

Evaluating Crystallinity in Thermoplastic Composites

Allison Christy, Sandi Miller

NASA Glenn Research Center, Cleveland, OH



Presentation overview

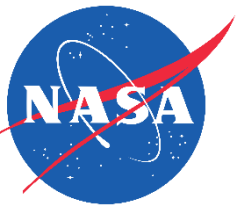
Benefits of Thermoplastics

Overview of Crystallinity in Thermoplastics

Characterizing Crystallinity

Crystallinity in Ultrasonically Welded Composites

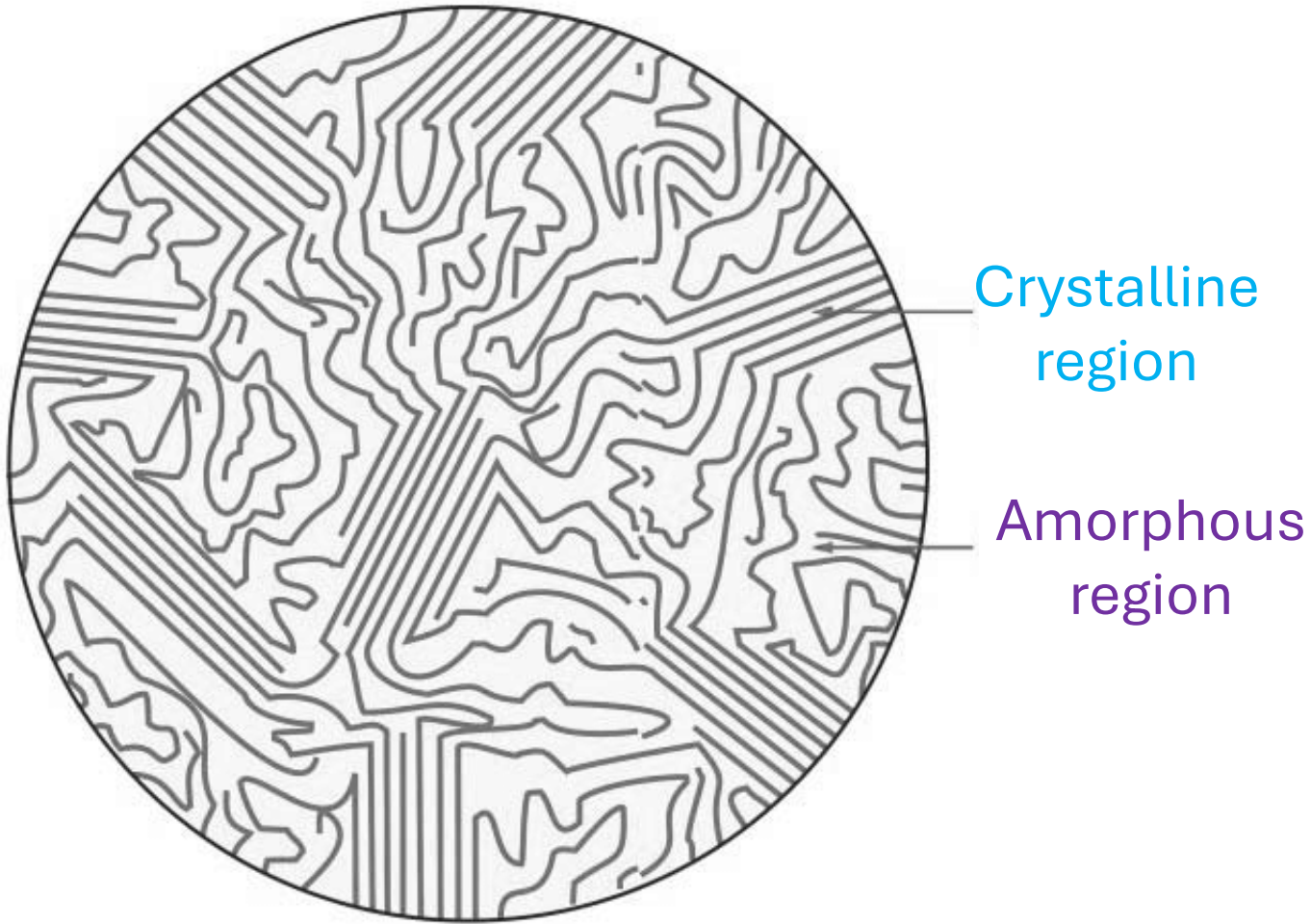
Next Steps



Benefits of Thermoplastics in Advanced Manufacturing

- Improved processing efficiency
- Enhanced mechanical properties
- Improved impact resistance, toughness, and fatigue
- Potential for in-situ repair and joining
- Recyclability
- Low temperature manufacturing

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

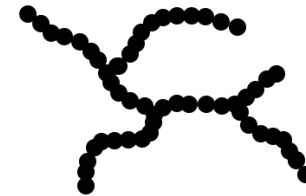
Factors Affecting Crystallinity

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

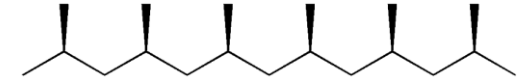
● = monomer unit



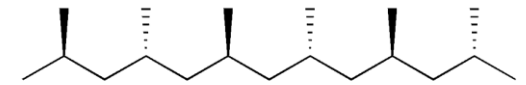
linear polymer chain



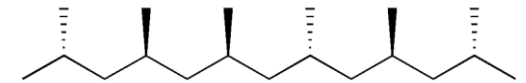
branched polymer chain



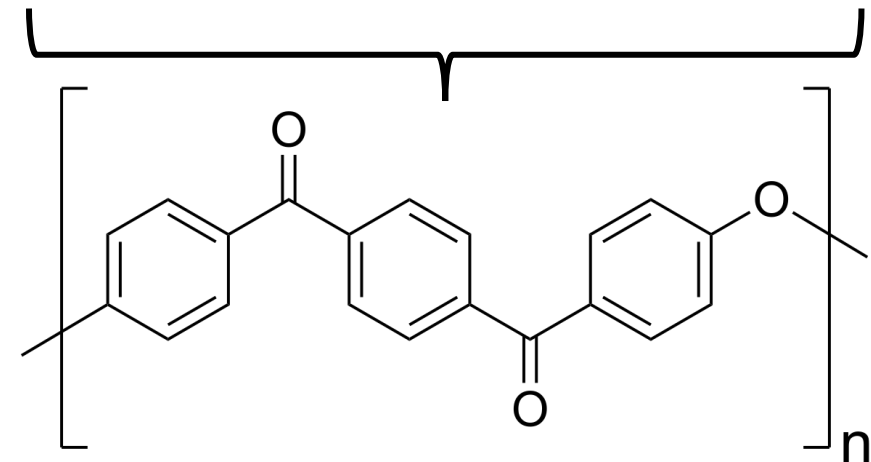
isotactic



syndiotactic



atactic



Poly(etherketoneketone) (PEKK)

Factors Affecting Crystallinity

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

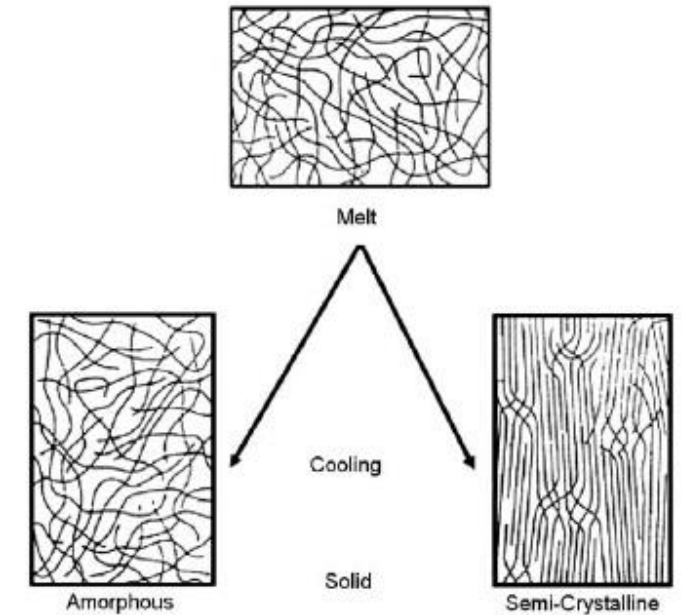
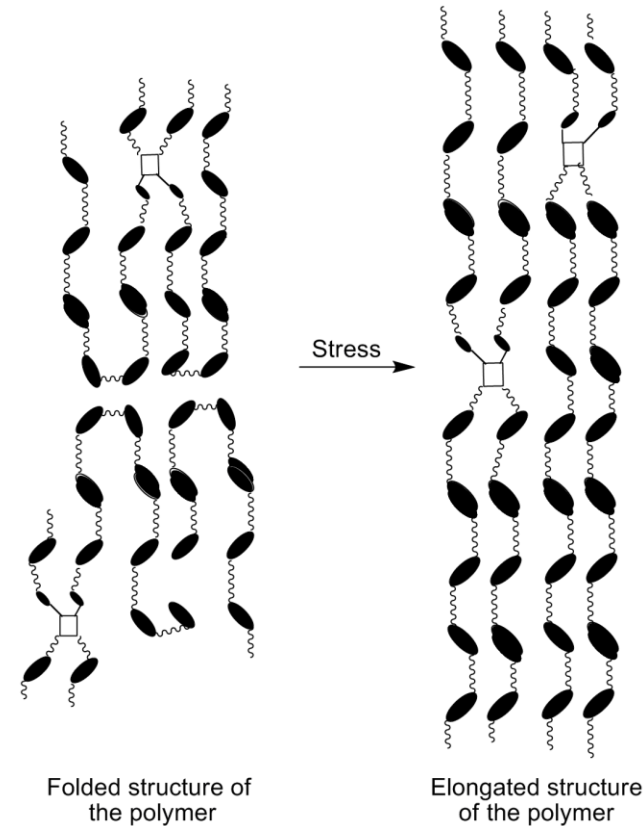
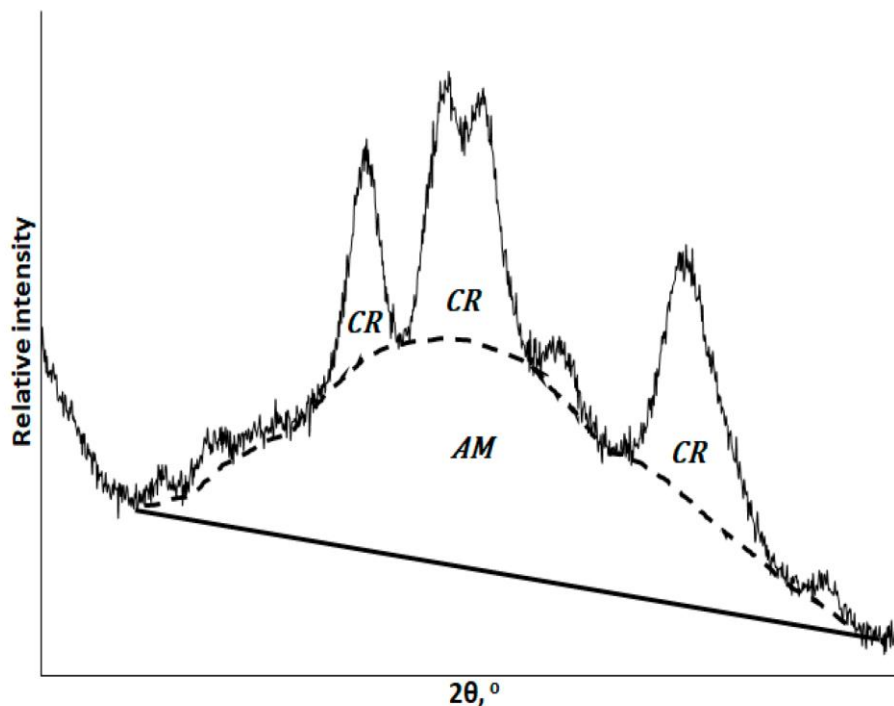
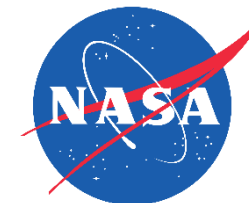


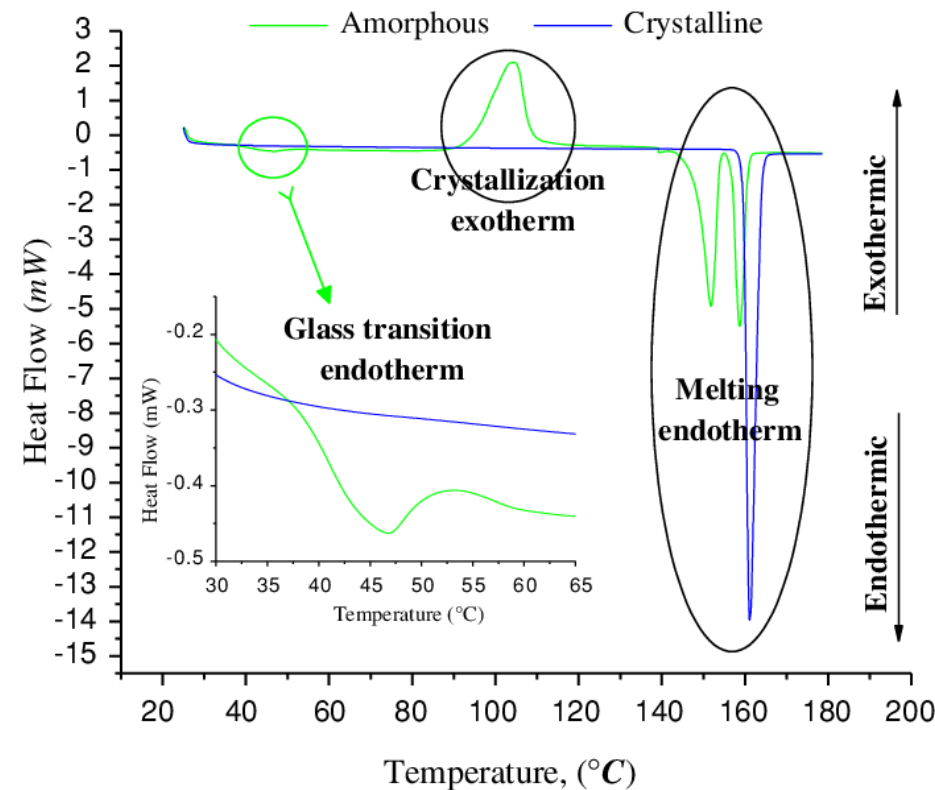
Figure 3.4 Cooling behaviour of materials

Will vary with increased manufacturing speeds and repair interphases

Conventional Methods to Evaluate Polymer Crystallinity



***X-Ray Diffraction
(XRD)***



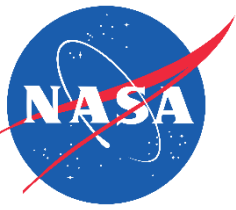
***Differential Scanning
Calorimetry (DSC)***

Crystallinity is Complex & Composites are Difficult to Evaluate

Varied
cooling
rates and
stress
exposure,



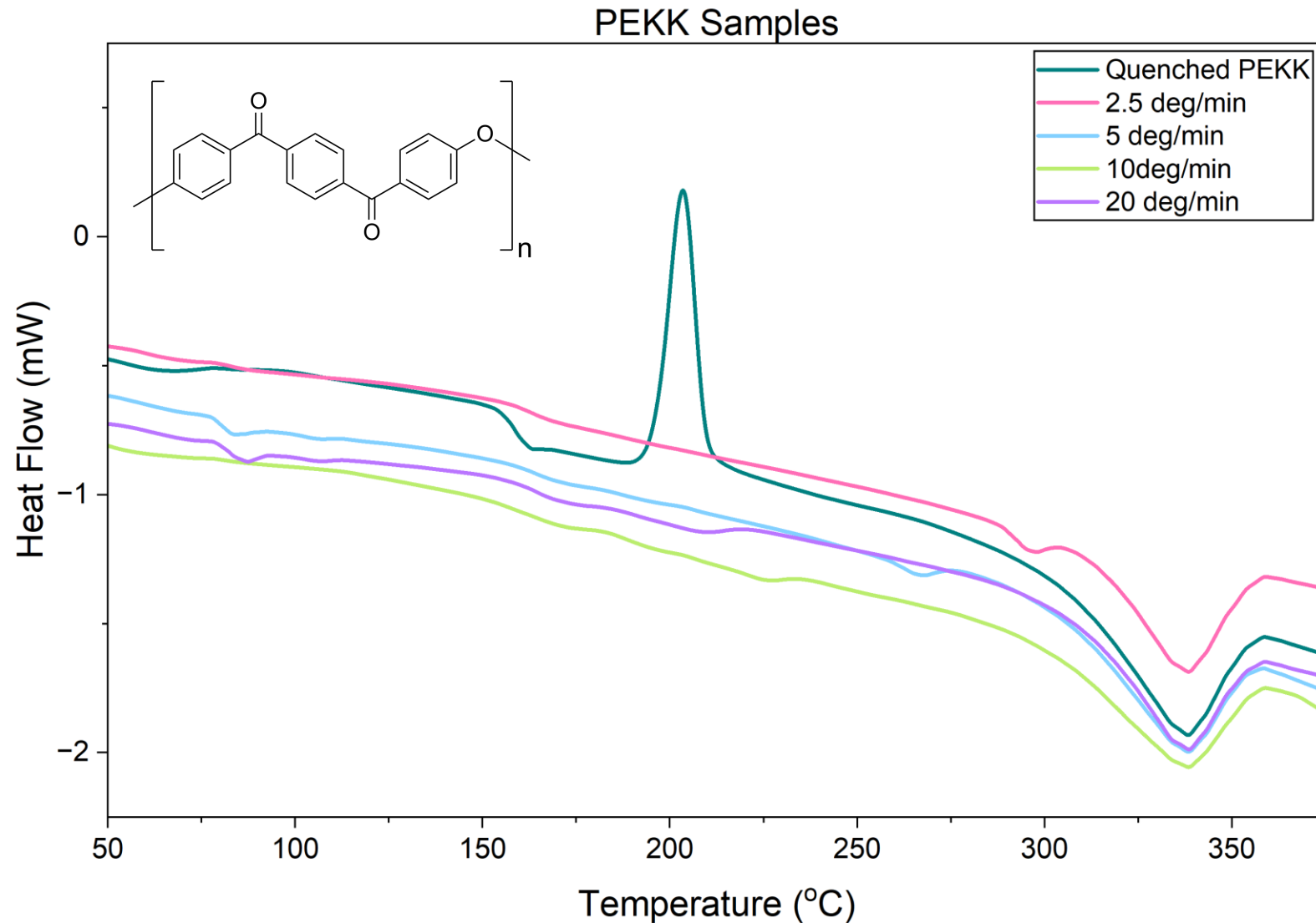
Varied
crystallinity
profile, varied
composite
properties



Experimental Approach – Carbon Fiber/PEKK Composites

1. Manufacture samples of various crystallinity profiles
2. Use DSC as benchmark for bulk crystallinity
3. Compare DSC values to FTIR data and cross-polarized microscopy as a feasibility study
4. Use methods to analyze

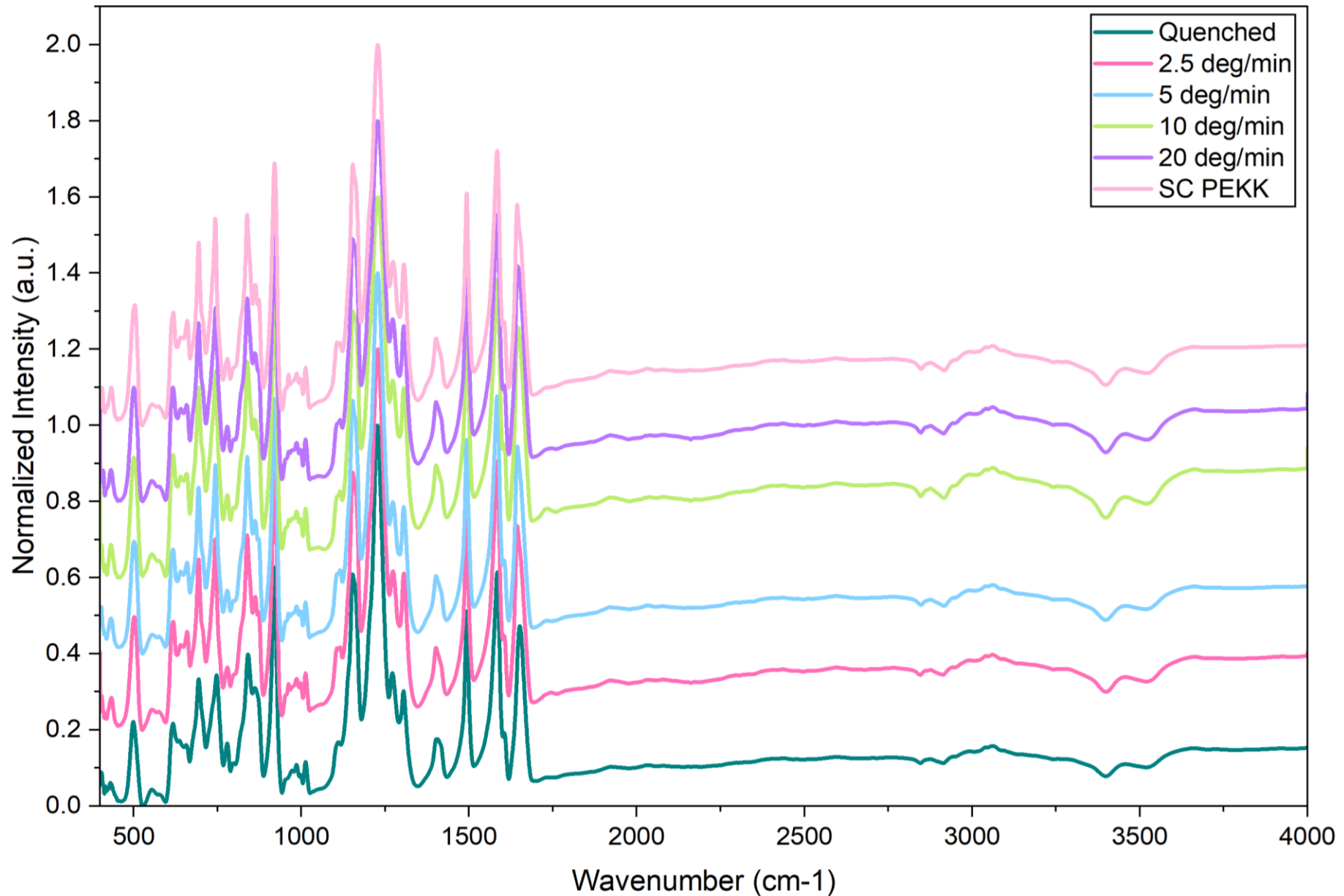
Differential Scanning Calorimetry of PEKK Resin



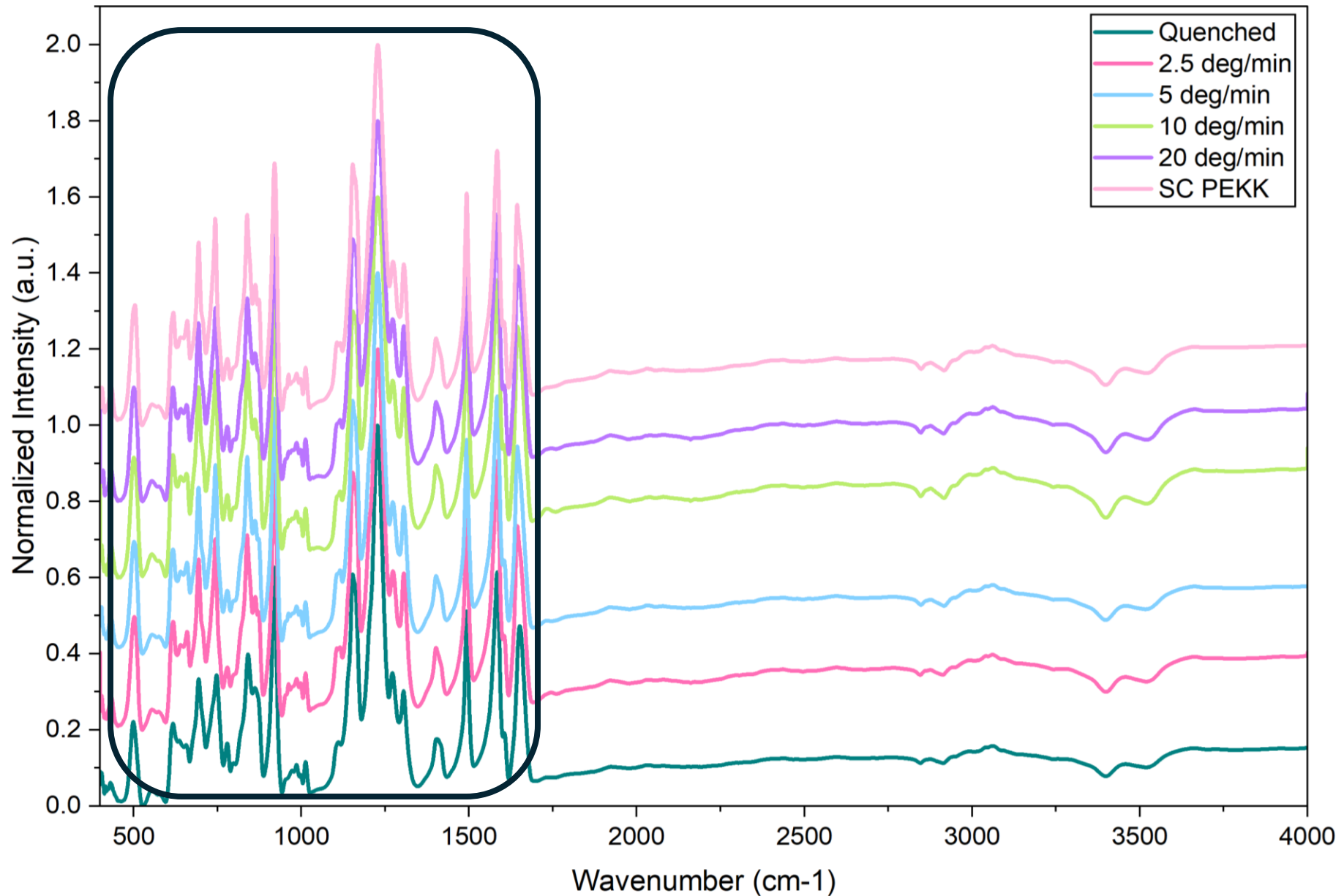
$$X_c = \frac{\Delta H_m}{\Delta H_{100\%}} \times 100$$

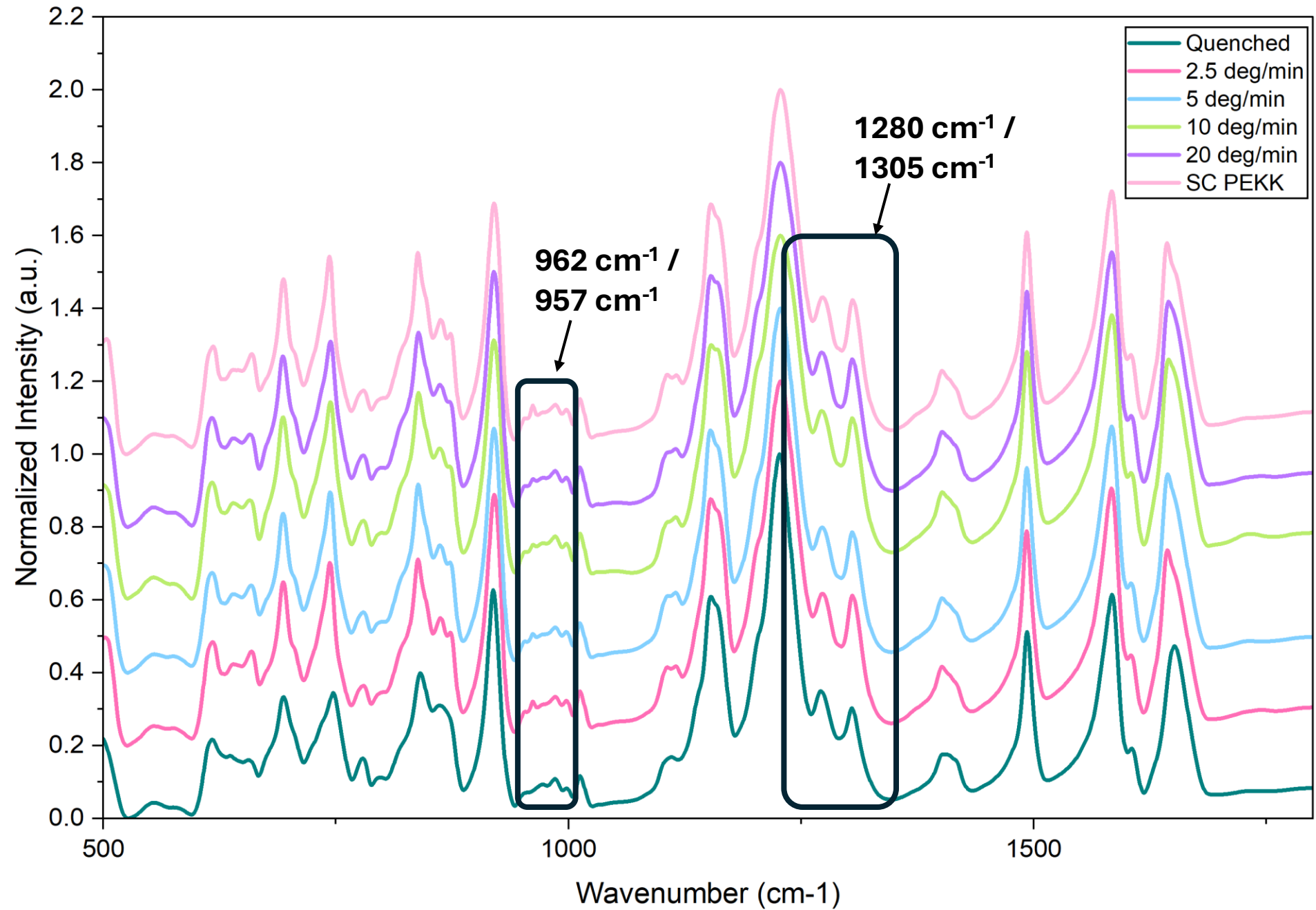
Sample	X_c
Quenched	11.75
2.5 deg/min	22.28
5 deg/min	27.35
10 deg/min	28.32
20 deg/min	27.14

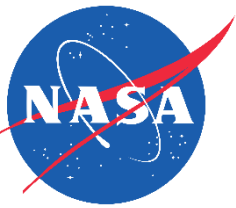
Fourier Transform Infrared (FTIR) Spectroscopy



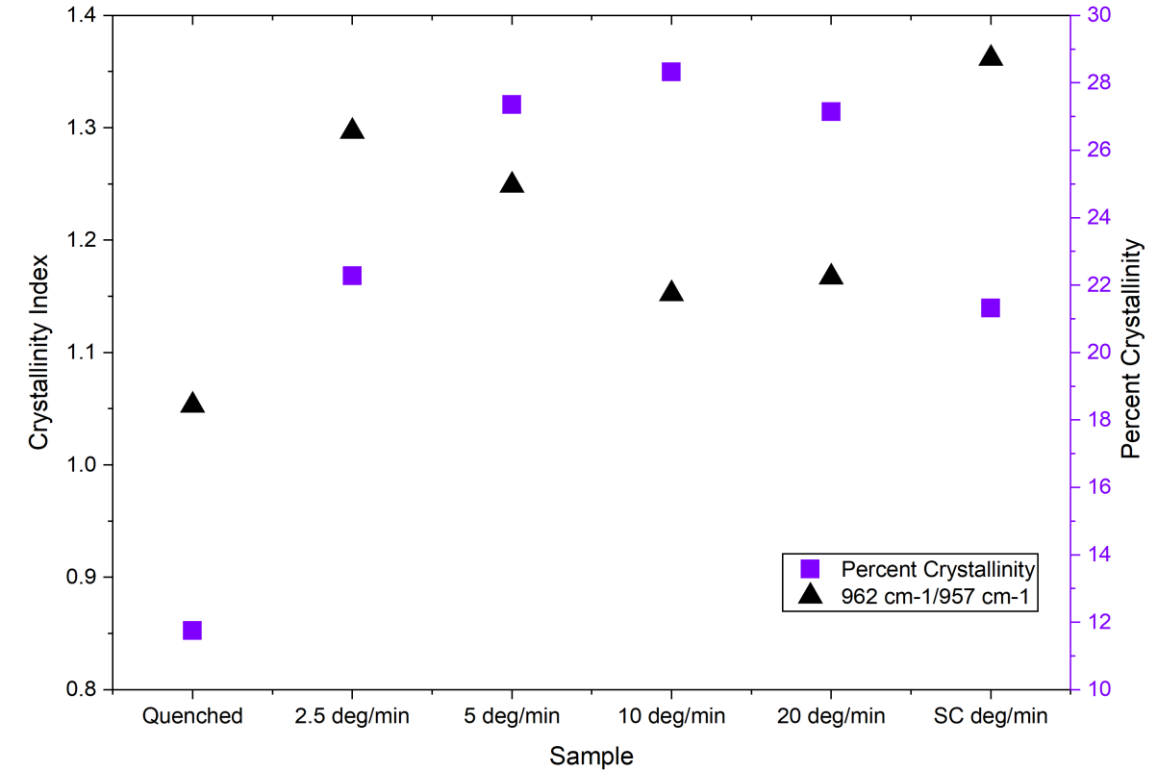
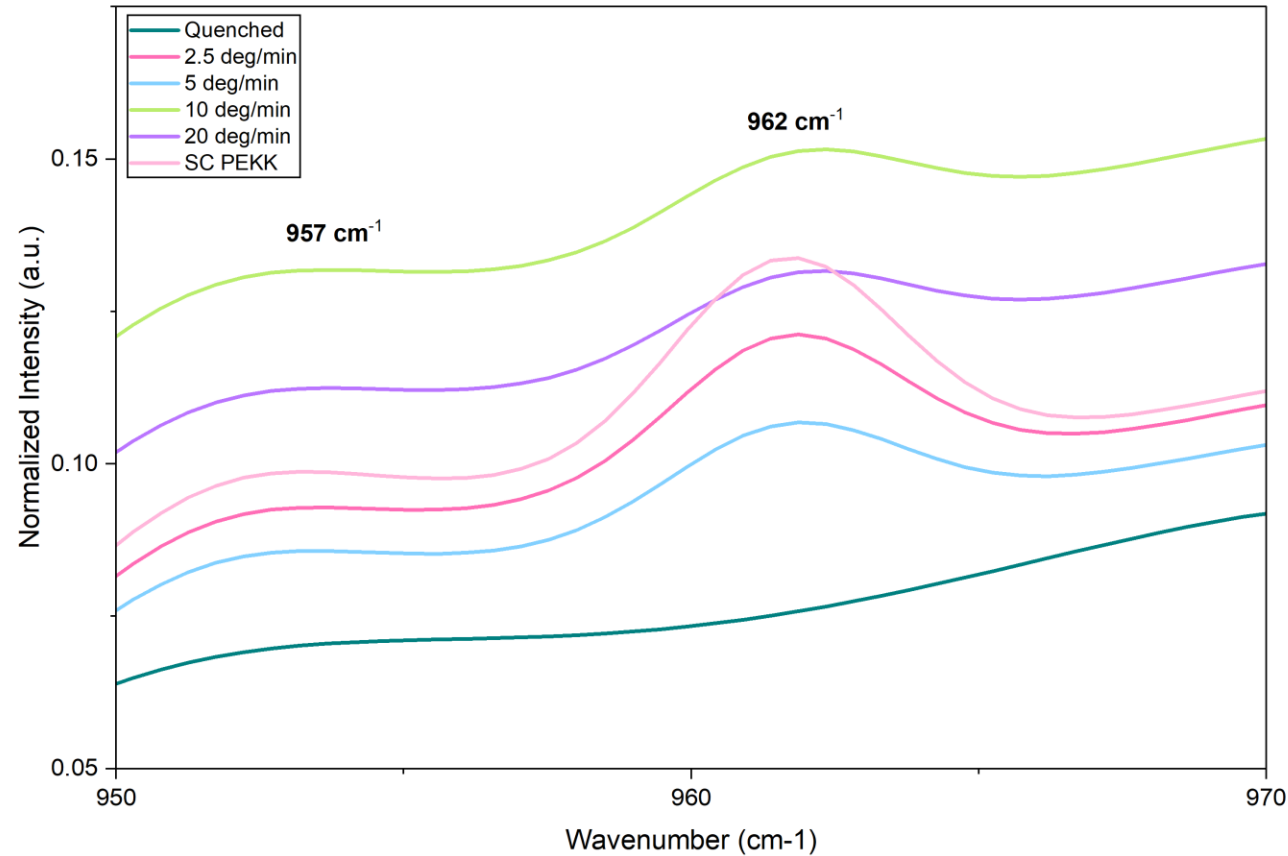
Fourier Transform Infrared (FTIR) Spectroscopy

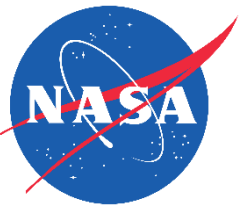




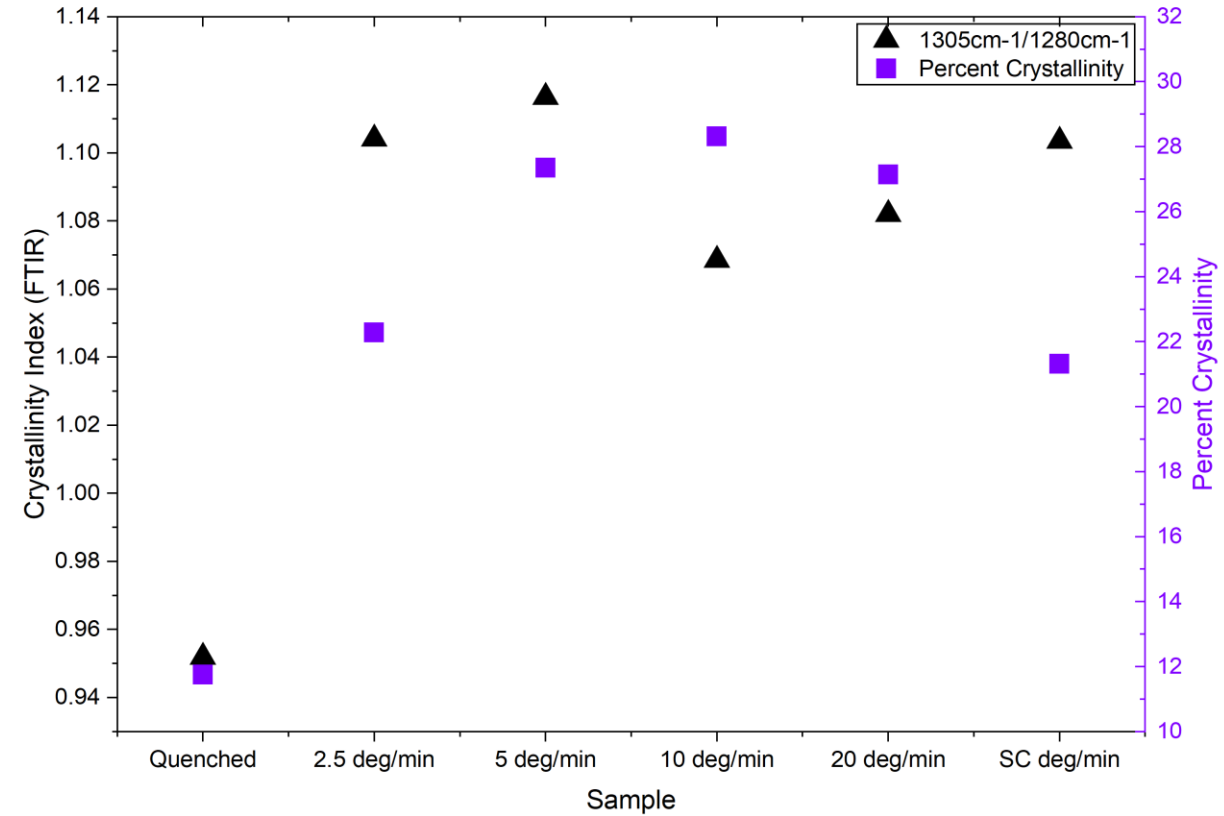
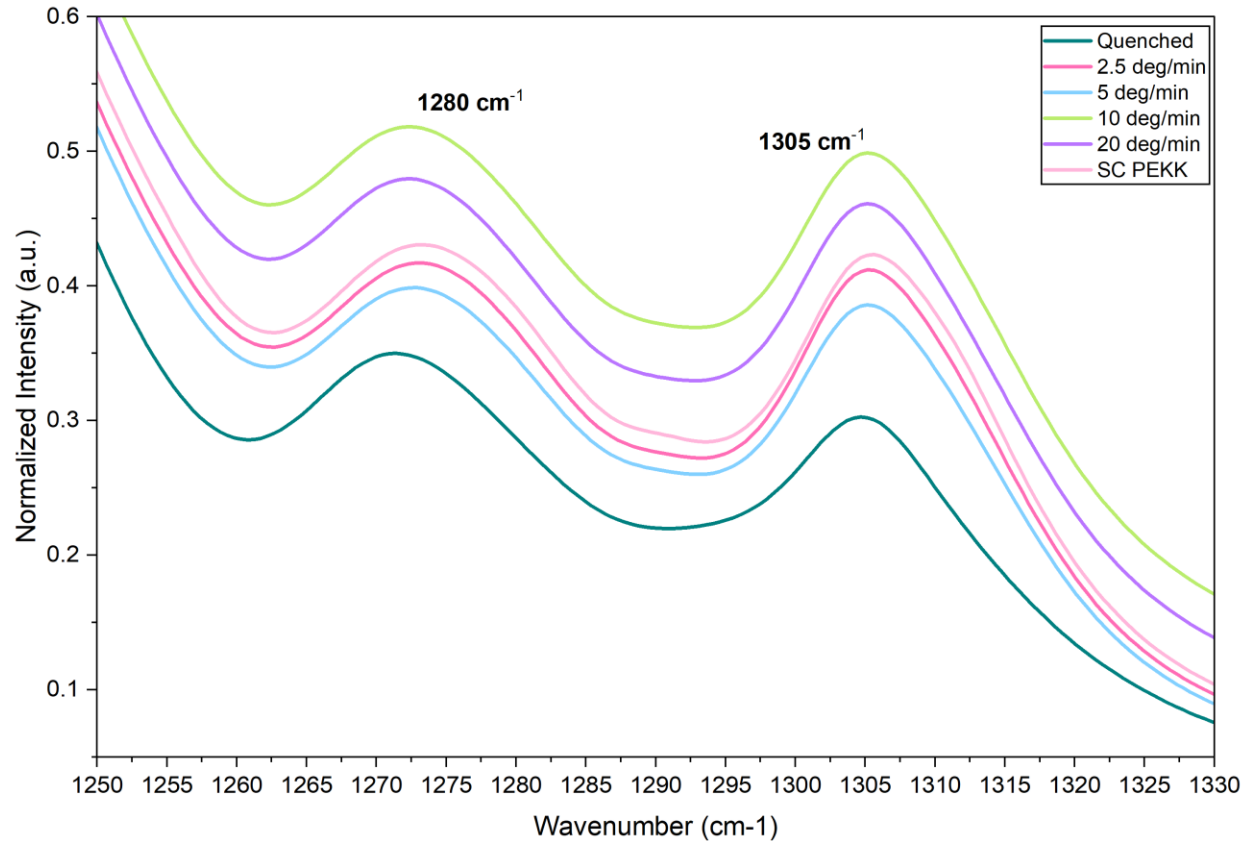


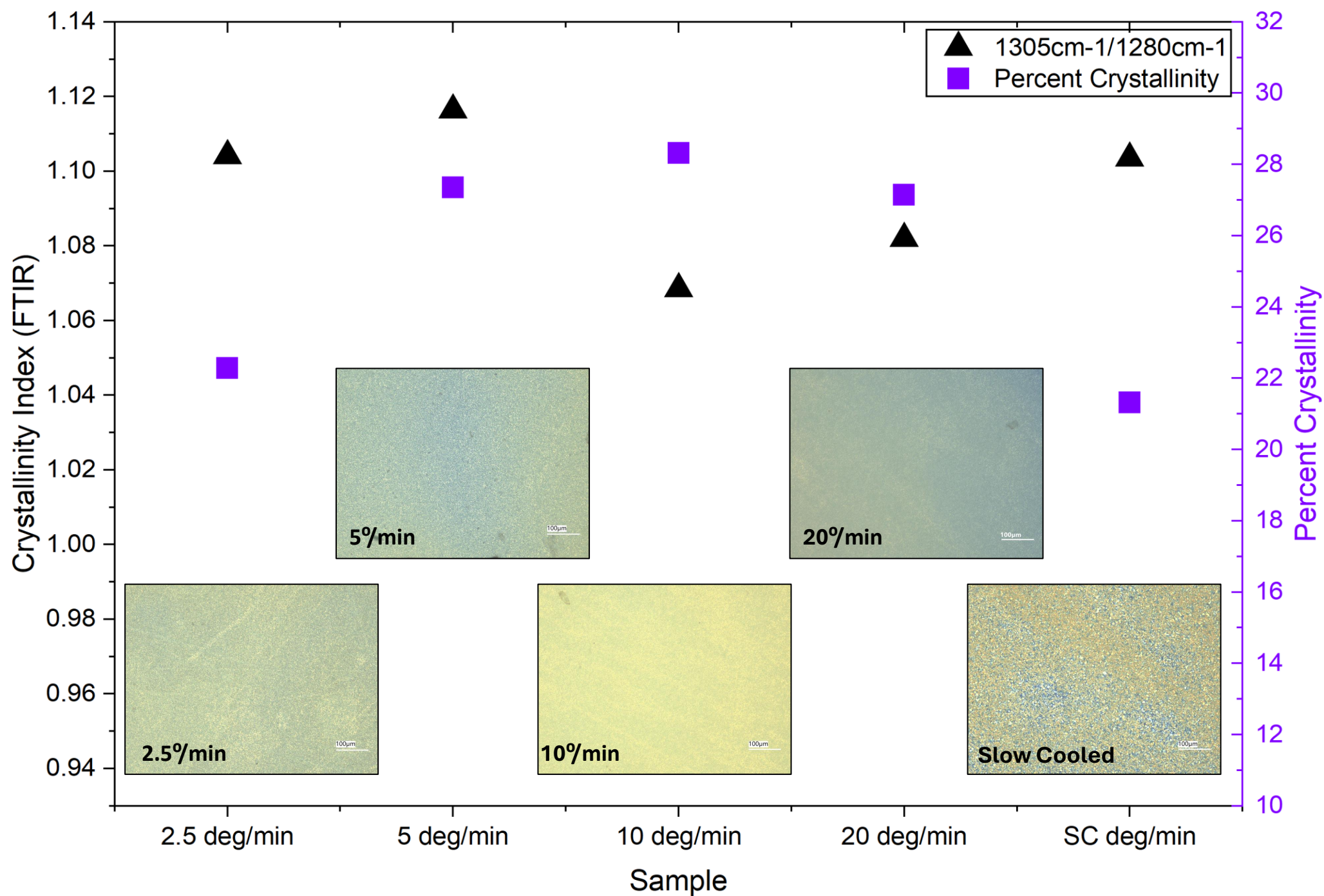
Fourier Transform Infrared (FTIR) Spectroscopy – Crystallinity Index



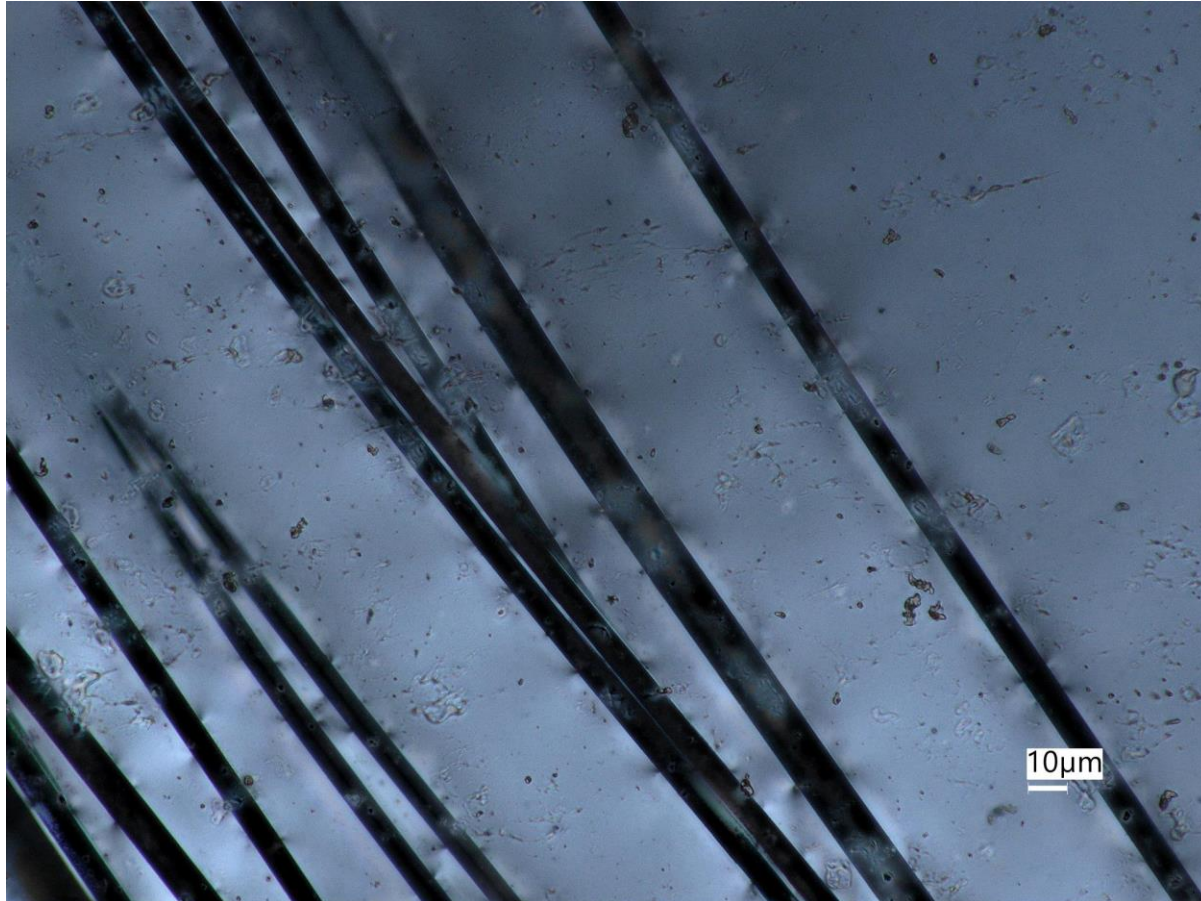


Fourier Transform Infrared (FTIR) Spectroscopy – Crystallinity Index

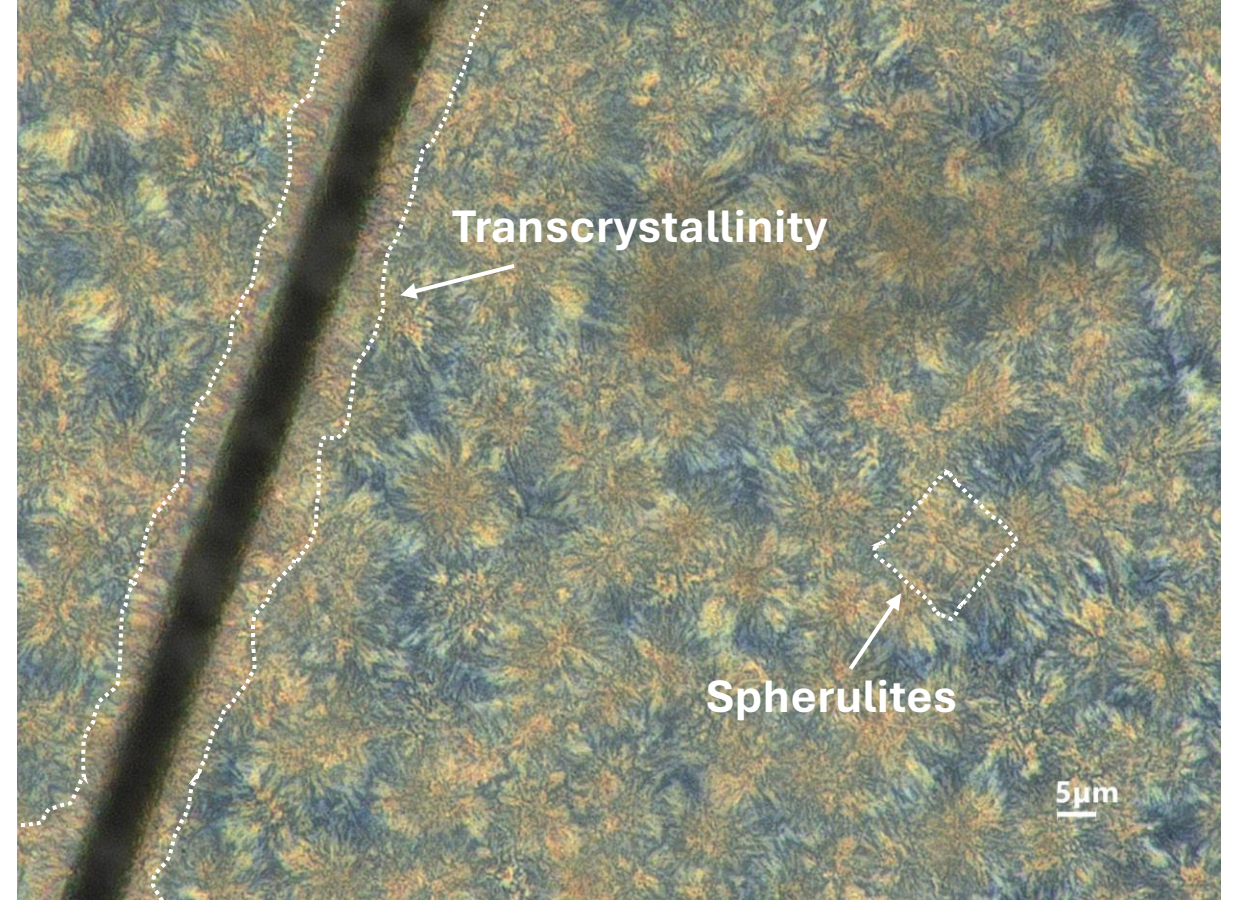




Differentiating Crystallites

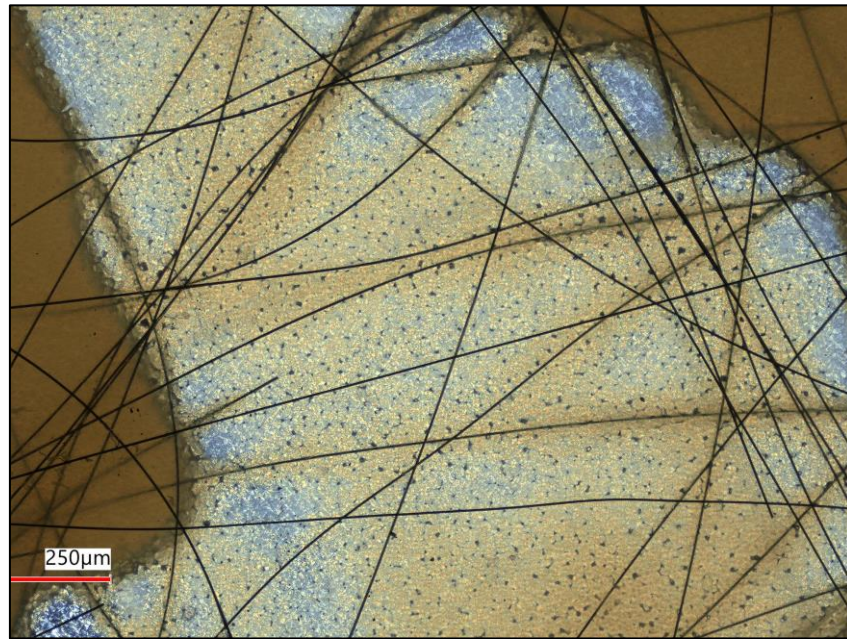
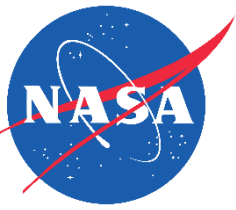


Quenched
 X_c : 11.75%



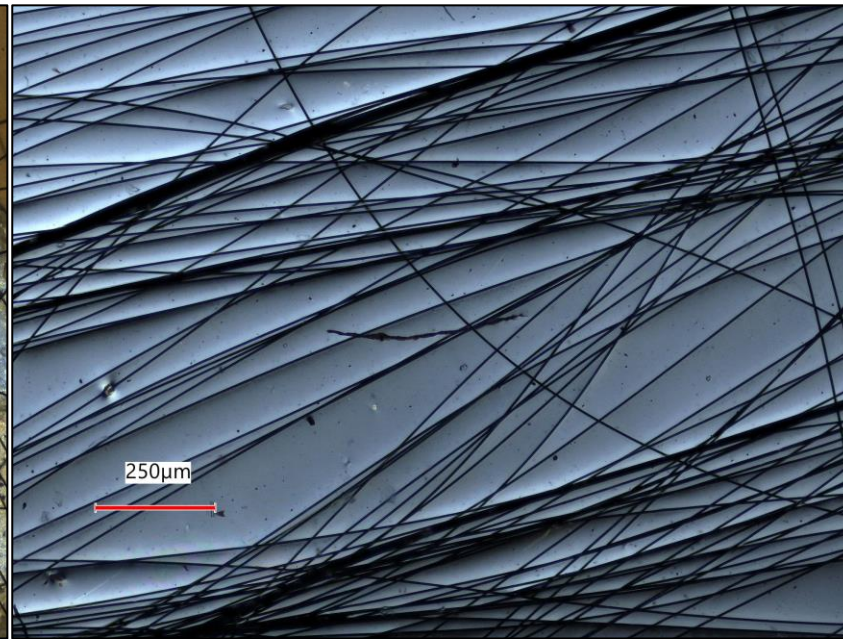
Slow Cooled
 X_c : 21.32%

Differentiating Crystallites

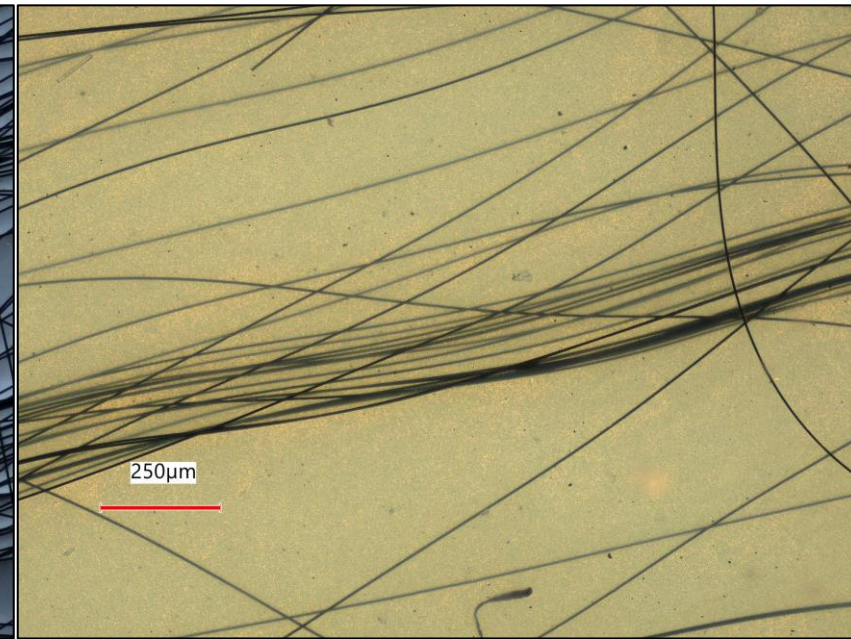


Slow Cooled PEKK

Spherulite dominated

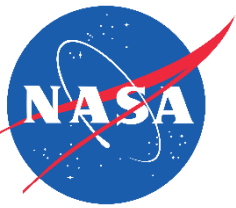


Quenched PEKK

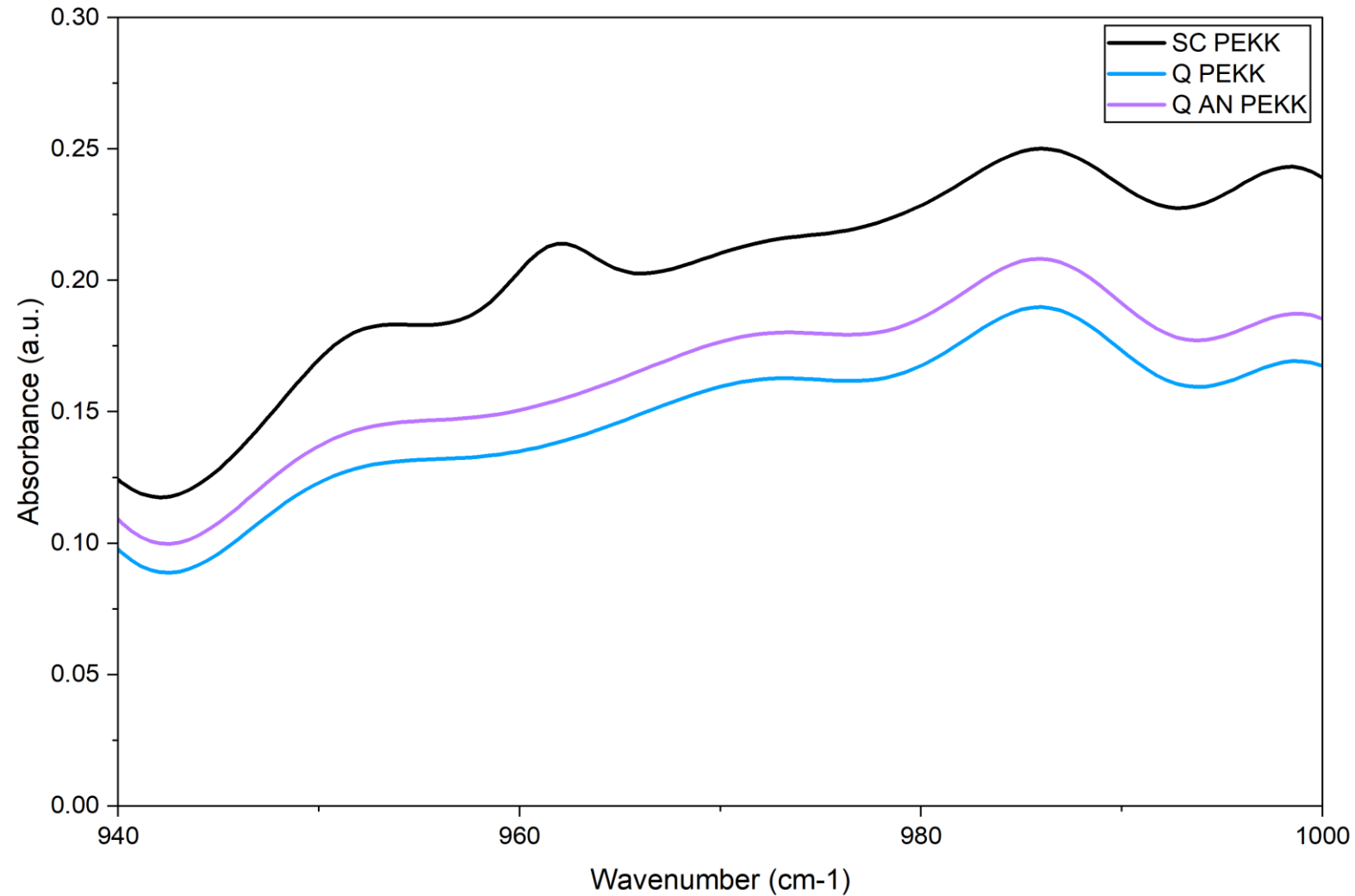


**Quenched PEKK
+ Annealed 1h 225°C**

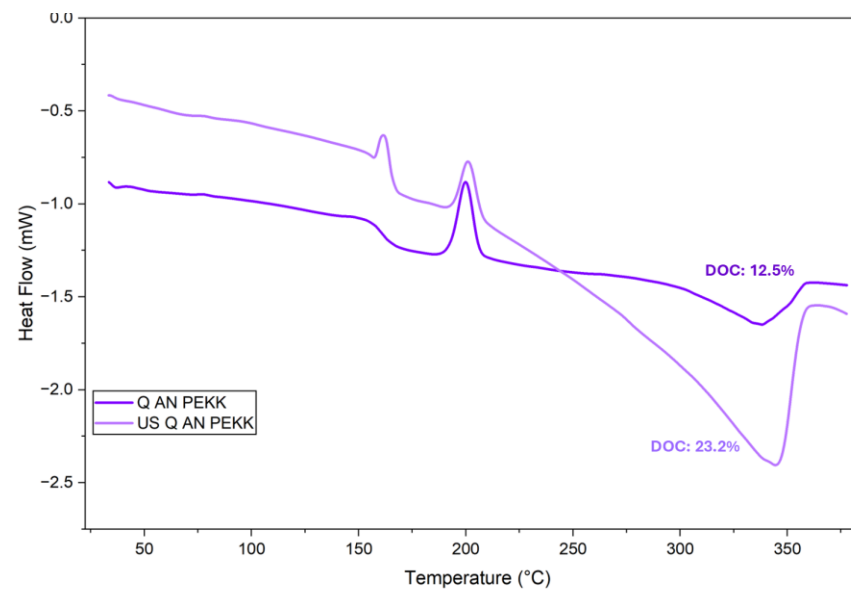
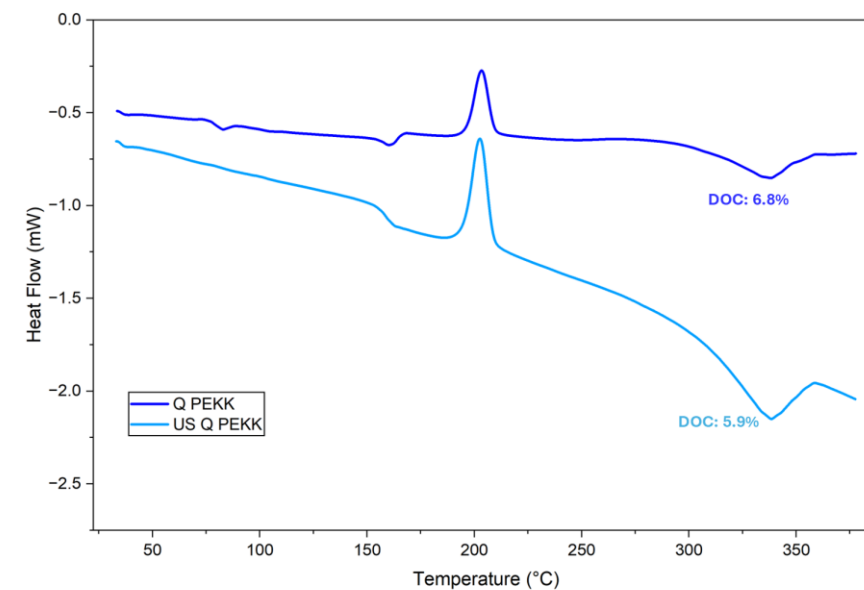
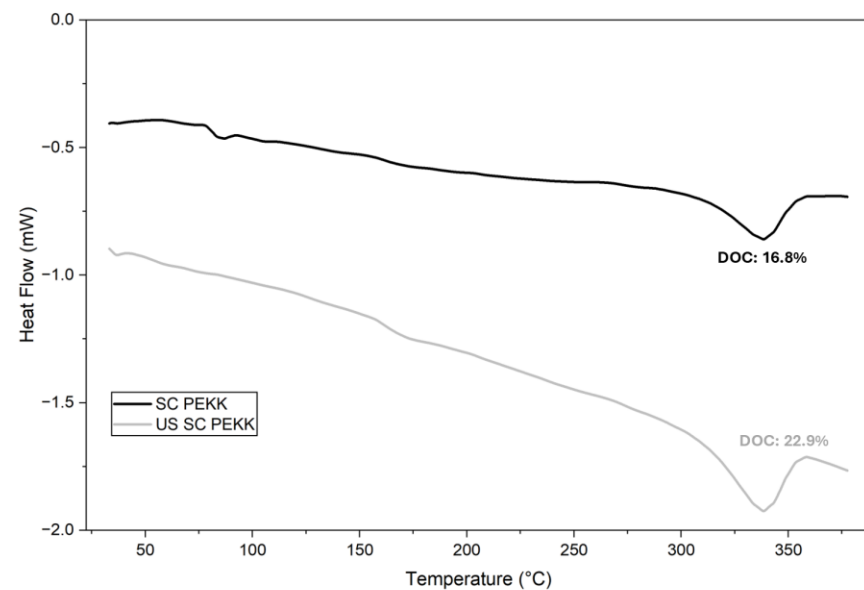
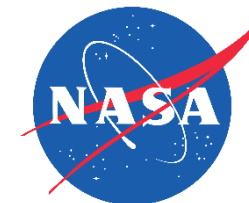
Transcrystalline dominated



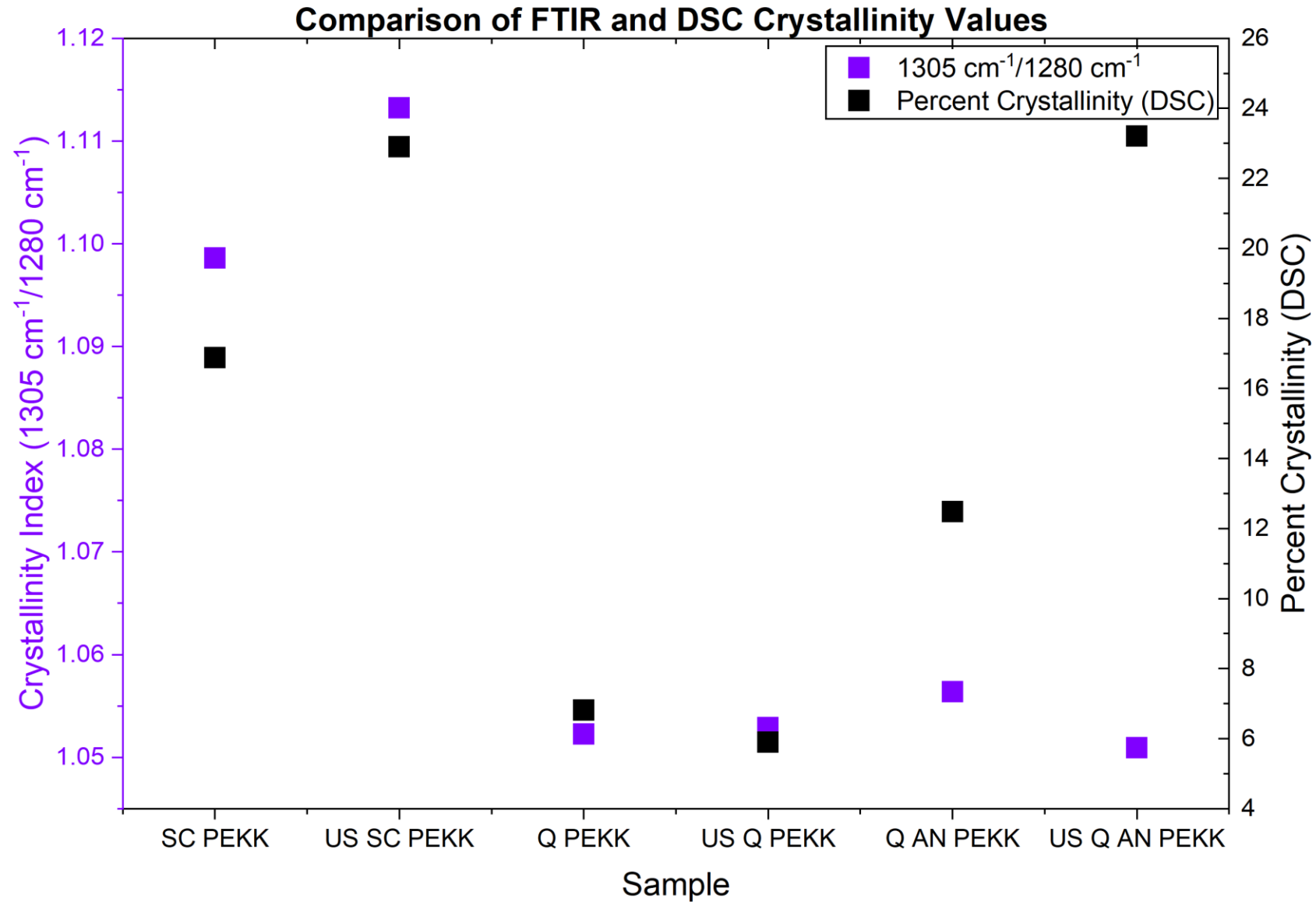
FTIR Analysis of Crystallites



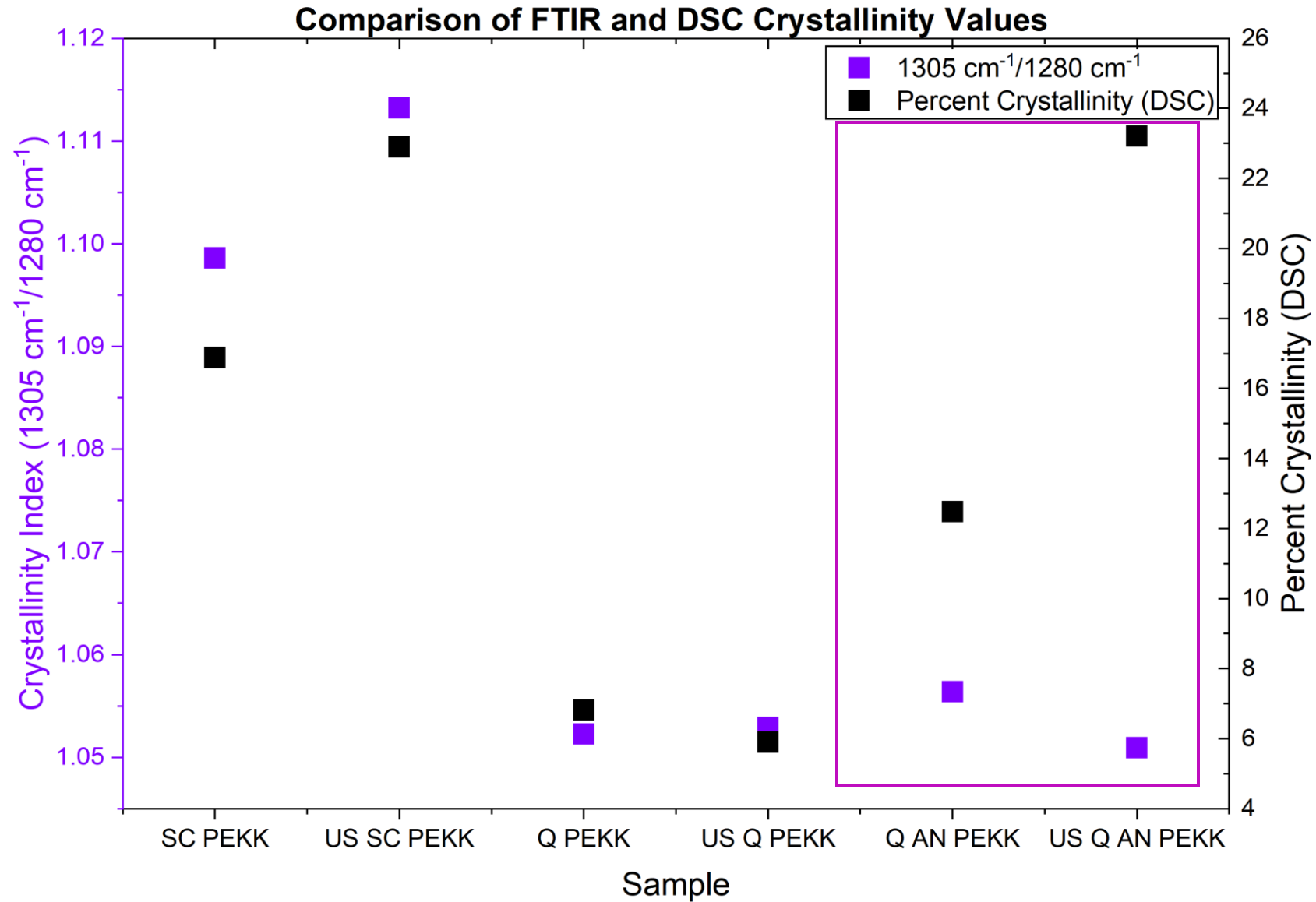
Crystallinity in Ultrasonically Welded Samples



Crystallinity in Ultrasonically Welded Samples

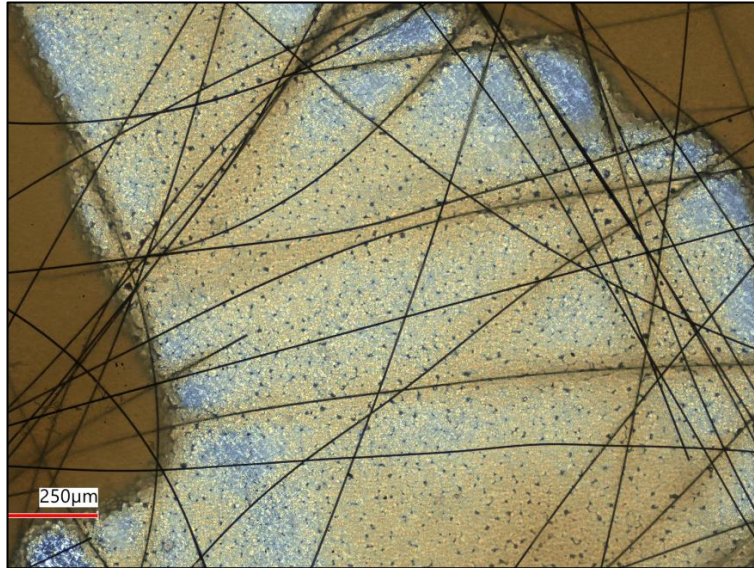


Crystallinity in Ultrasonically Welded Samples

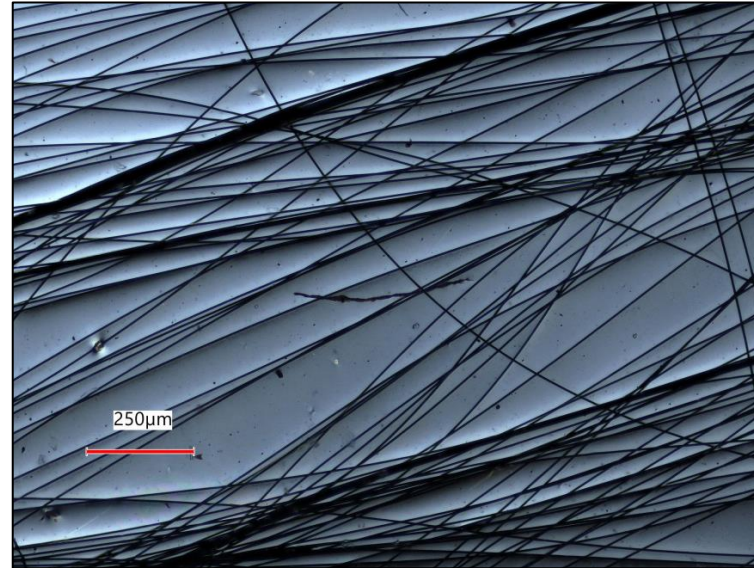


Crystallinity in Ultrasonically Welded Samples

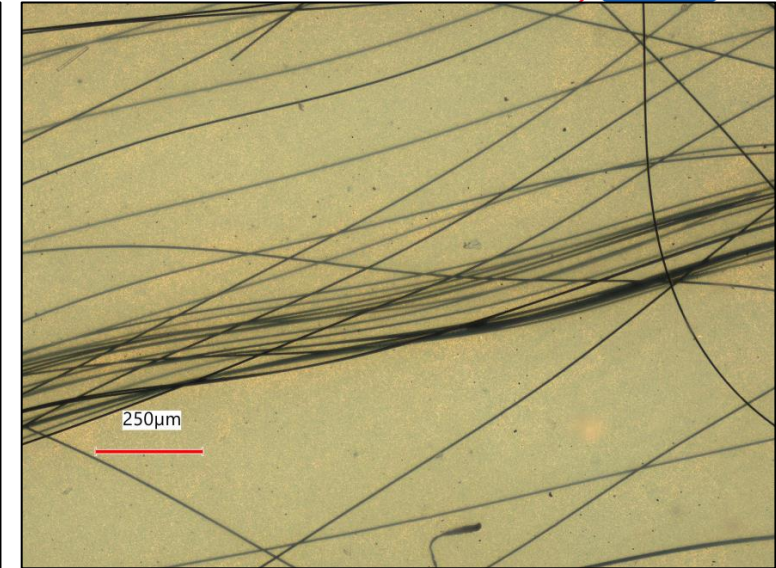
Before
welding



Slow Cooled PEKK

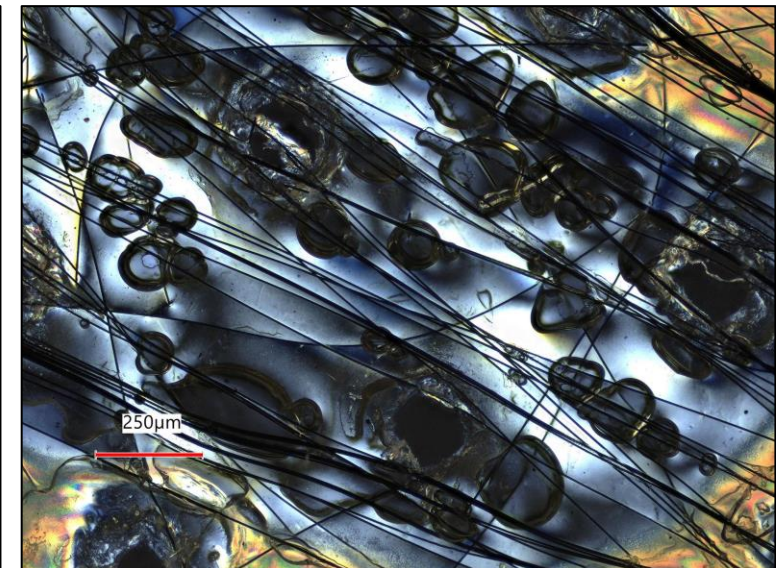
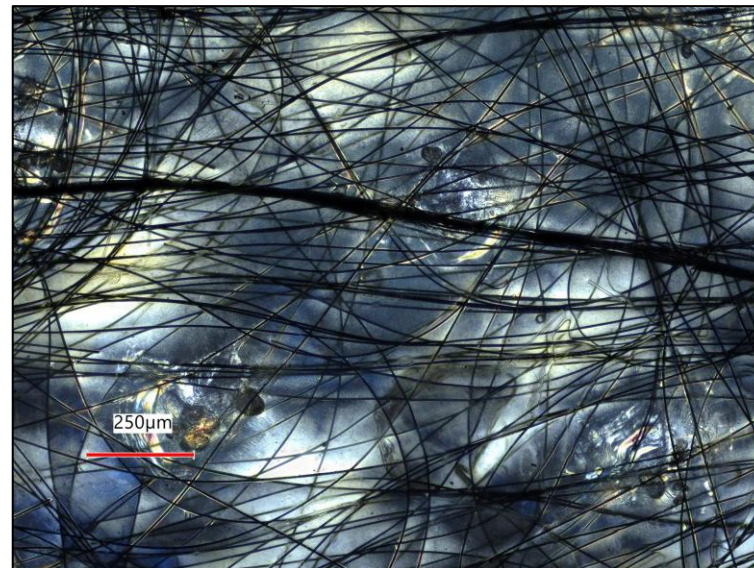


Quenched PEKK

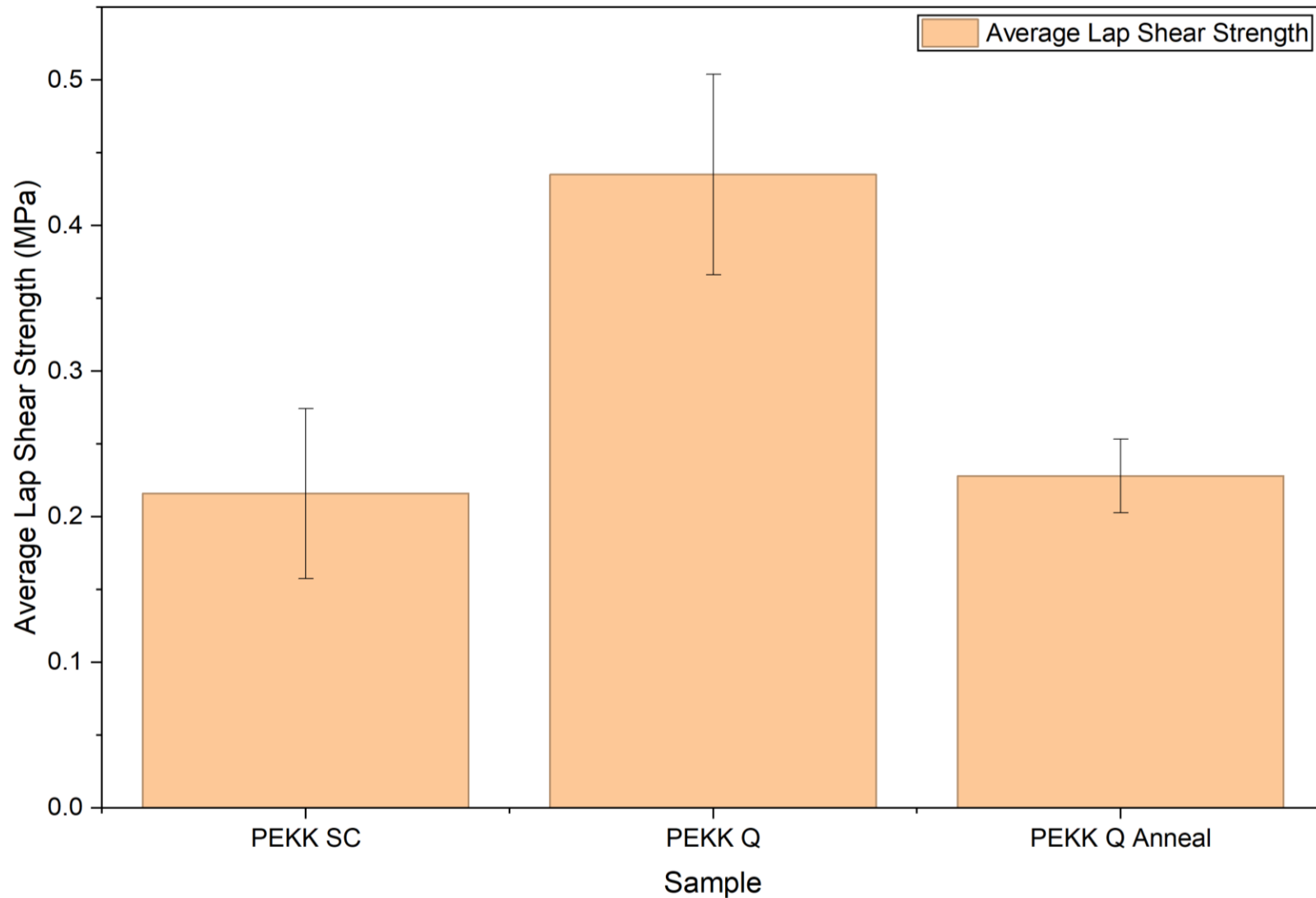
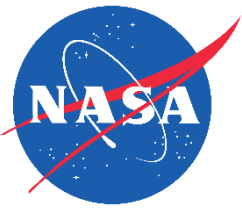


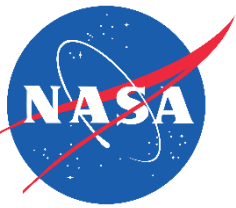
Annealed PEKK

After
welding



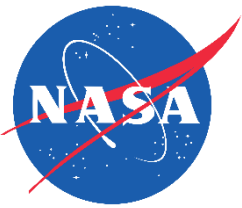
Crystallinity Effects on Ultrasonically Welded Samples





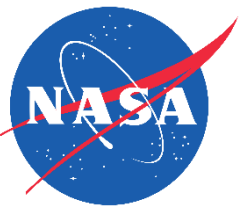
Key Takeaways

- **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**



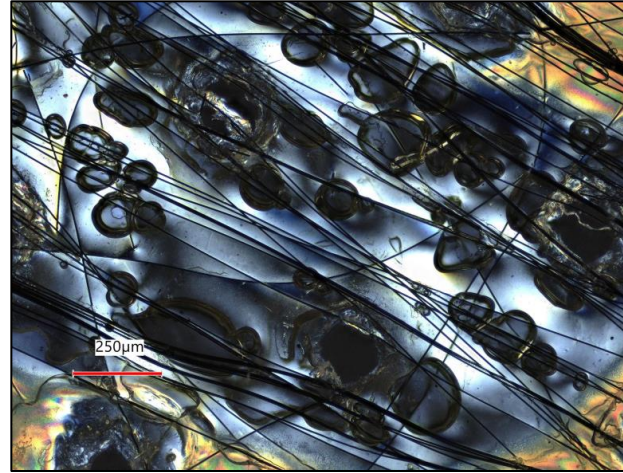
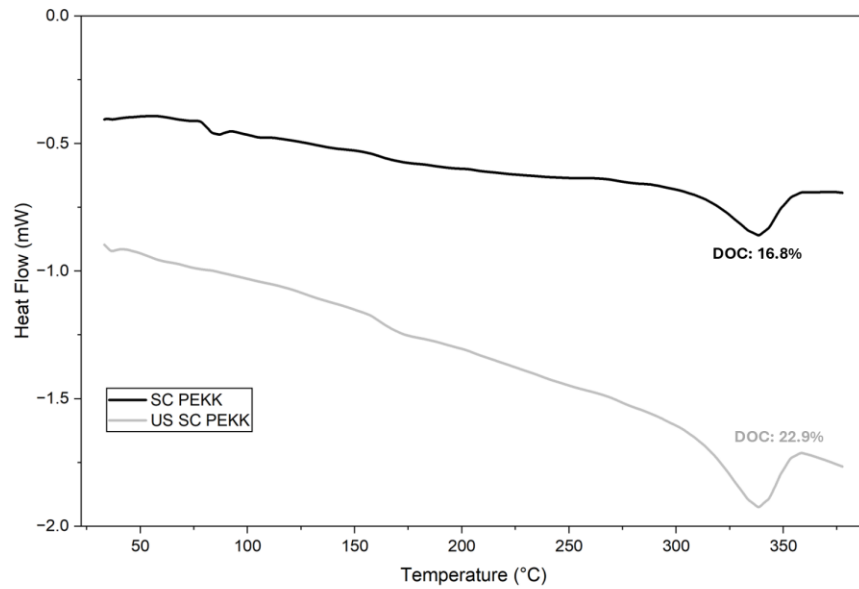
Key Takeaways & Next Steps

- 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 weld strength and performance for in-situ repair**
- **Use fundamental data to guide modeling efforts for predicting material performance**

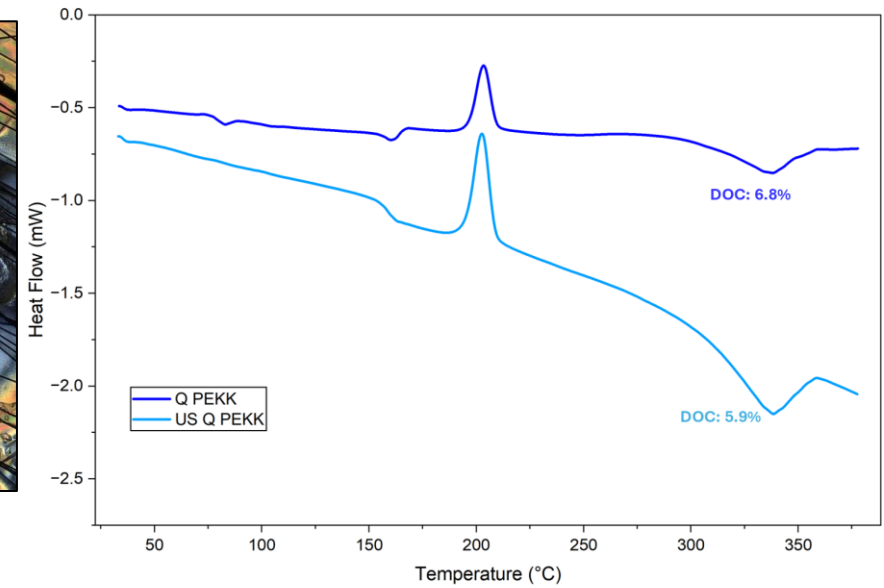


Backup Slides

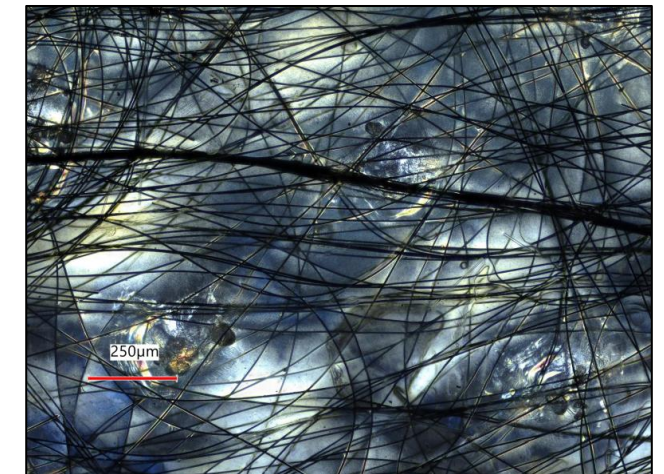
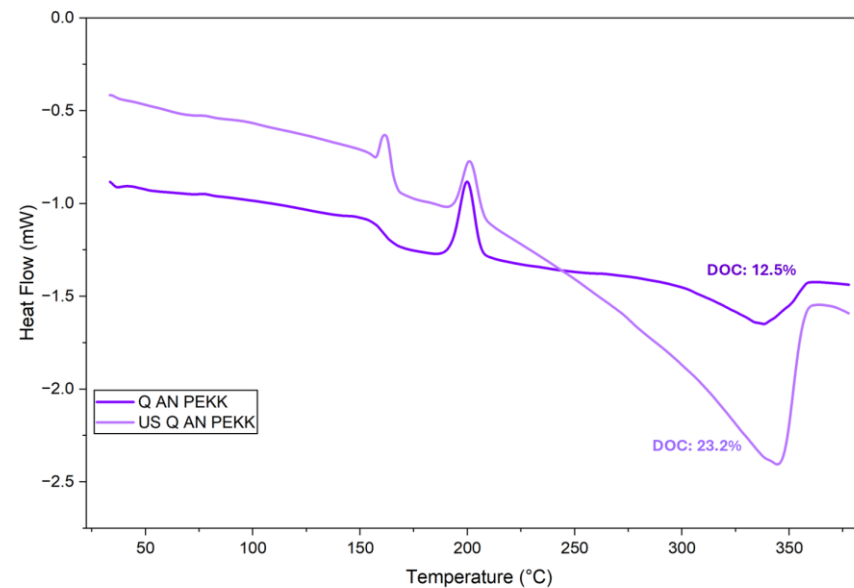
Crystallinity on Ultrasonically Welded Samples



US Annealed



US Slow Cooled



US Quenched

Cross Polarized Light Optical Microscopy

