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Using AFM as a High Resolution Technique to Measure Elastic Modulus Changes on a Thermally Aged Epoxy System

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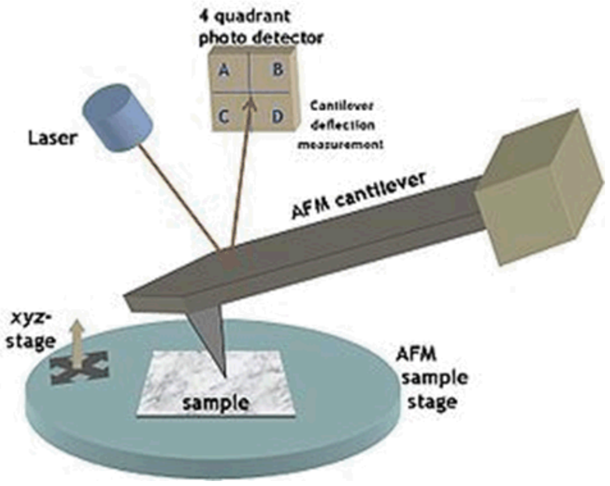
Outline

- What is AFM
- Background
- AFM vs. Conventional Nanoindentation
- AFM Constraints
- Experiment /Sample Prep
- Results
- Conclusion

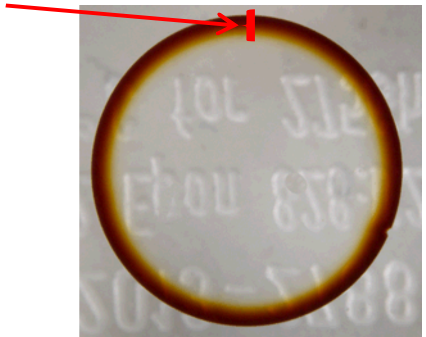
Atomic Force Microscopy (AFM)

- Ultra fine tip (10 - 35nm radius) attached to a cantilever
- Tip physically interacts with sample surface
- Laser beams focused on backside of cantilever tracks cantilever movement
- Contact Mode
- Tapping Mode



Background

- Develop a method to physically measure degree of change in a thermally aged polymer
  - Needs:
    - High Resolution/Sensitivity
- AFM has been used to measure:
  - Thin Films
  - Nanocomposite/Fiber materials
- Goal: Use AFM Nanoindenting to measure degree of change in a thermally aged epoxy material
- sdf



AFM vs. Conventional Nanoindentation

- Conventional Nanoindentation
  - Depth-Sensing Indentation (DSI)
    - Tip size ~200nm
    - Indentation depth ~
    - System compliance is low for soft materials (< 1GPa – polymers)
    - Analysis based on elasticity (polymers are viscoelastic)
    - Calibration uncertain for polymers
  - Interfacial Force Microscope (IFM)
    - Tip sizes 45 – 500nm
    - Indentation depth few nanometers
    - No compliance at fast rates – eliminates elasticity problem

- sdf

AFM Limitations

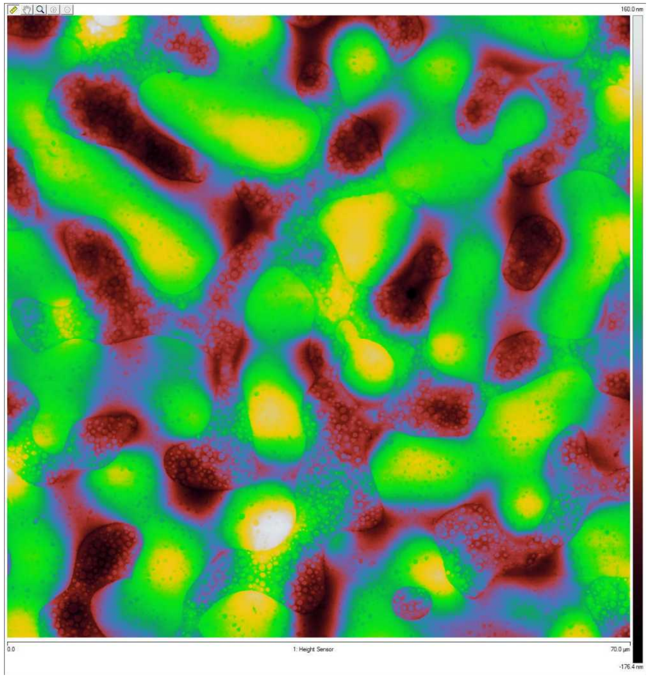
- Surface measurement
- Spring constant is poorly calibrated
- Difficulty measuring tip shape
- Tip shape changes
- Polymer constraints

- AFM indentation yields relative measurements

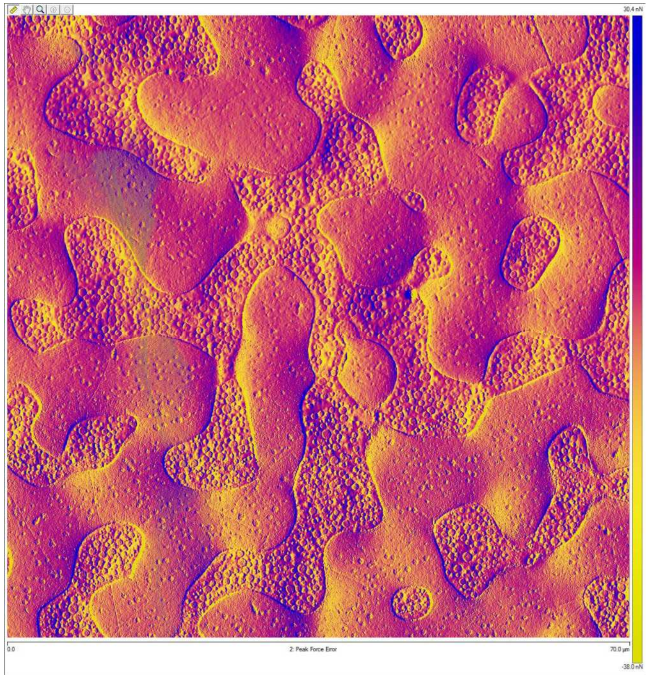
AFM Advantage

- Ability to image topography easily
- Surface Measurement nm range
- High resolution – tip size

Two phase epoxy system



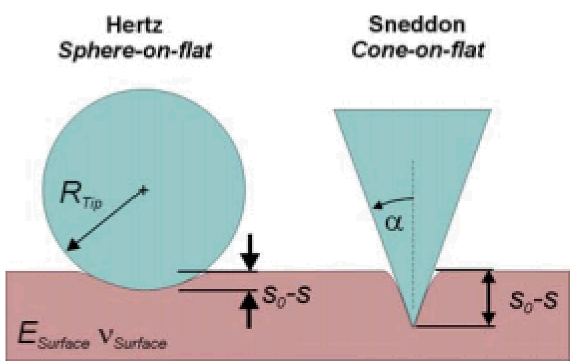
Height Sensor



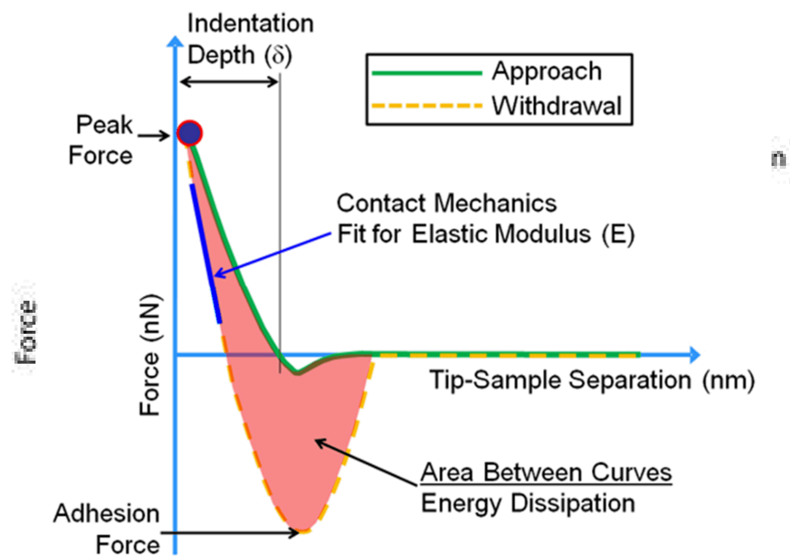
Peak force Error

QNM/Nanoindenting

- Each interaction between tip and sample generates force-distance curves
- Nanomechanical properties at each interaction

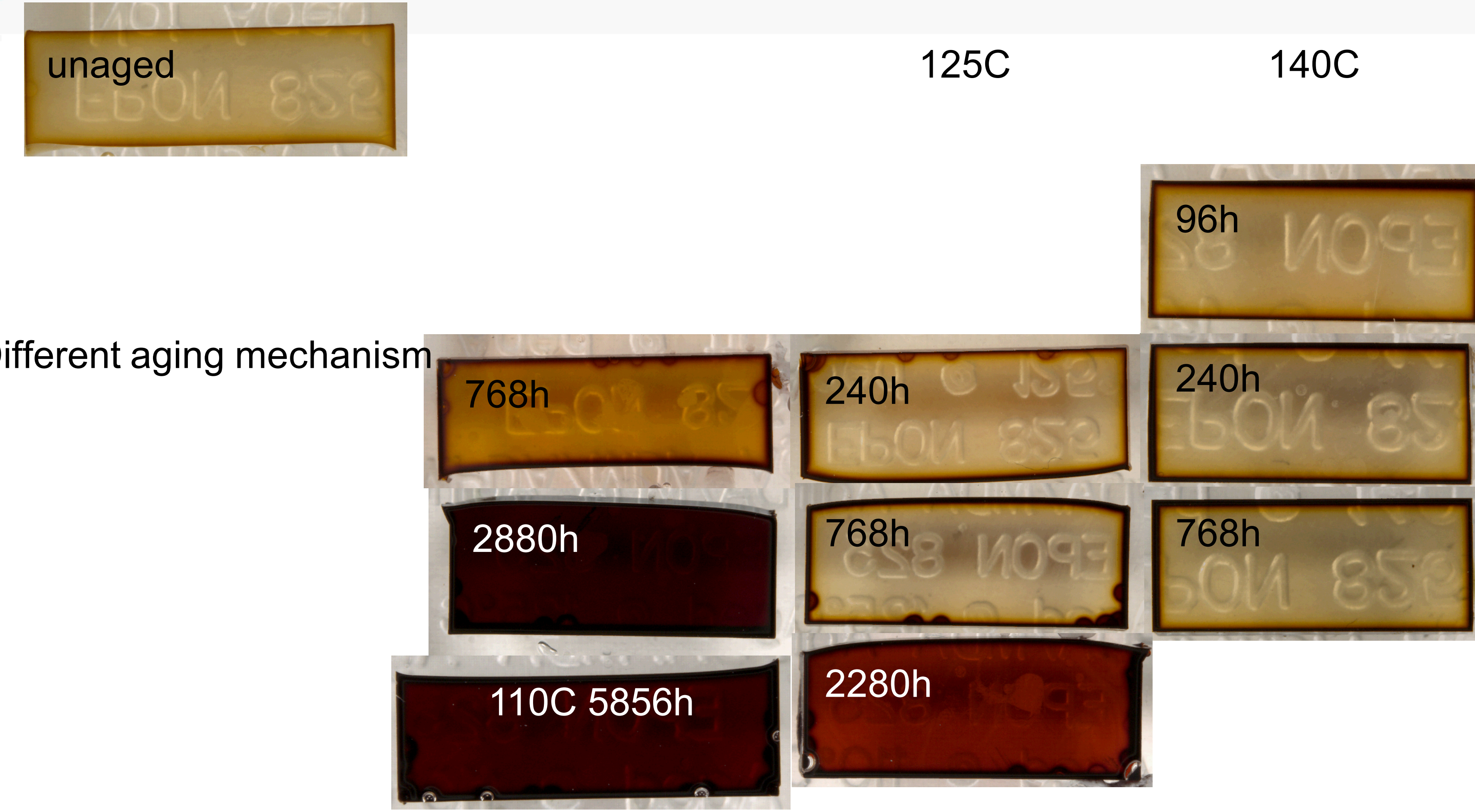


$$F_{Hertz} = \frac{4}{3}\frac{E_{surface}}{(1-\nu^2_{surface})}\sqrt{R_{tip}}(s_0 - s)^{3/2} \quad F_{Sneddon} = \frac{2}{\pi}\frac{E_{surface}}{(1-\nu^2_{surface})}\tan(\alpha)(s_0 - s)^2$$



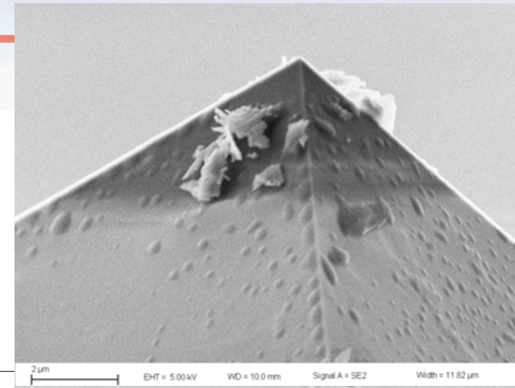
Ignores viscoelastic behavior

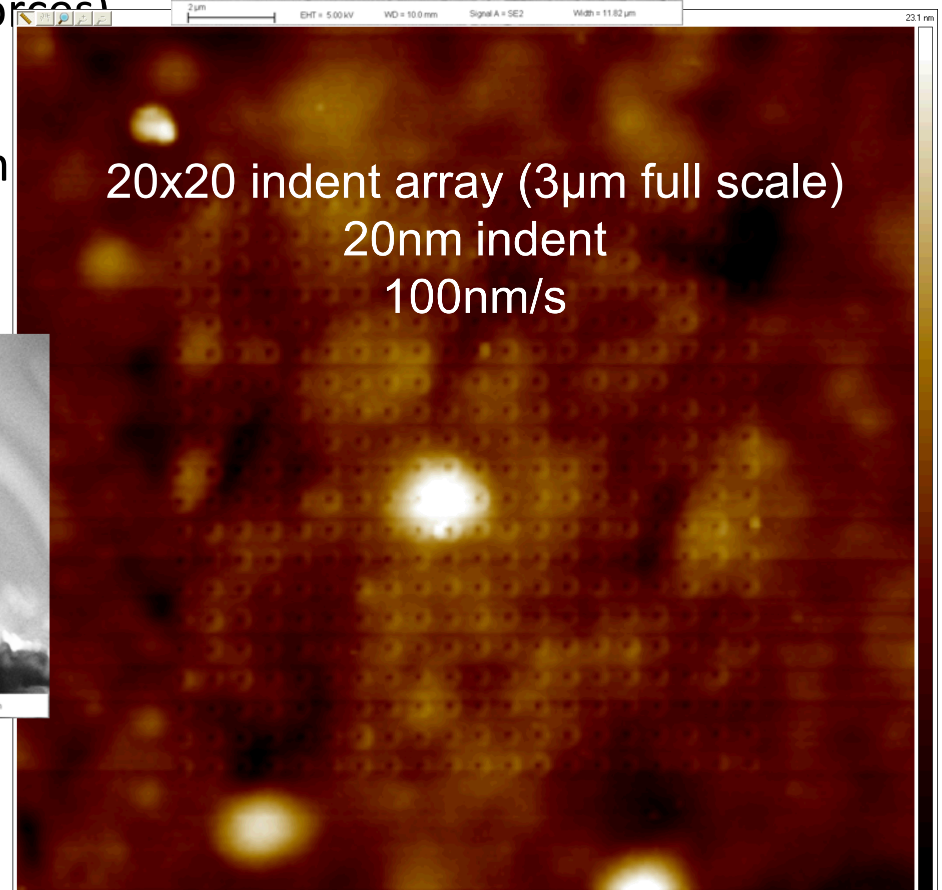
Thermally Aged Epoxy

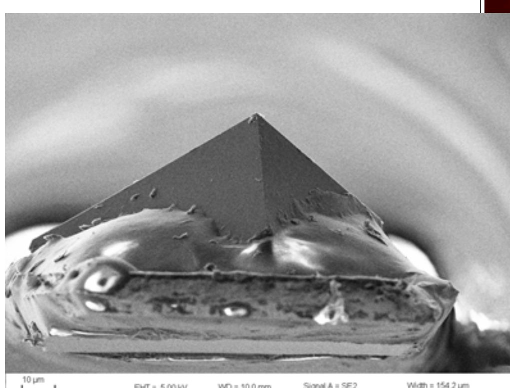


Parameter Optimization

- Bruker Dimension Icon7
- probe used
- Force (more than 10% adhesive for
- Speed
- Compliance calibration on diamon
- Image area
- Curve fit
- X rotation
- cleaning

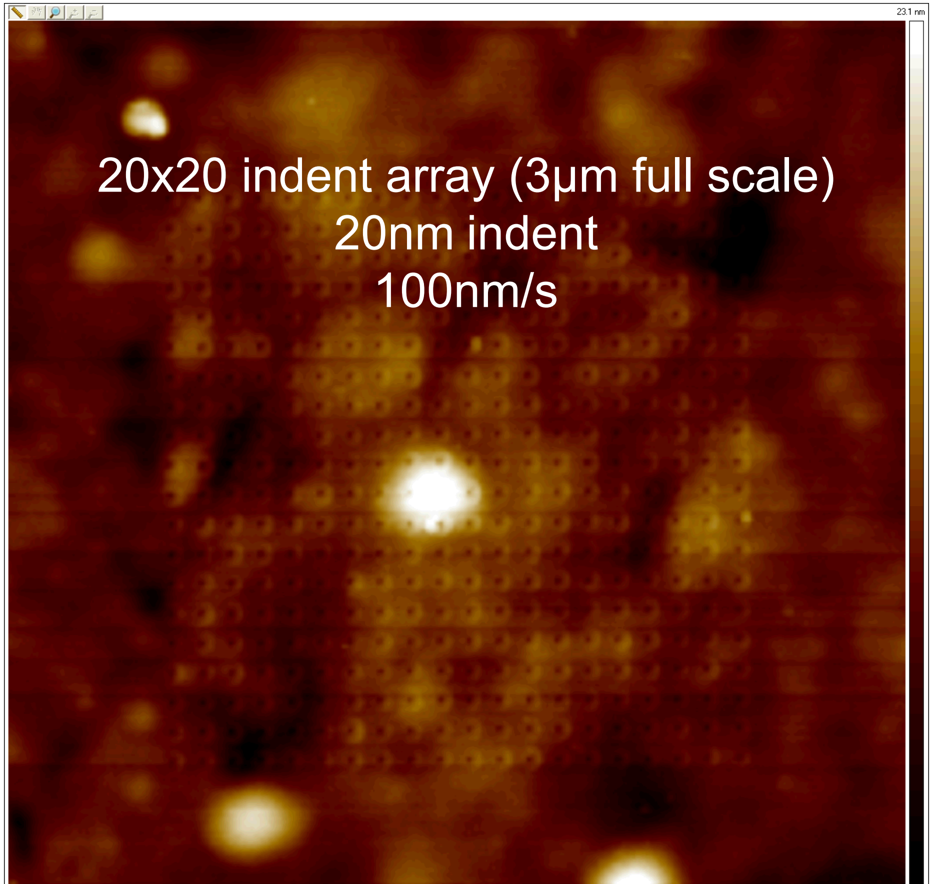




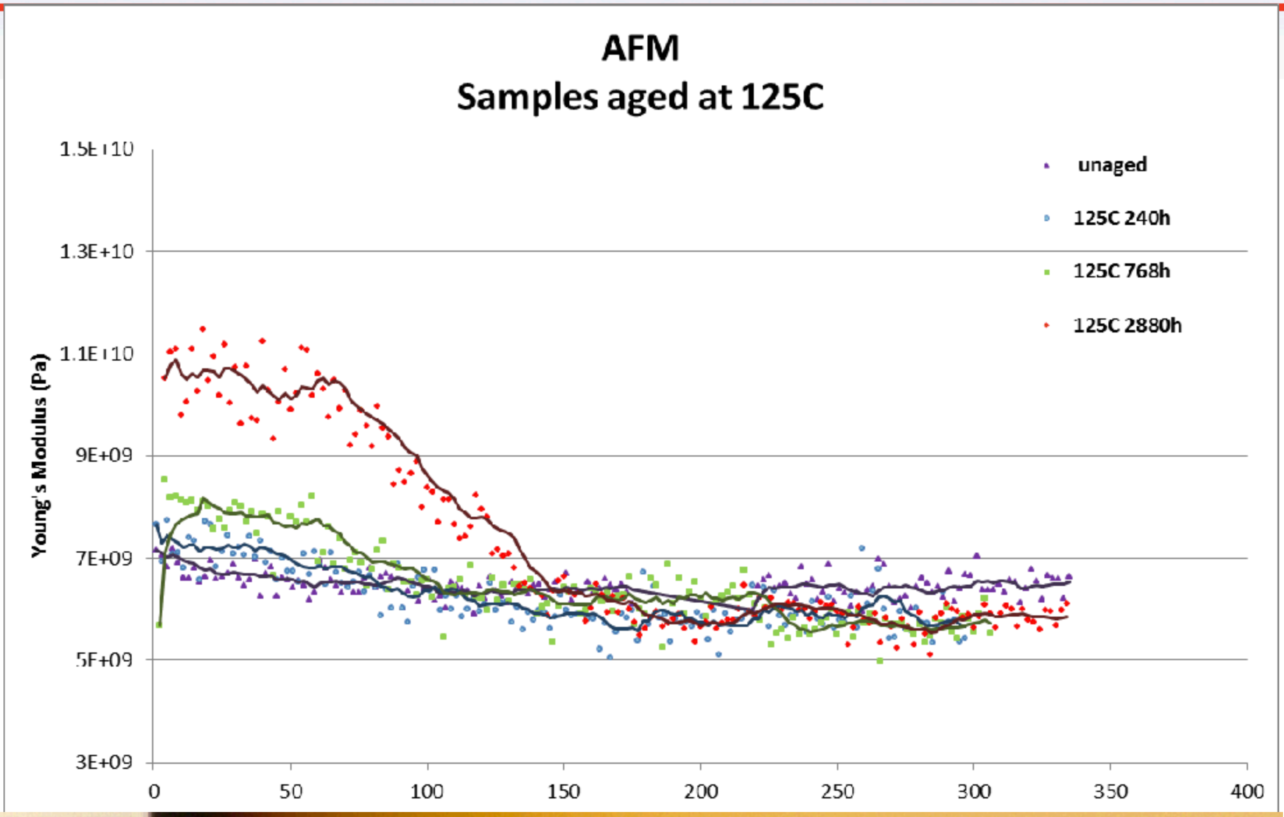


Procedure

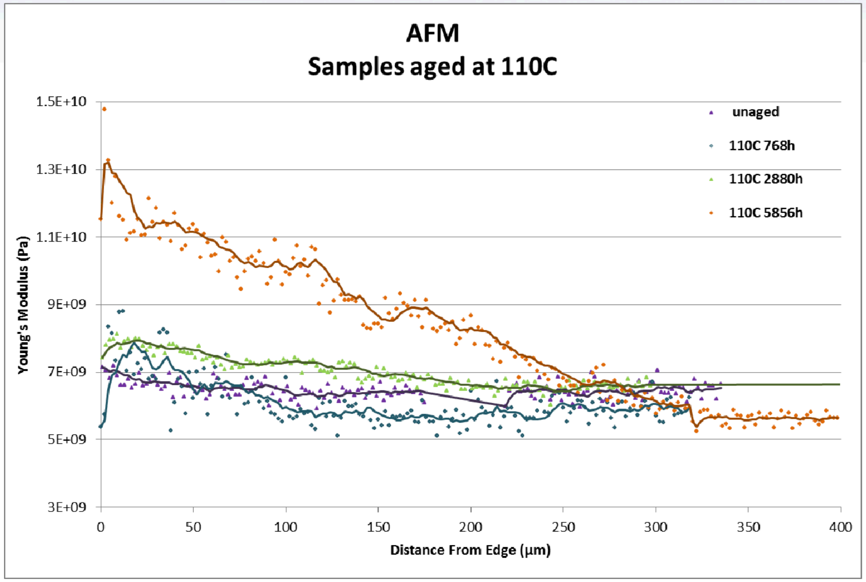
- Diamond nanoindenting probe used
  - 35nm radius
  - 145k spring constant
- Force
- Speed
- Compliance calibration on diamon
- Image area
- Mounting & Polish

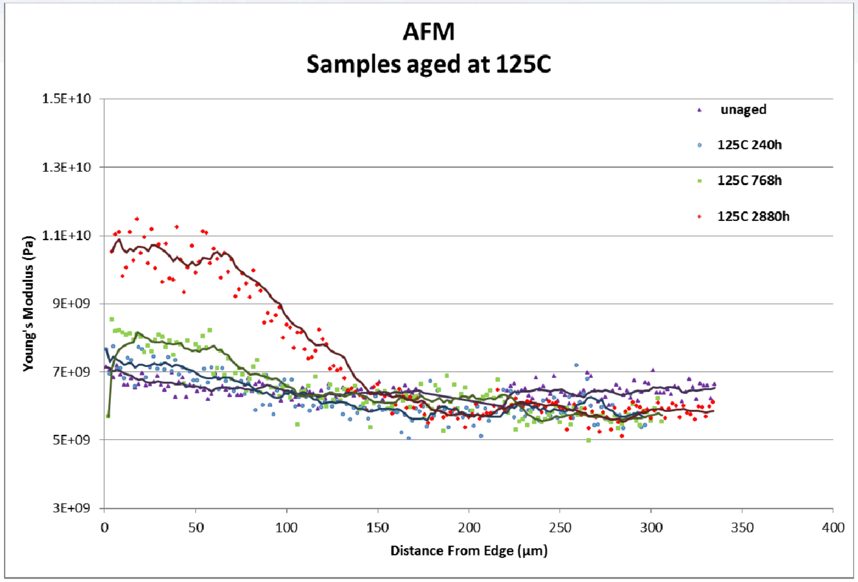


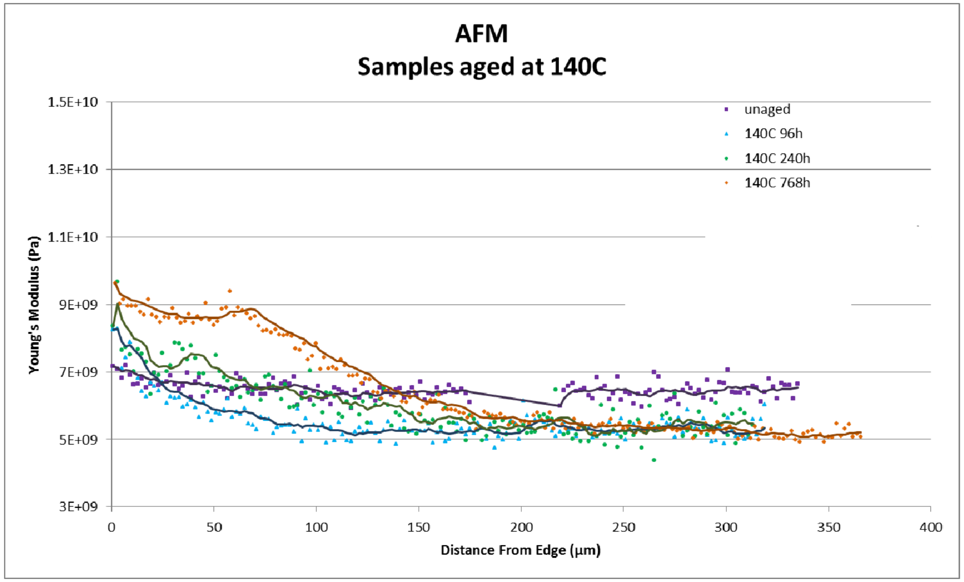
Results


AFM
Samples aged at 125C
Young's Modulus (Pa)
1.5E+10
1.3E+10
1.1E+10
9E+09
7E+09
5E+09
3E+09
0
50
100
150
200
250
300
350
400
unaged
125C 240h
125C 768h
125C 2880h

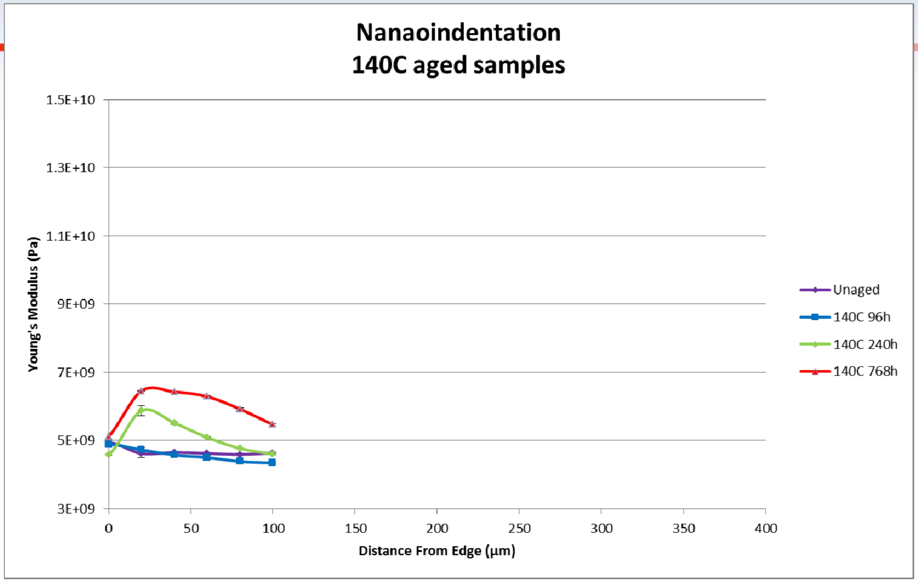
Results

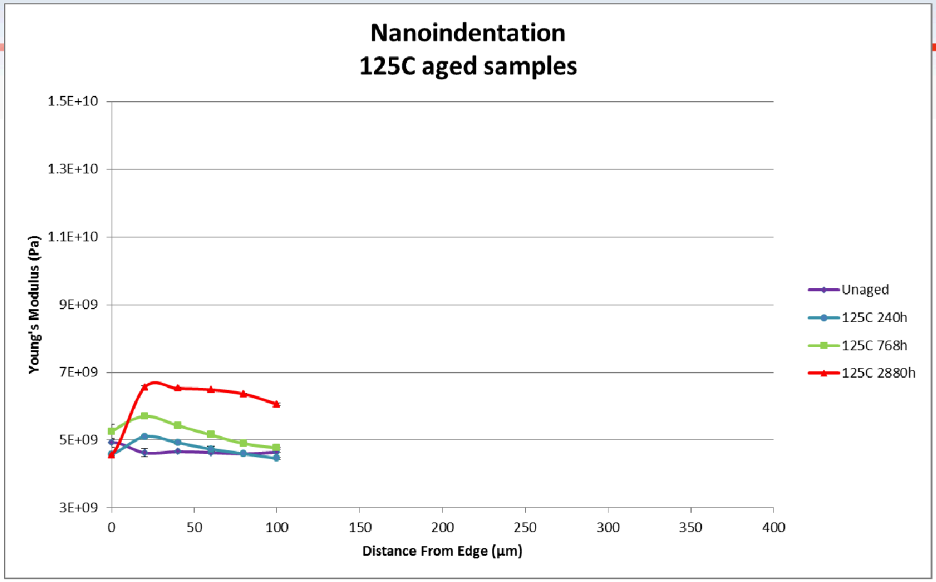

AFM
Samples aged at 110C
1.5E+10
1.3E+10
1.1E+10
9E+09
7E+09
5E+09
3E+09
Young's Modulus (Pa)
0
50
100
150
200
250
300
350
400
Distance From Edge (µm)
unaged
110C 768h
110C 2880h
110C 5856h

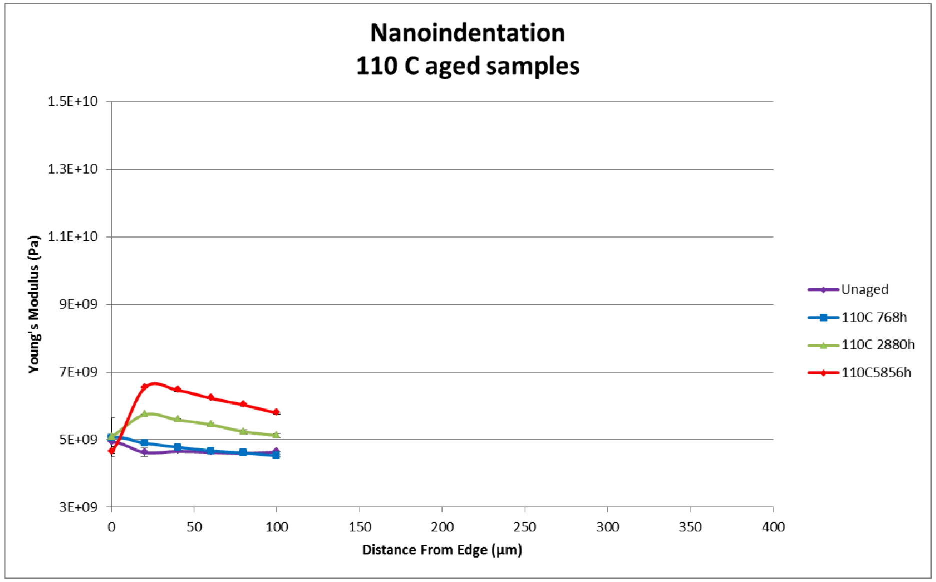

AFM
Samples aged at 125C
1.5E+10
1.3E+10
1.1E+10
9E+09
7E+09
5E+09
3E+09
Young's Modulus (Pa)
0
50
100
150
200
250
300
350
400
Distance From Edge (µm)
unaged
125C 240h
125C 768h
125C 2880h


AFM
Samples aged at 140C
1.5E+10
1.3E+10
1.1E+10
9E+09
7E+09
5E+09
3E+09
Young's Modulus (Pa)
0
50
100
150
200
250
300
350
400
Distance From Edge (µm)
unaged
140C 96h
140C 240h
140C 768h

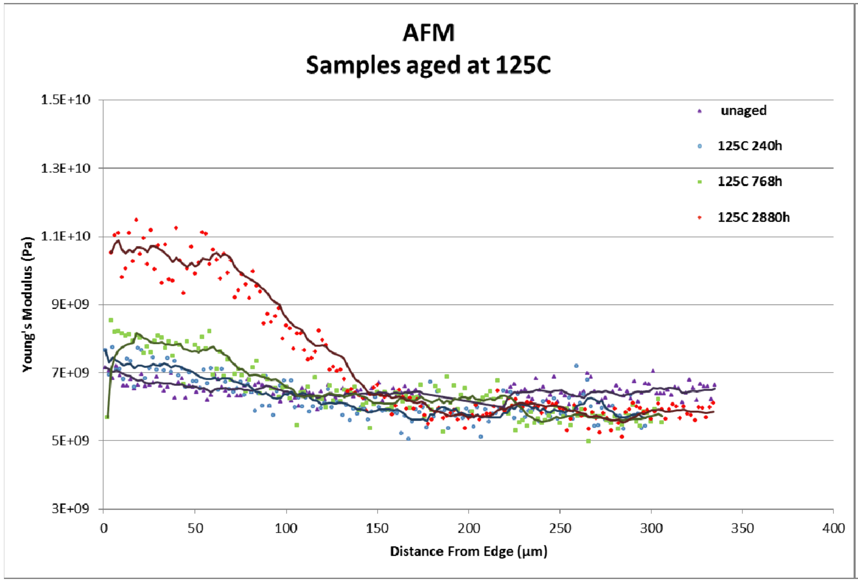
DSI Results

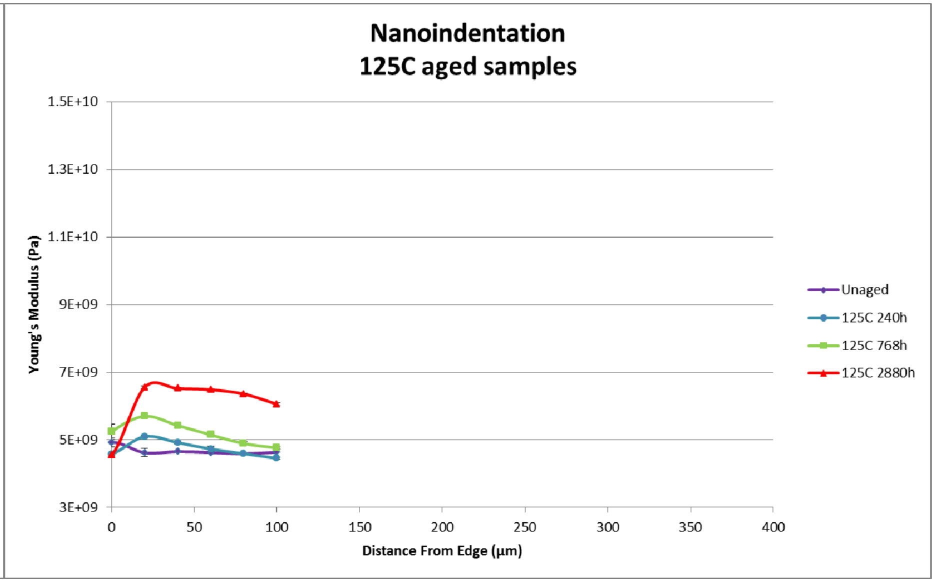

Nanaoindentation
140C aged samples
1.5E+10
1.3E+10
1.1E+10
9E+09
7E+09
5E+09
3E+09
Young's Modulus (Pa)
0
50
100
150
200
250
300
350
400
Distance From Edge (µm)
Unaged
140C 96h
140C 240h
140C 768h


Nanoindentation
125C aged samples
1.5E+10
1.3E+10
1.1E+10
9E+09
7E+09
5E+09
3E+09
Young's Modulus (Pa)
0
50
100
150
200
250
300
350
400
Distance From Edge (µm)
Unaged
125C 240h
125C 768h
125C 2880h


Nanoindentation
110 C aged samples
1.5E+10
1.3E+10
1.1E+10
9E+09
7E+09
5E+09
3E+09
Young's Modulus (Pa)
0
50
100
150
200
250
300
350
400
Distance From Edge (µm)
Unaged
110C 768h
110C 2880h
110C5856h

Comparison Results





Conclusions

- Optimized sample preparation
- Found appropriate probe
- Optimized nanoindentation parameters
- Established relative modulus measurements for a thermally aged epoxy gradient using nanoindentation on AFM
- Comparable with DSI values

Acknowledgements

- Tony Ohlhausen
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