



Measurement of Percent Crystallinity of Polyaryletherketone Composites using Fourier Transform Infrared Spectroscopy

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Research Context & Motivation



- Aerospace industry needs lightweight, high-performance materials
- NASA HiCAM project: developing rapid composite aircraft manufacturing
- Thermoplastic polyaryletherketone (PAEK) materials offer key advantages:
 - Rapid consolidation and reformability
 - Excellent fracture toughness
- Challenge: Material properties directly linked to crystallinity
- Need reliable methods to measure and control crystallinity

PAEKs and the Crystallinity Challenge



- Materials studied: polyetheretherketone (PEEK), polyetherketoneketone (PEKK), Low-melt PAEK (LM-PAEK)
- Crystallinity affects:
 - Stiffness, strength, toughness
 - Thermal expansion, solvent resistance
- Processing conditions influence crystallinity:
 - Tool temperature, placement speed, cooling rates
- Goal: Homogeneous crystallinity distribution for consistent performance

Three Methods for Measuring Crystallinity

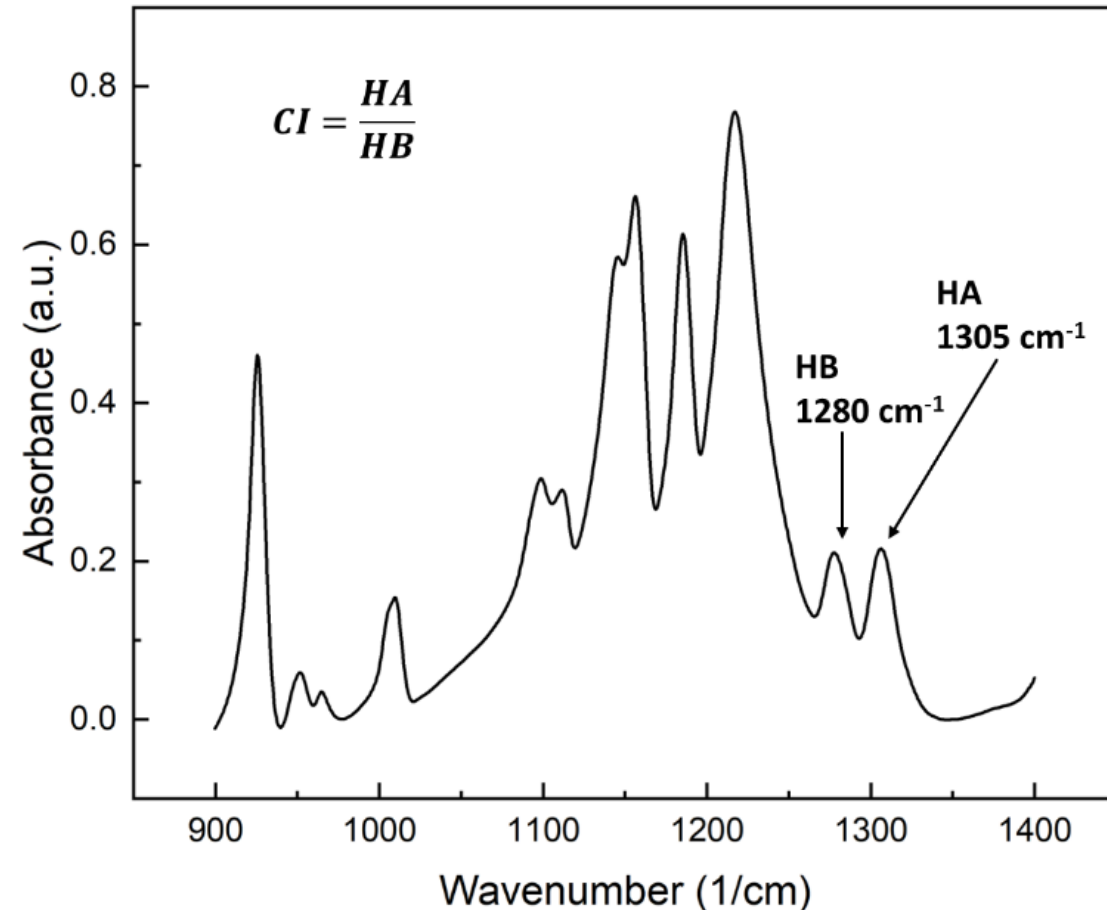


- DSC (Differential Scanning Calorimetry):
 - Measures bulk thermal transitions
 - Industry standard for crystallinity
- FTIR (Fourier Transform Infrared):
 - Surface crystalline regions via absorption peaks
 - Ratio of 1305 cm^{-1} to 1280 cm^{-1} peaks (using ASTM F2778)
- XRD (X-ray Diffraction):
 - Crystallographic structure analysis
 - Kilian & Jenckel method applied

FTIR Crystallinity Index Approach



- Following ASTM F2778 standard
- Extended from PEEK to other PAEK materials
- Metric: crystallinity index (CI)

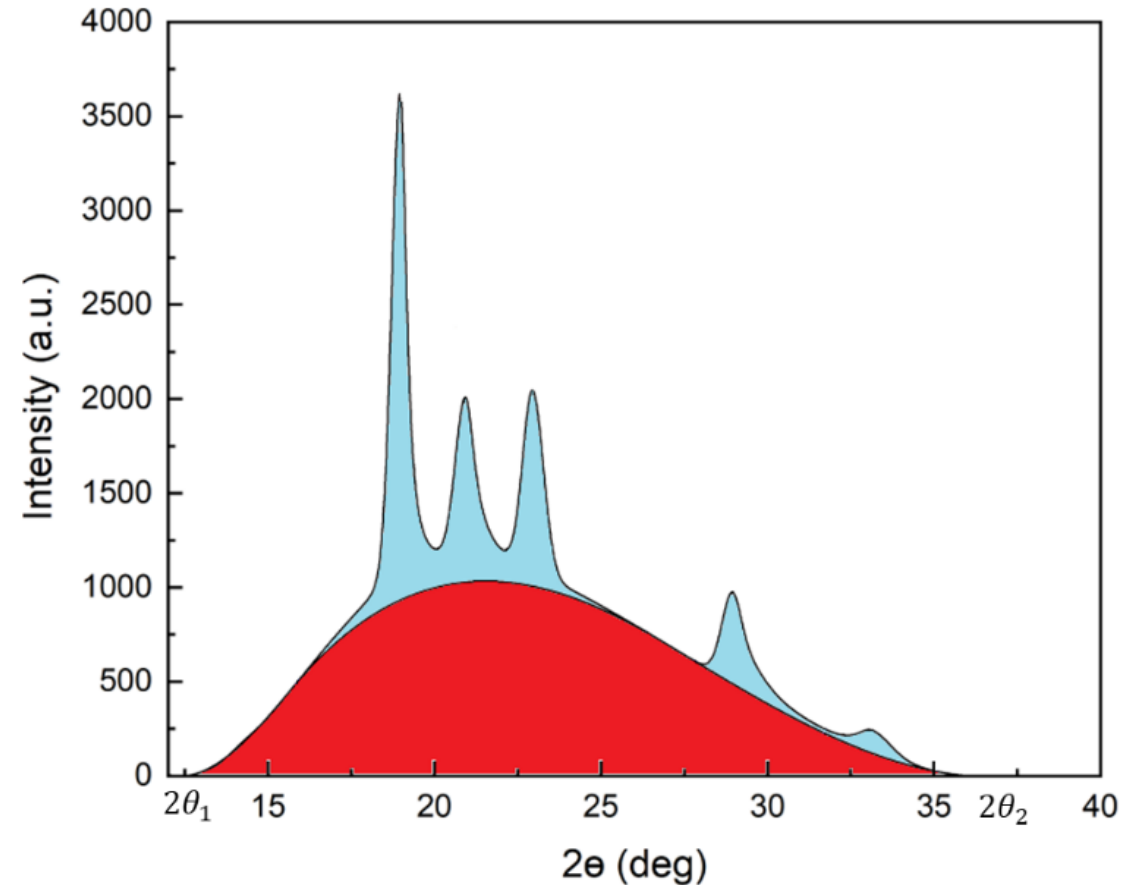


Kilian & Jenckel Method



- The degree of crystallinity can be determined by the ratio of the area of the crystalline peaks to the total area (which includes the amorphous phase)
- I_c : crystallinity intensity
- I_t : total intensity

$$\chi(\%) = \frac{\int_{2\theta_1}^{2\theta_2} I_c d\theta}{\int_{2\theta_1}^{2\theta_2} I_t d\theta} \times 100$$



Materials and Test Conditions

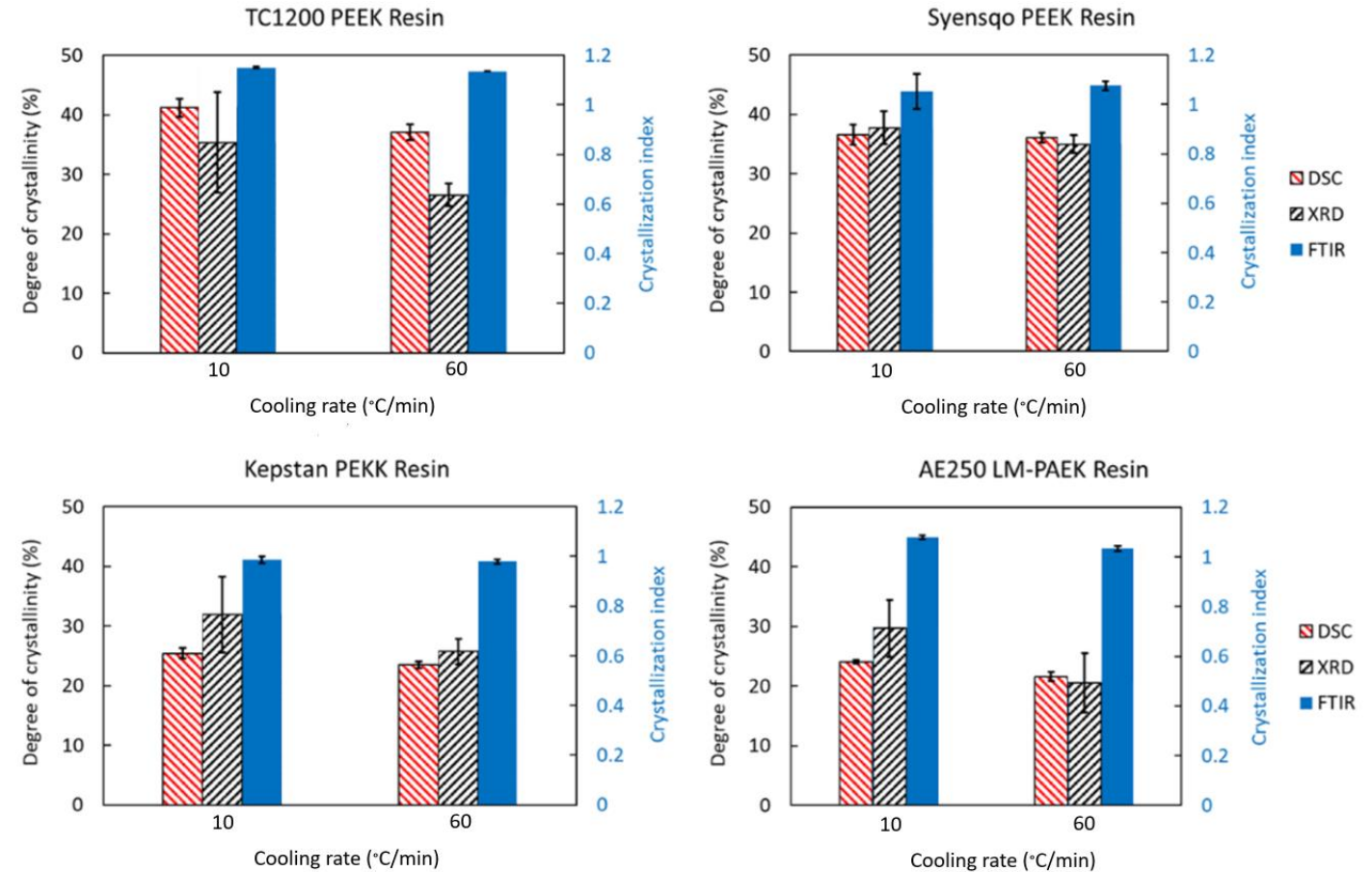


- Four PAEK materials tested:
 - Two PEEK variants (Toray TC1200, Syensqo KT-800P)
 - PEKK (Kepstan 7002)
 - LM-PAEK (Viktrex AE 250)
- Test conditions: Two cooling rates (10°C/min, 60°C/min)
- All three techniques applied to same samples for comparison

Key Results - Technique Comparison



- Good correlation: DSC and XRD for most materials
- DSC vs FTIR: Comparable results for PEEK materials
- Exception: TC1200 PEEK showed XRD divergence
 - Suggests localized amorphous regions
- FTIR CI: Remained similar despite different cooling rates among materials
 - Useful to compare crystallinity within the same material system



PEEK vs. Other PAEKs



- PEEK consistently higher crystallinity than PEKK and LM-PAEK
- Reason: PEEK molecular structure promotes:
 - Efficient chain packing
 - Regular arrangements
 - Stronger intermolecular forces
- PEKK and LM-PAEK: Structural irregularities inhibit crystallization
- Processing sensitivity: All materials are affected by thermal history

Technique Strengths & Limitations



- DSC: Most robust for quantitative bulk crystallinity
 - Direct enthalpy measurements
 - Works across different polymer types
- XRD: Excellent for structure analysis
 - Can show localized variations
- FTIR: Good for local molecular order
 - Limitation: Not directly comparable between polymer types
 - Requires calibration against DSC for quantitative work

Conclusions & Recommendations



- DSC recommended as primary quantitative technique
- Complementary approach using multiple methods provides complete picture
- PEEK shows superior crystallization compared to other PAEKs
- Process control critical: Cooling rates affect final properties
- Quality assurance: Understanding crystallinity essential for consistent manufacturing

Supporting Rapid Composite Manufacturing



- Process optimization: Understanding crystallinity-property relationships
- Quality control: Reliable measurement methods for production
- Material selection: Data-driven choices between PAEK types
- Future work: Extending to in-situ monitoring during automated fiber placement (AFP) processing
- Goal: High-rate, high-quality composite aircraft production

Questions?



- Discussion
- Thank you for your attention

- **Support**
 - NASA HiCAM Project

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