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Characterization of Glass-Ceramic to Metal Interfaces via Nano-Indentation

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Outline

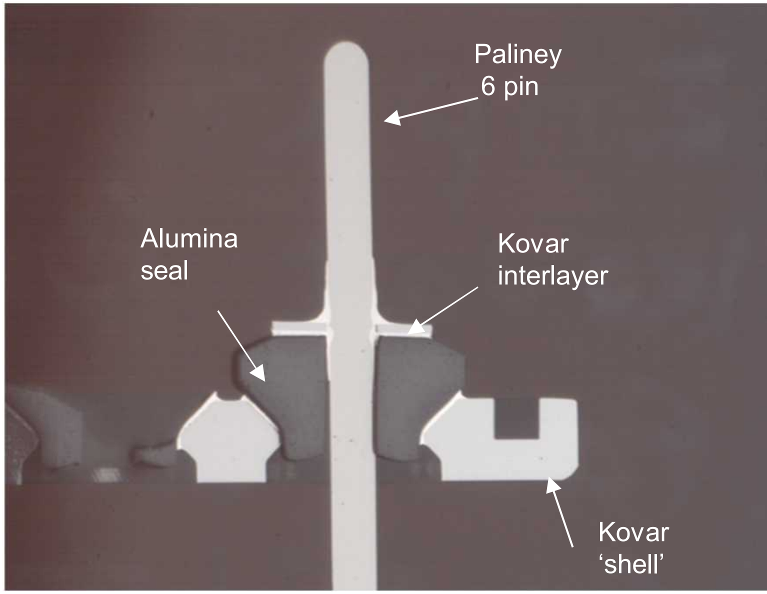
- Objective
- Formation of glass-ceramics
- Finite element analysis (FEA) for robust, reliable design component design
- Material property determination via indentation
- Nano-indentation experimental results
- Conclusions and future work

Objective: Glass-Ceramic Seal Materials for Robust, Reliable, Hermetic Feedthroughs

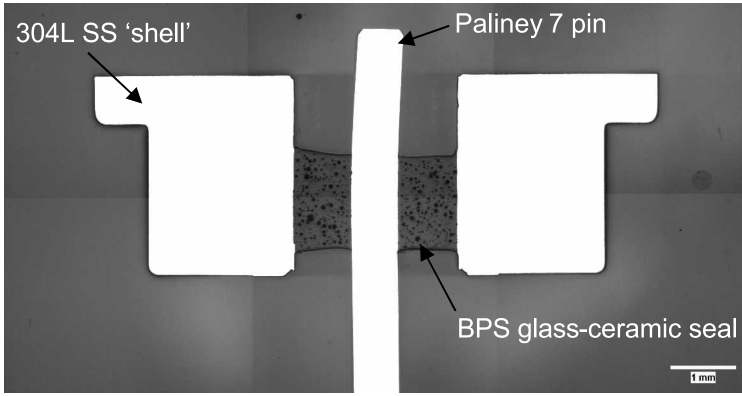
- Feedthrough allows electrical signal to conduct through pin while maintaining atmospheric isolation across the seal

Driving force for developing alternatives to brazed ceramic sealed feedthroughs:

- Reduction of cost
- Reduced vulnerability to handing damage
- Increased productivity: fewer & simpler manufacturing steps
    - 4 major fabrication steps required vs. 10 for brazed ceramic feedthroughs



Optical micrograph of cross-sectioned brazed Alumina hermetic seal



Micrograph of cross sectioned glass-ceramic-to-metal seal

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Formation of Glass-Ceramics (G-C)

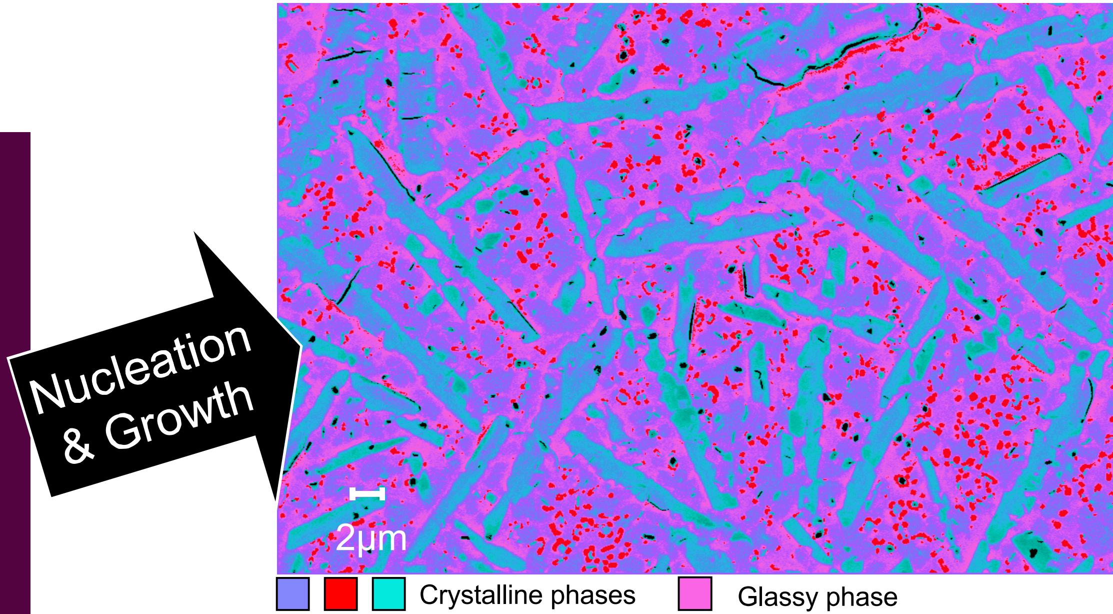
- Definition: *Polycrystalline ceramic material prepared by the controlled bulk crystallization of suitable glasses*

- Complex multiphase microstructure results after nucleation and crystalline growth heat treatment

- **Properties of glass-ceramic are dependent on the proportion of phases present**

Monolithic BPS parent glass

BPS Glass-ceramic



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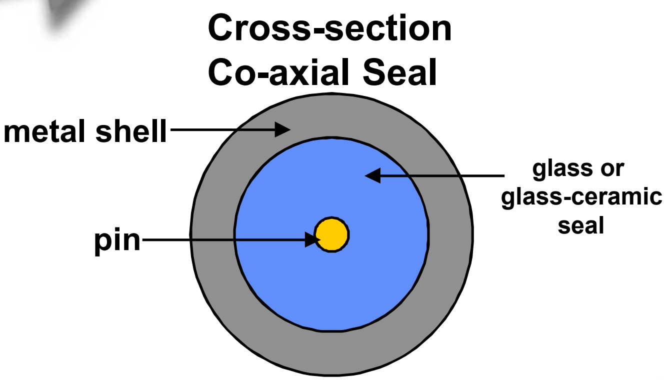
Accurate FEA Stress Prediction for Robust, Reliable, Hermetic Feedthroughs Materials Requires Representative Material Properties



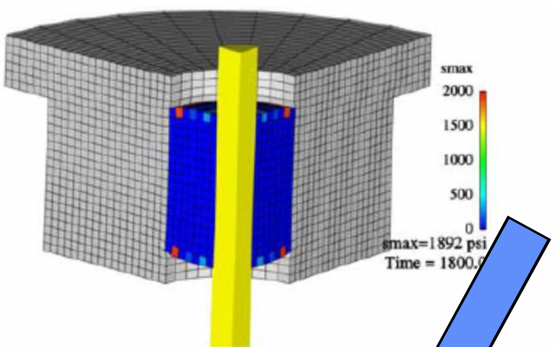
Materials Characterization

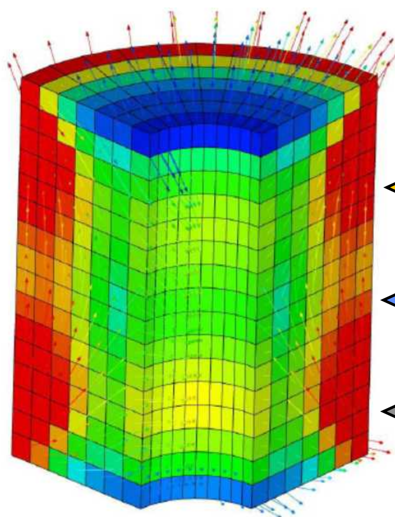
<u>Underlined</u> properties require characterization

Pin: **E, H, ν, ρ**, <u>α</u>, **σ_{ys}, σ_{ult}**

G/G-C: <u>E</u>, <u>ν</u>, <u>ρ</u>, <u>α</u>, <u>T_{set}</u>

Shell: **E, ν, H, ρ**, <u>α</u>, **σ_{ys}, σ_{ult}**





Pin: elastic-plastic

G-C: linear-elastic

Shell: elastic-plastic

Finite Element Stress Analysis for Component

How do the predicted stresses relate to the failure criteria?

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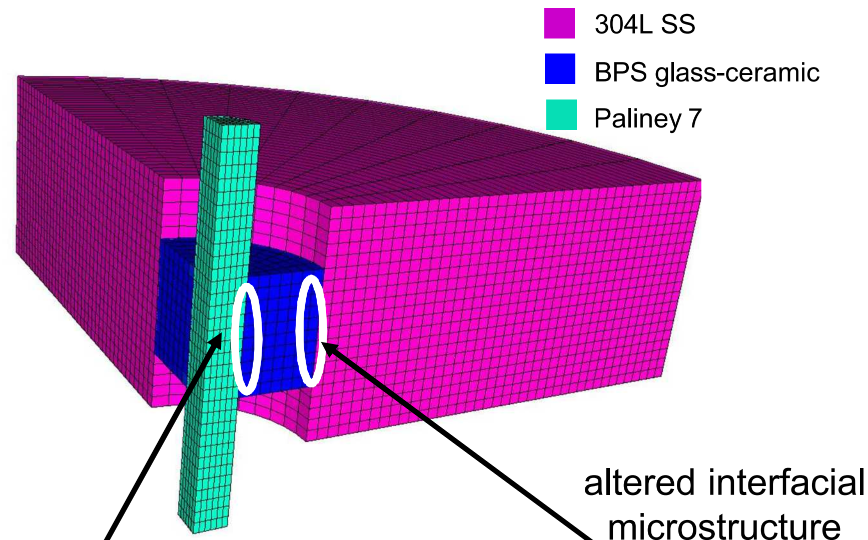
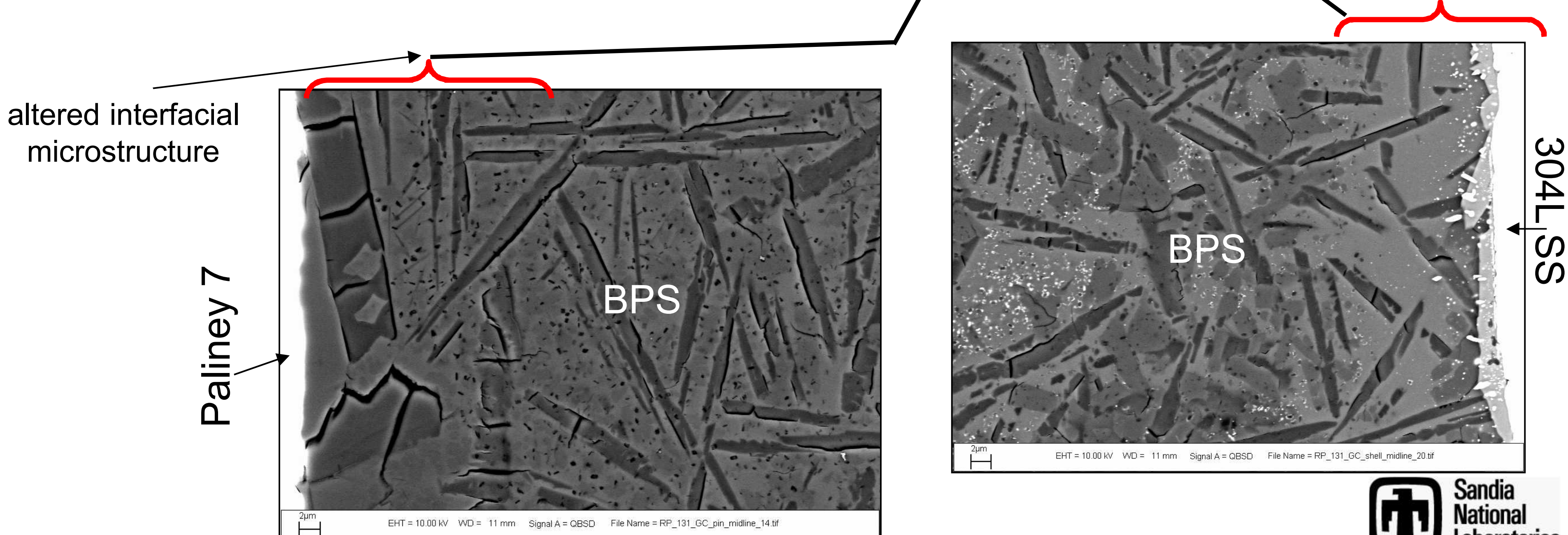
Accurate FEA Stress Analysis Should Account for Complex G-C to Metal Interfaces

- Near interface, phase type and proportion changes
- Failure often occurs at the interface rather than bulk
- **Indentation can be used to determine local material properties in heterogeneous microstructures**





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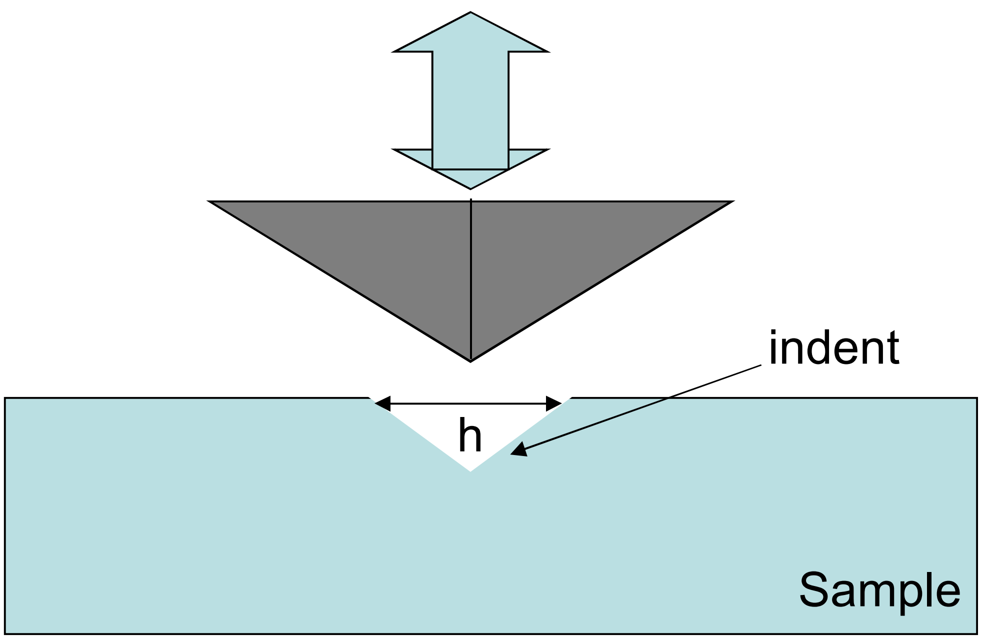
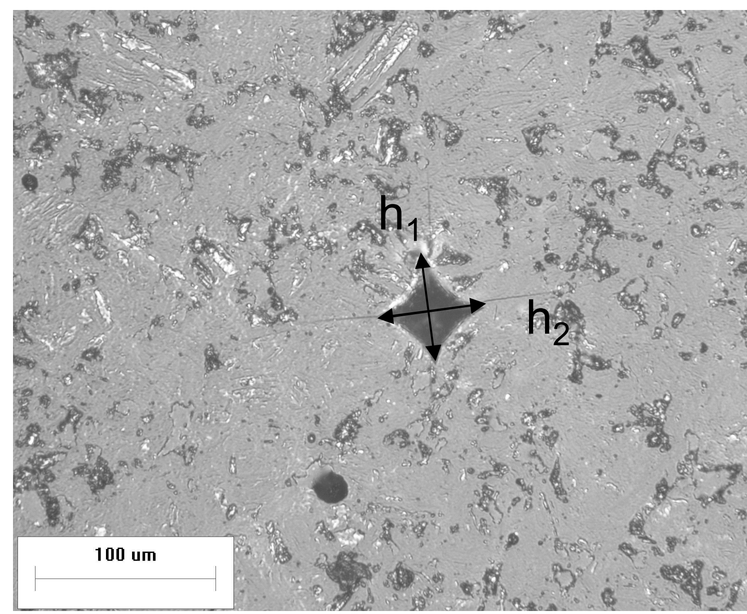
Determination of Interfacial Elastic Properties: Indentation Basics

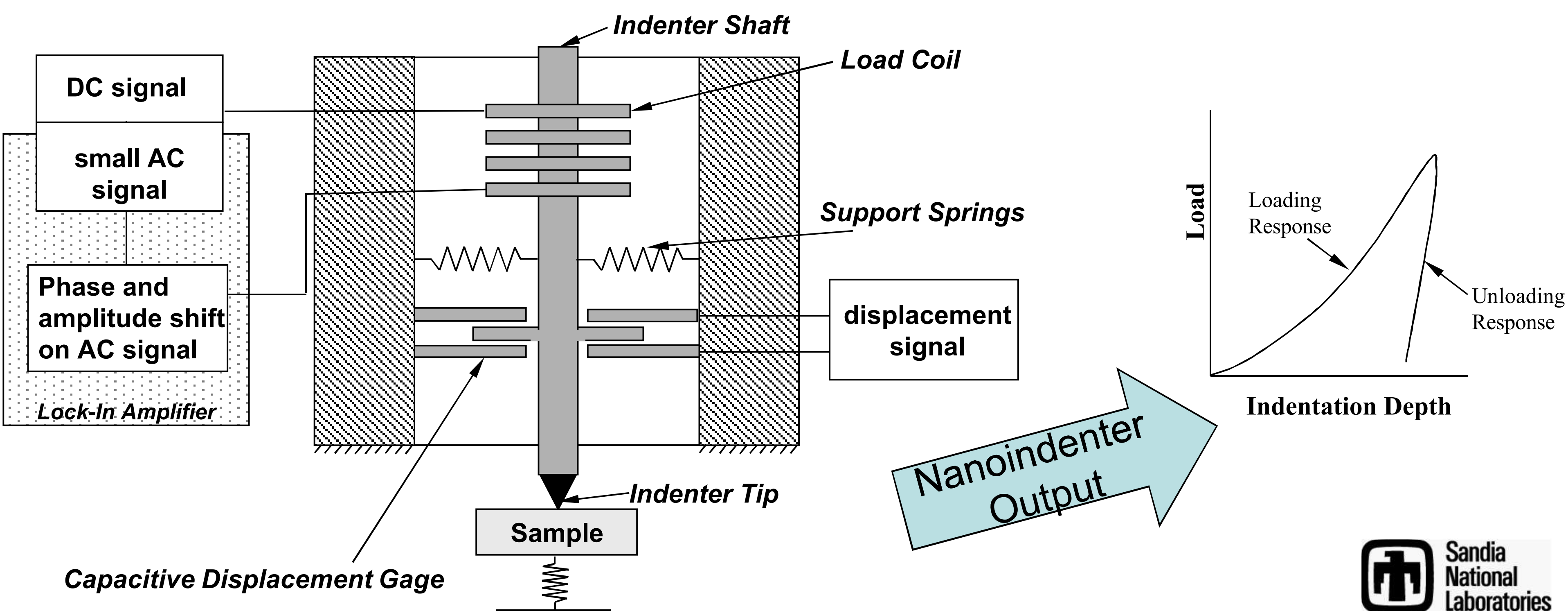
1. Small sharp diamond indenter tip is pressed into the sample surface at applied load, P
2. Tip is retracted leaving a residual indentation
3. Measure indent area (A)
4. From indent area, mechanical properties such as hardness (H) are determined



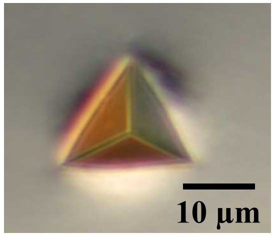


Determination of Interfacial Elastic Properties: Nanoindentation

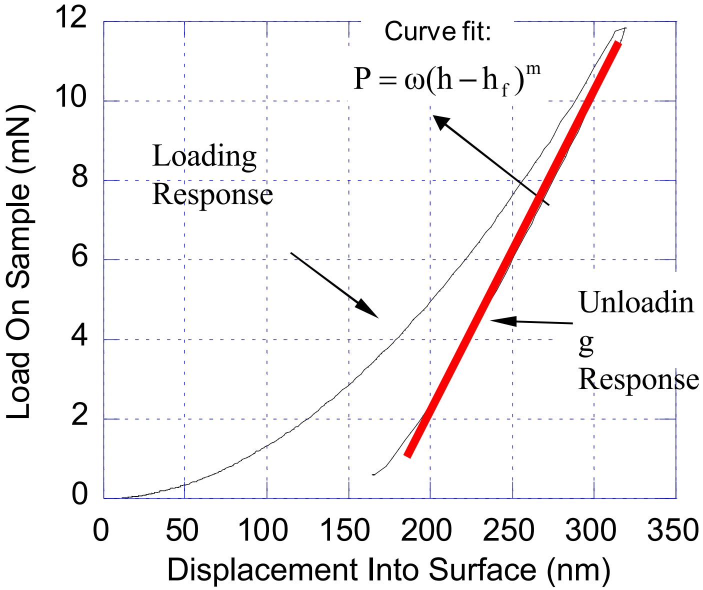
- For very small indentations, accurate determination of A is difficult
- Nano indentation obtains A from load and indent depth sensing
- Determination of A (and subsequently mechanical properties) is achieved without 'seeing' indent



Determining E and H via Nanoindentation



Large Berkovich indent in Ni



Determining Young's Modulus (E) & Hardness (H)

1) Curve fit unloading data using:

$$P = A(h - h_f)^m$$

where:
P=Load on sample (y variable)
h=Displacement into surface (x variable)
ω, m, h_f = curve fitting parameters

2) Obtain unloading stiffness (S) by differentiating P(h) at P_{max}:

$$S = \frac{dP}{dh}(P_{max}) = m\omega(h_{max} - h_f)^{m-1}$$

3.) Determine hardness (H) by determining contact depth, h_c

$$h_c = h_{max} - 0.75\left(\frac{P_{max}}{S}\right)$$

4.) Determine contact area, A_c, for given indenter geometry

$$A_c = 24.504\left(h_c^2\right)$$

5.) Determine Hardness (H)

$$H = \frac{P}{A_c}$$

6.) Determine Young's Modulus (E):

ν = Poisson's Ratio
ß = Indenter tip geometrical factor

$$E = \frac{S}{2\beta\left(1 - \nu^2\right)}\sqrt{\frac{\pi}{A_c}}$$

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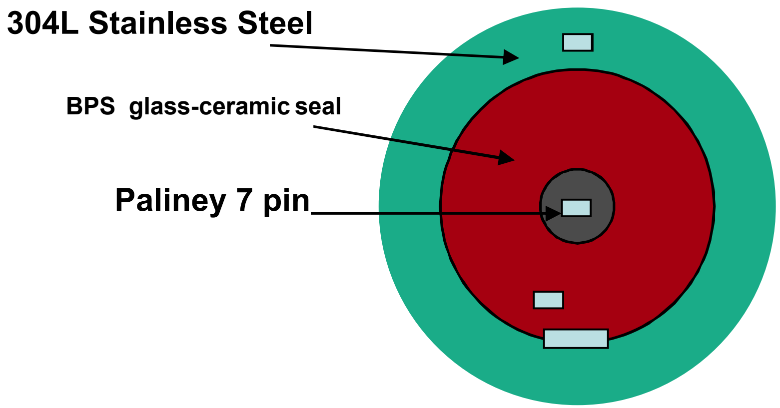
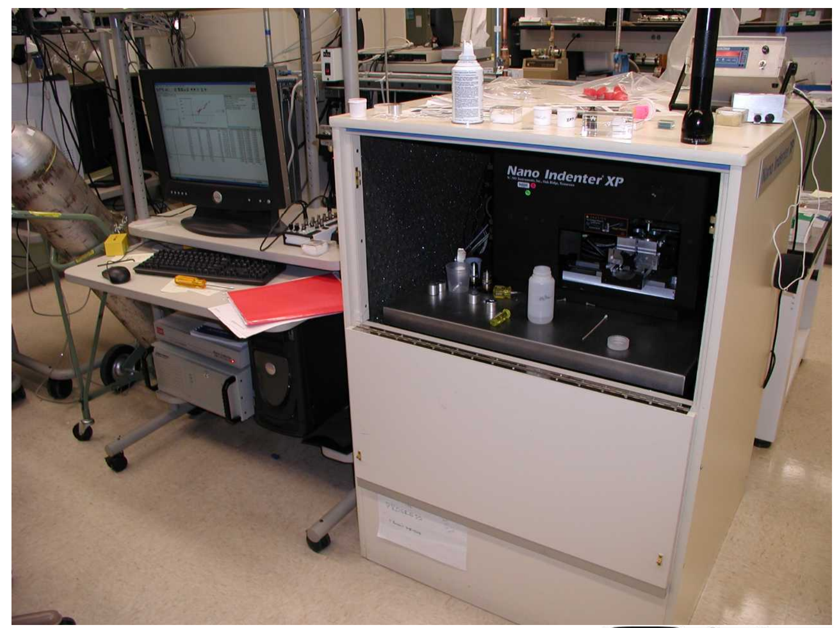
Nanoindentation: Experimental Procedure



transverse cross section

Relative locations of indent arrays

- Nano-Indenter XP (MTS Systems Corp.)
  - Berkovich diamond indenter (~7:1 width:depth)
- Arrays contained at least 10 indentations
- Displacement controlled indents
- Target indent depth: 250 & 1000 nm
- Spacing between indents: 10 & 25 μm



MTS Nano-Indenter XP

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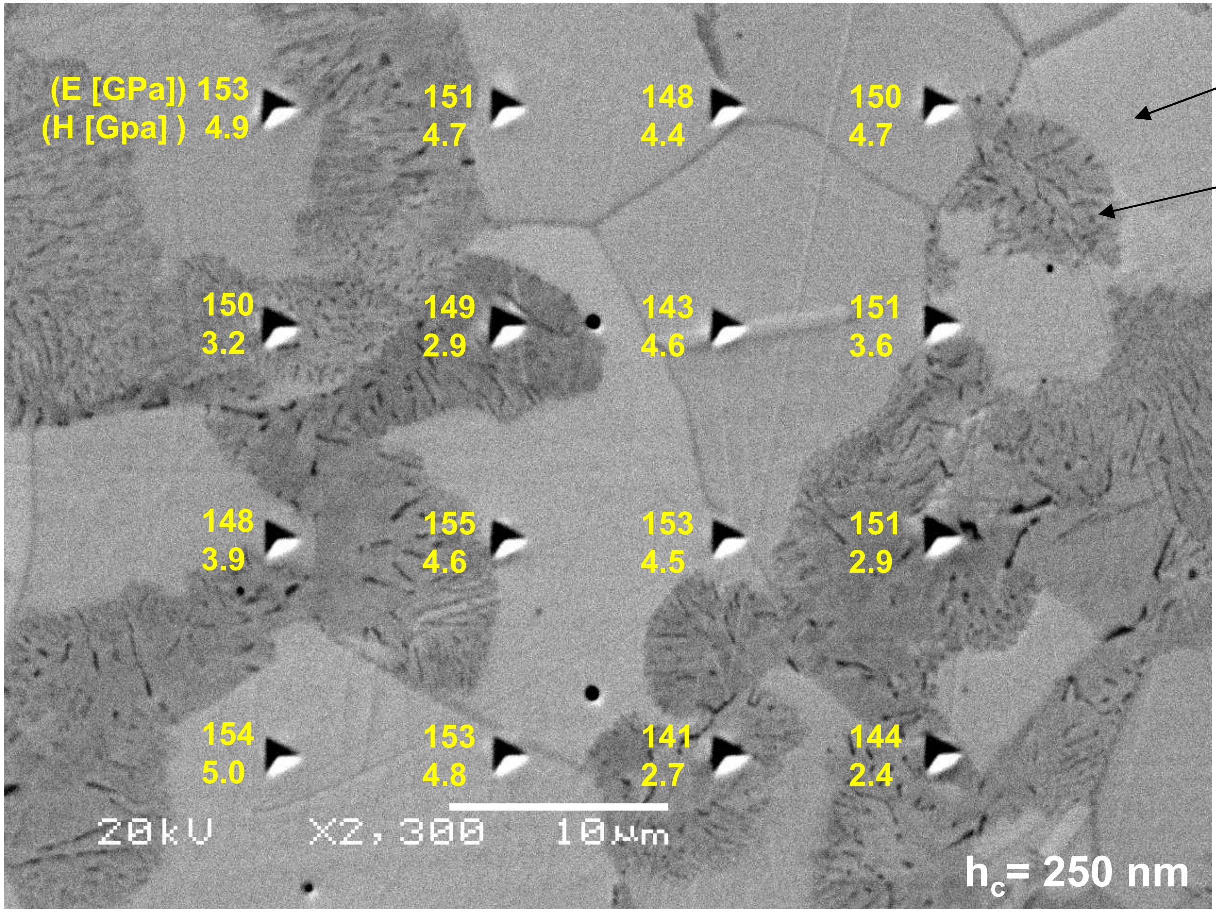
Indentation Results – Bulk Paliney 7

- High dwell time BS SEM micrograph reveals a multiphase Paliney microstructure
- Indent array covers compositionally-varied region of the sample



E_{avg}= 150 ± 4 GPa

E (as per ASTM B540)= 117 GPa

•Discrepancy possibly due to indenter ‘pile-up’

H_{light}= 4.5 ± 0.4 GPa

H_{dark}= 2.8 ± 0.2 GPa

Bulk H (ASTM B540)= 3.2-3.9 GPa

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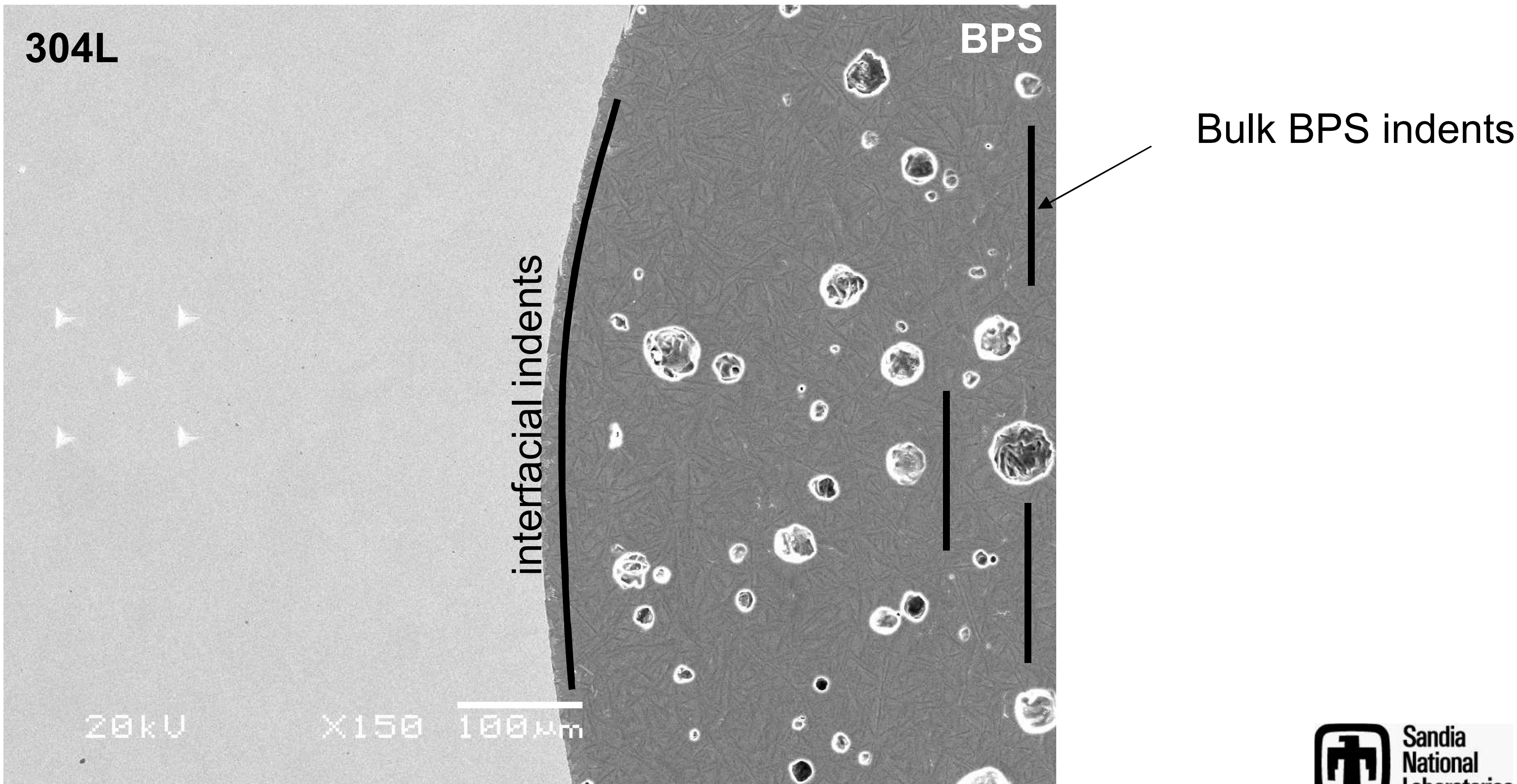
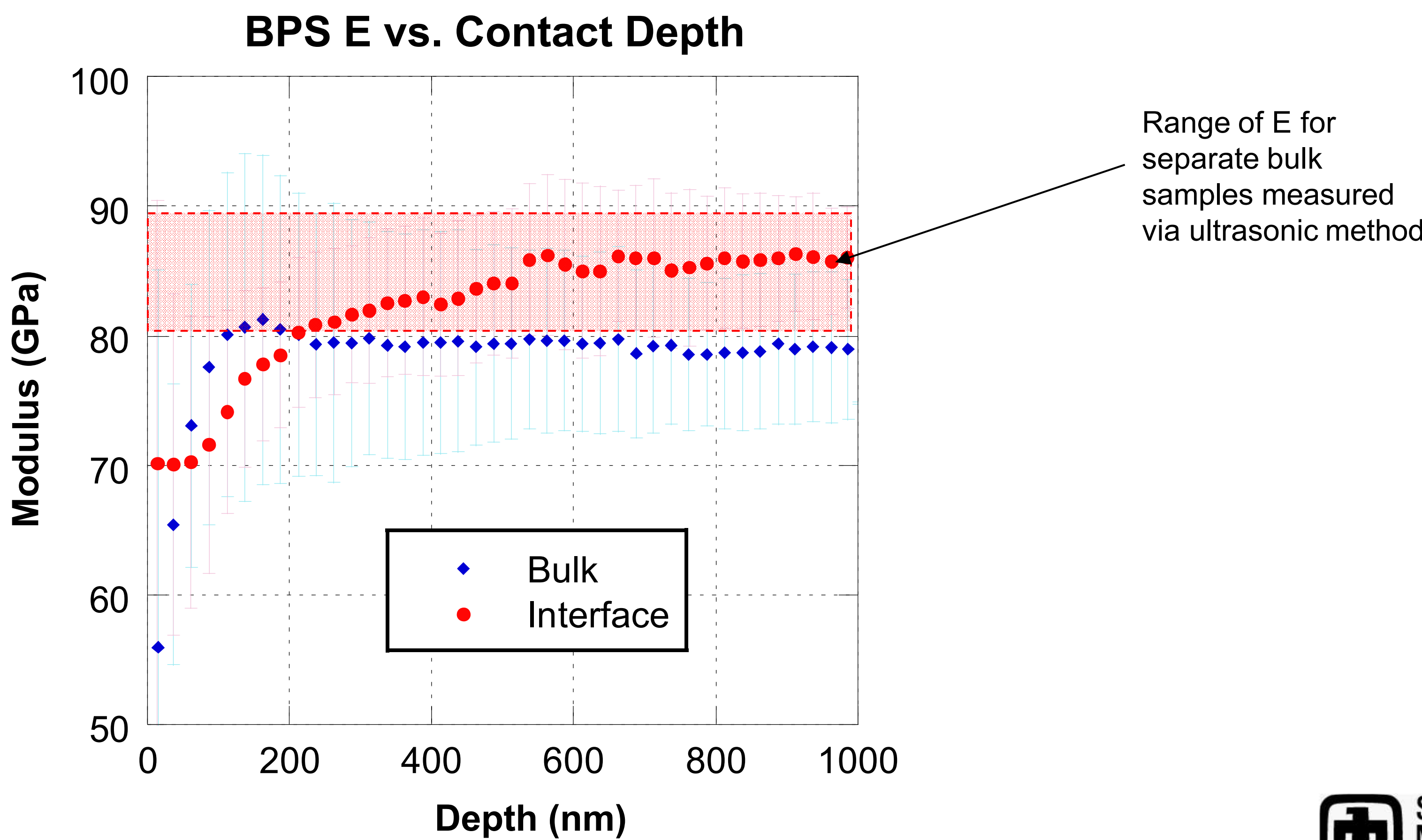
Indentation Results – BPS G-C and 304L SS (h_c= 1μm)

- 1μm deep indents placed in bulk 304L and BPS g-c as well as in BPS g-c ~10μm near interface



Indentation Results – BPS G-C near 304L SS (h_c= 1µm)

- Near interface, increase in E likely due to indent interaction from 304L SS
- E away and near interface corresponds to value measured for separate bulk samples via ultrasonic resonance

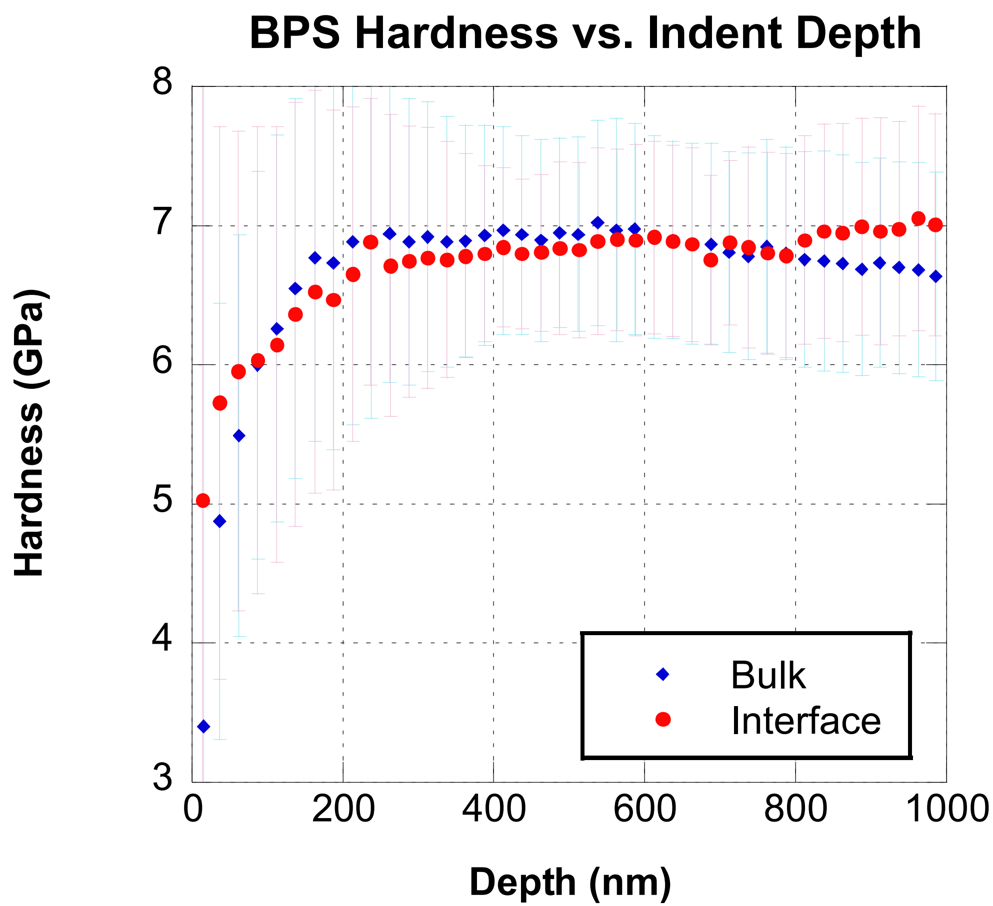


Indentation Results – BPS G-C near 304L SS (hc= 1µm)

•Slight increase in H near interface may be interaction of residual stress near interface

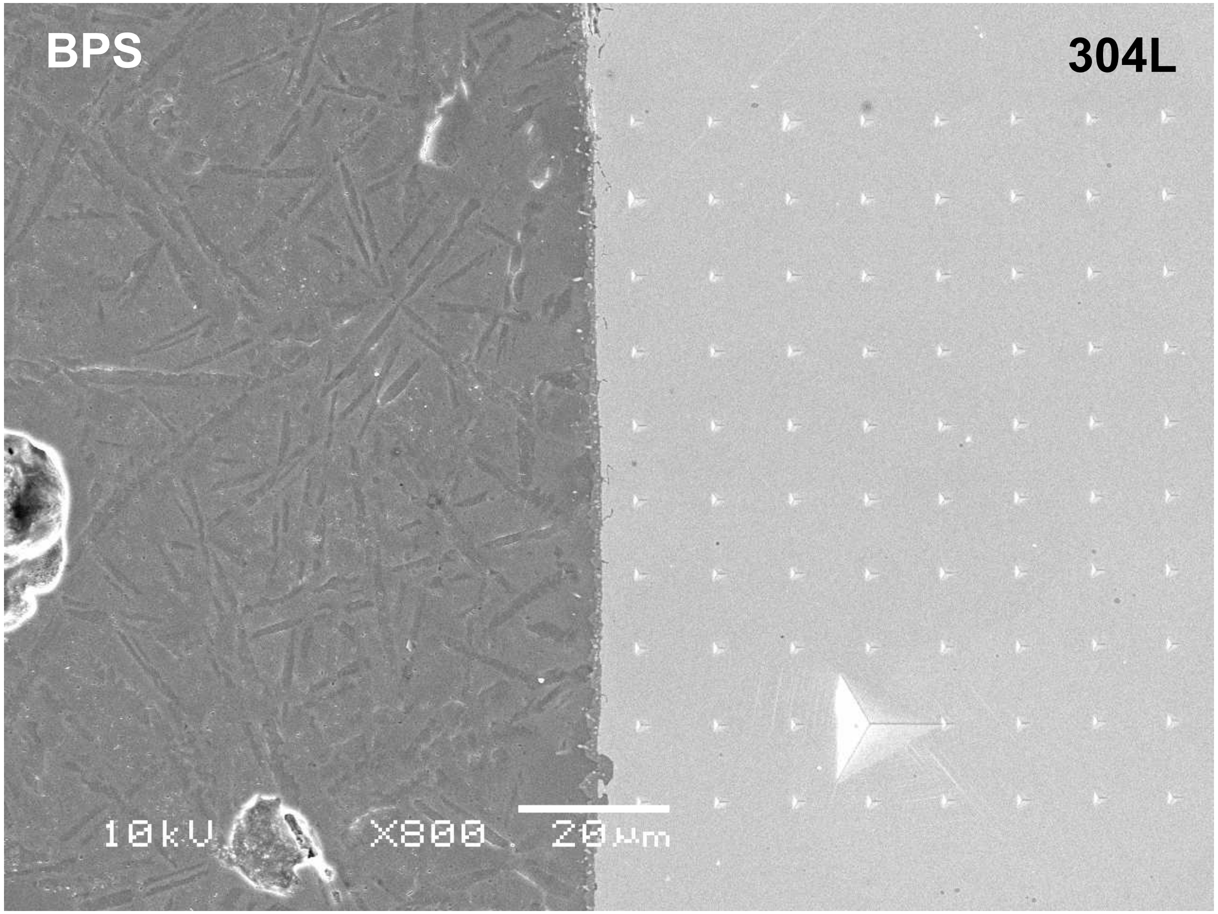
•Expected increase in H as h_c increases

  •No formation of plastic zone at very low h_c



Indentation Results – BPS Glass-Ceramic (h_c= 250nm)

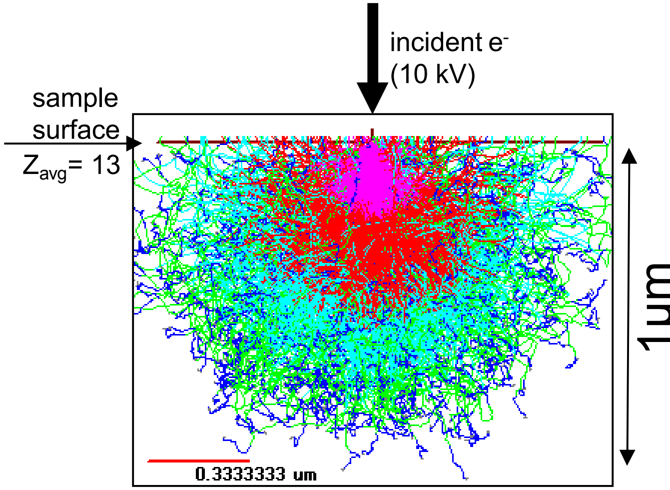
- Shallow indents (hc= 250nm) placed in BPS glass ceramic from interface to bulk
  - Shallow indents sample less material volume therefore results are more sensitive to localized material variances
- Varied microstructure of BPS g-c coupled with shallow indents leads to wide scatter in indentation results
  - Difficulties encountered correlating results with indent position on sample



<u>BPS g-c</u>

E_{avg}= 76 ± 11 GPa

H_{avg}= 7.1 ± 0.9 GPa



Electron interaction volume effects obscures g-c indents

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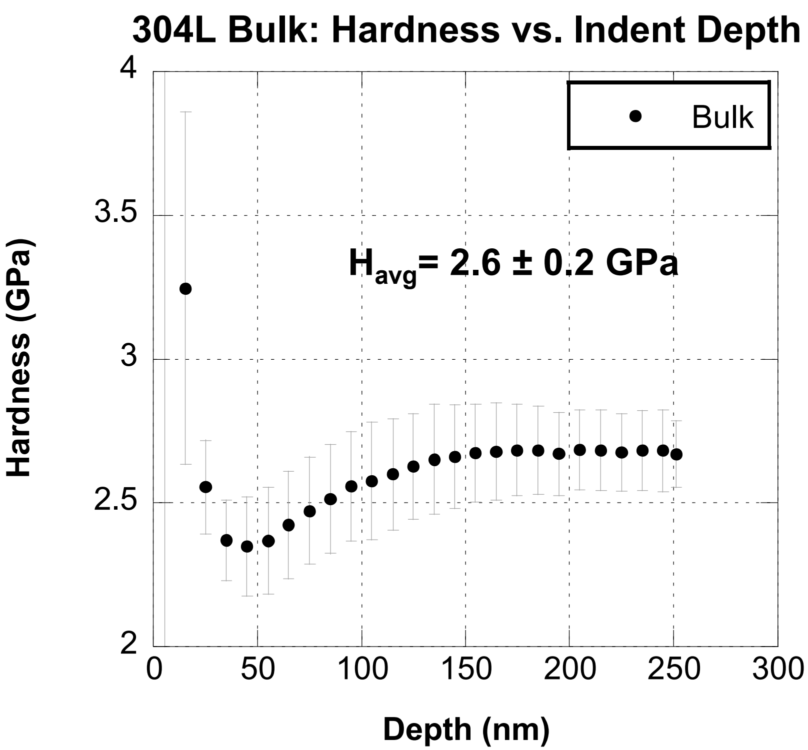
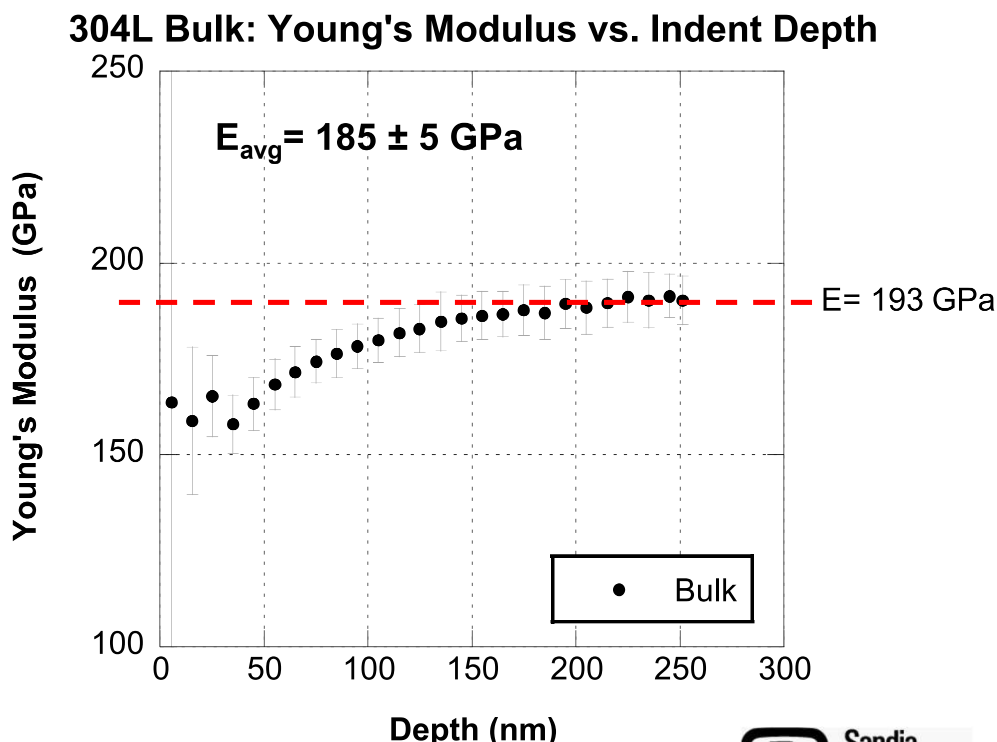
Indentation Results – Bulk 304L SS (hc= 250nm)

- Bulk E corresponds to nano-indentation-measured values
- Increase in hardness of stainless steel <50µm from surface
  - Localized increase in H possibly due to passivation layer
  - Possible tip sharpness/calibration issue





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Conclusions

- Paliney 7
  - Nano-indentation was able to differentiate dissimilar phases based on hardness results
  - Quoted value of E is 22% lower than measured possibly due to indenter ‘pile-up’
- BPS g-c
  - E measured away from interface within range of past ultrasonic bulk measurements (1 μm indents)
  - Slight increase in H near interface possibly due to slight interfacial stress (1 μm indents)
  - 250 nm deep indents difficult to resolve and correlate to results
- 304L SS
  - Measured E corresponds to quoted E of 193 GPa
  - Measured hardness value significantly higher than typical hardness values (2.6 GPa measured vs. 1.9 GPa expected)

Nano-indentation Future Work

- Refinement of indentation experimental procedure to reduce variability in results
  - *Use of sharper tip indenter geometries*
  - *Use of different indent arrays configurations to minimize possible external effects*
- Use of very low accelerating voltage scanning electron microscopy to resolve shallow indents in BPS g-c
  - *Will result in better correlation of data to microstructural features*
- Use other diamond tip geometries to induce cracking to verify residual stress states at interfaces

Questions?