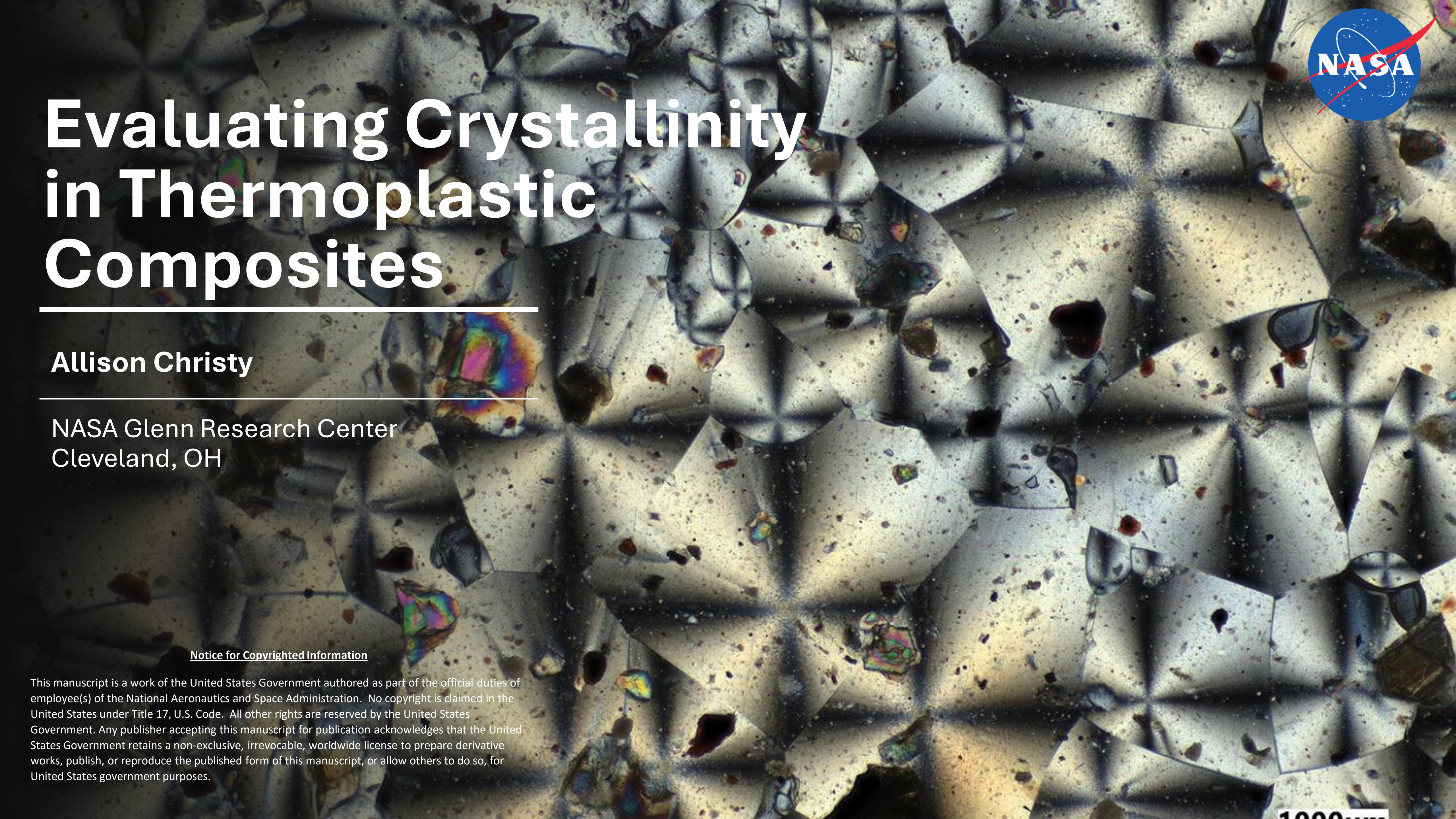


Evaluating Crystallinity in Thermoplastic Composites

Allison Christy

NASA Glenn Research Center
Cleveland, OH

Presentation overview

Overview of Crystallinity in Thermoplastics

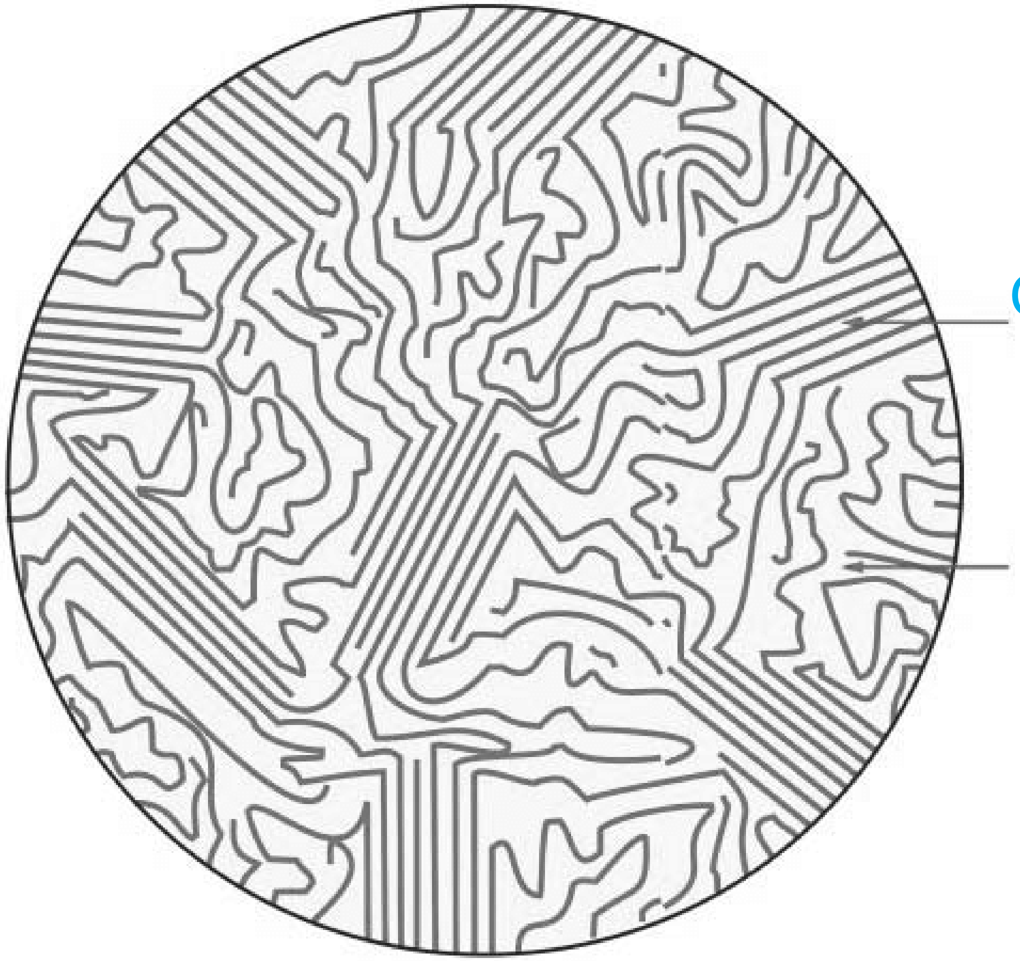
Regolith/PHB Composites for In-Space Manufacturing

Ultrasonic Welding of Carbon Fiber Composites

Injection Molded Composites

Next Steps

Role of Crystallinity in Thermoplastics



Highly crystalline regions have…

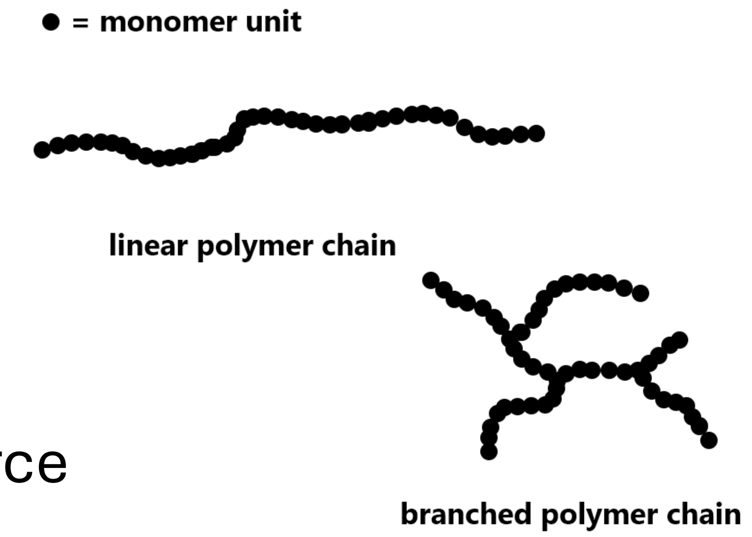
- Increased storage modulus
- Increased chemical resistance
- Increased stiffness and strength
- Increased heat deflection temperature

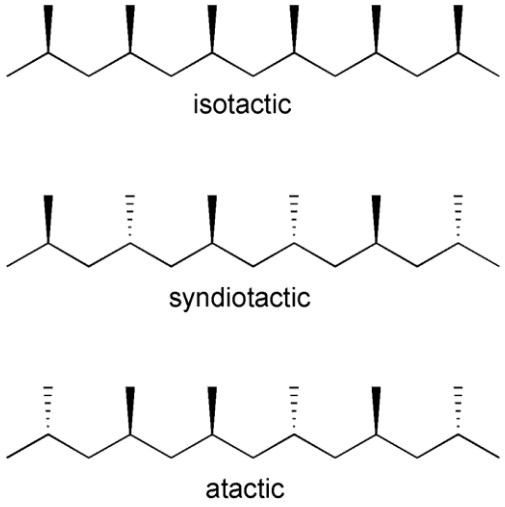
Amorphous regions have…

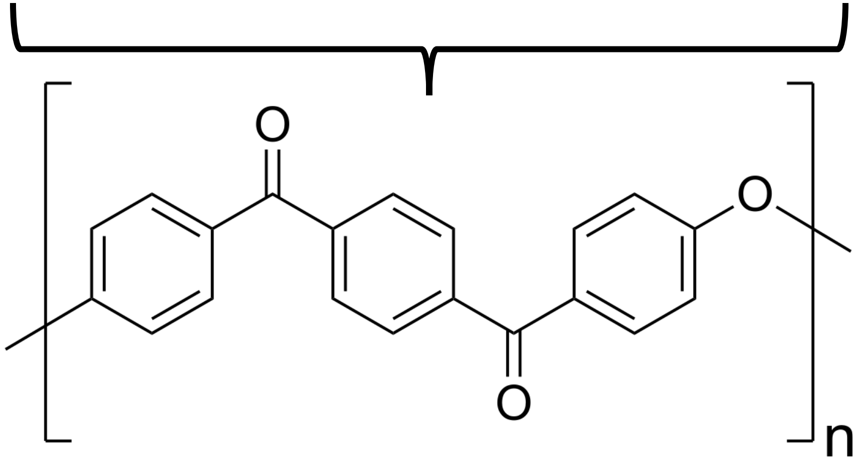
- Increased flexibility
- Optical transparency
- Decreased shrinkage
- Increased gas permeability

Factors Affecting Crystallinity

1. **Chemistry and tacticity**
2. **Polymer structure**
3. Mechanical stress and force
4. Thermal processing



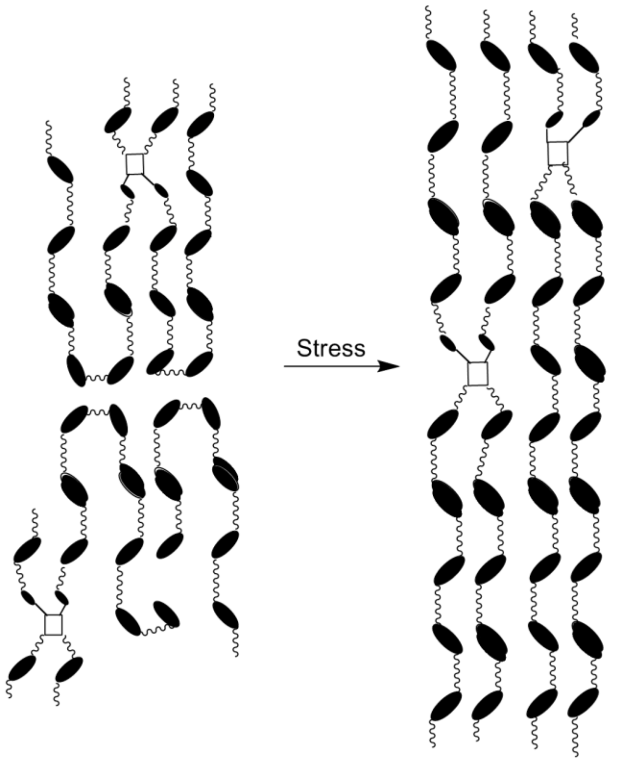




Poly(etherketoneketone) (PEKK)

Factors Affecting Crystallinity

1. Chemistry and tacticity
2. Polymer structure
3. **Mechanical stress and force**
4. **Thermal processing**



Folded structure of the polymer

Elongated structure of the polymer

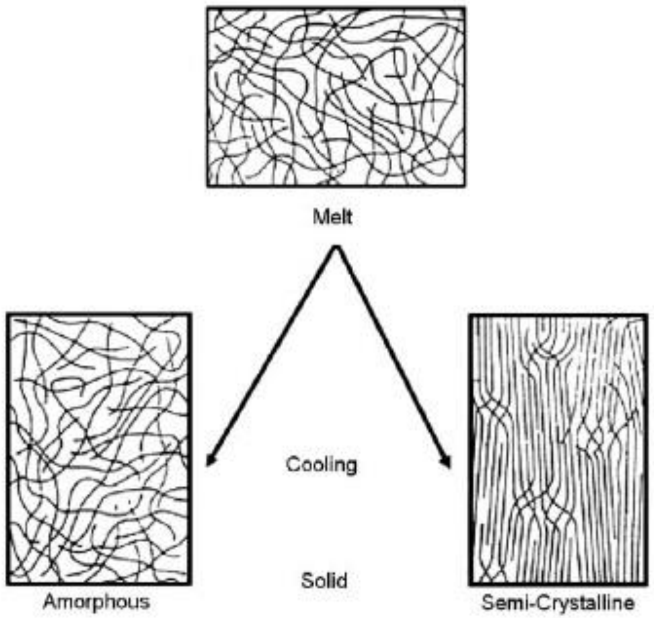
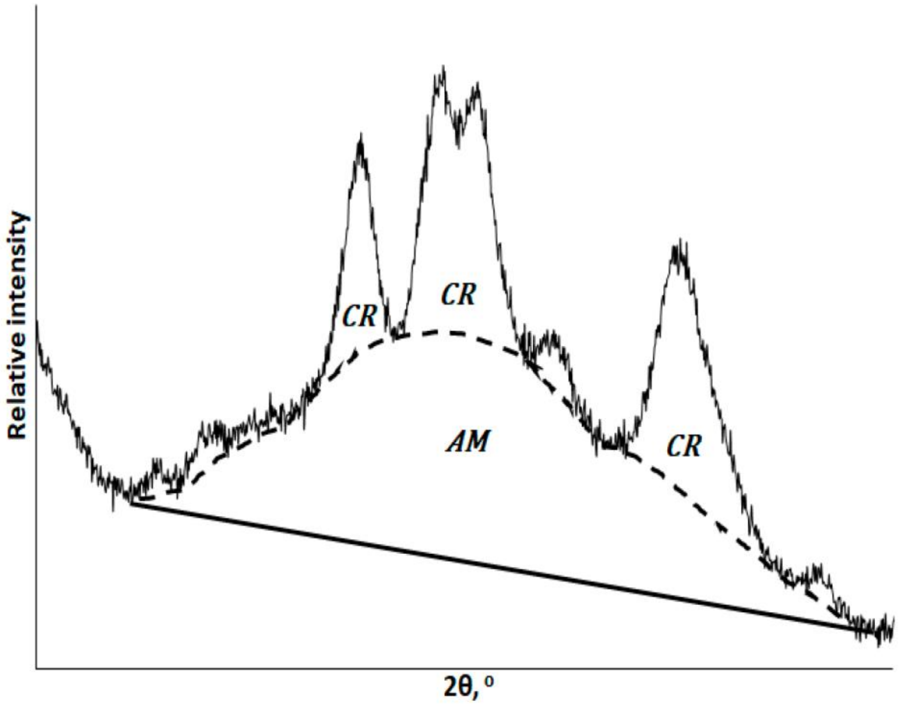


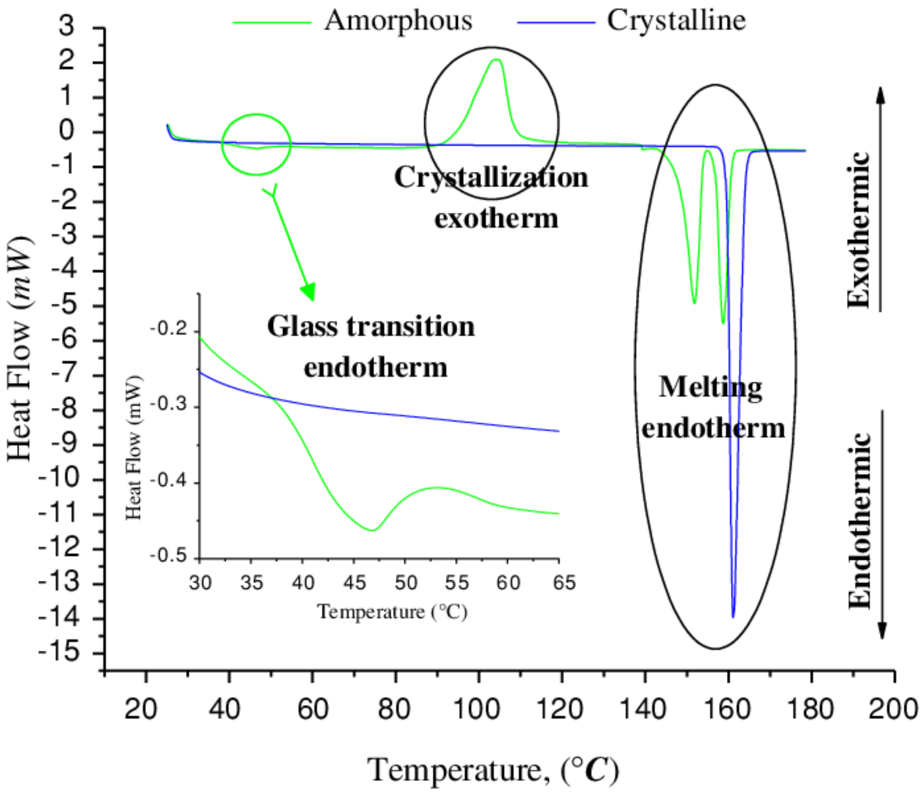
Figure 3.4 Cooling behaviour of materials

Will vary with increased manufacturing speeds and repair interphases

Conventional Methods to Evaluate Polymer Crystallinity

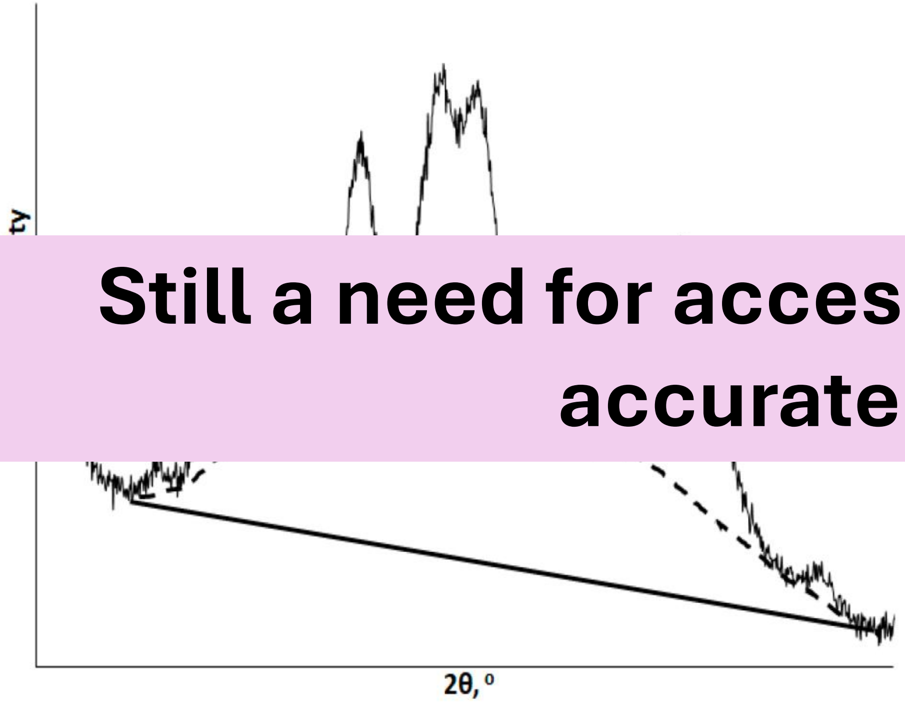


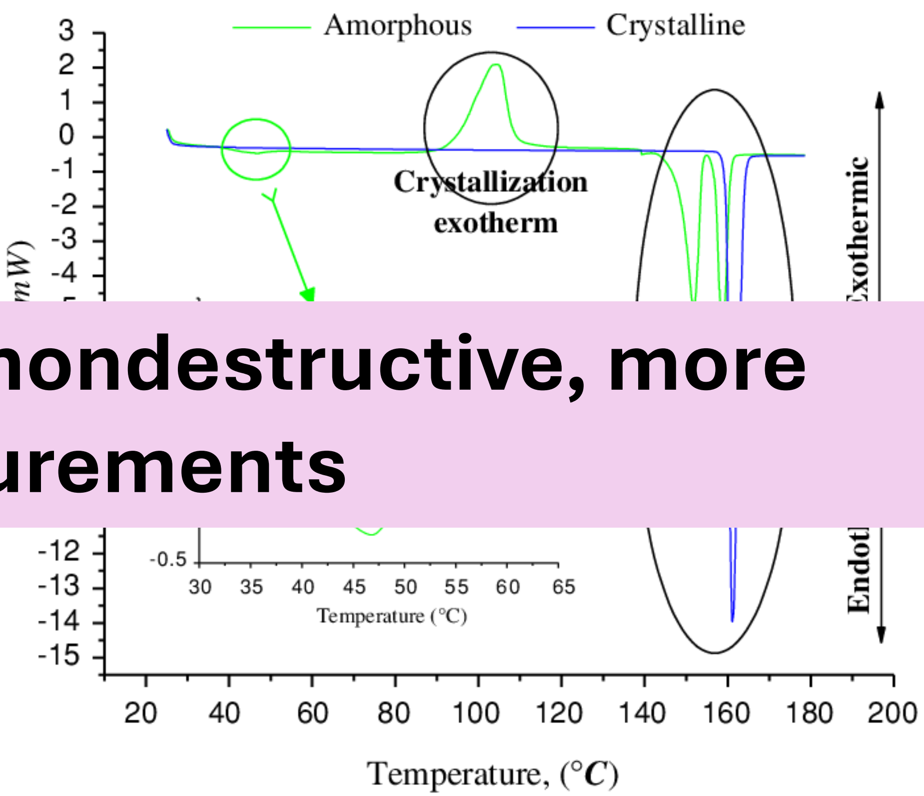
X-Ray Diffraction (XRD)
WAXS/SAXS



Differential Scanning Calorimetry (DSC)

Conventional Methods to Evaluate Polymer Crystallinity



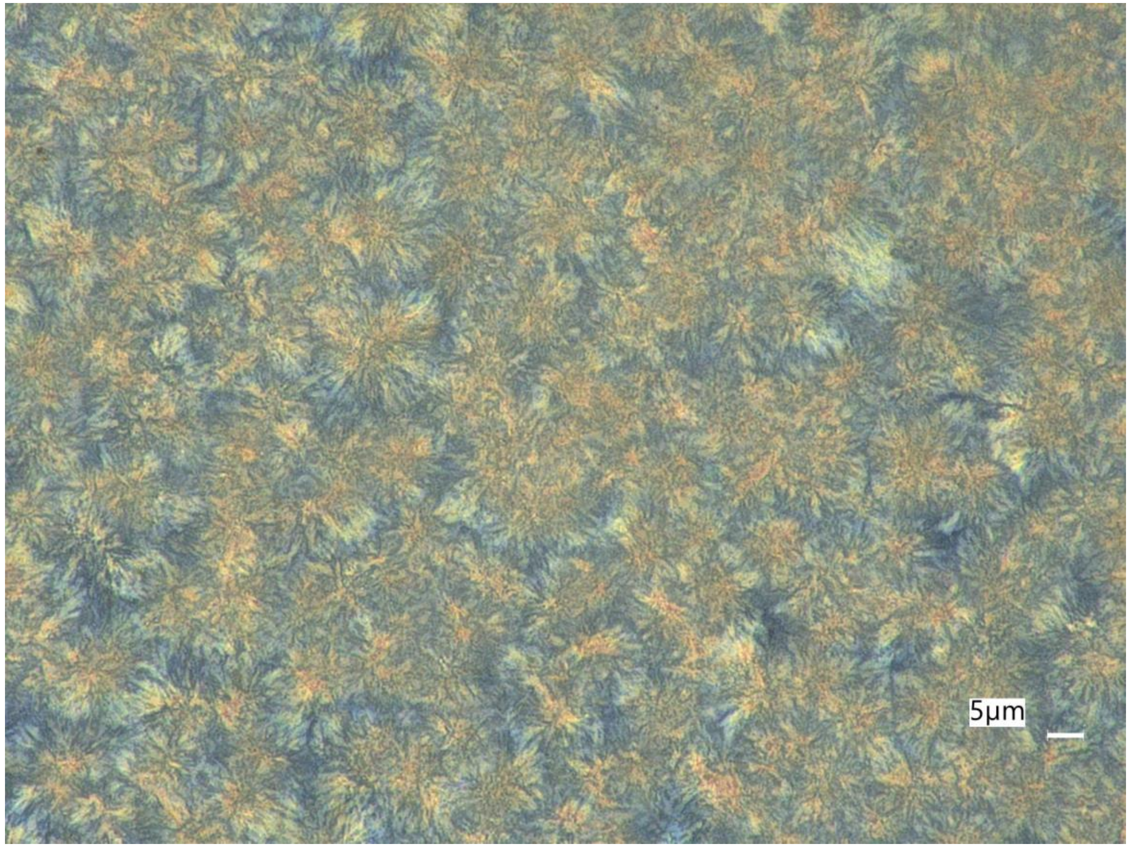


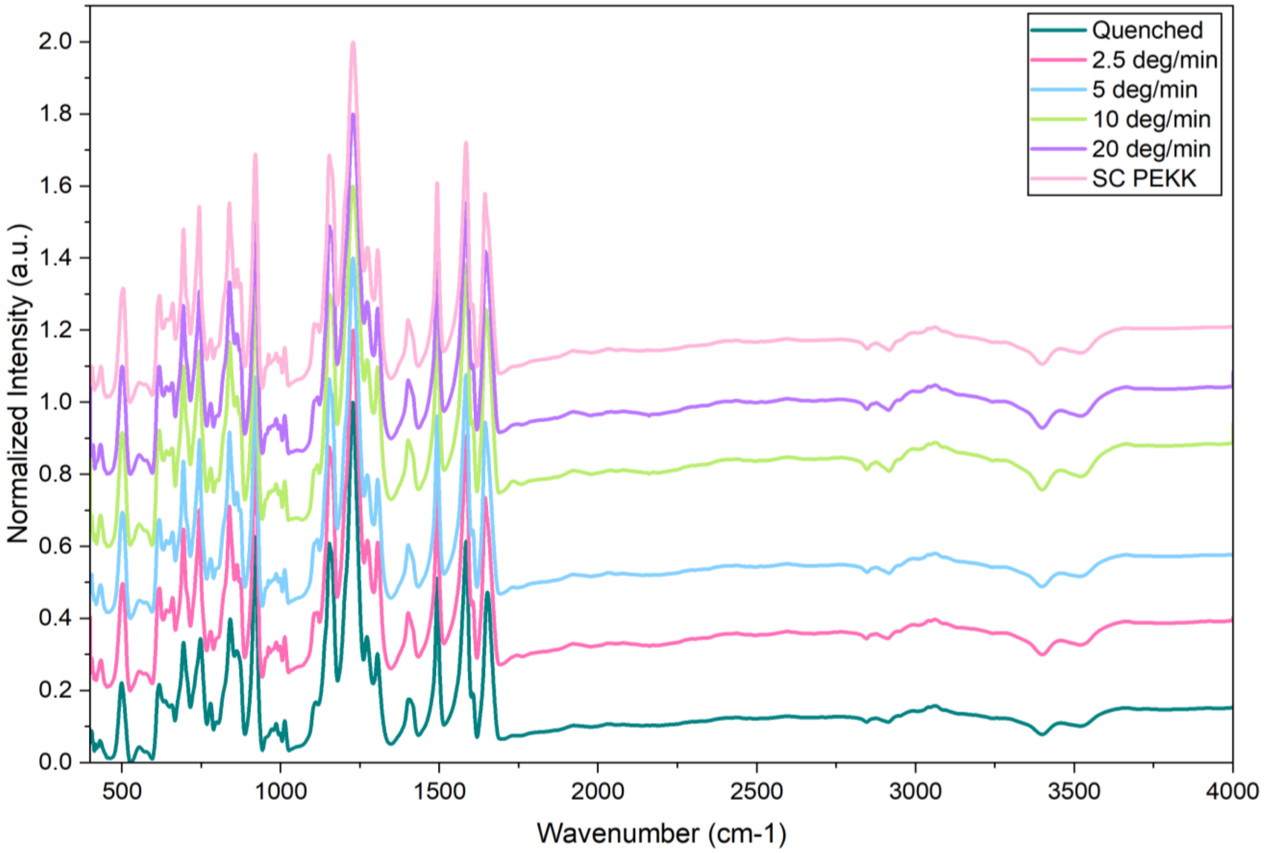
Still a need for accessible, nondestructive, more accurate measurements

X-Ray Diffraction (XRD) WAXS/SAXS

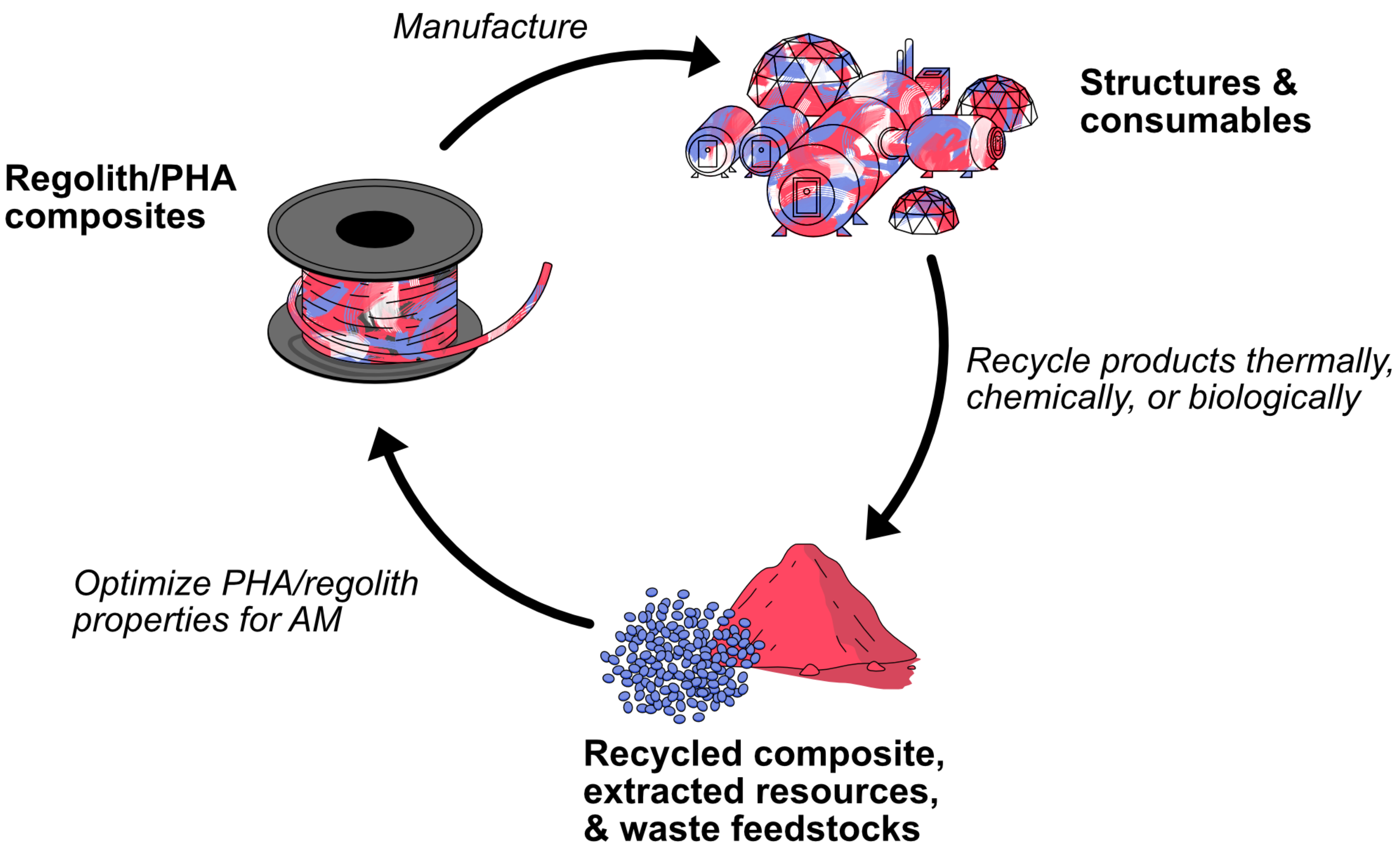
Differential Scanning Calorimetry (DSC)

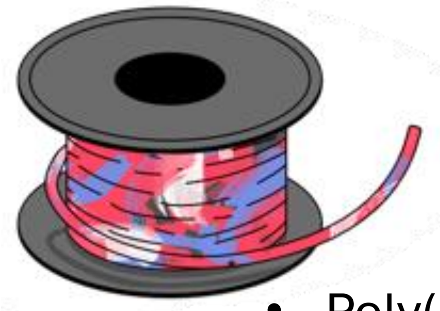
Polarized Light Optical Microscopy and Fourier Transform Infrared (FTIR) Spectroscopy





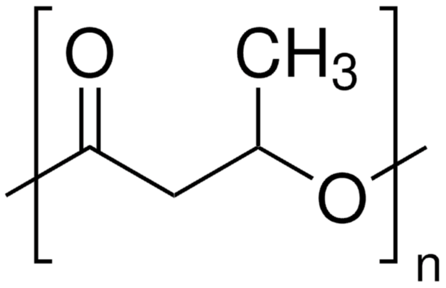
Overview of Closed-Loop ISM System

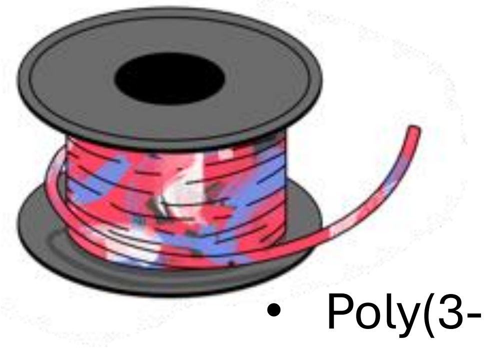




Regolith/Resin Composites

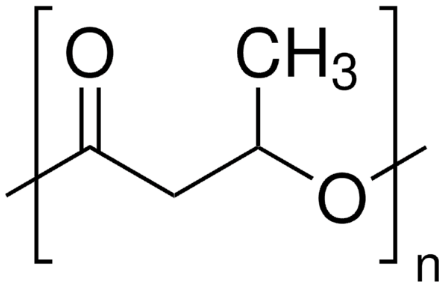
- Poly(3-hydroxybutyrate) (PHB) is a bacterially synthesized resins that use various sources of carbon (including waste products, CO_2, polymers etc.) to create more material
- PHB is highly crystalline but crystallizes very slowly
  - Poor mechanical performance
  - Poor processability





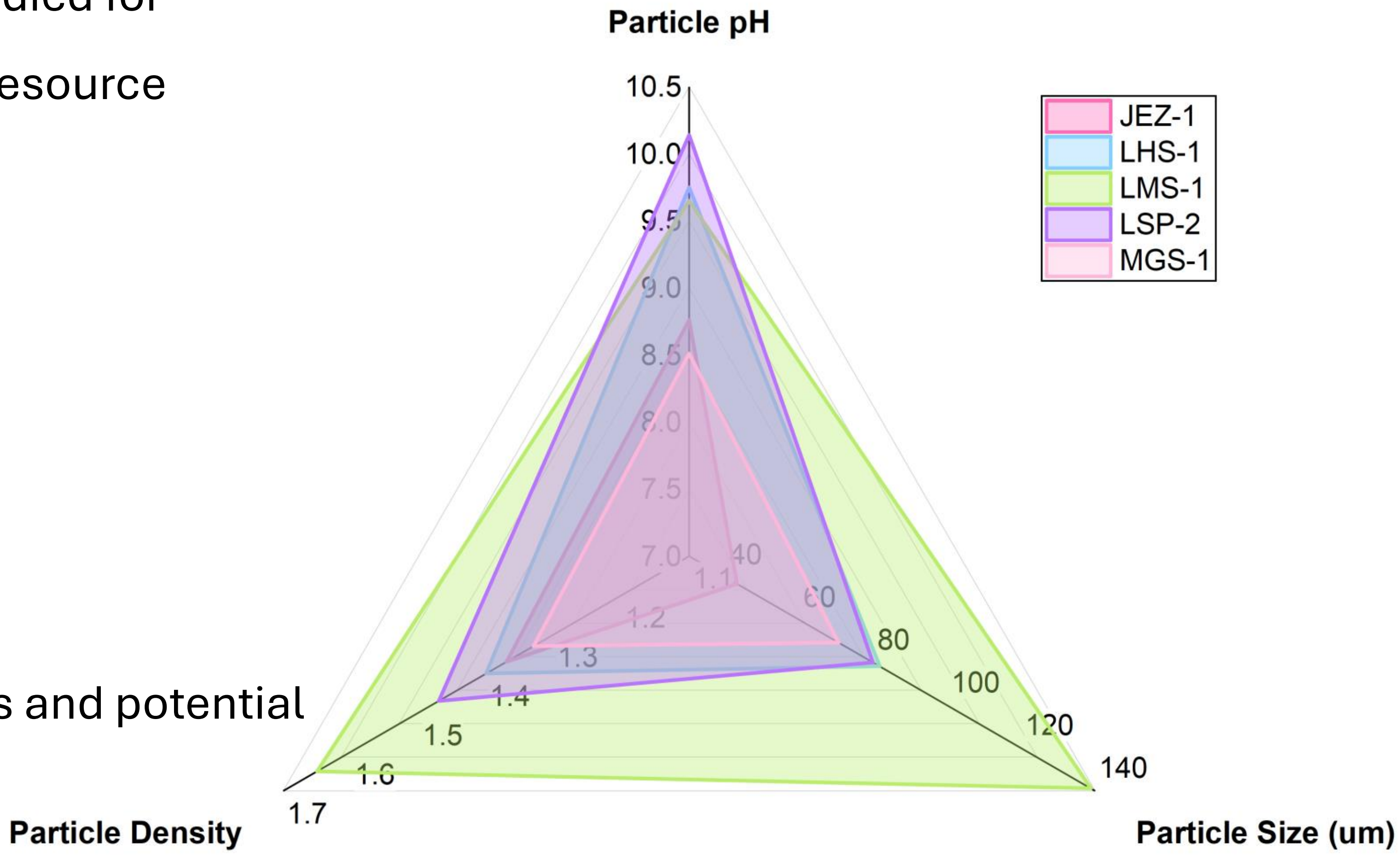
Regolith/Resin Composites

- Poly(3-hydroxybutyrate) (PHB) is a bacterially synthesized resins that use various sources of carbon (including waste products, CO_2, polymers etc.) to create more material
- PHB is highly crystalline but crystallizes very slowly
    - Poor mechanical performance
    - Poor processability
- **Can we use regolith to alter PHB crystallinity?**
- **Does this play a role on material performance?**

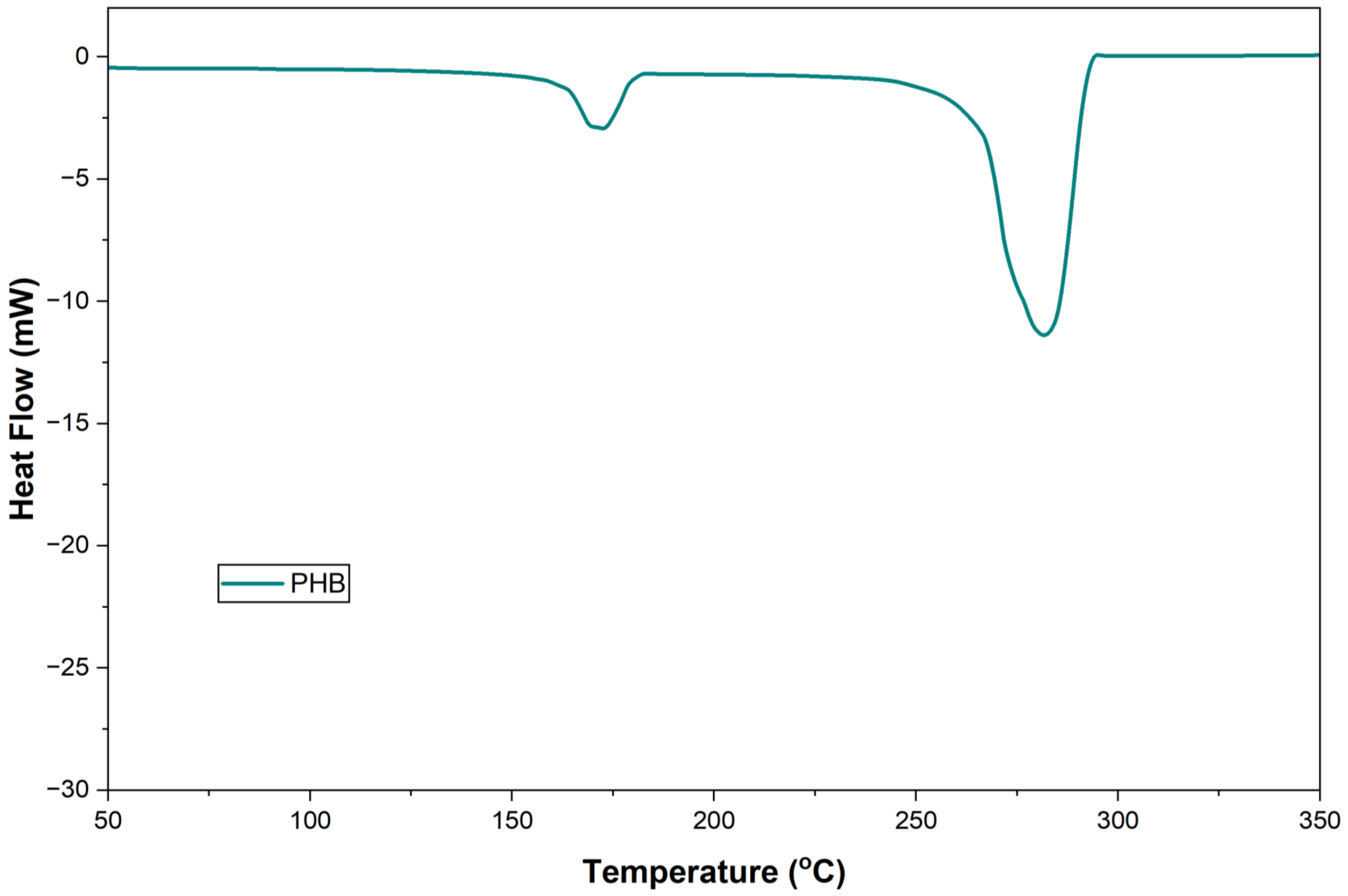


Regolith/PHB Composite Development

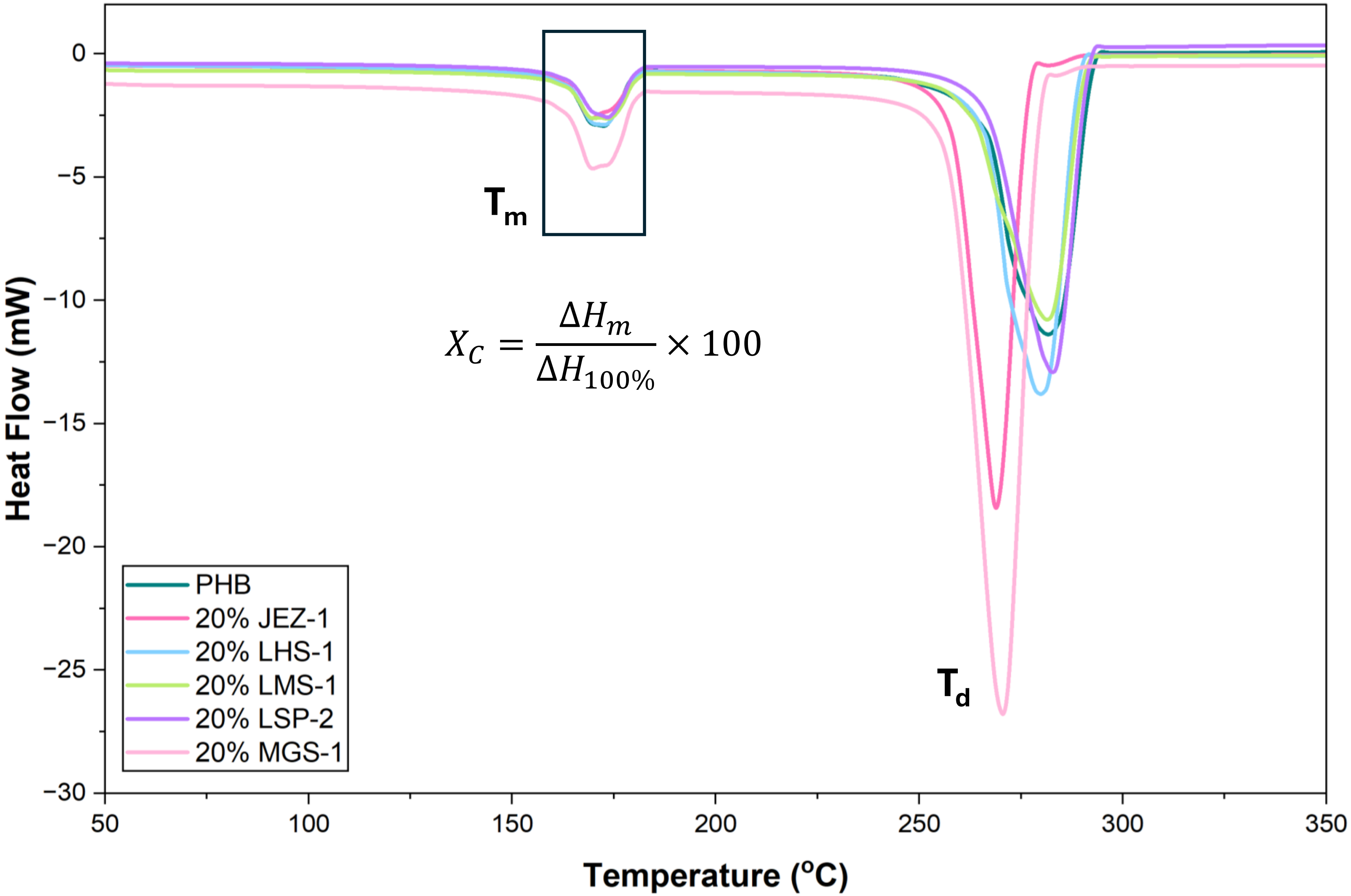
- 5 candidate regolith simulants studied for materials screening from Space Resource Technologies
  - Mars Jezero Delta (JEZ-1)
  - Lunar Highlands (LHS-1)
  - Lunar Mare (LMS-1)
  - Lunar South Pole (LSP-2)
  - Mars Global (MGS-1)
- Design system for as many regions and potential applications as possible



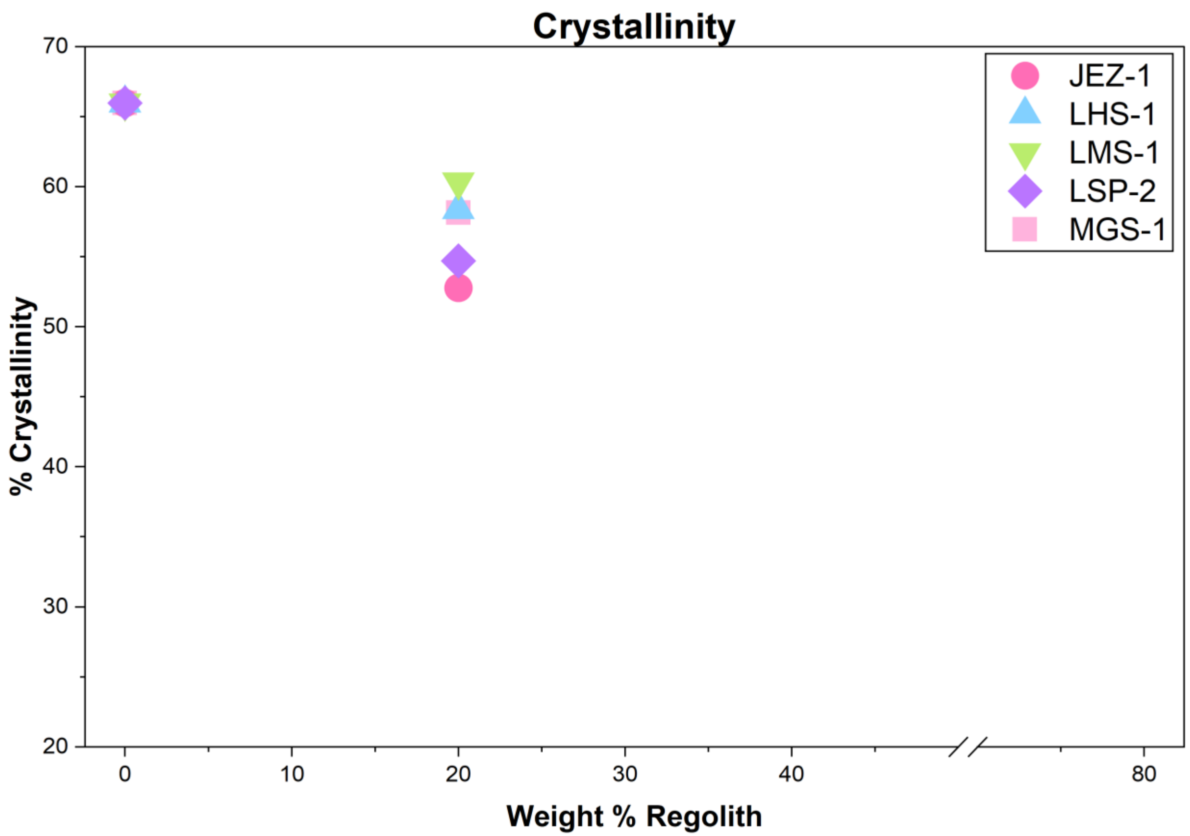
Composites with 20 wt% Regolith

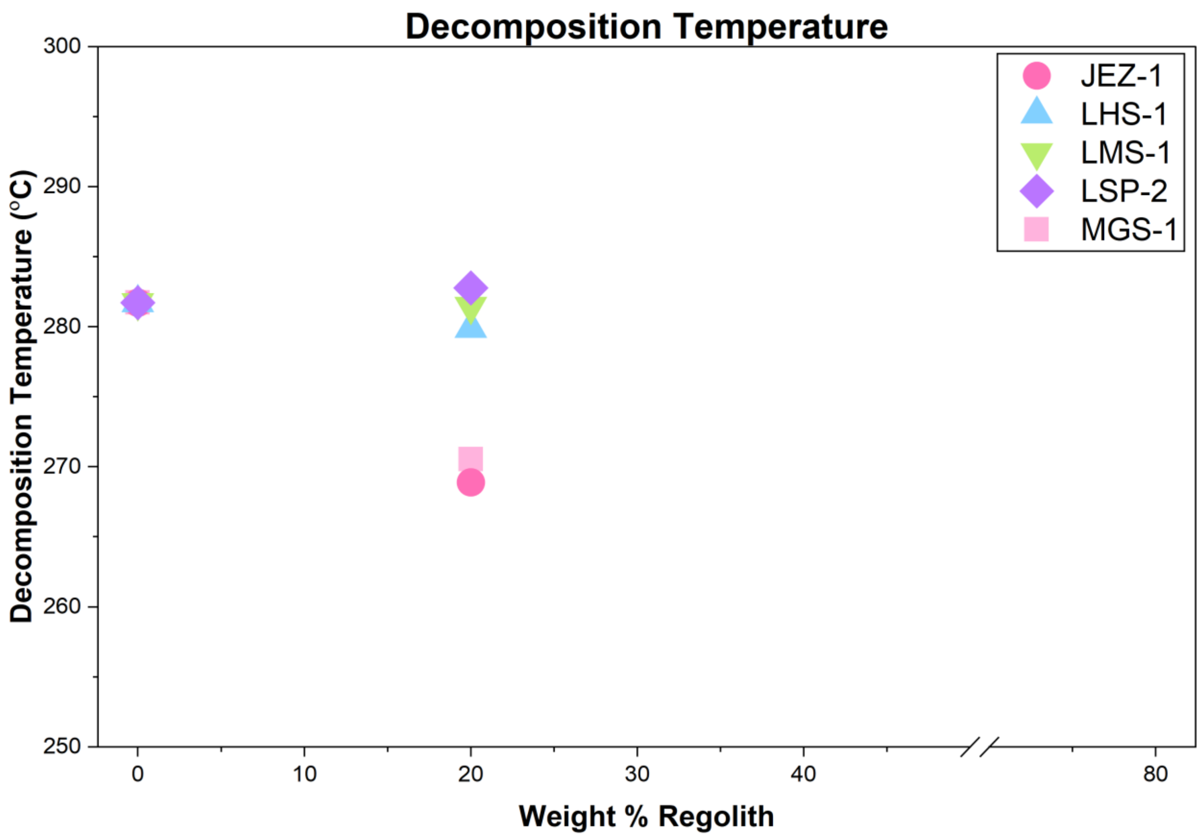


Composites with 20 wt% Regolith

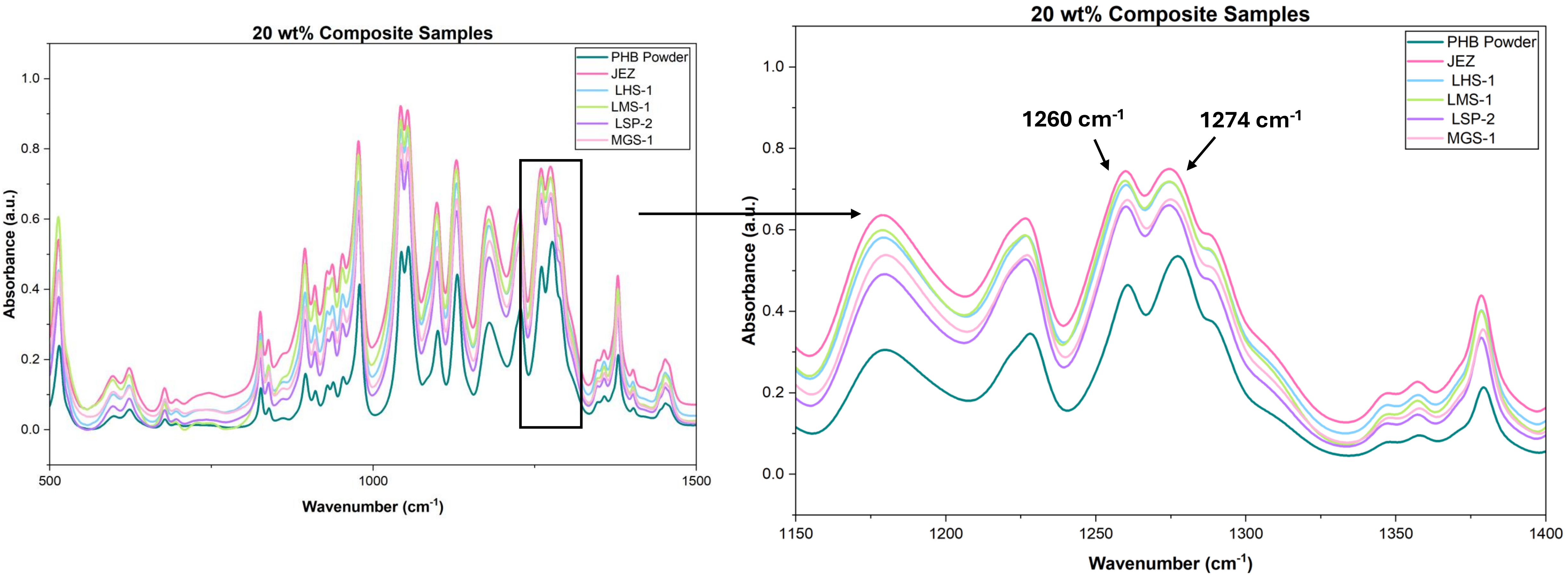


Composites with 20 wt% Regolith

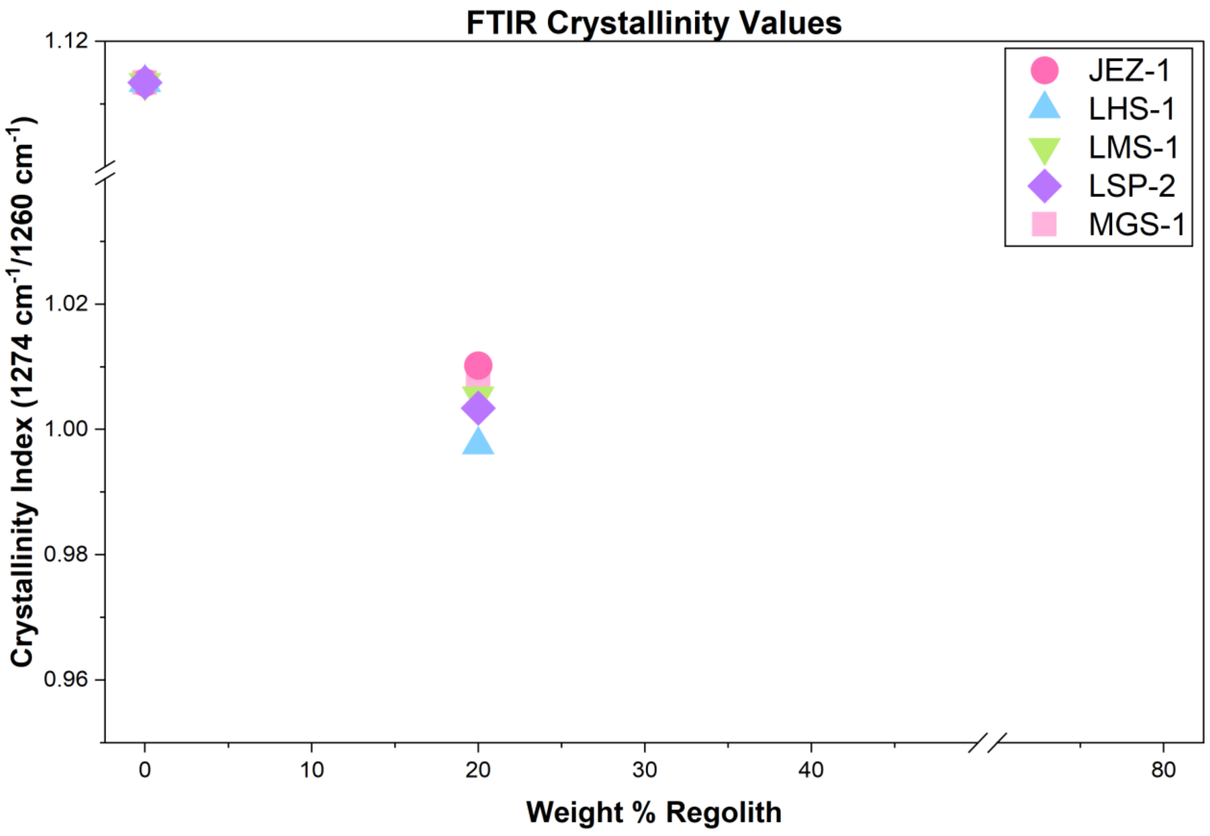


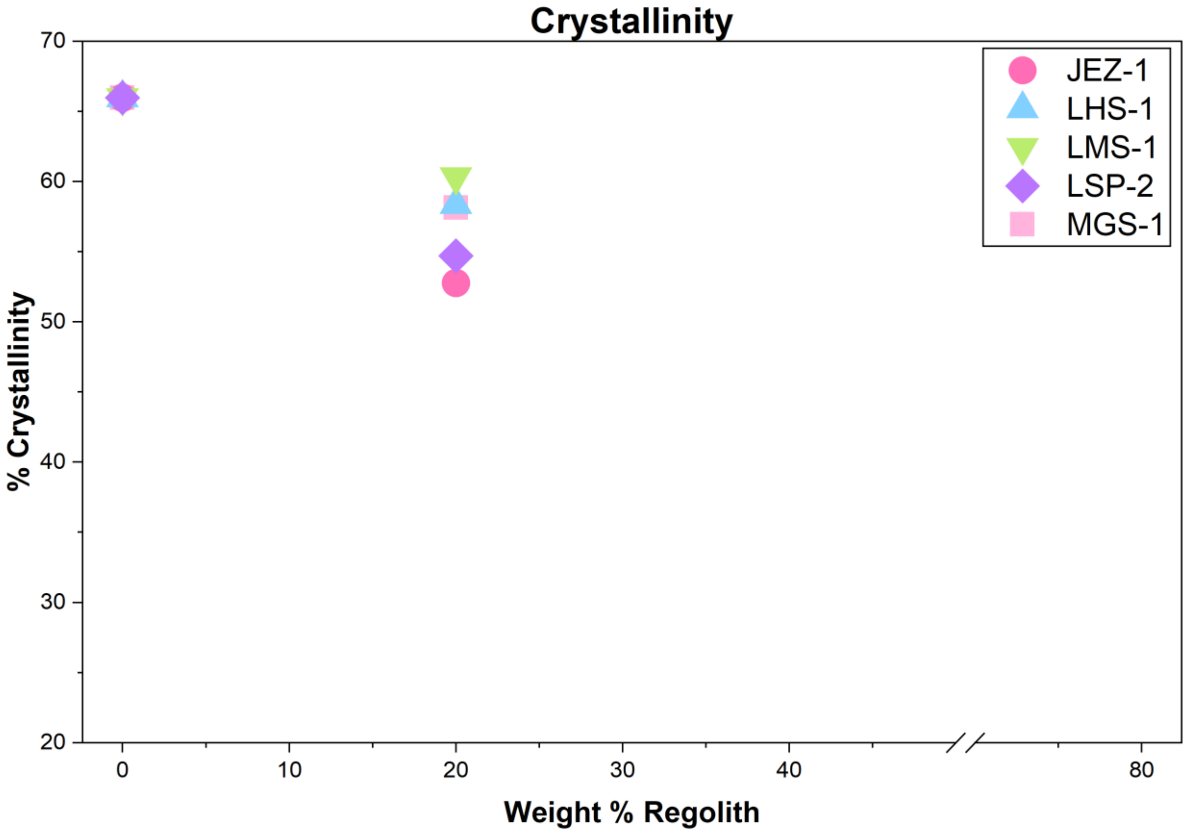


Composites with 20 wt% Regolith



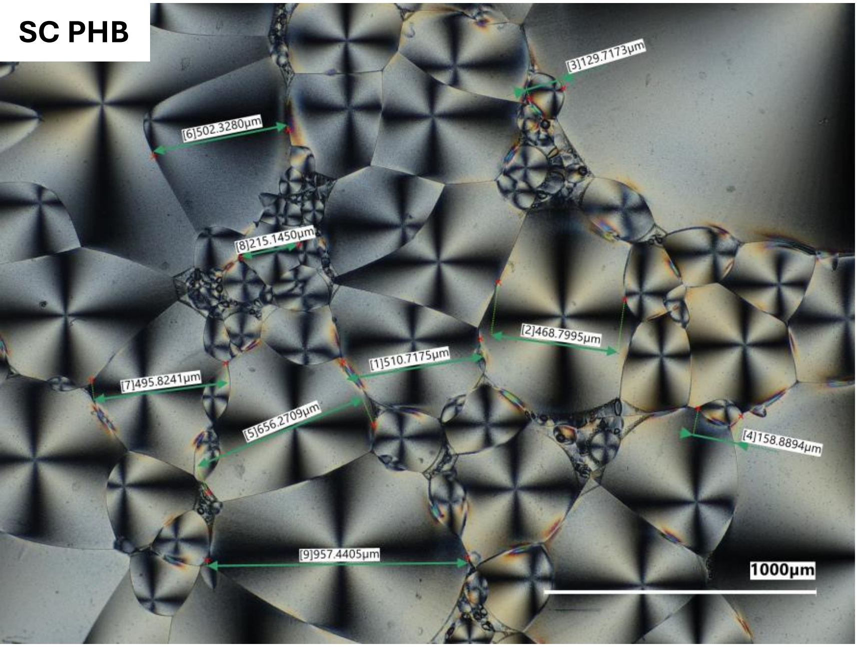
Composites with 20 wt% Regolith



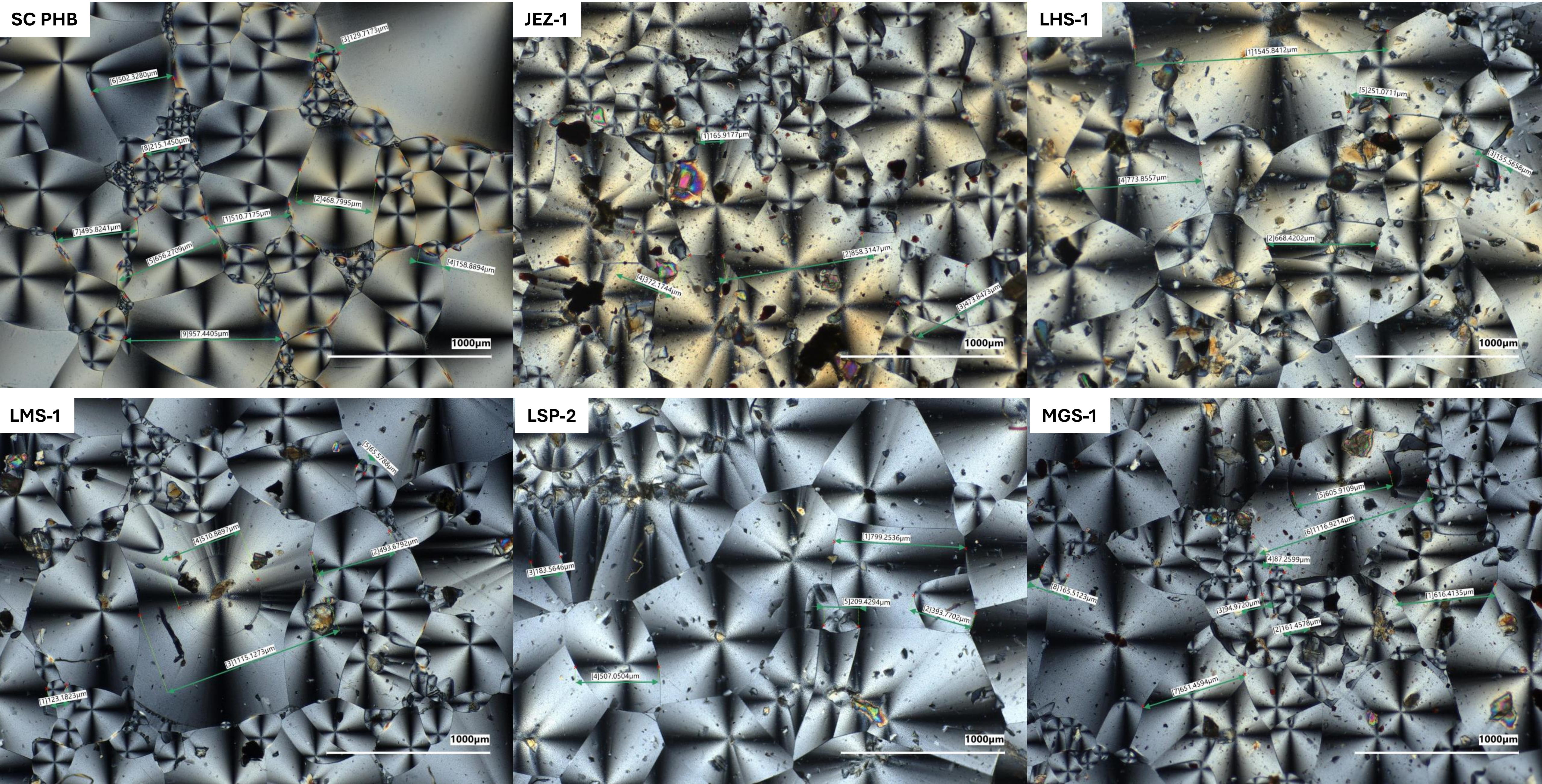


Composites with 20 wt% Regolith

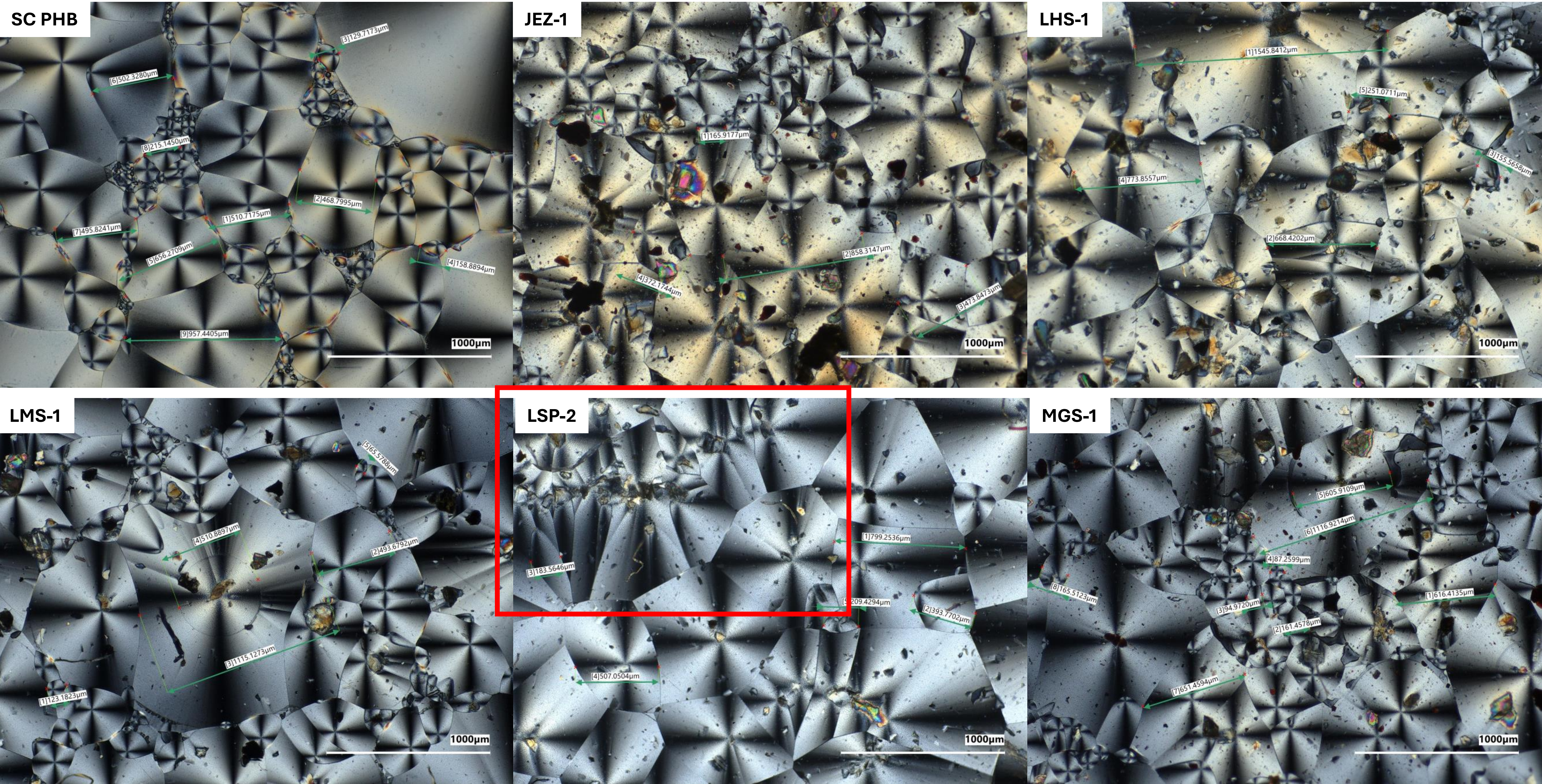
SC PHB



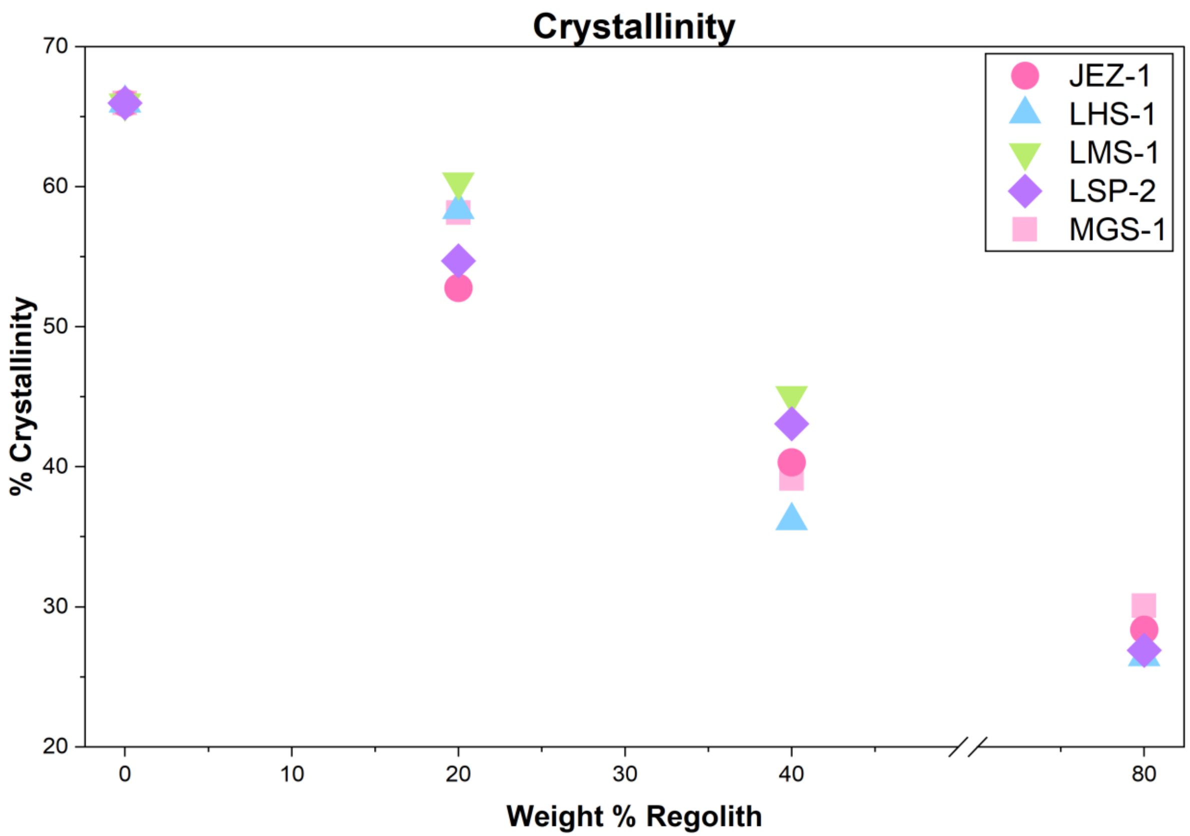
Composites with 20 wt% Regolith

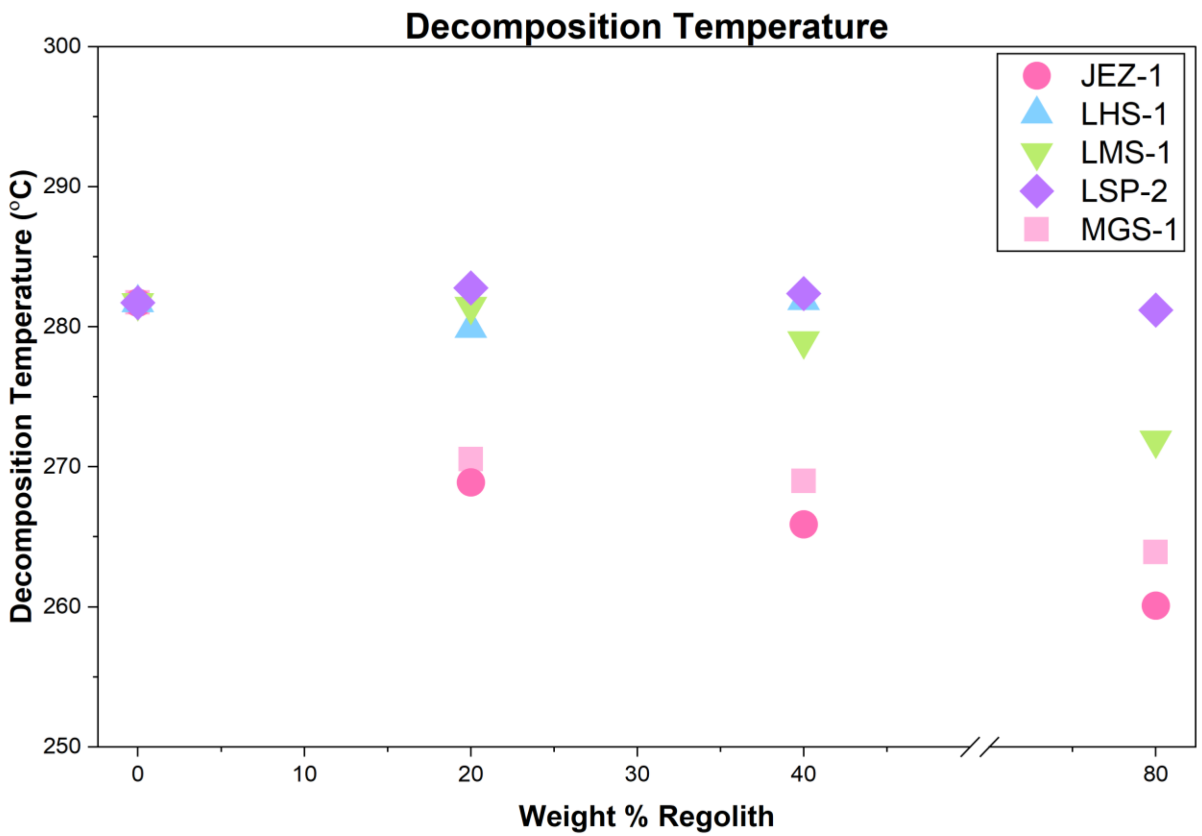


Composites with 20 wt% Regolith

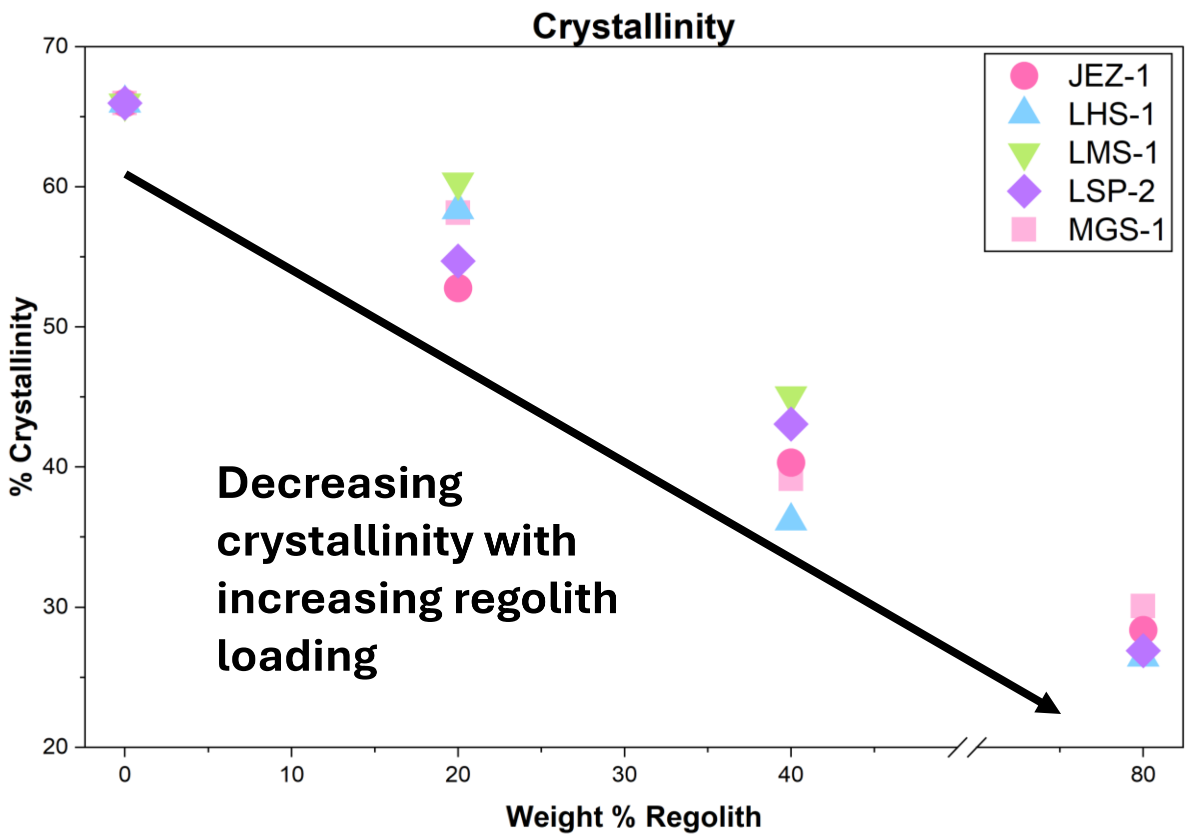


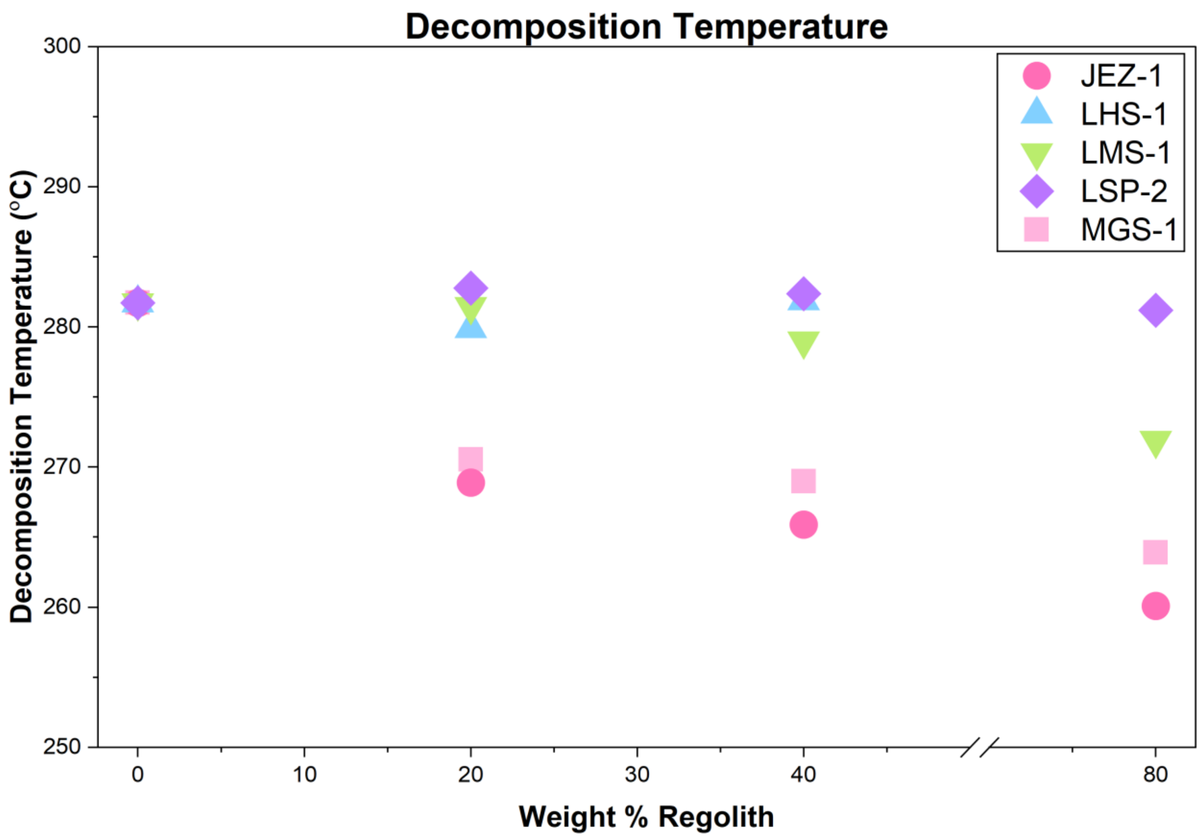
Effect of Regolith Loading





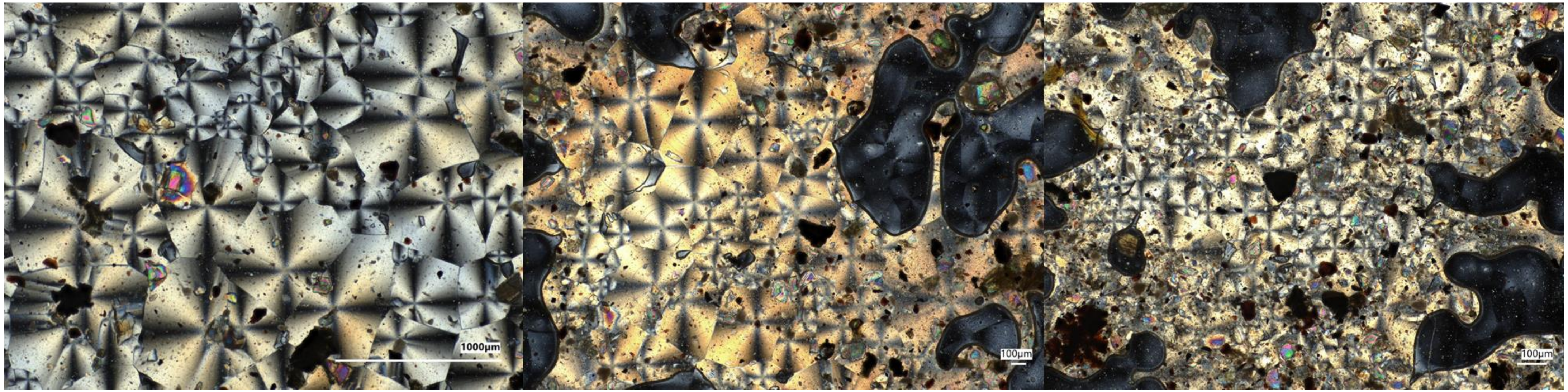
Effect of Regolith Loading – *Differential Scanning Calorimetry*



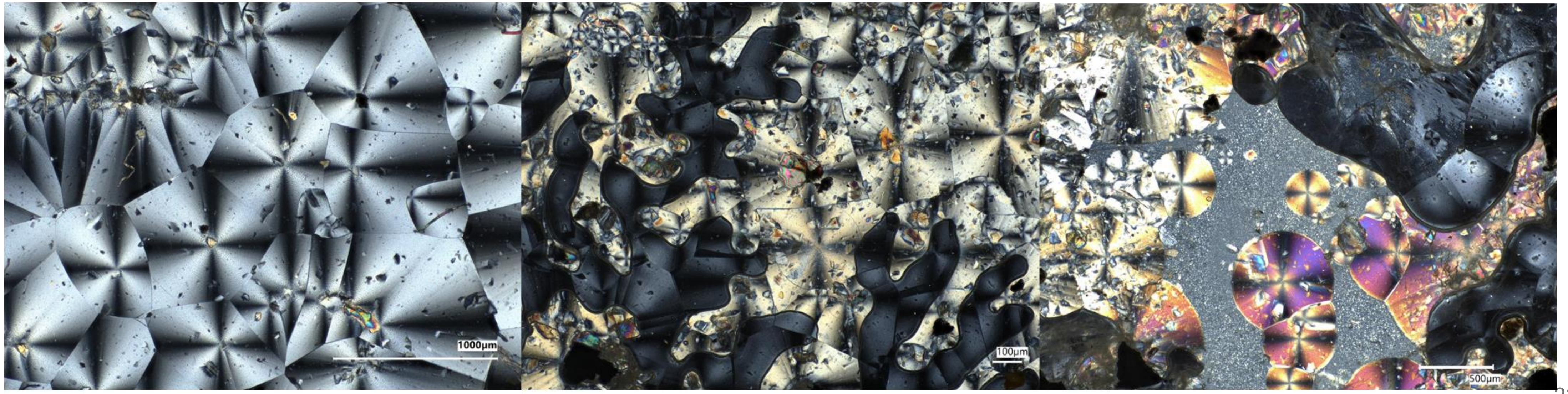


Effect of Regolith Loading – *Differential Scanning Calorimetry*

JEZ-1

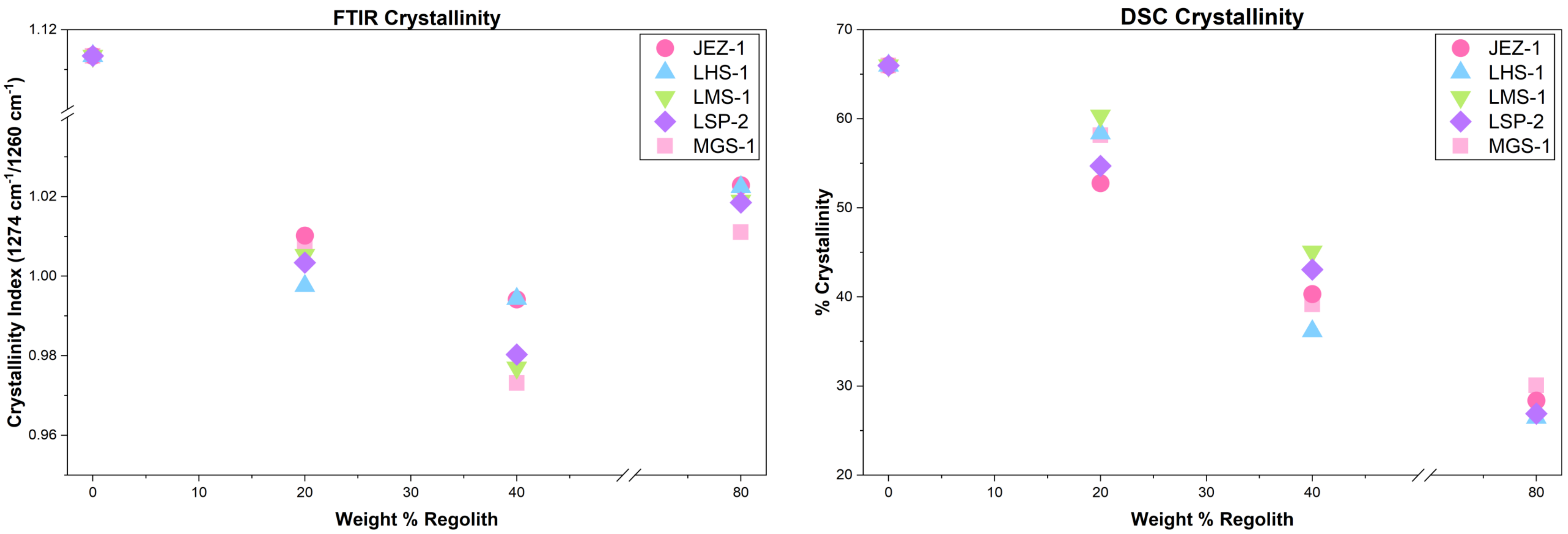


LSP-2



20% | 40% | 80%

Effect of Regolith Loading –
Differential Scanning Calorimetry

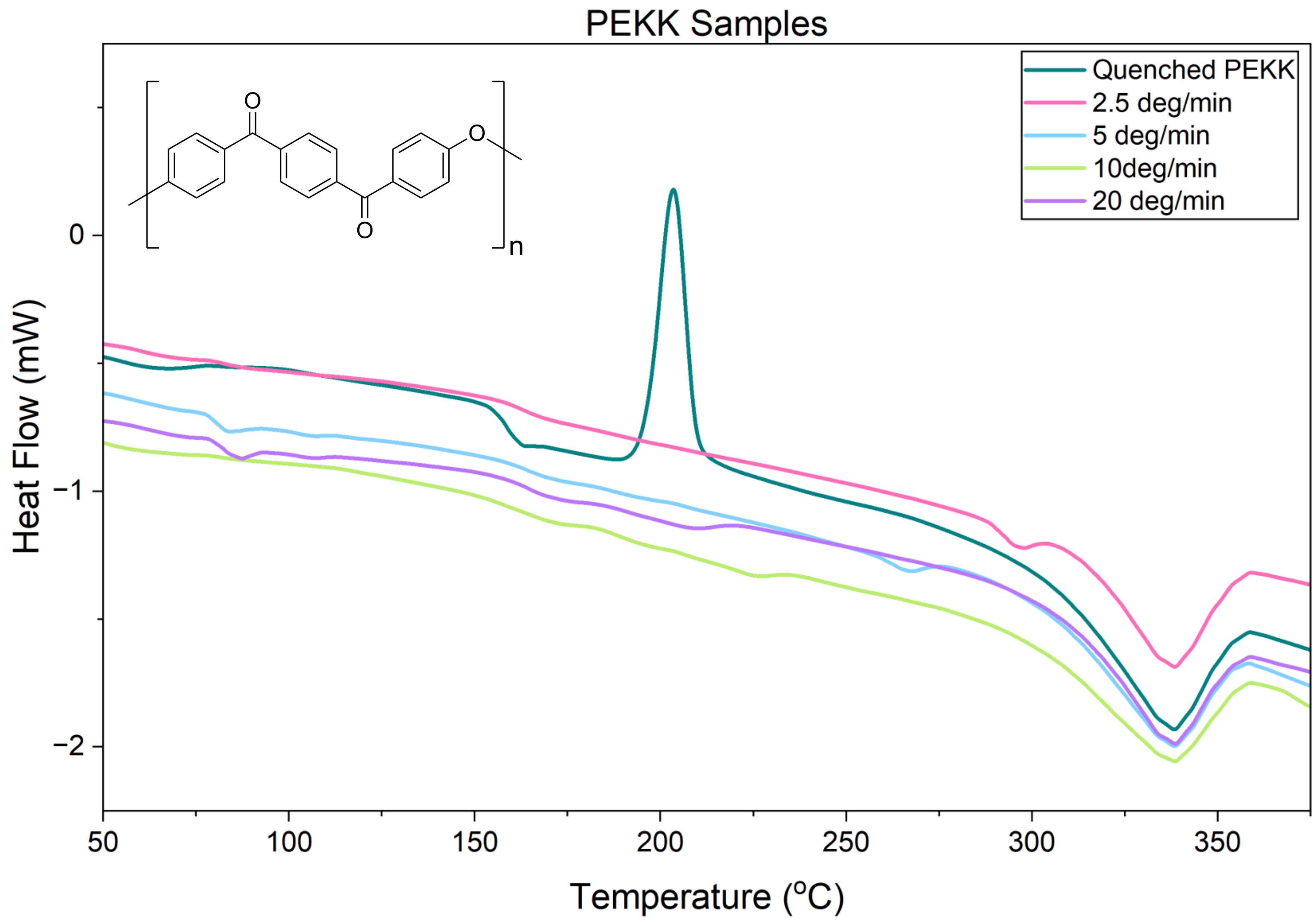


Crystallinity is Complex & Composites are Difficult to Evaluate

Varied cooling rates and stress exposure,

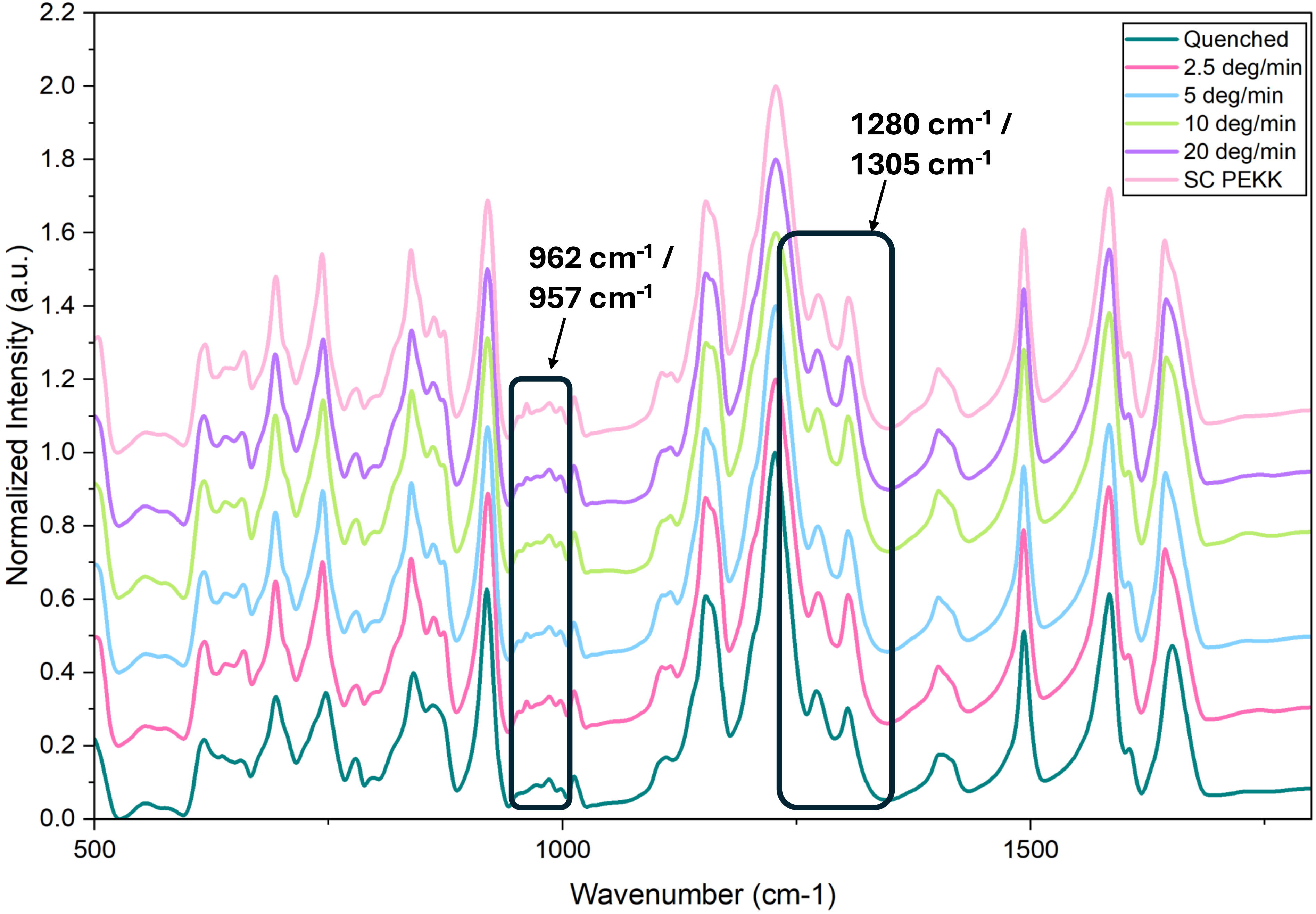
Varied crystallinity profile, varied composite properties

Differential Scanning Calorimetry of PEKK Resin

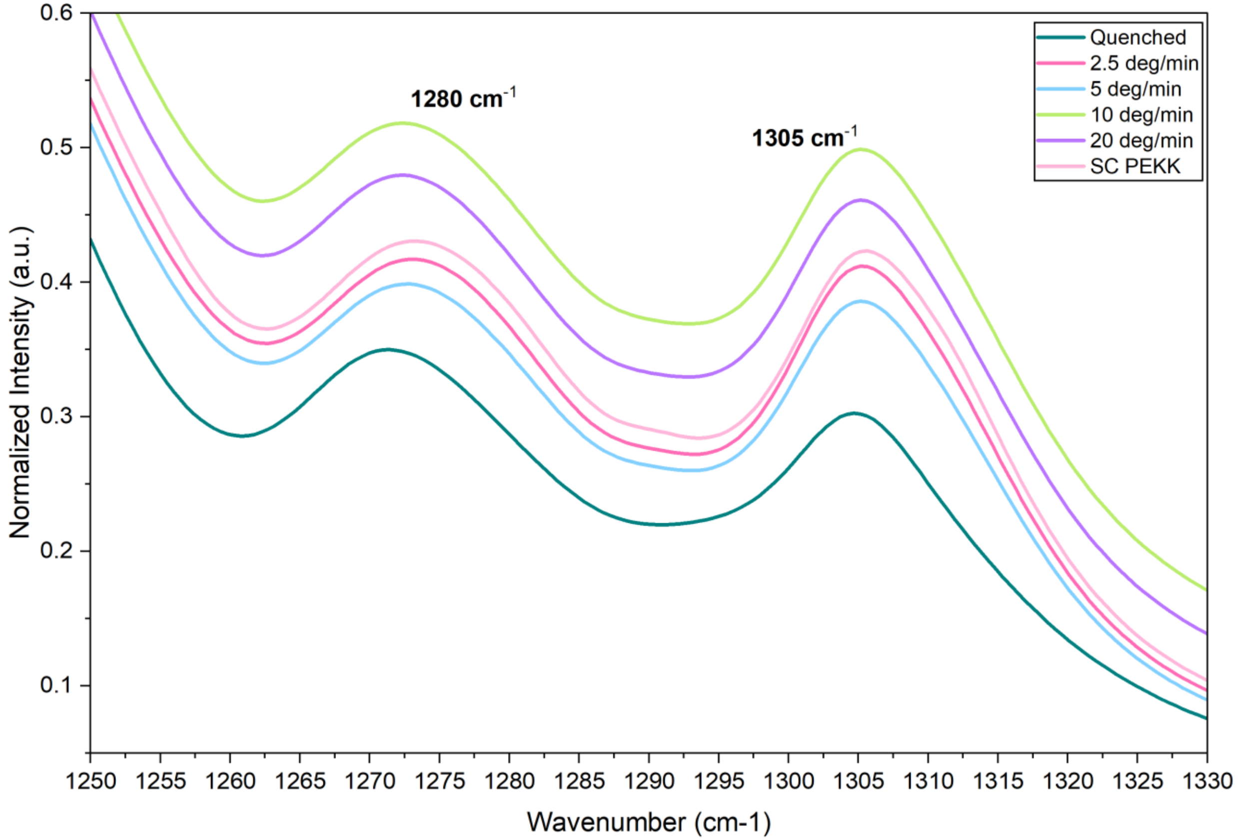


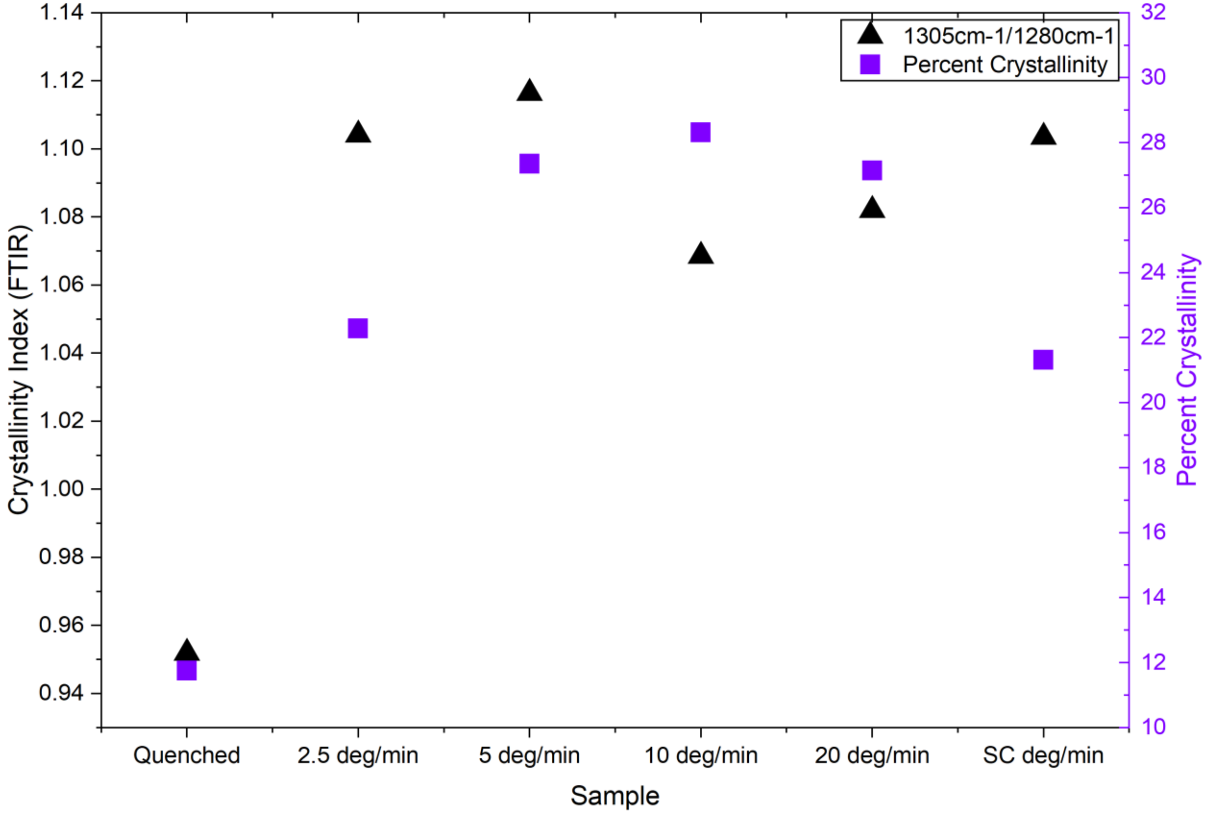
$$X_C = \frac{\Delta H_m}{\Delta H_{100\%}} \times 100$$

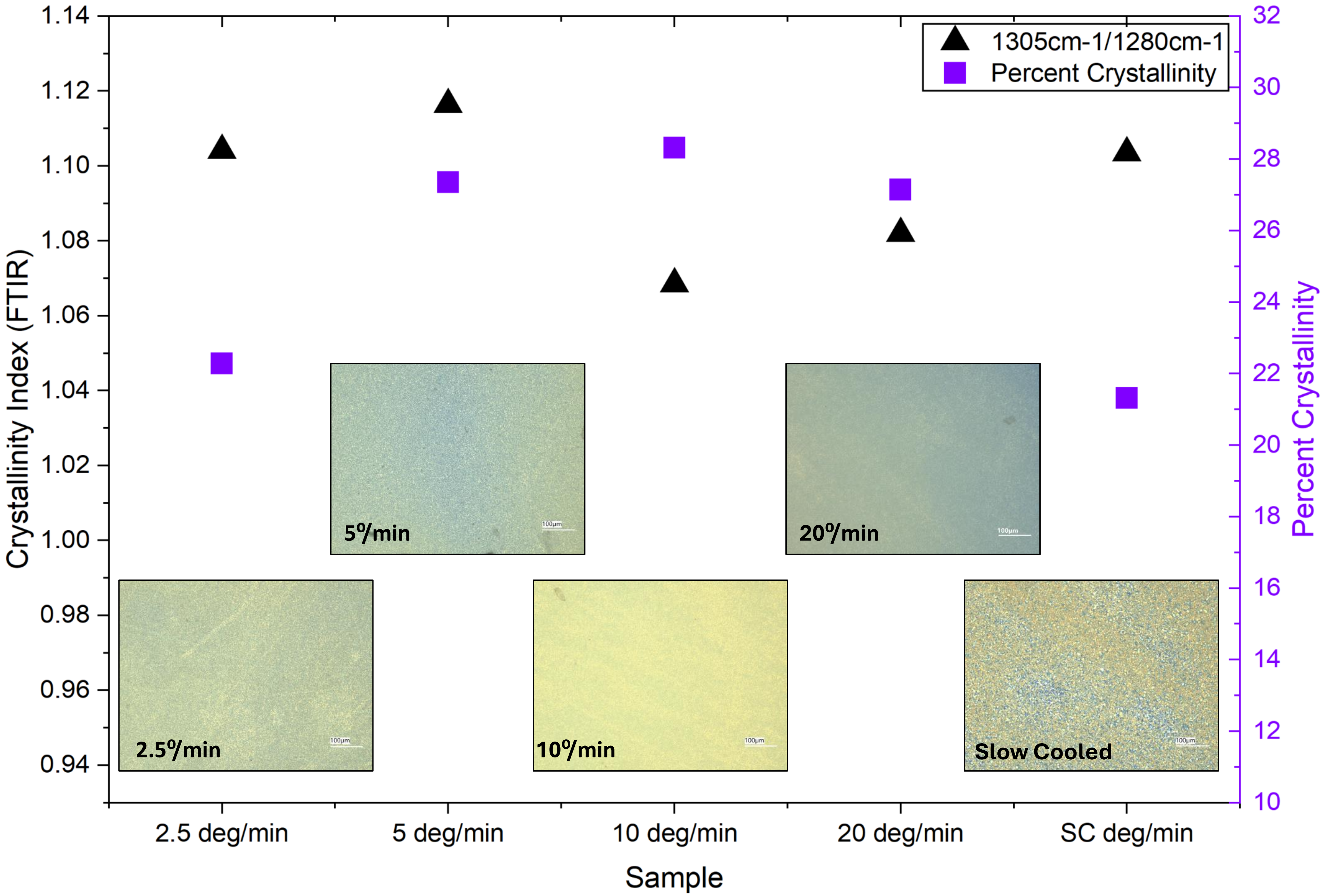
Sample	X_C
Quenched	11.75
2.5 deg/min	22.28
5 deg/min	27.35
10 deg/min	28.32
20 deg/min	27.14


Quenched
2.5 deg/min
5 deg/min
10 deg/min
20 deg/min
SC PEKK
962 cm-1 / 957 cm-1
1280 cm-1 / 1305 cm-1
Normalized Intensity (a.u.)
Wavenumber (cm-1)
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
500
1000
1500

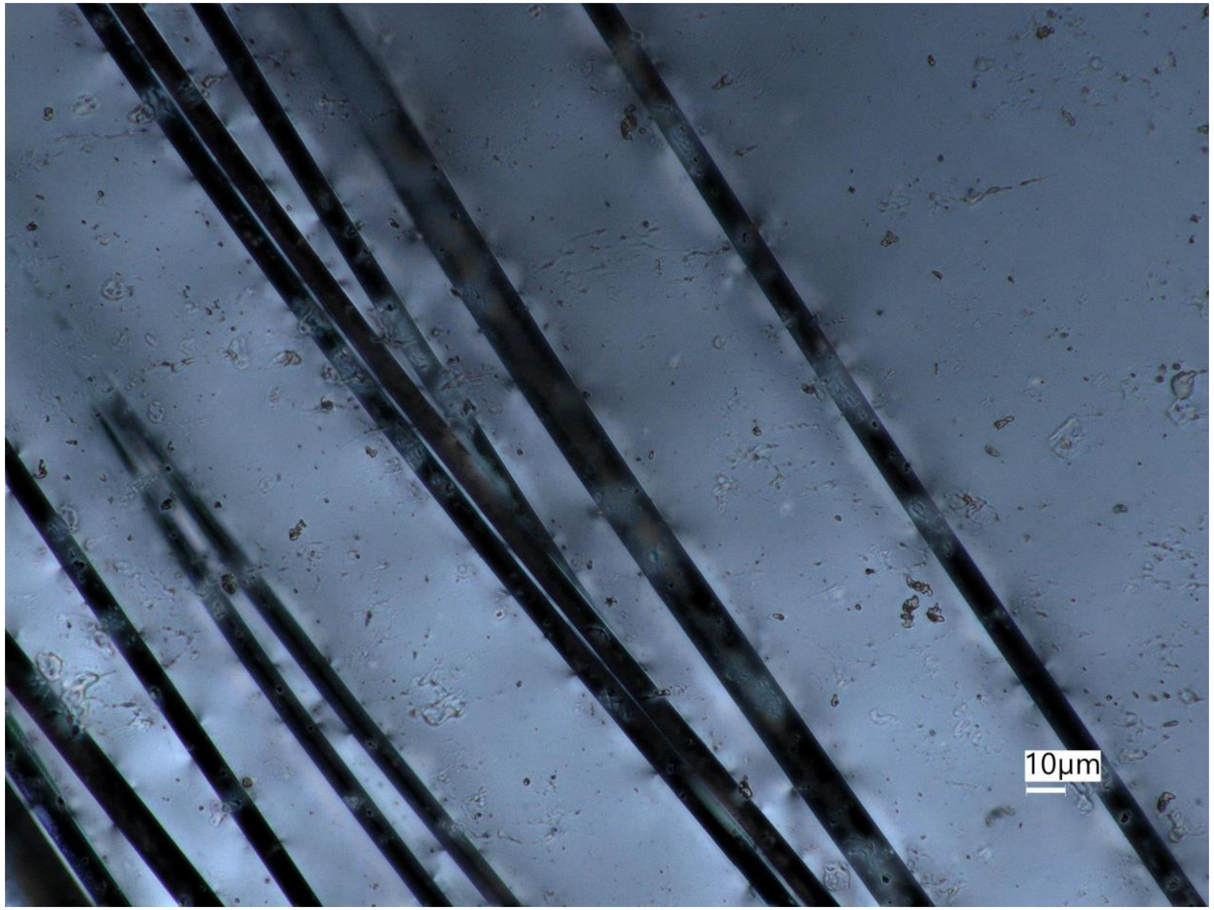
Fourier Transform Infrared (FTIR) Spectroscopy – Crystallinity Index



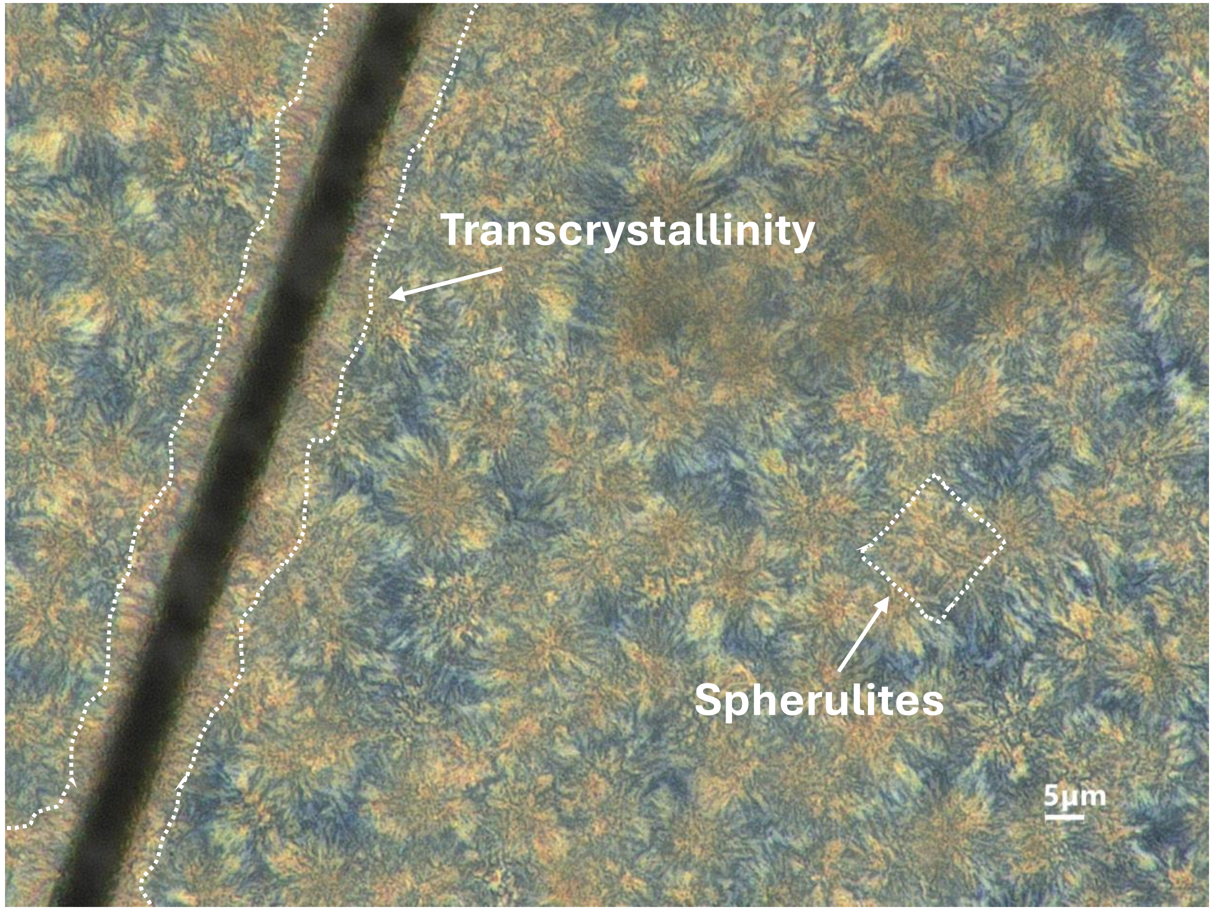



1305cm-1/1280cm-1
Percent Crystallinity
Crystallinity Index (FTIR)
Percent Crystallinity
Sample
2.5 deg/min
5 deg/min
10 deg/min
20 deg/min
SC deg/min
1.14
1.12
1.10
1.08
1.06
1.04
1.02
1.00
0.98
0.96
0.94
32
30
28
26
24
22
20
18
16
14
12
10
5°/min
20°/min
2.5°/min
10°/min
Slow Cooled

Differentiating Crystallites

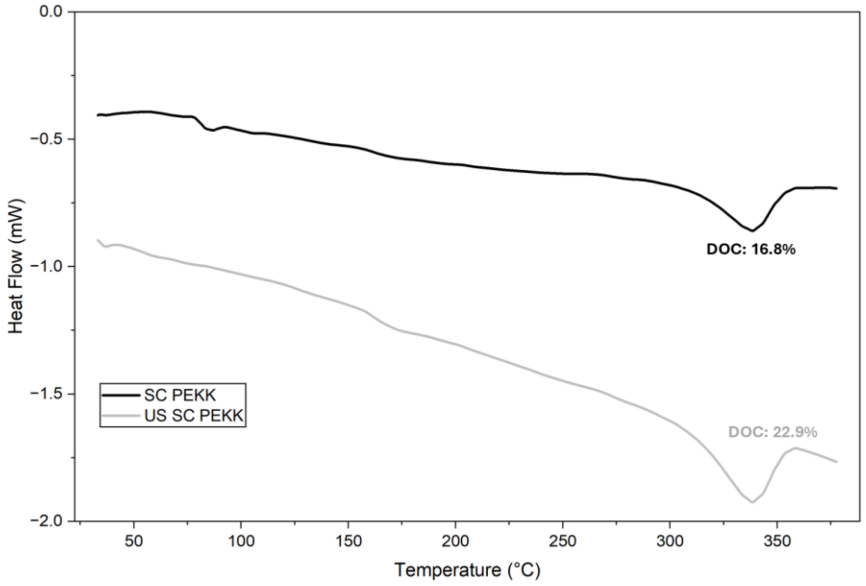


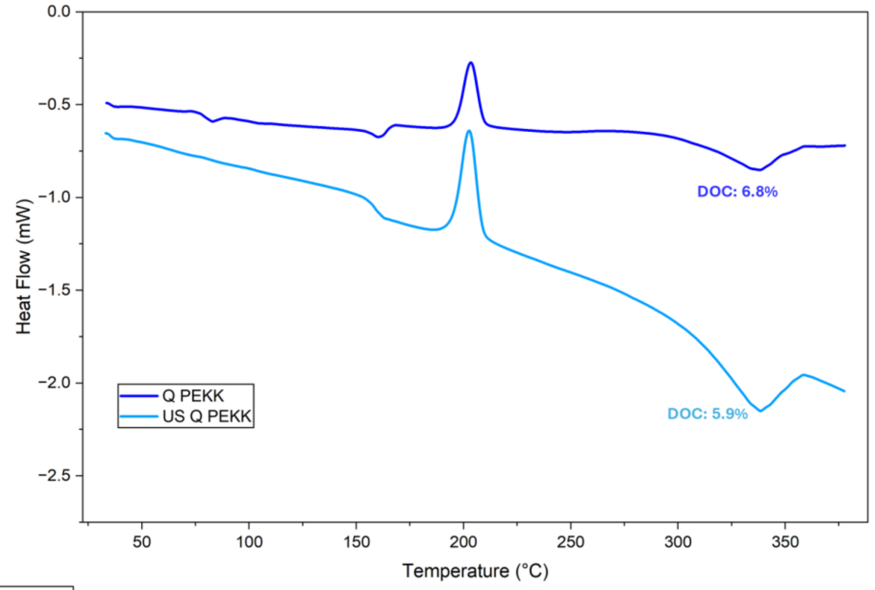
Quenched
X_C: 11.75%

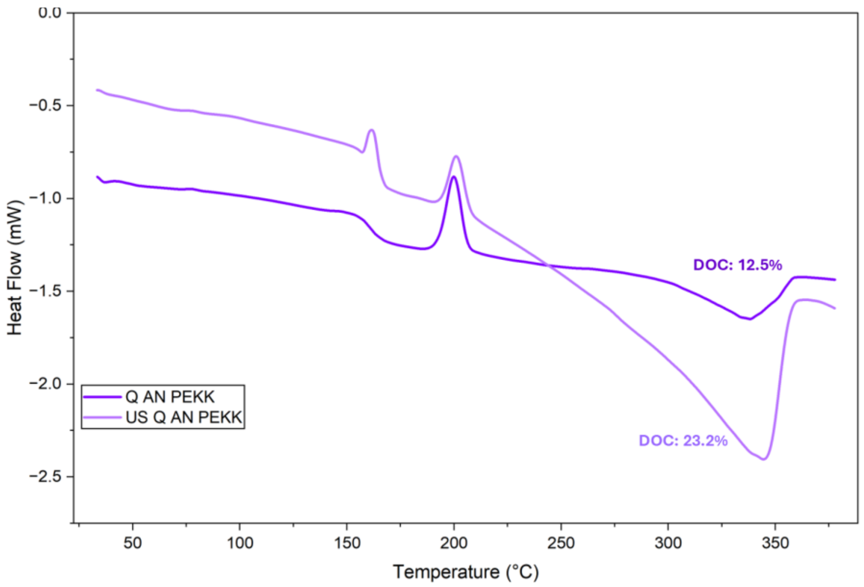


Slow Cooled
X_C: 21.32%

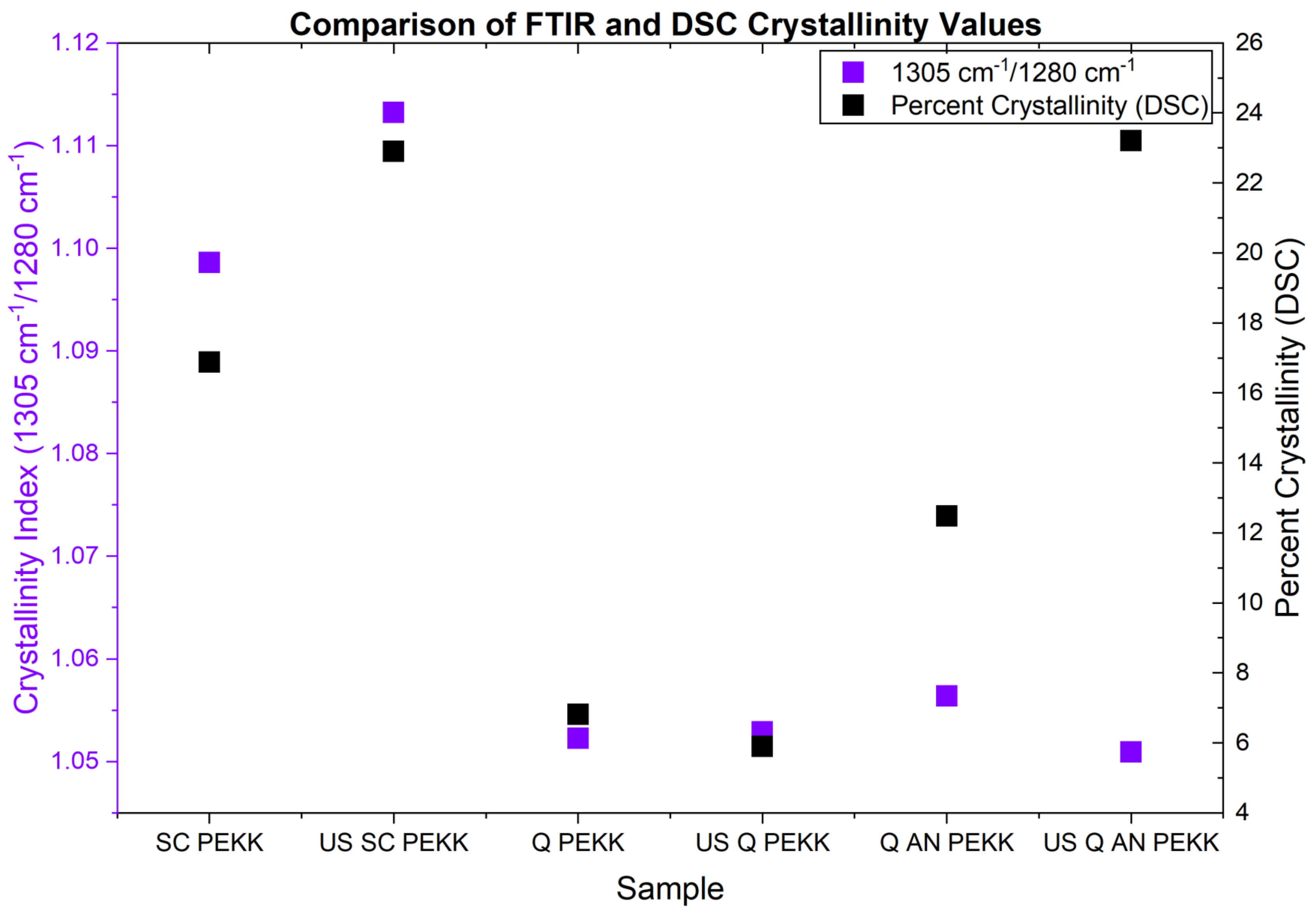
Crystallinity in Ultrasonically Welded Samples



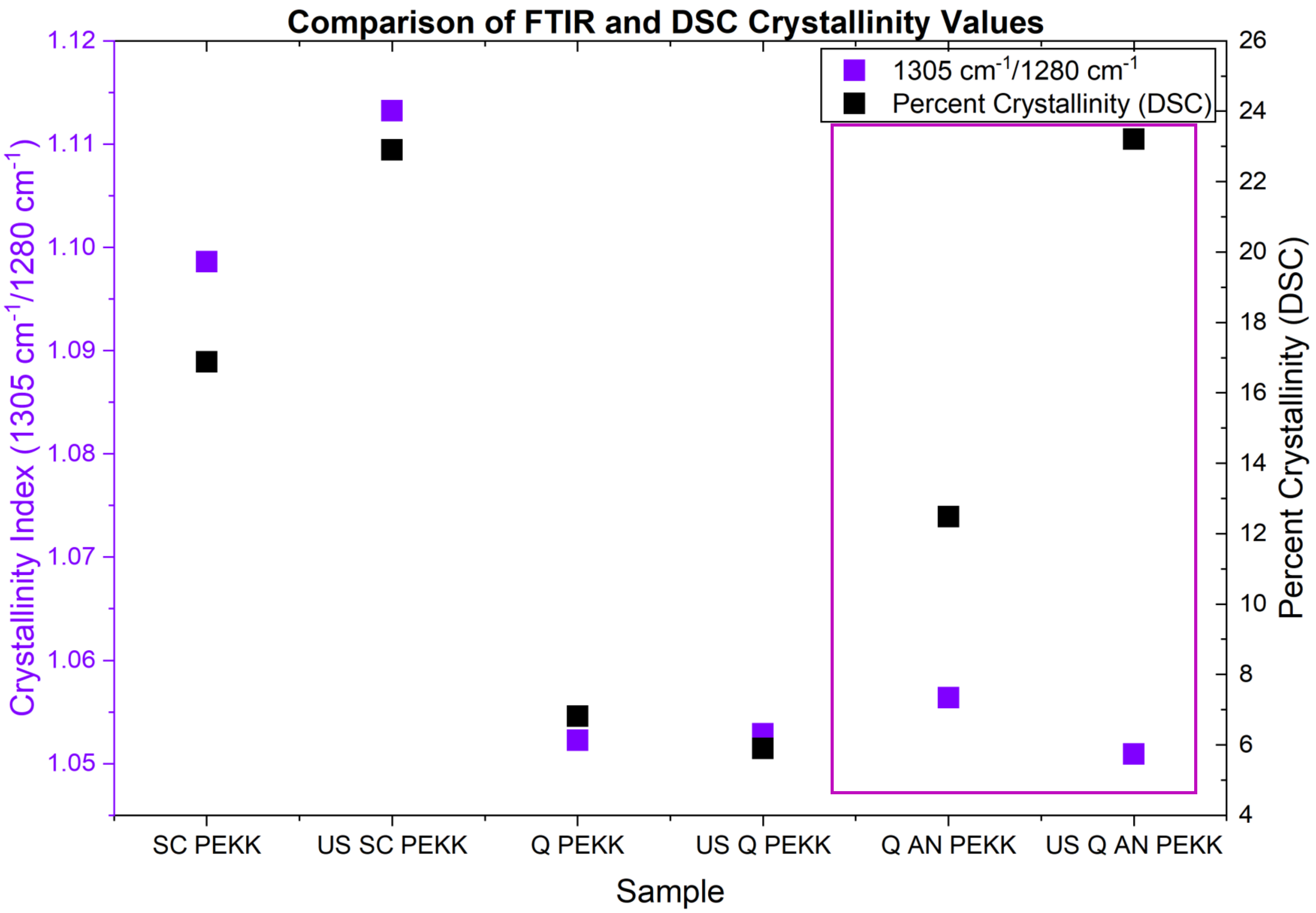




Crystallinity in Ultrasonically Welded Samples

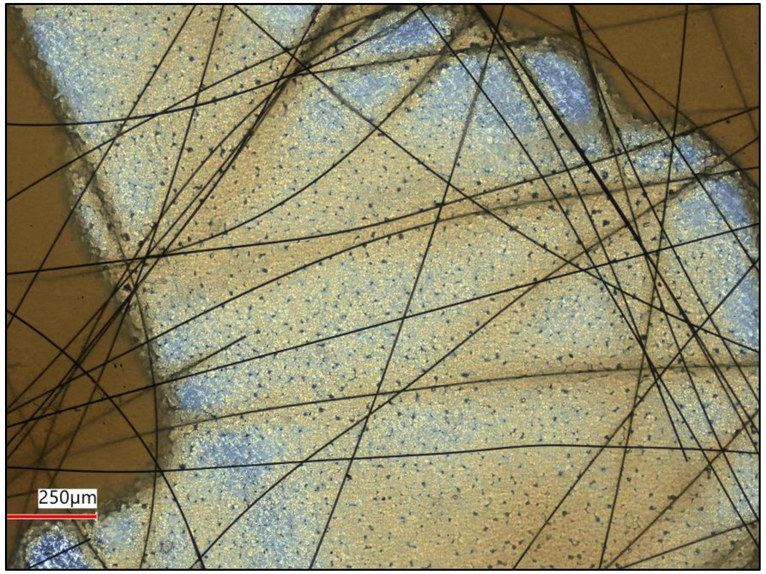


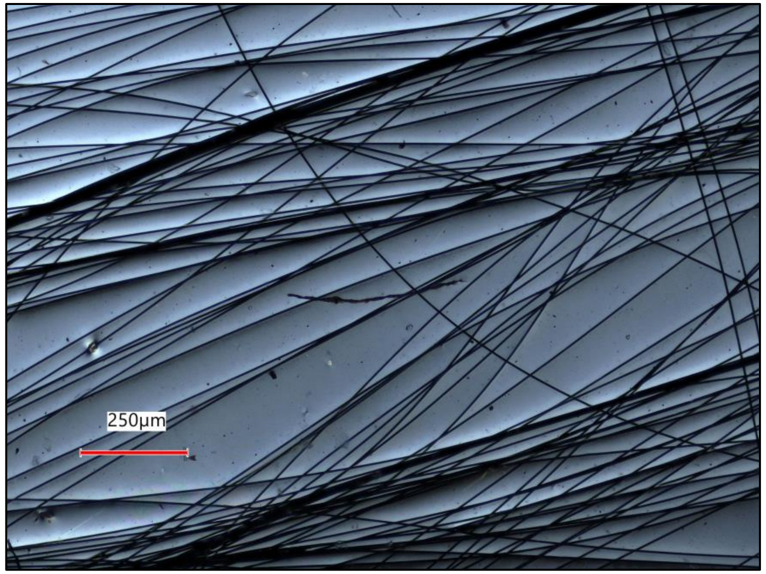
Crystallinity in Ultrasonically Welded Samples

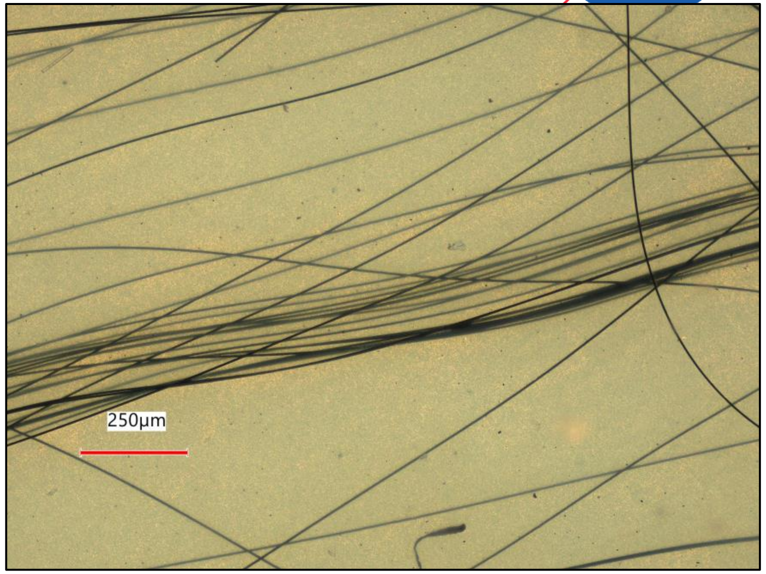


Crystallinity in Ultrasonically Welded Samples

Before welding

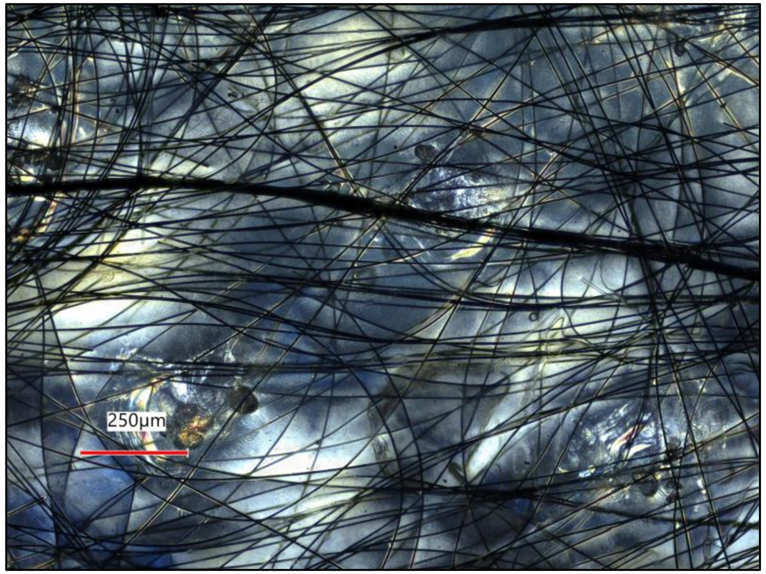


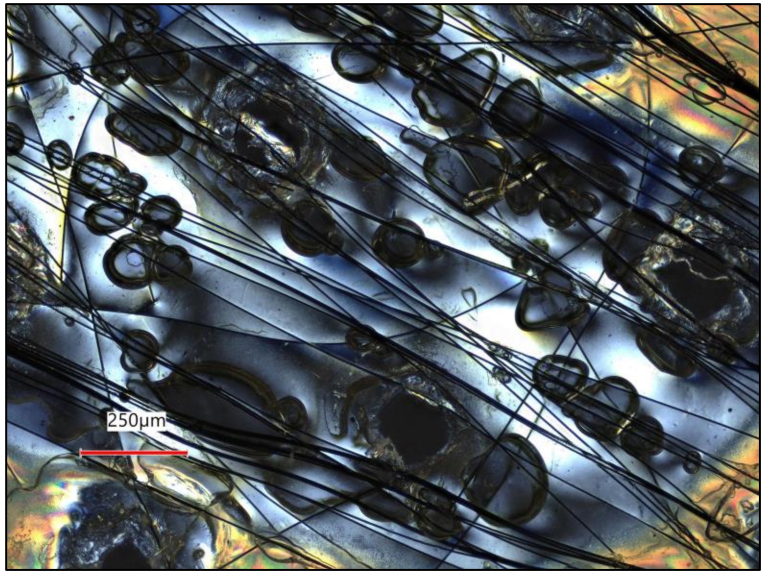




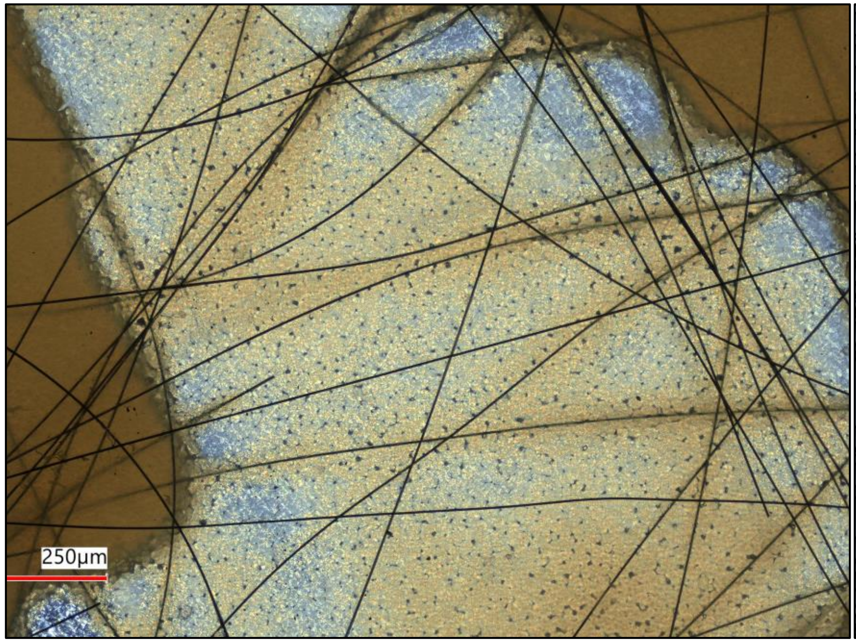
Slow Cooled PEKK | **Quenched PEKK** | **Annealed PEKK**

After welding



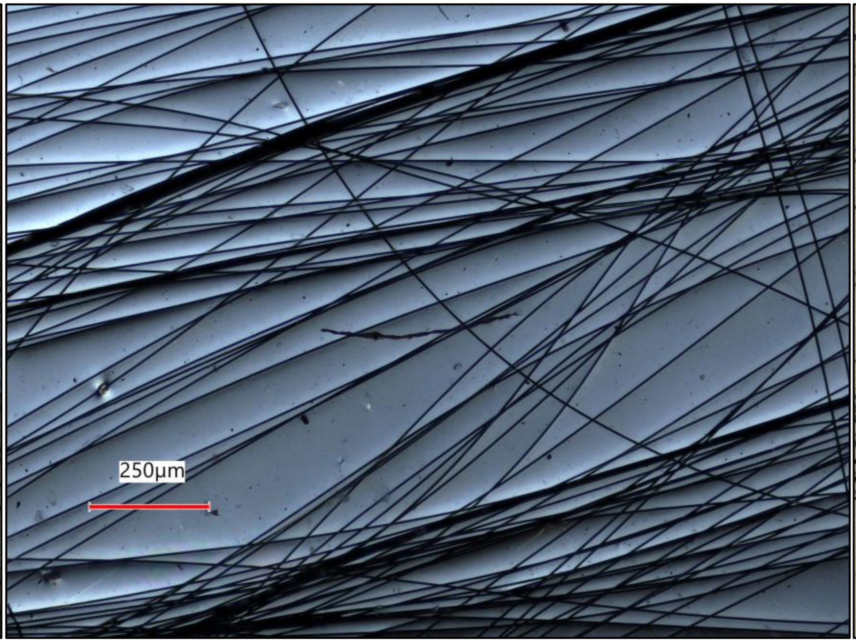


Differentiating Crystallites

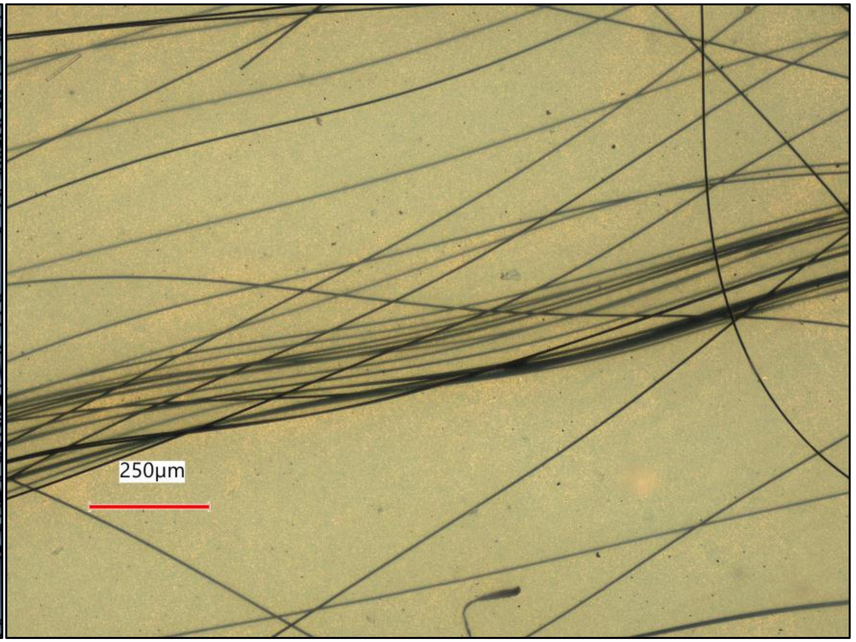


Slow Cooled PEKK

Spherulite dominated



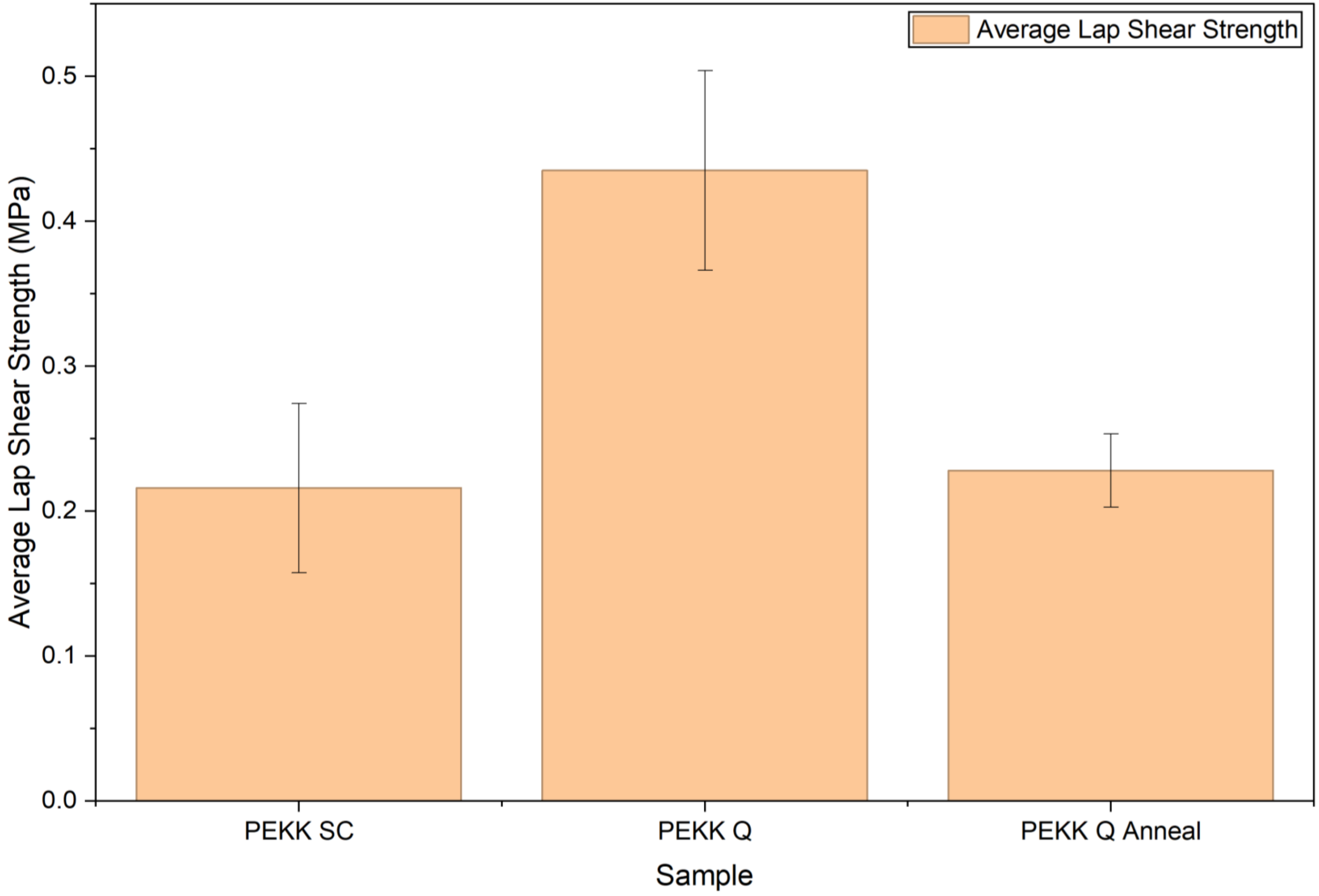
Quenched PEKK



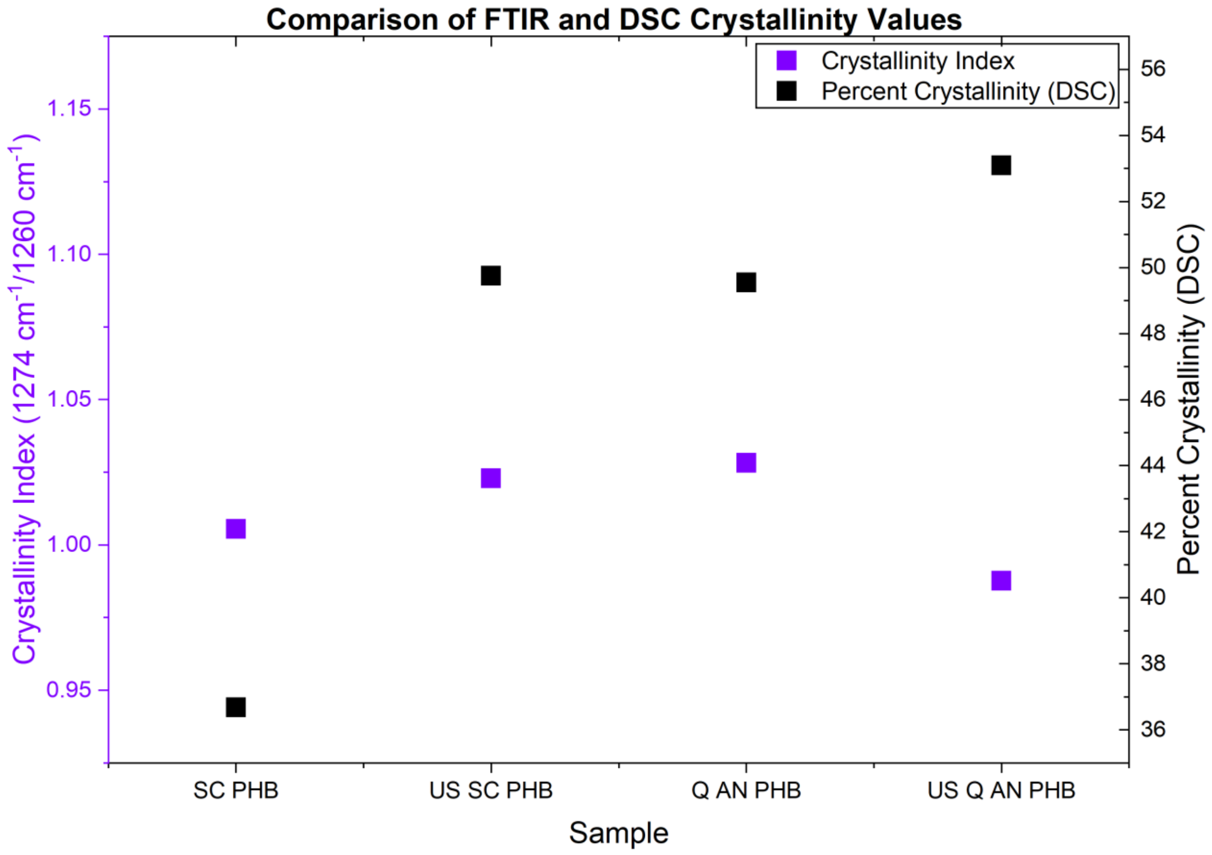
Quenched PEKK
+ Annealed 1h 225^{o}C

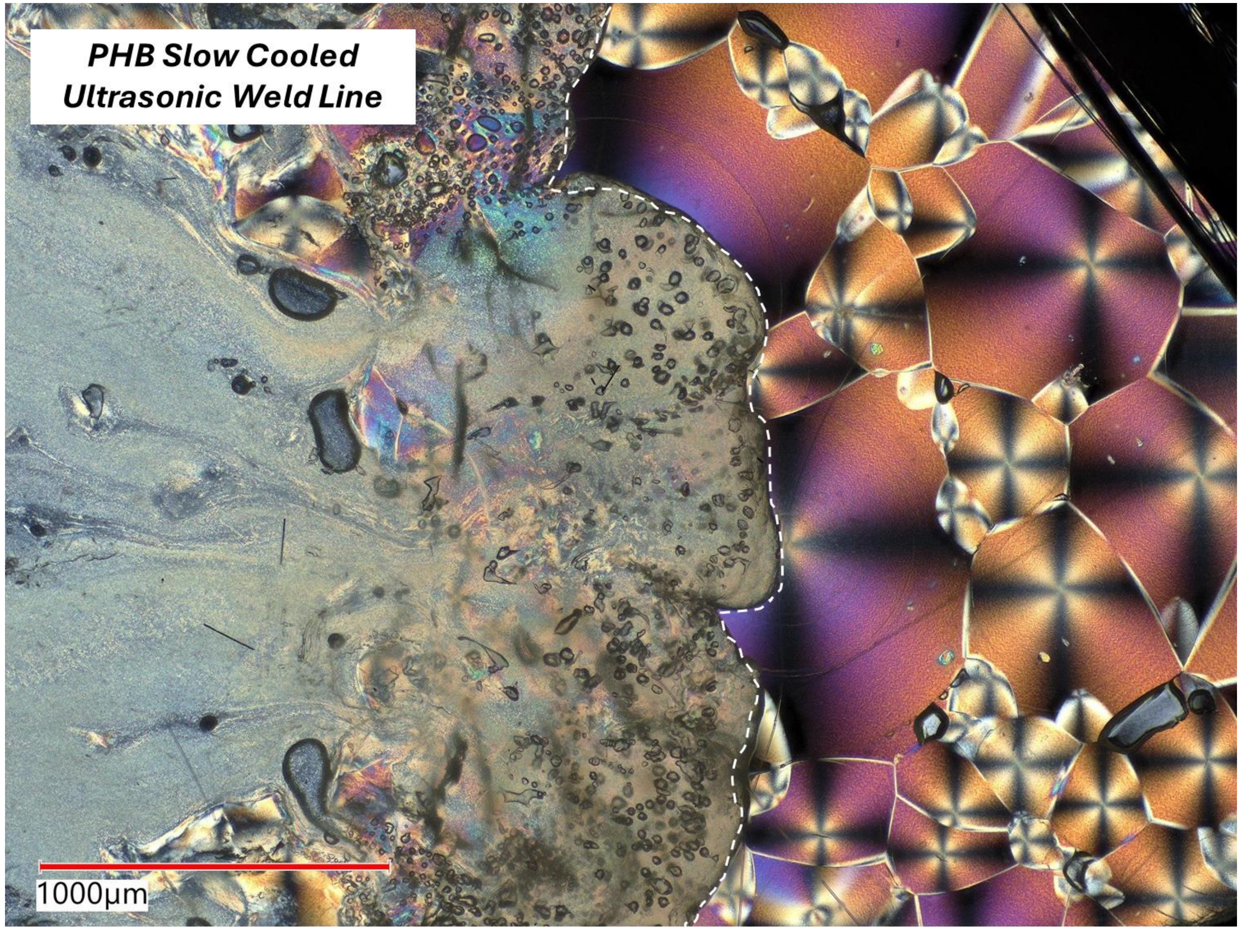
Transcrystalline dominated

Crystallinity Effects on Ultrasonically Welded Samples

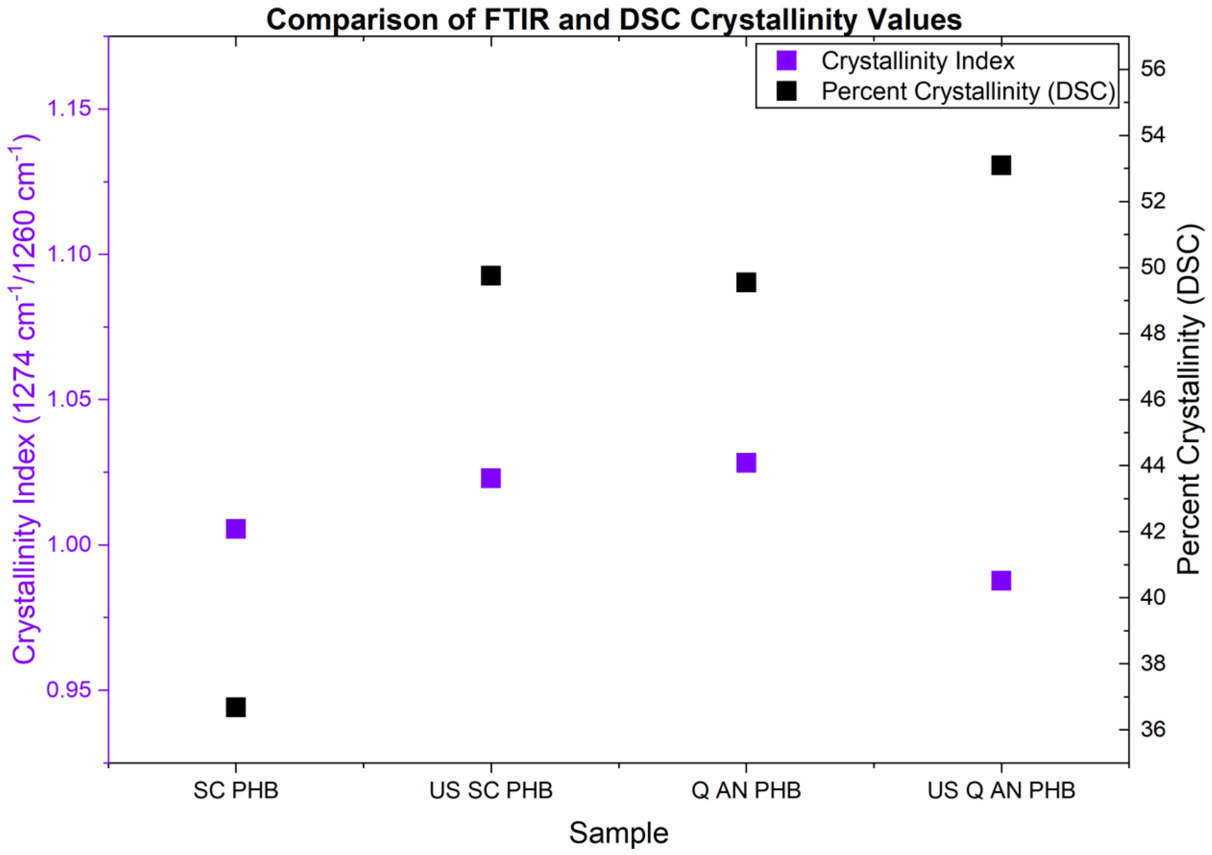


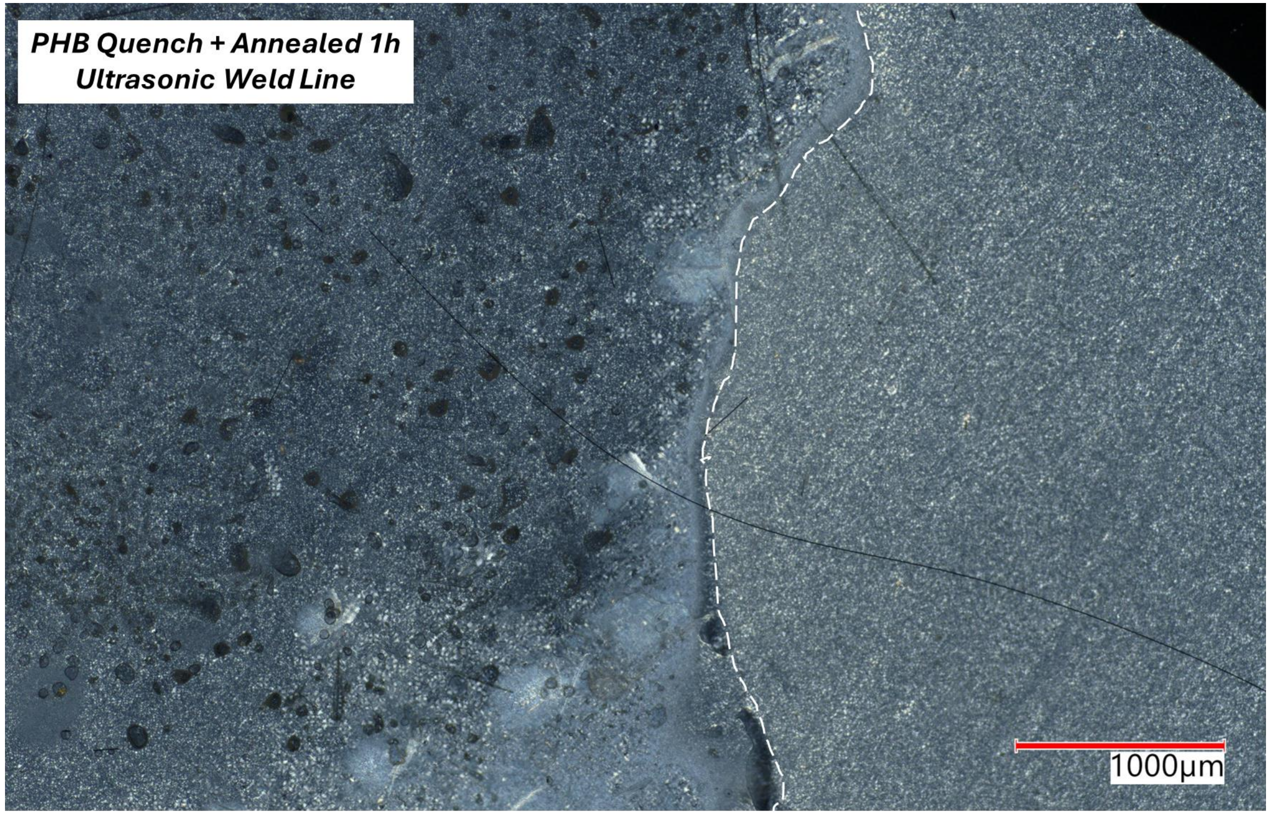
Crystallinity Effects on Ultrasonically Welded Samples



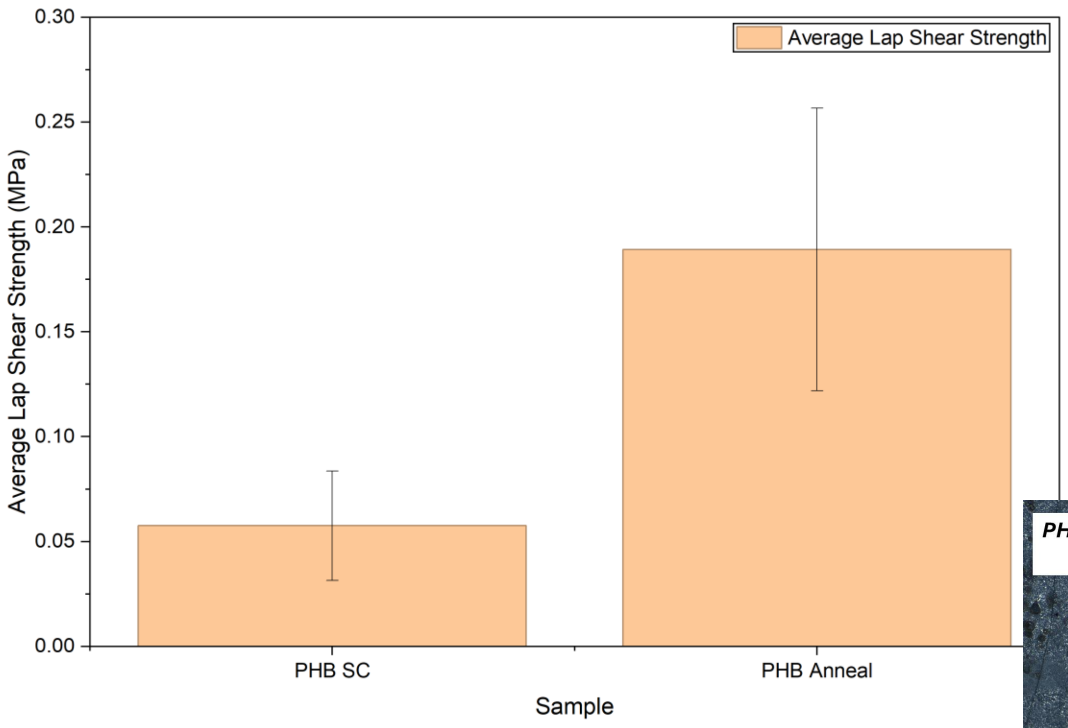


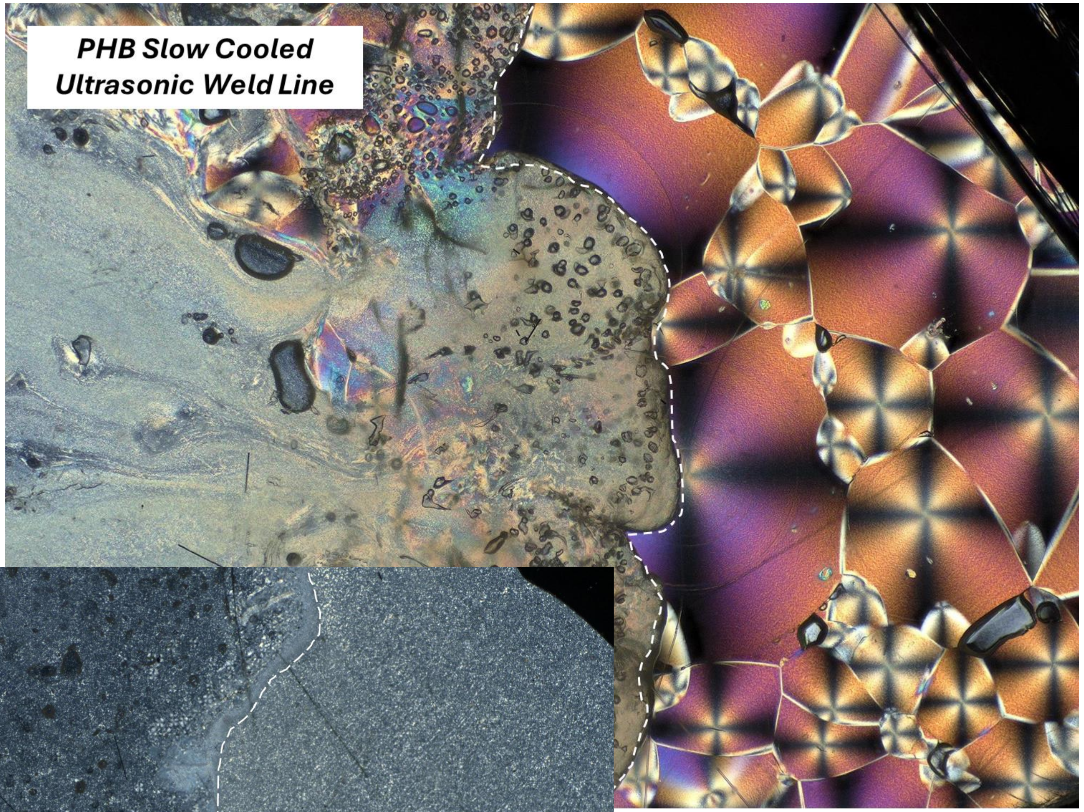
Crystallinity Effects on Ultrasonically Welded Samples

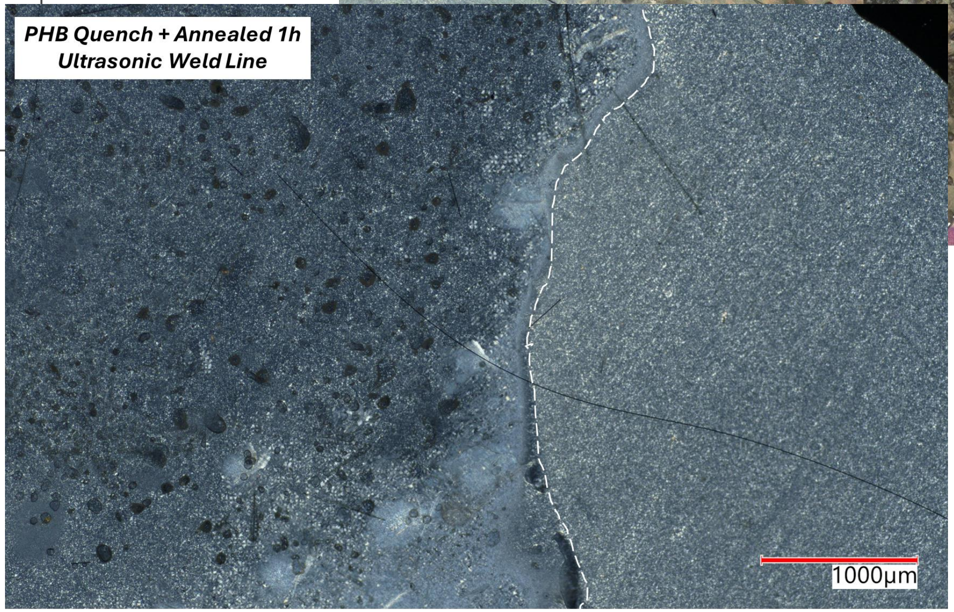




Crystallinity Effects on Ultrasonically Welded Samples







Key Takeaways

- Crystallinity matters, but is still difficult to analyze
- FTIR is viable non-destructive method for analyzing crystallinity
- Bulk crystallinity measurements don’t always capture the whole story, and it matters

Key Takeaways & Next Steps

- Crystallinity matters, but is still difficult to analyze
- FTIR is viable non-destructive method for analyzing crystallinity
- Bulk crystallinity measurements don't always capture the whole story, and it matters
- **Obtain WAXS and SAXS and compare to other methods**
- **Conduct broader study on the effects of crystallinity on strength and performance for in-situ manufacturing & repair**
- **Use fundamental data to guide modeling efforts for predicting material performance**

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Questions?