PHASE TRANSFORMATION OF COMPOSITES VIA RESISTIVE HEATING

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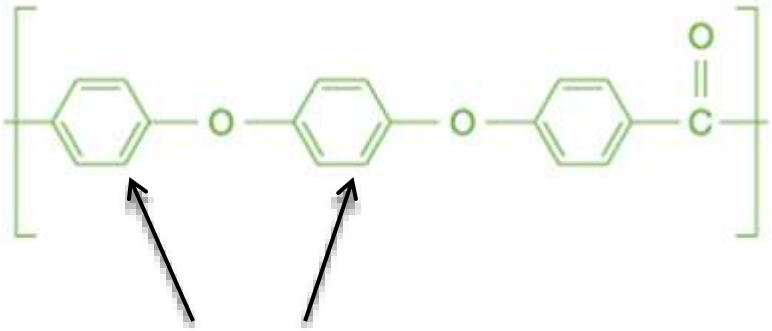
Polyetheretherketone (PEEK)

- Excellent mechanical properties
  - Strength
  - Stiffness
- Chemically resistant
- Aromatic (containing phenyl rings)
- High glass transition temperature → Good thermal stability

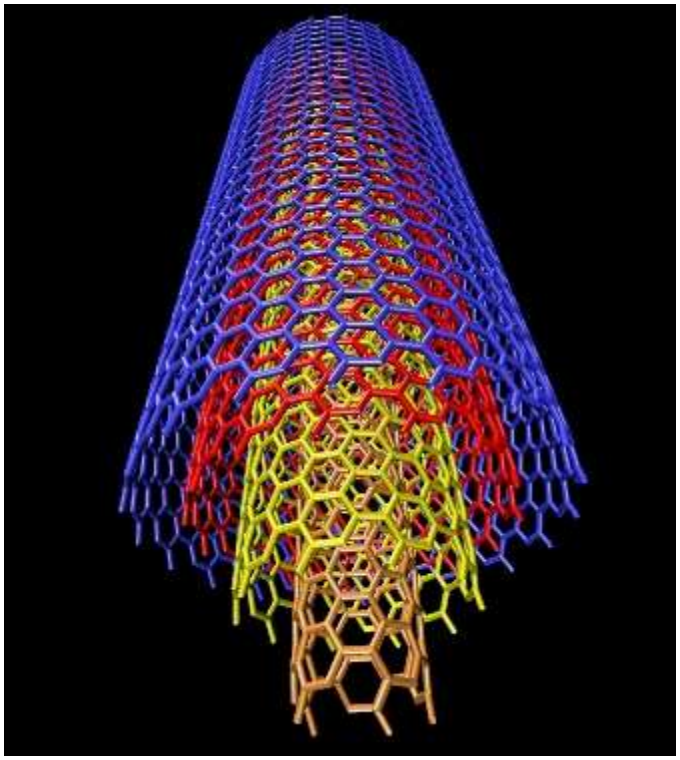


Phenyl Rings

Spinal Implants Manufactured from PEEK

Multi Walled Carbon Nanotubes (MWCNTs)

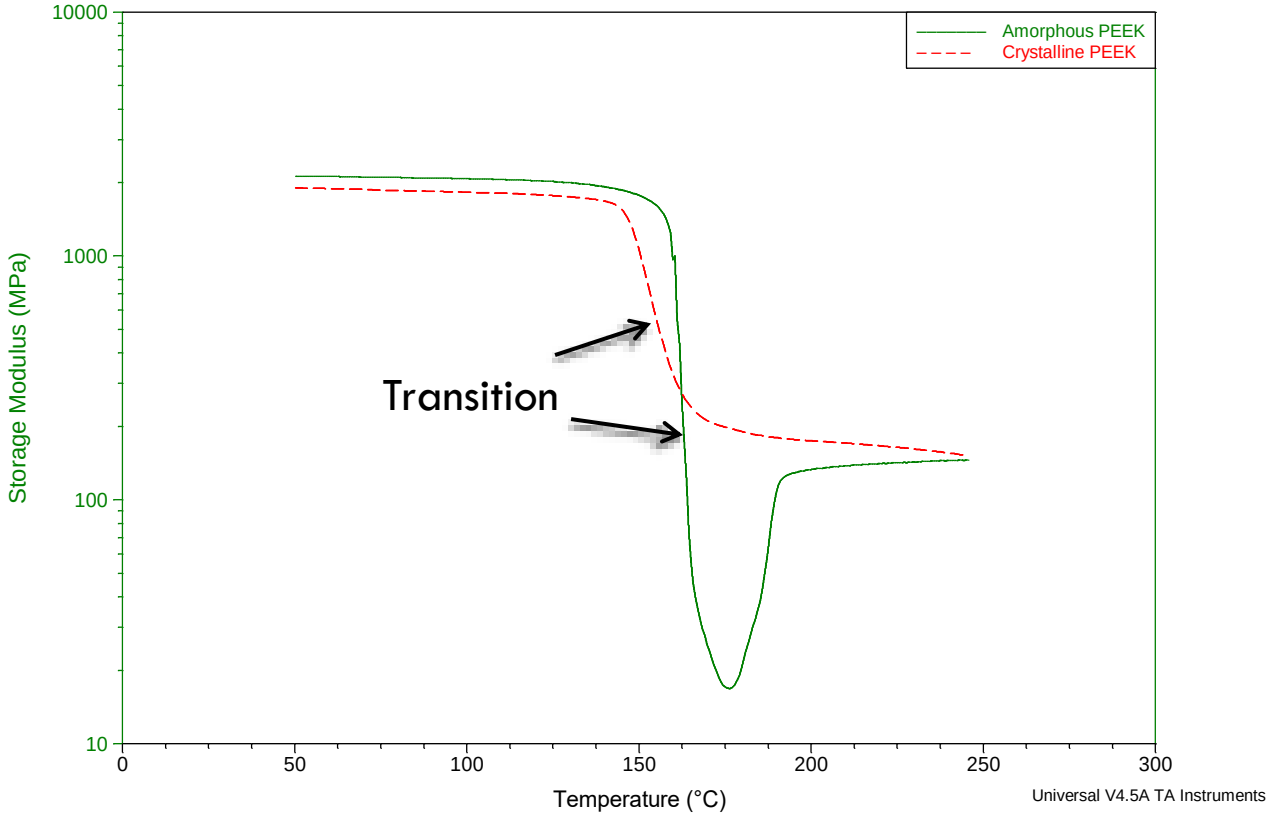
- Excellent conductivity
- Functional surfaces
  - -OH Functionalized
    - Improve dispersion
  - Non-Functionalized
- Addition of MWCNTs to PEEK will increase strength and stiffness
- Provide a current path for resistive heating
  - Induce phase transition



MWCNT Model – (helixmaterials.com)

Amorphous and Crystalline

- Crystalline
- Amorphous
- Glass transition temperature (T_g)



Goal of Research

Establish a reliable composite manufacturing technique.

- Based on preliminary framework for pure PEEK

Resistively induce an amorphous-to-crystalline transition.

- Non-traditional phase transition induction (without applied heat)
- Characterization for future use

Manufacturing - Amorphous PEEK

- Equipment Specifications
  - WABASH hydraulic press

    5 ksi (34.5 MPa) and 750°F (400°C)
  - Two thin circular plates with rectangular mold

WABASH Hydraulic Press

- Process Specifications
  - 1 Tablespoon of powder
  - Plates in press for 10 minutes
  - Water/Ice bath for quenching

* Non-homogeneous → Pockets of various nucleating amorphous patterns

Manufacturing - Amorphous PEEK/MWCNT Composite

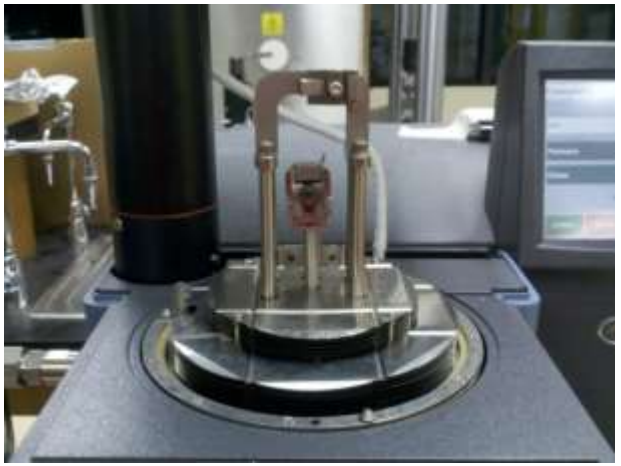
- Material procurement
  - US Research Nanomaterials Inc.
- 5.0 vol. % MWCNTs to PEEK
  - Volume percent based on preliminary framework by Amy DiRienzo
- Ethanol Suspension
  - 24 hrs on hot plate at 203°F (95 °C)
  - 12 hrs in vacuum oven
- Powder sintering technique similar to pure PEEK

Testing - Phase Transition

- Resistive Heating
  - Powerstat Variable Autotransformer
  - Heat composite through T_g

Resistive Heating Apparatus

- Dynamic Mechanical Analysis
  - TA Instruments Q Series 800 DMA
  - Show amorphous/crystalline behavior



TA Instruments Q Series 800 DMA

Testing - Mechanical Behavior

- Tensile Tests
  - 858 Mini Bionix II Load Cell
  - Show strength and strain to failure

858 Mini Bionix II Load Cell

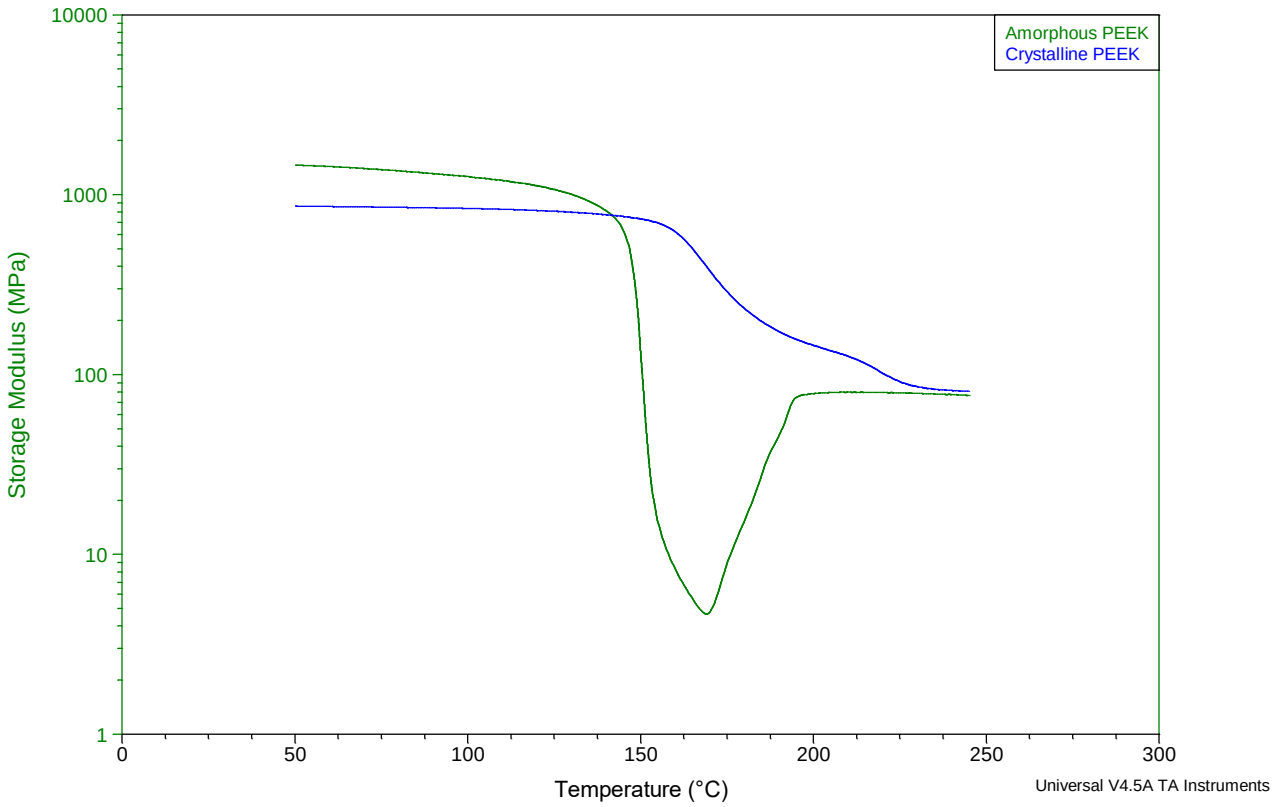
- Fracture Analysis
  - Scanning Electron Microscopy
  - Show MWCNT dispersion on fractured surface

Results – Resistive Heating

- Early sample destruction
  - Ramp voltage at 10volts/2min
- Recovery
  - Dial immediately to higher voltage

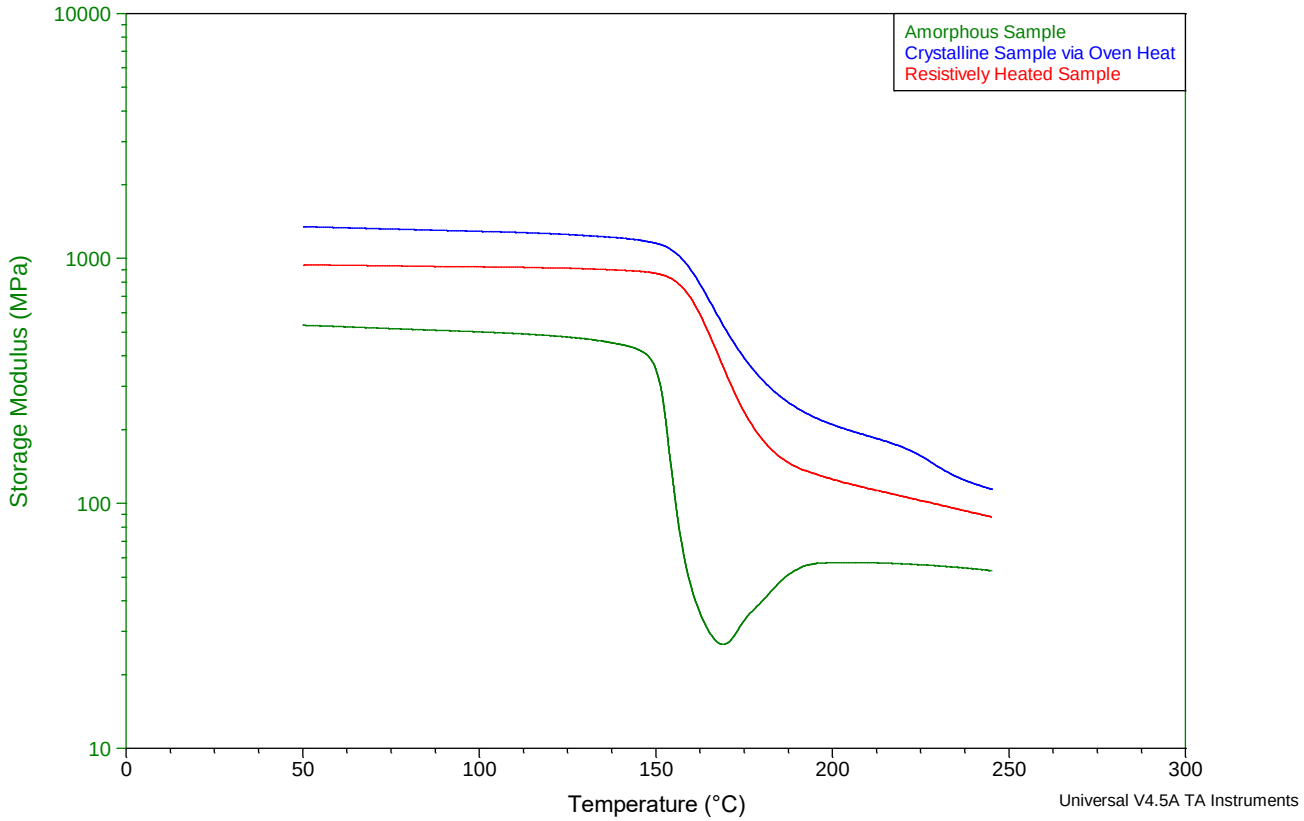
Results – DMA Testing of PEEK

- Amorphous behavior of manufactured samples
- Amorphous to Crystalline transition observed after oven heating



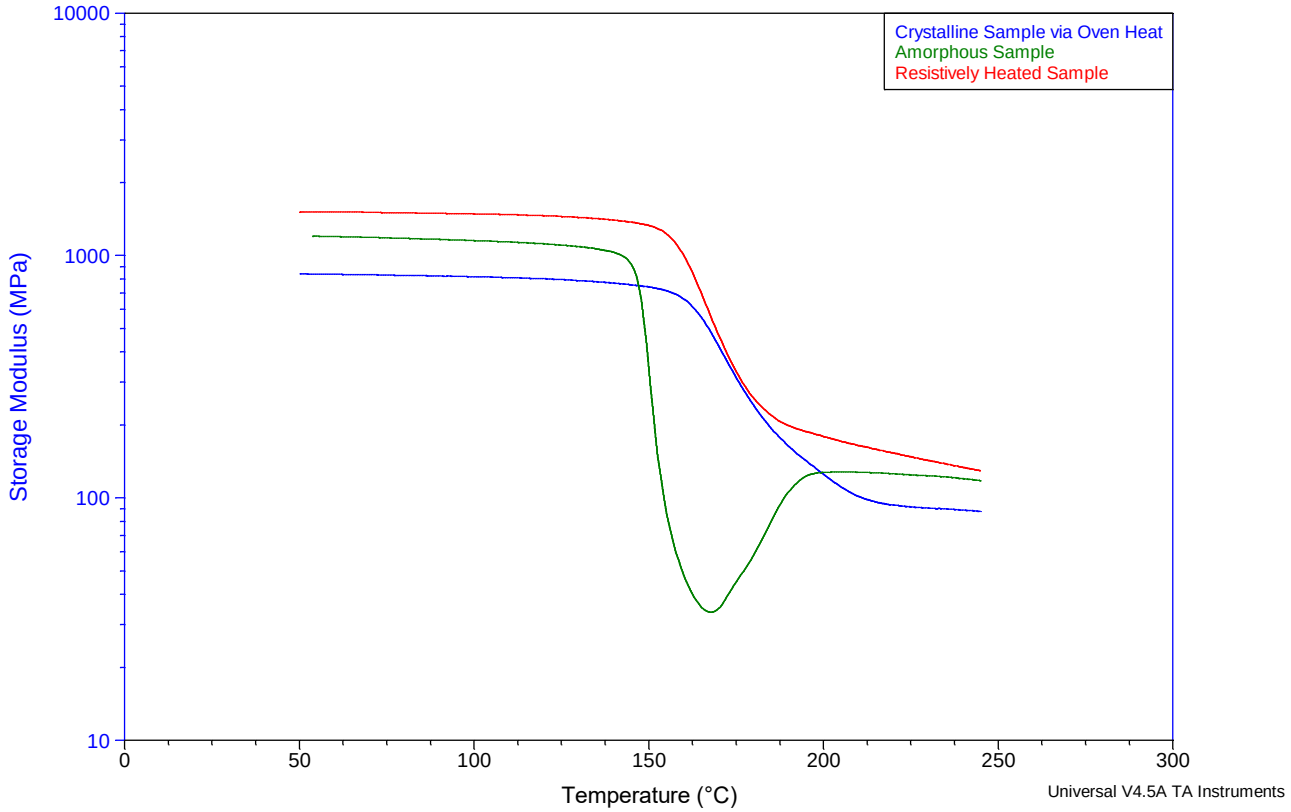
Results – DMA Testing of Non-Functionalized Composites

- Amorphous behavior of manufactured samples
- Crystalline behavior after oven heating
- Crystalline behavior after resistive heating



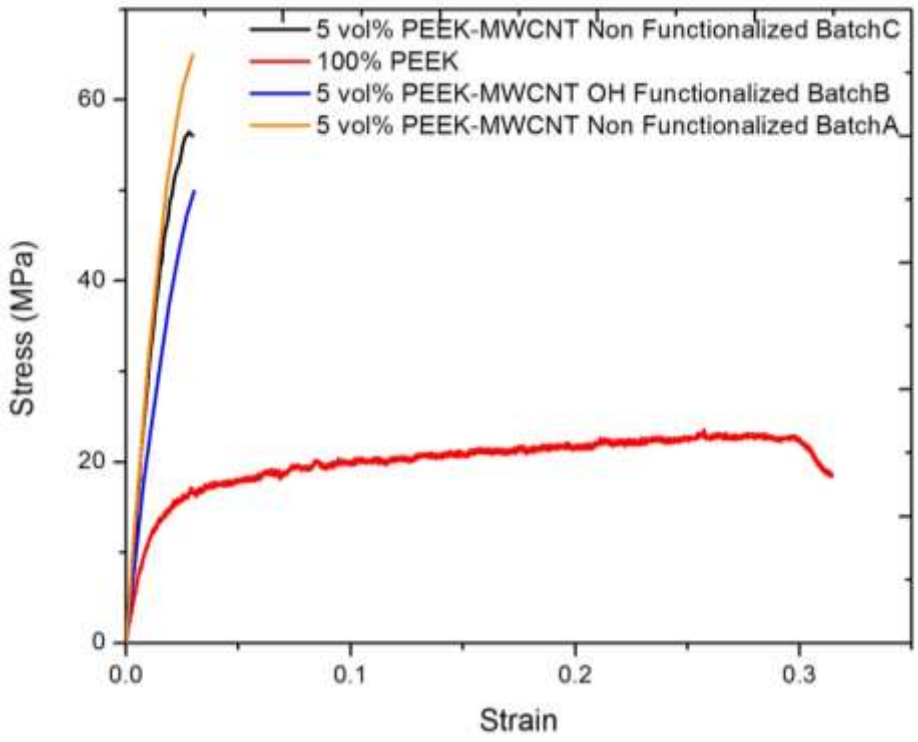
Results – DMA Testing of –OH Functionalized Composites

- Amorphous behavior of manufactured samples
- Crystalline behavior after oven heating
- Crystalline behavior after resistive heating

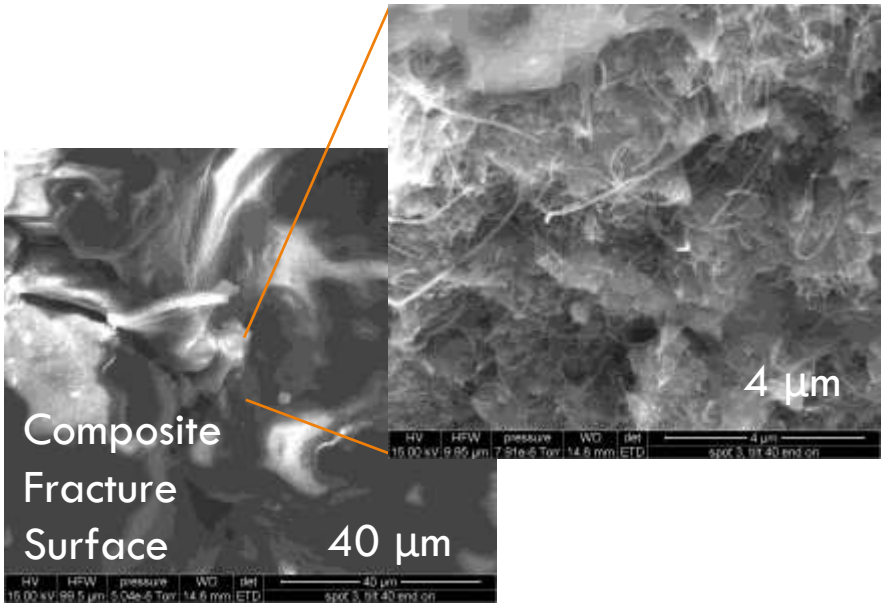


Results – Tensile Tests

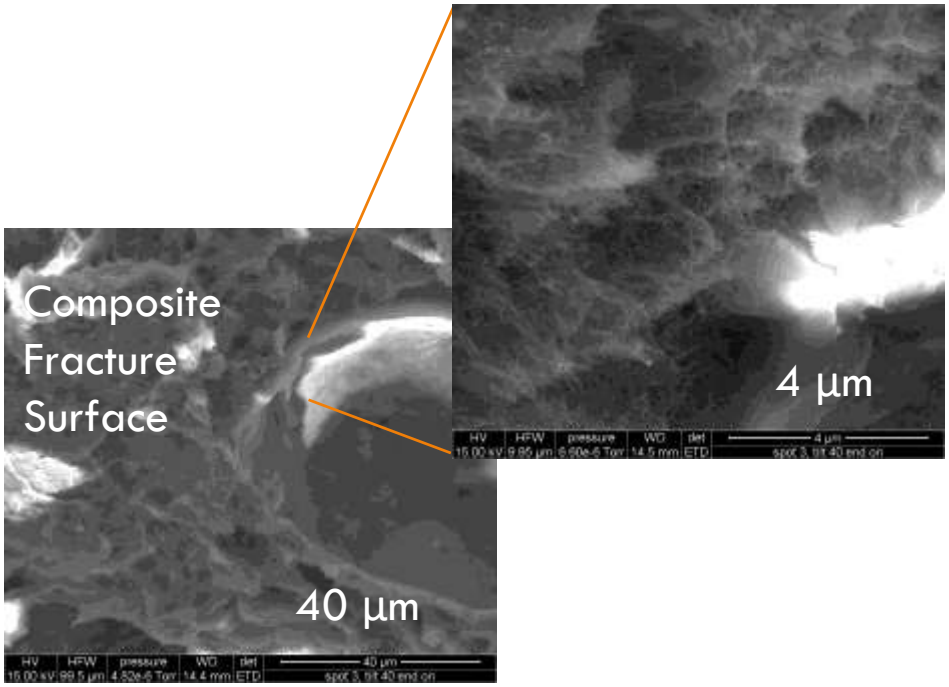
- Each composite showed higher strength and less ductility than pure PEEK



Results – Fracture Analysis



Non-Functionalized Fracture Surface



-OH Functionalized Fracture Surface

Conclusion

- Successful phase transformation via resistive heating
  - Early resistive heating quantification tests ended in failure of sample
    - Temperature/voltage relationship could be time dependent
    - Underestimating temperature measurements with current set-up
  - Fast voltage ramp rate for phase transition

- SEM and tensile tests
  - Good dispersion of MWCNTs
    - Good contact for current path
  - MWCNT effective reinforcement

Conclusion

- Future Work
  - Basis for Master's Thesis
    - Apply resistive heating framework to other networks
  - Quantify destructive resistive heating behavior

Questions