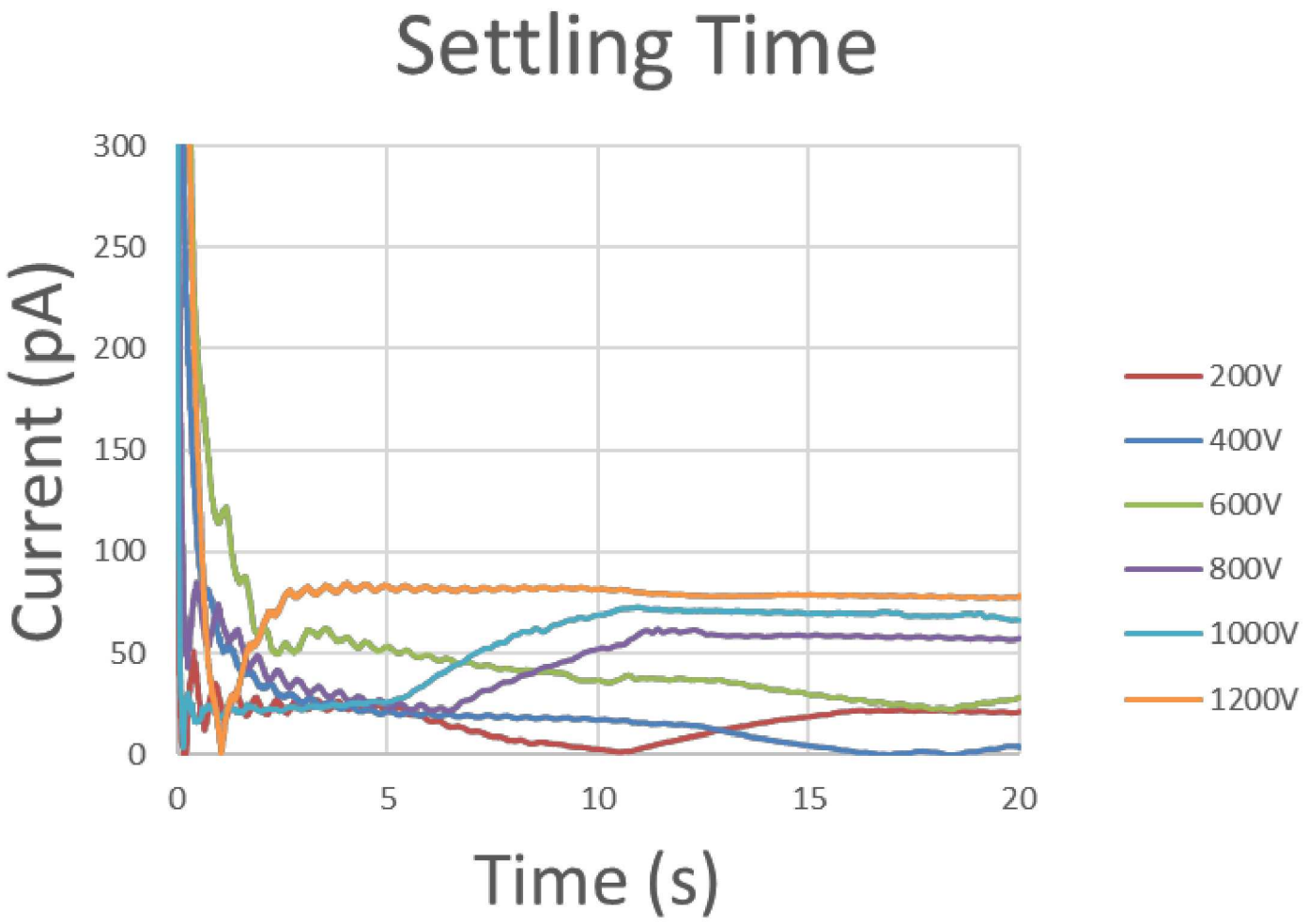
- Thank you to all collaborators who have worked on this project!
- Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Future Work

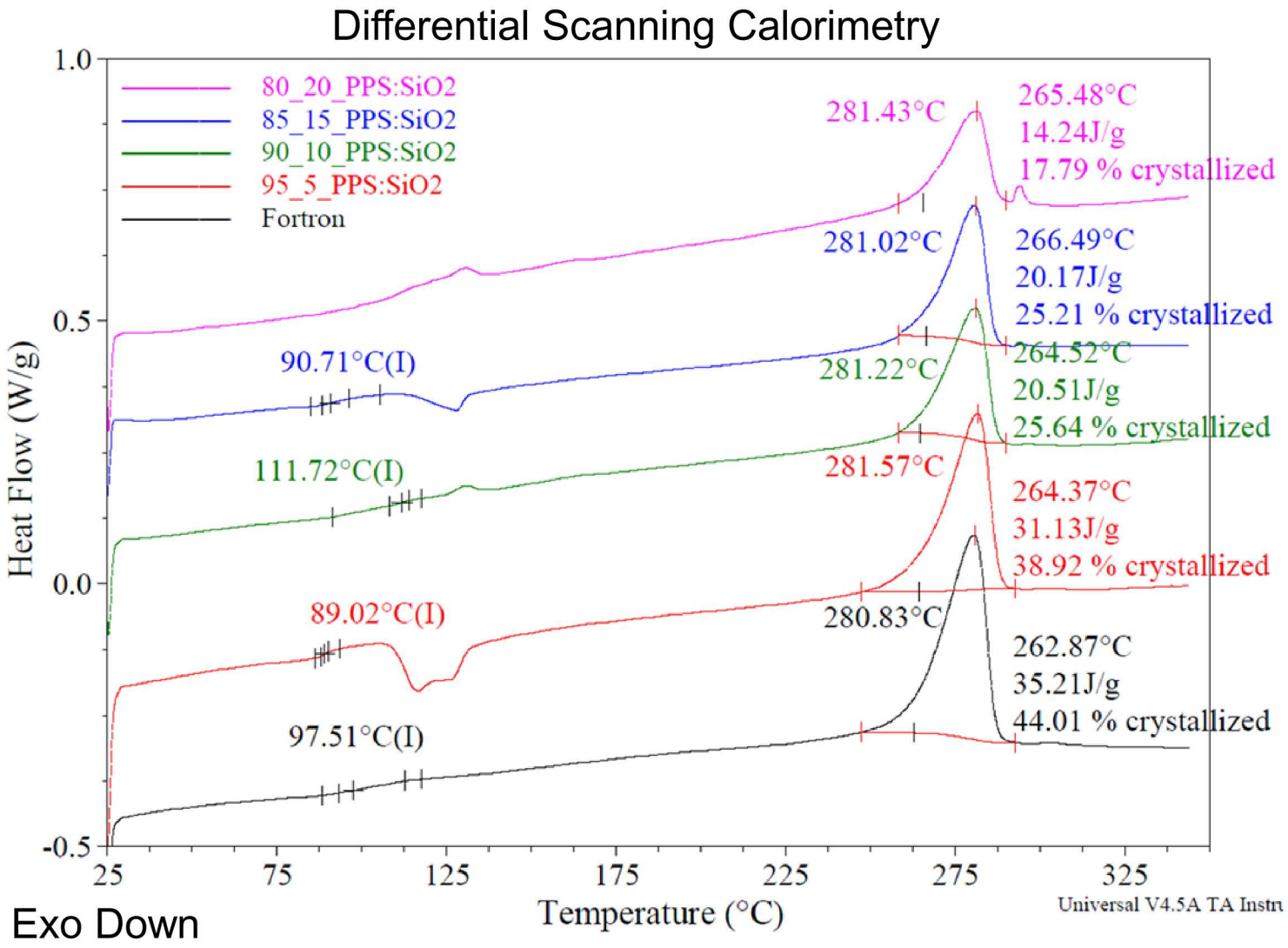
- Refine filament composition and processing conditions for optimal properties and tolerances
- Gain understanding of print parameters (speed, temperatures, layer height, etc.) on material properties
- Similar structure and properties characterization as performed on 3DXTech filaments and coupons
  - Microstructure
  - Mechanical
  - Thermal
  - Electrical
  - Print quality



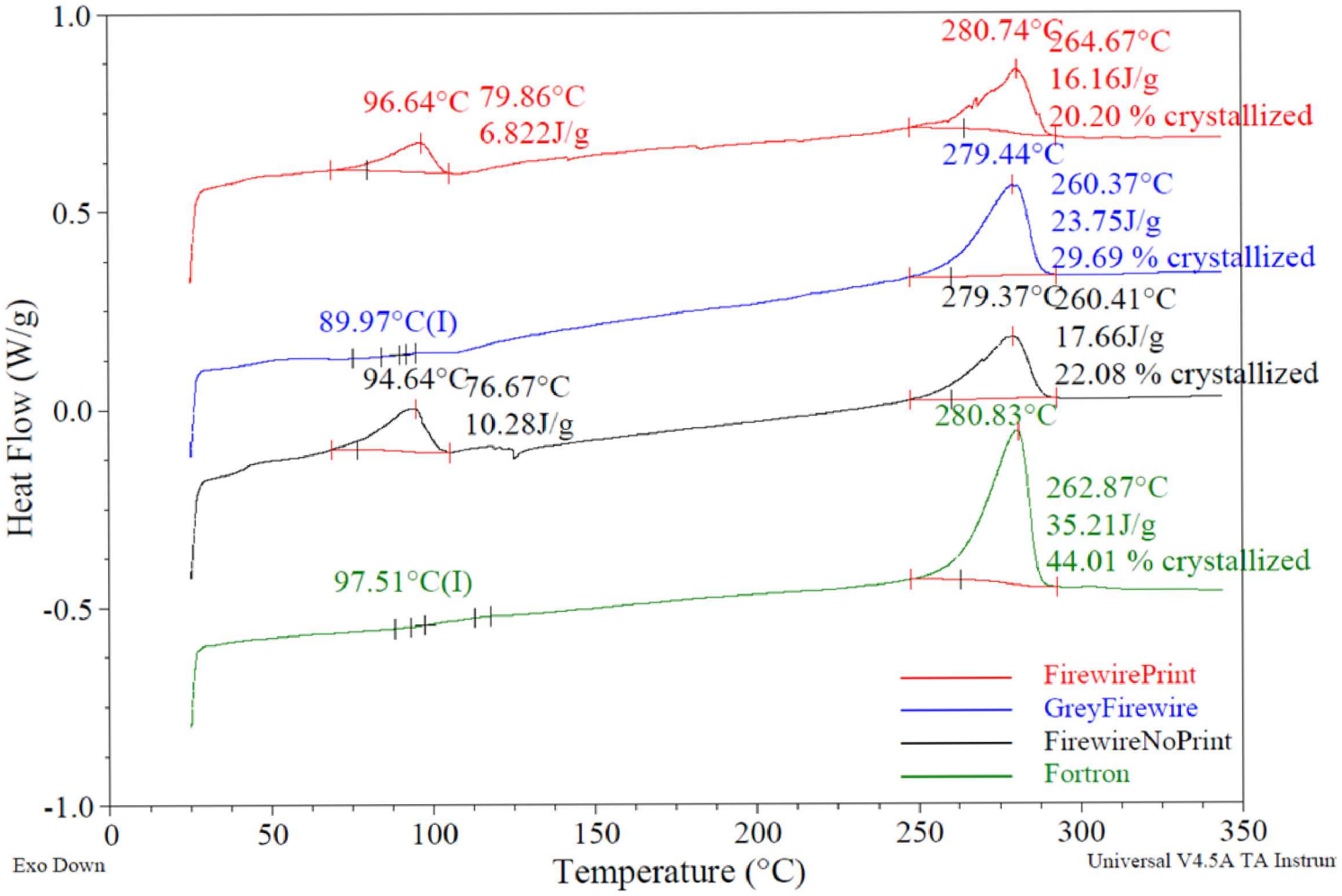
Electrical Properties



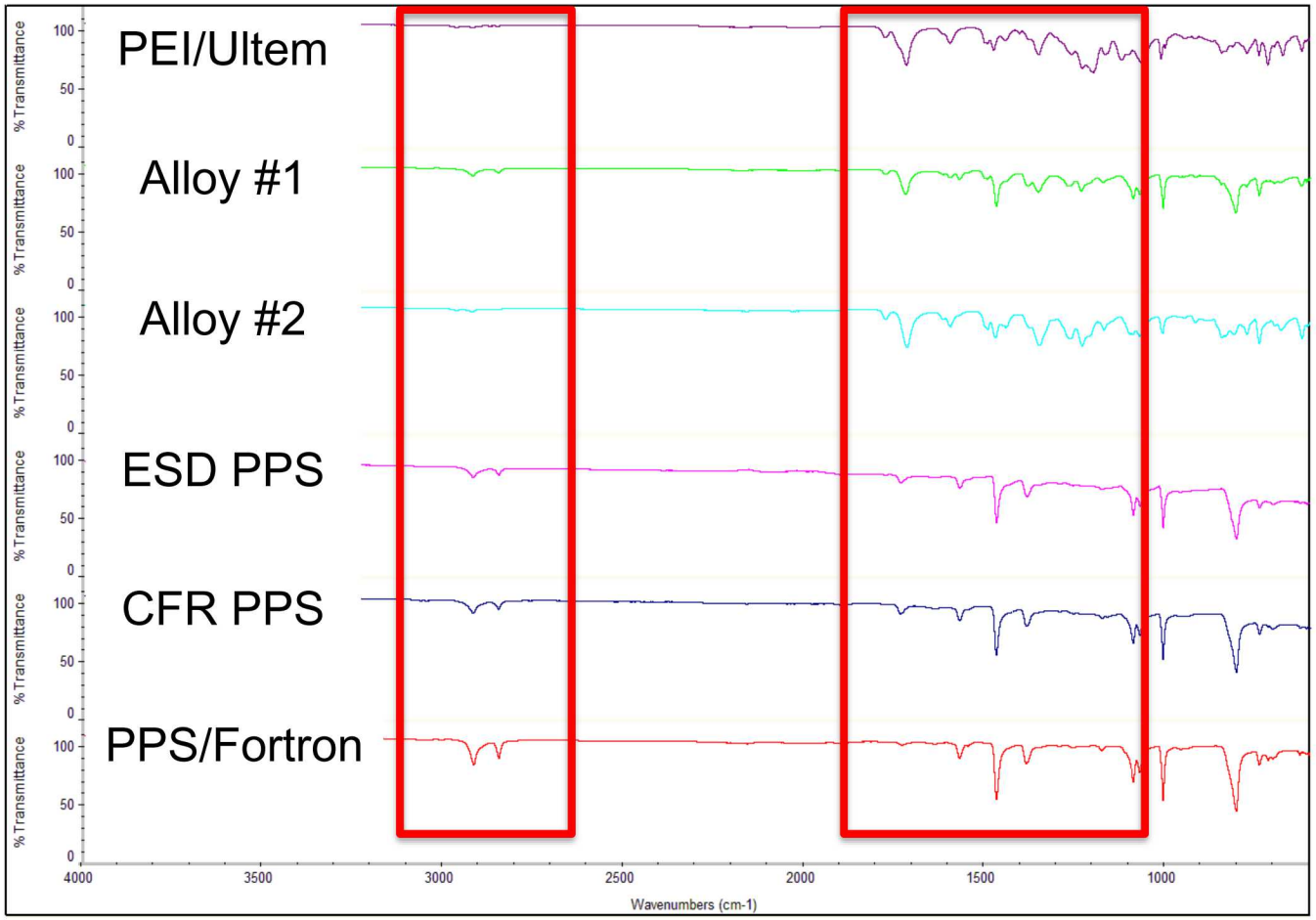
DSC


Differential Scanning Calorimetry
80_20_PPS:SiO2
85_15_PPS:SiO2
90_10_PPS:SiO2
95_5_PPS:SiO2
Fortron
281.43°C
265.48°C
14.24J/g
17.79 % crystallized
281.02°C
266.49°C
20.17J/g
25.21 % crystallized
90.71°C(I)
281.22°C
264.52°C
20.51J/g
25.64 % crystallized
111.72°C(I)
281.57°C
264.37°C
31.13J/g
38.92 % crystallized
89.02°C(I)
280.83°C
262.87°C
35.21J/g
44.01 % crystallized
97.51°C(I)
Heat Flow (W/g)
1.0
0.5
0.0
-0.5
25
75
125
175
225
275
325
Temperature (°C)
Universal V4.5A TA Instr
Exo Down

DSC


1.0
0.5
0.0
-0.5
-1.0
Heat Flow (W/g)
0
50
100
150
200
250
300
350
Temperature (°C)
Exo Down
Universal V4.5A TA Instrun
96.64°C
79.86°C
6.822J/g
280.74°C
264.67°C
16.16J/g
20.20 % crystallized
279.44°C
260.37°C
23.75J/g
29.69 % crystallized
89.97°C(I)
279.37°C
260.41°C
17.66J/g
22.08 % crystallized
94.64°C
76.67°C
10.28J/g
280.83°C
262.87°C
35.21J/g
44.01 % crystallized
97.51°C(I)
FirewirePrint
GreyFirewire
FirewireNoPrint
Fortron

Compositional Analysis: FTIR (3DXTech)



Ultem → Alloy#2 → Alloy#1 → Fortron

Increasing PPS Concentration

Reduction in mechanical properties while retaining electrical properties when printed

PPS Properties	Printed	Reported [2]
Density (g/cc)	1.25	1.35
Elastic Modulus (GPa)	1.3-1.5	4.0
Strength (MPa)	40-45	65
Permittivity	3.5	3.3
Resistivity (ohm-cm)	10^{15}	10^{15}
Dielectric Breakdown (V/mil)	>850	523