



Diffusion Couple Approach to Alloy Development in Stainless Steels

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Applying the Diffusion Multiple Approach to Cladding Steels

How can we increase the operating temperature and radiation resistance of structural alloys?

Work by Klueh (ORNL) suggests that refractory metals will increase high temperature strength of fuel pin cladding...

316L stainless steel

- Used in thermal/fast reactor structural components
- Austenitic (FCC)
- Well-studied system
- Readily available

HT9 stainless steel

- Prototypical cladding steel in current use for fast reactor cladding
- Less widely available

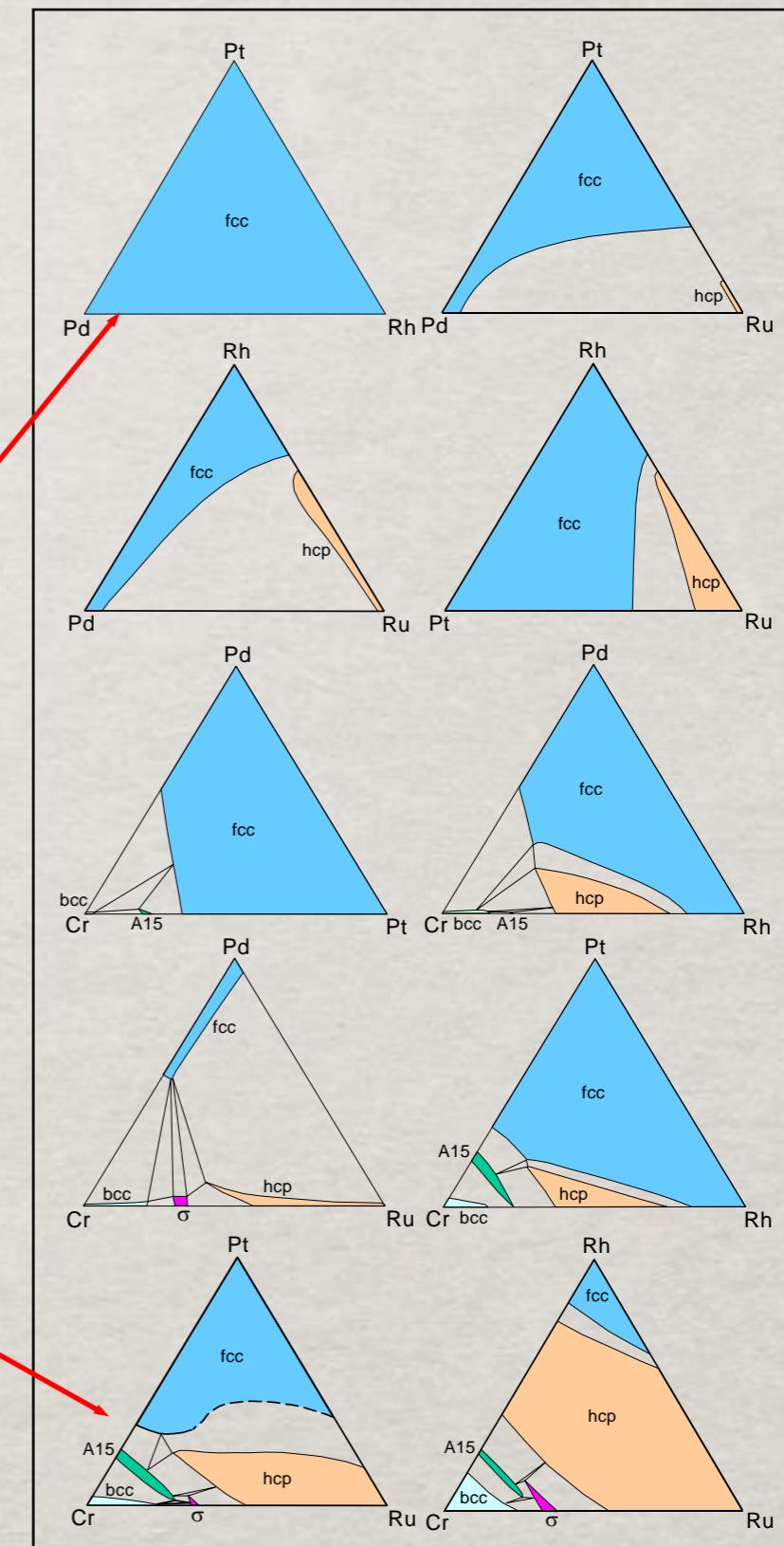
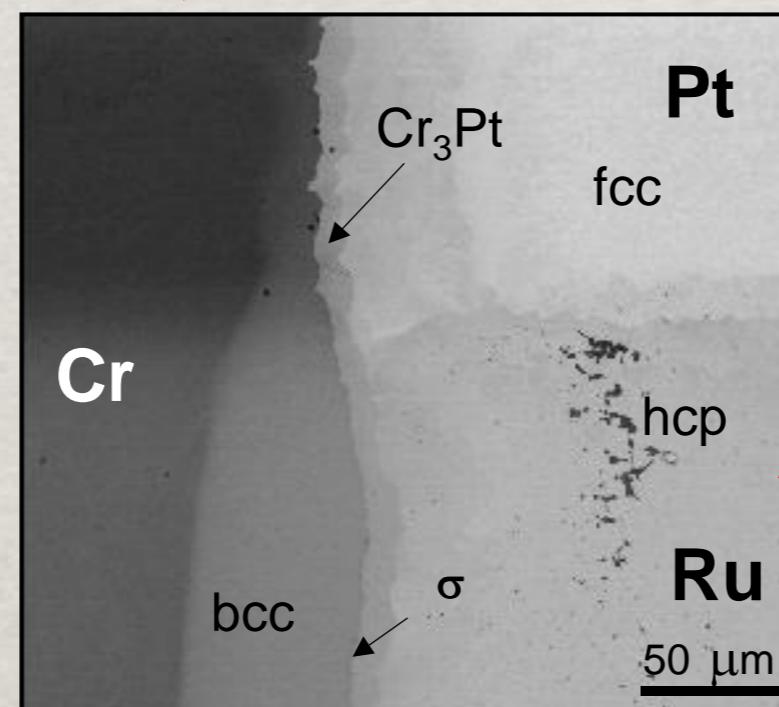
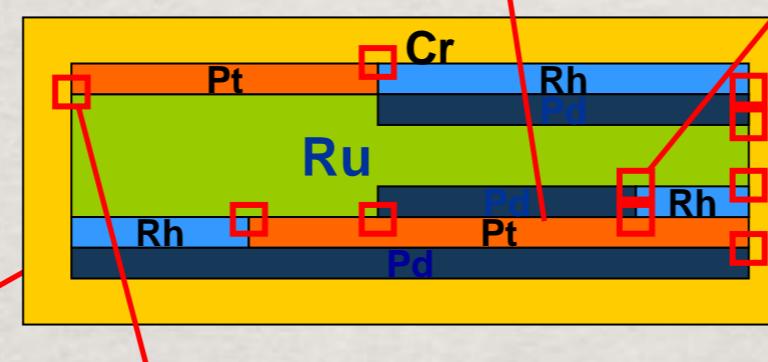
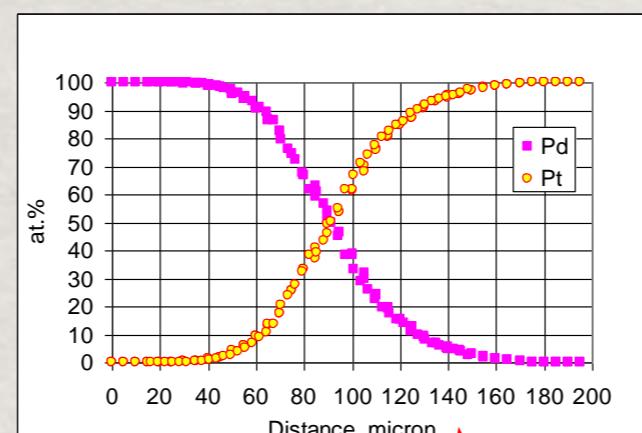
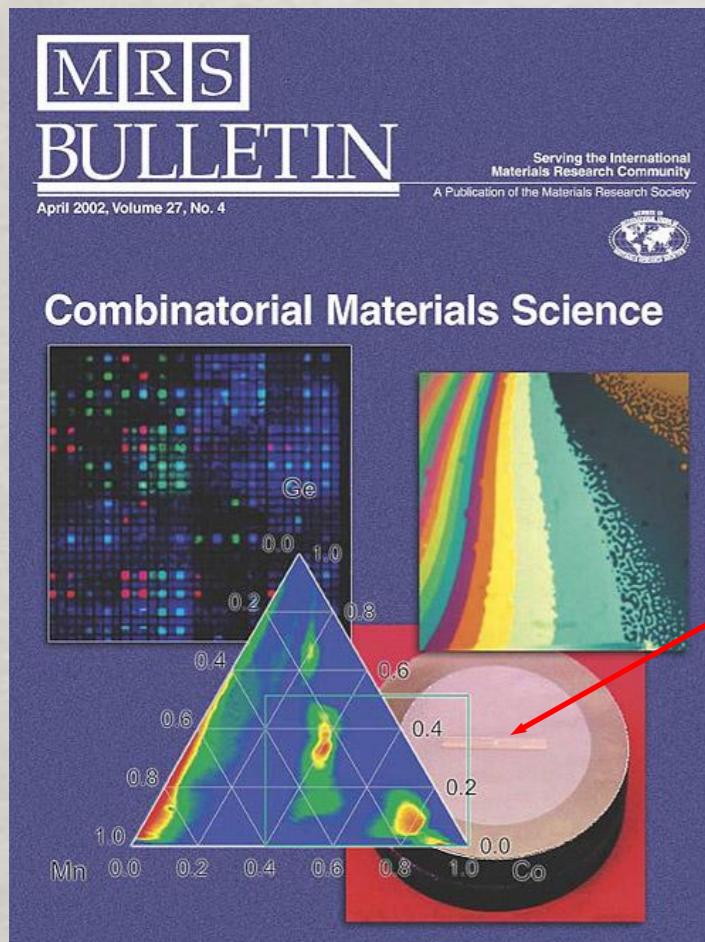
Changes in refractory level—will lead to changes in:

Strength
Phase stability/Fracture toughness
Elastic modulus

- Higher temperatures
- Longer burns
- Exotic chemistries
- Certification challenges

Alloy design programs can be expensive and take a long time...how can we reduce the composition space that we need to investigate?

Diffusion Multiple Approach to Rapid Alloy Development

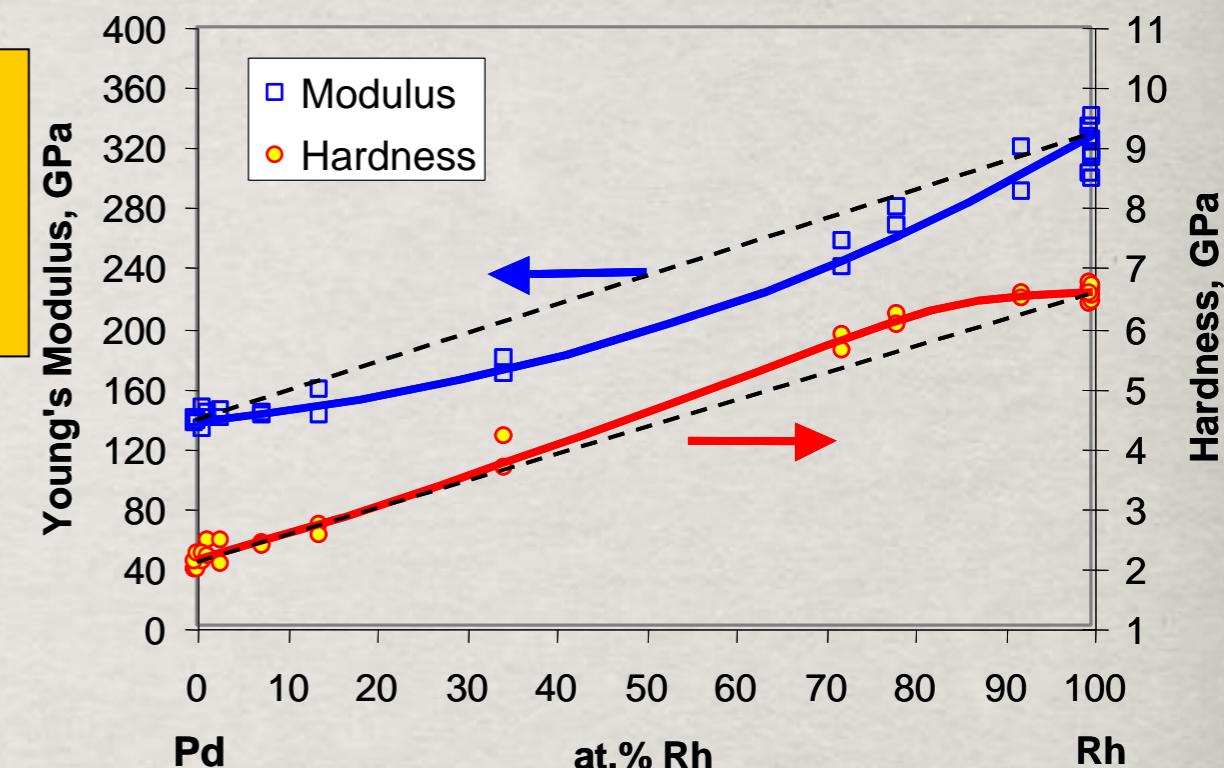
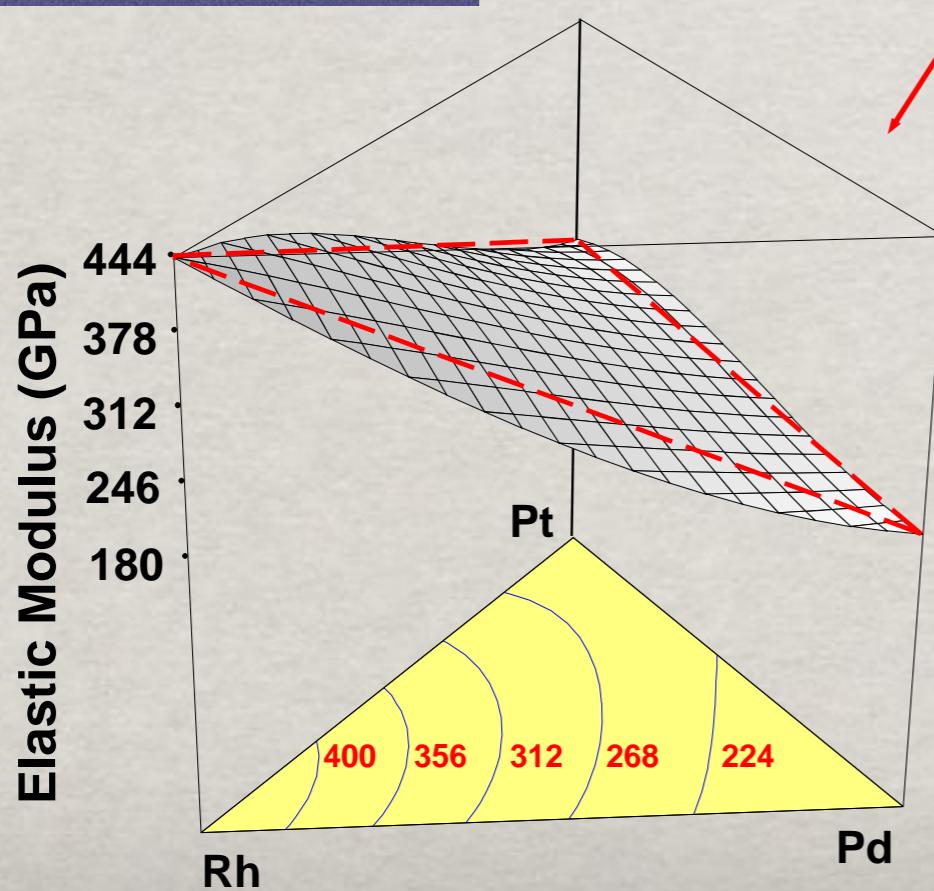
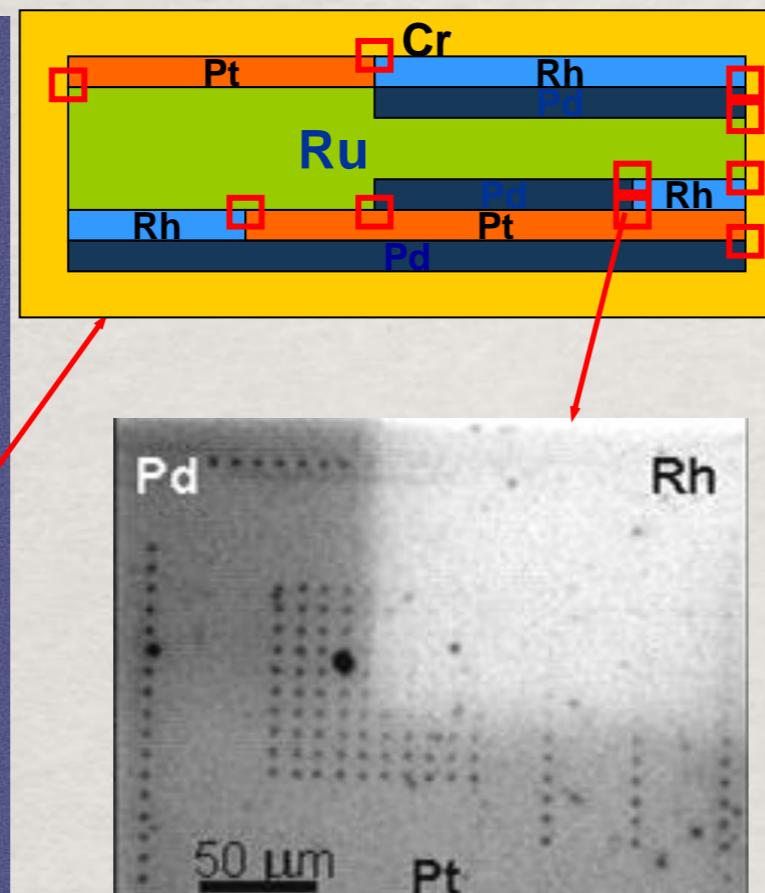
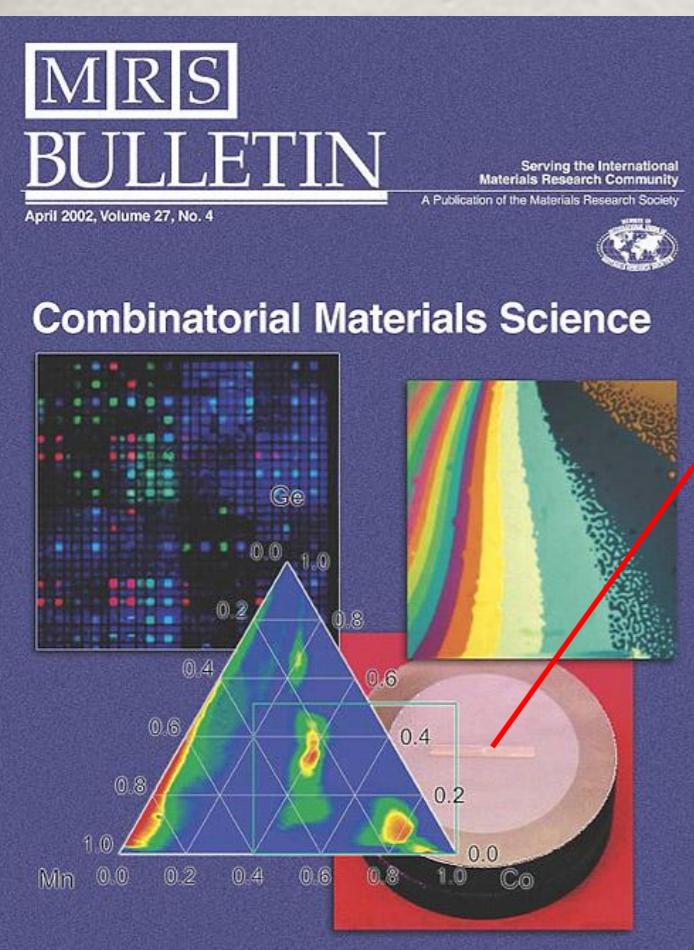


10 Phase diagrams from a single sample

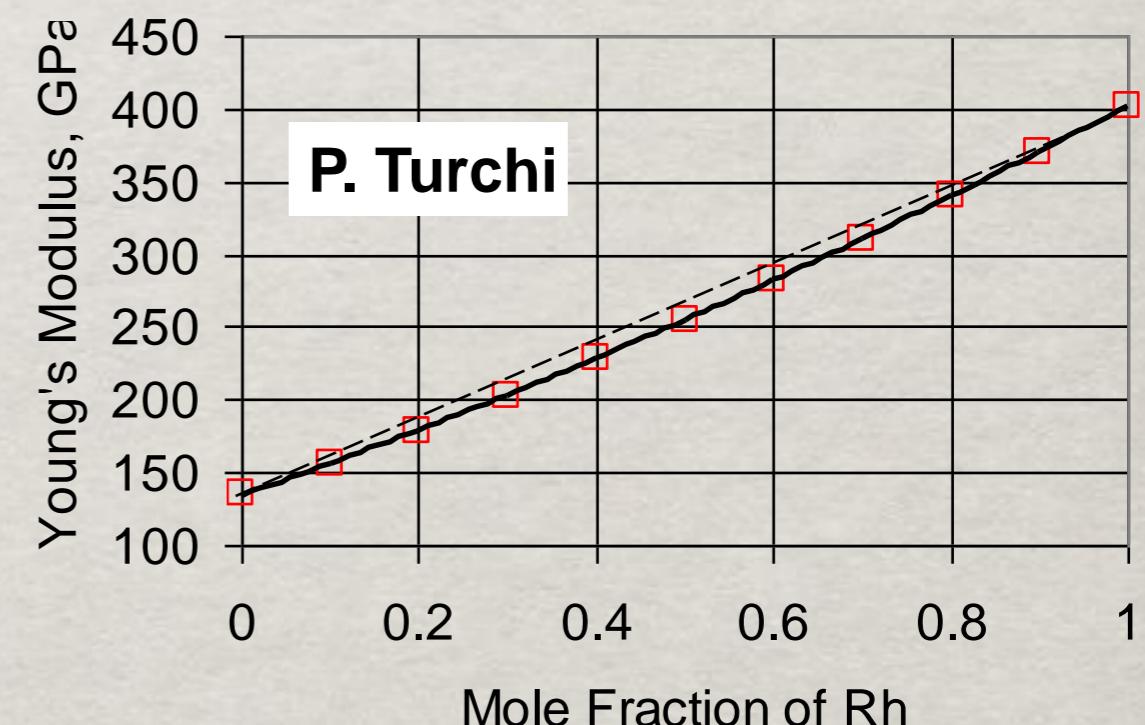
Slide courtesy, J.C. Zhao,
Ohio State University

Zhao, Jackson, Peluso, Brewer: JOM 2002;54(7):42.

Hardness and Modulus



Check against theoretical predictions

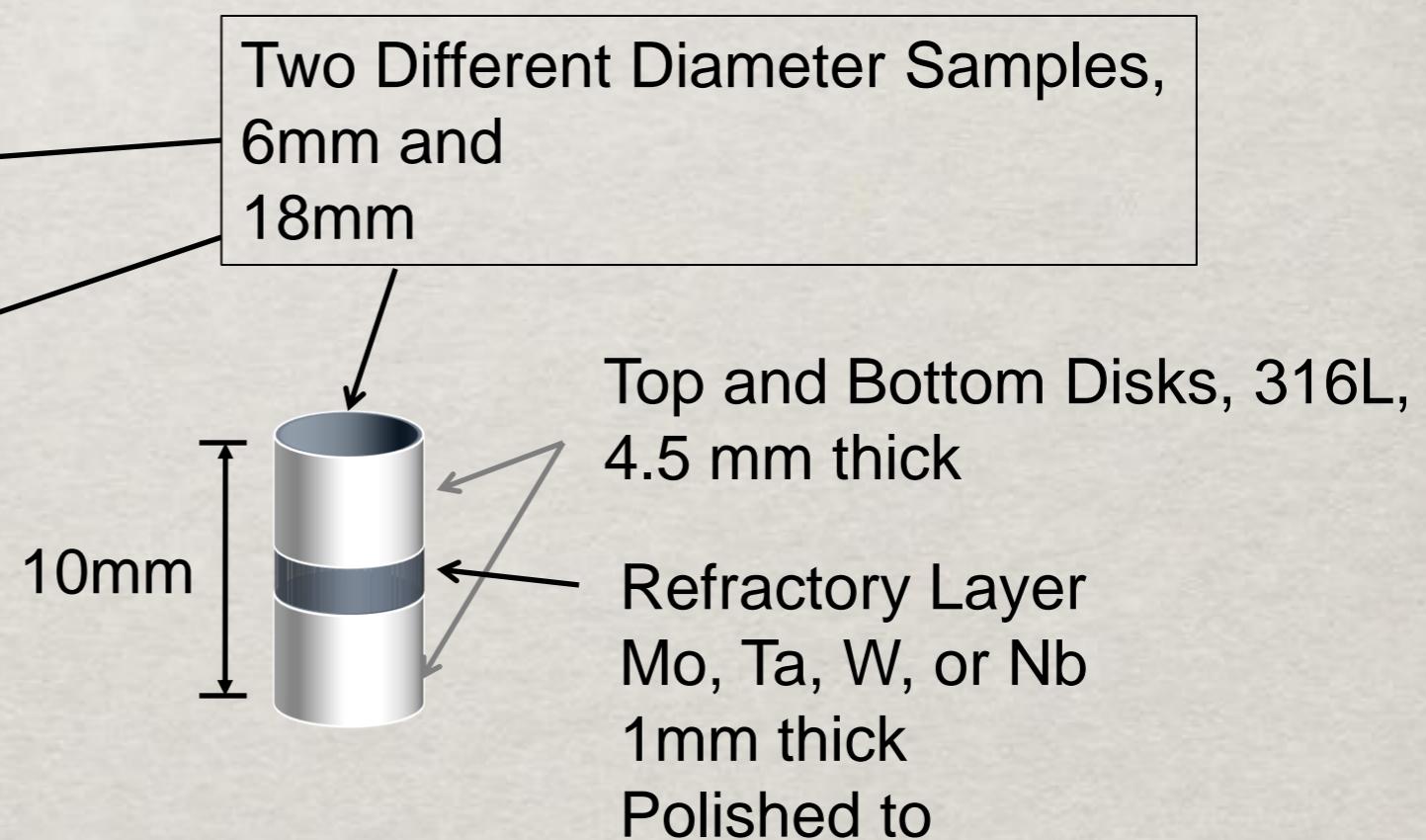
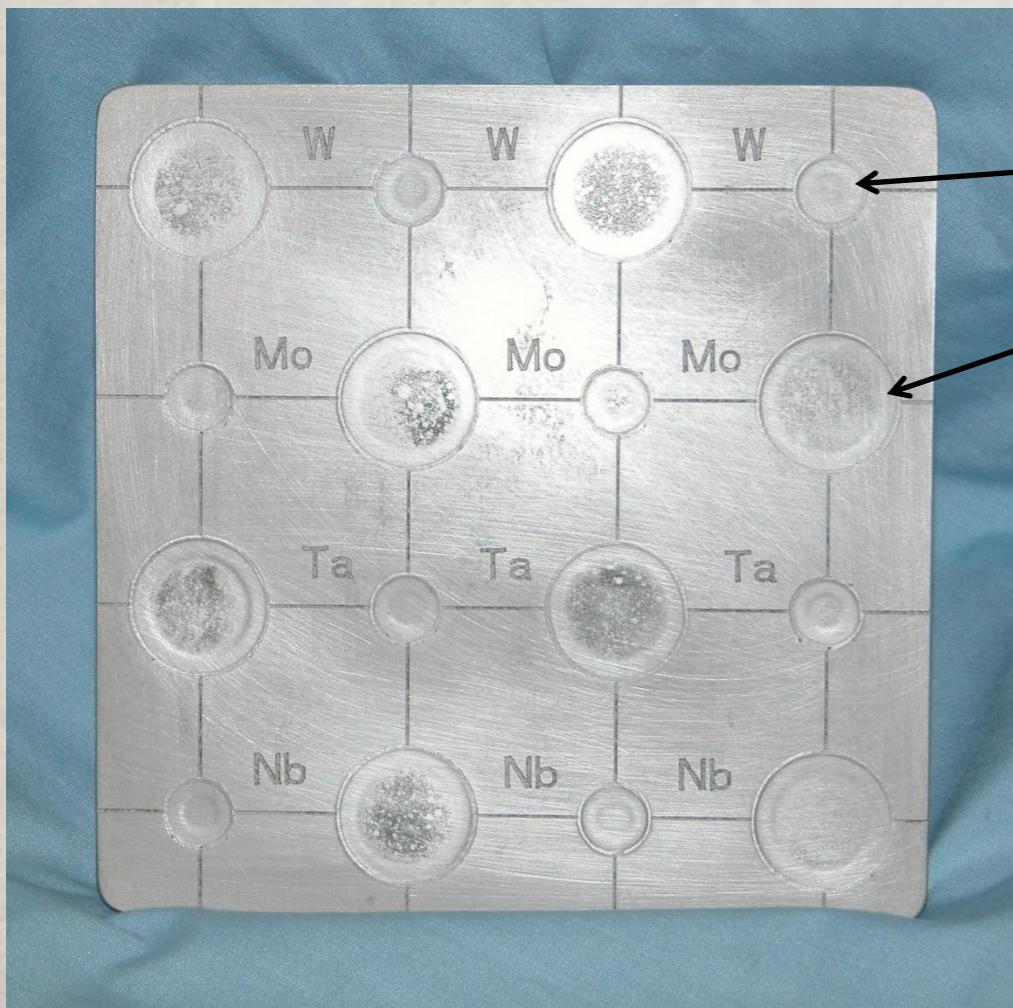


Slide courtesy, J.C. Zhao,
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Processing 316 SS/Refractory Diffusion COUPLES

Fe-0.019C-1.321Mn-0.024P-0.008S-0.610Si-16.34Cr-10.03Ni-2.03Mo-0.025N

- 2 Different Disk Stackups for each material combination
 - 316L (surface ground) to W, Mo, Ta, or Nb – Metallographic finish
- Disks assembled in 316L base plate and welded closed in vacuum, 10-5
- The disks were then bonded via hot isostatic pressing (HIP) at 1100C for 2 hours @ 100 MPa
- Entire plate was then post-HIP heat treated at 1100C for 400 hours
- Samples were then sectioned from plate via EDM

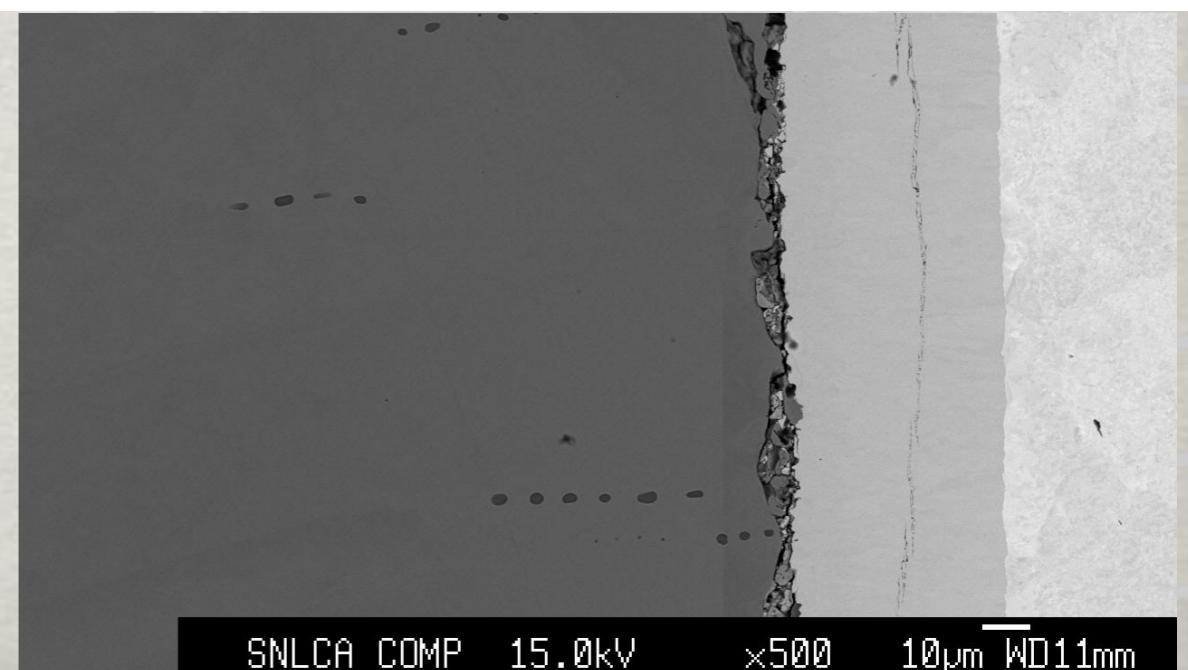
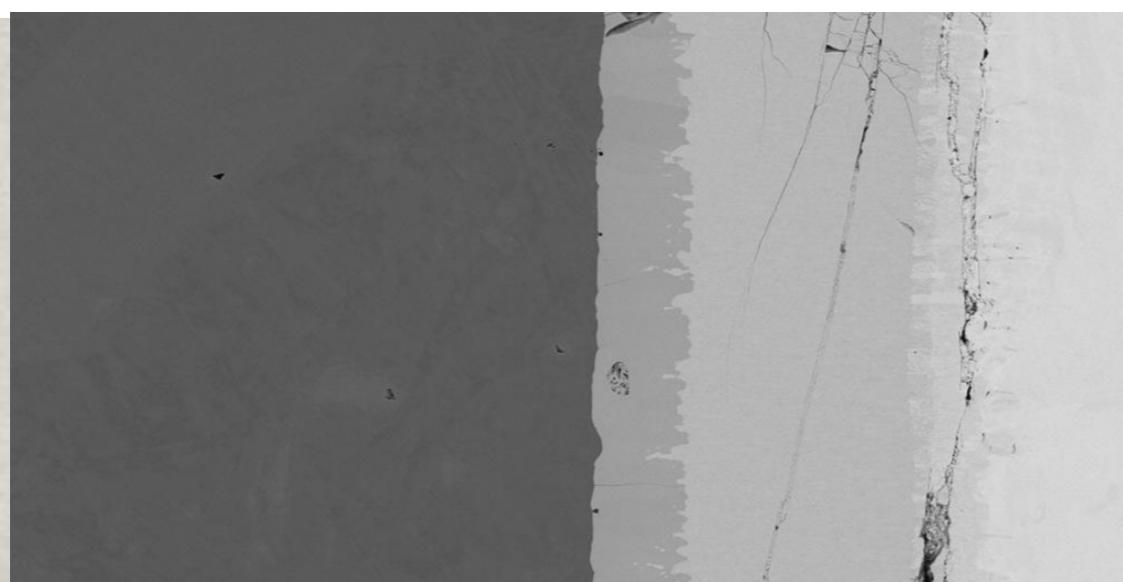
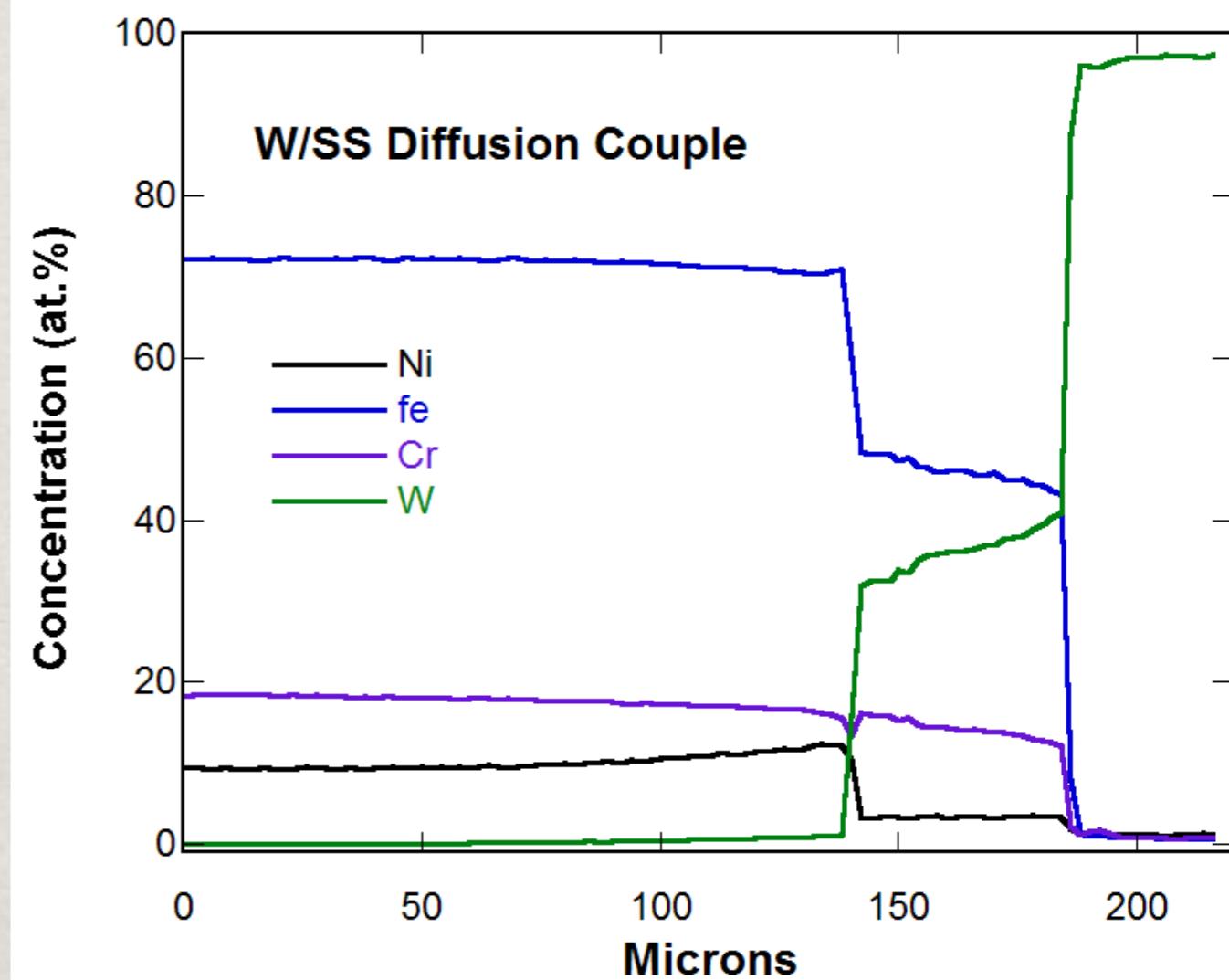
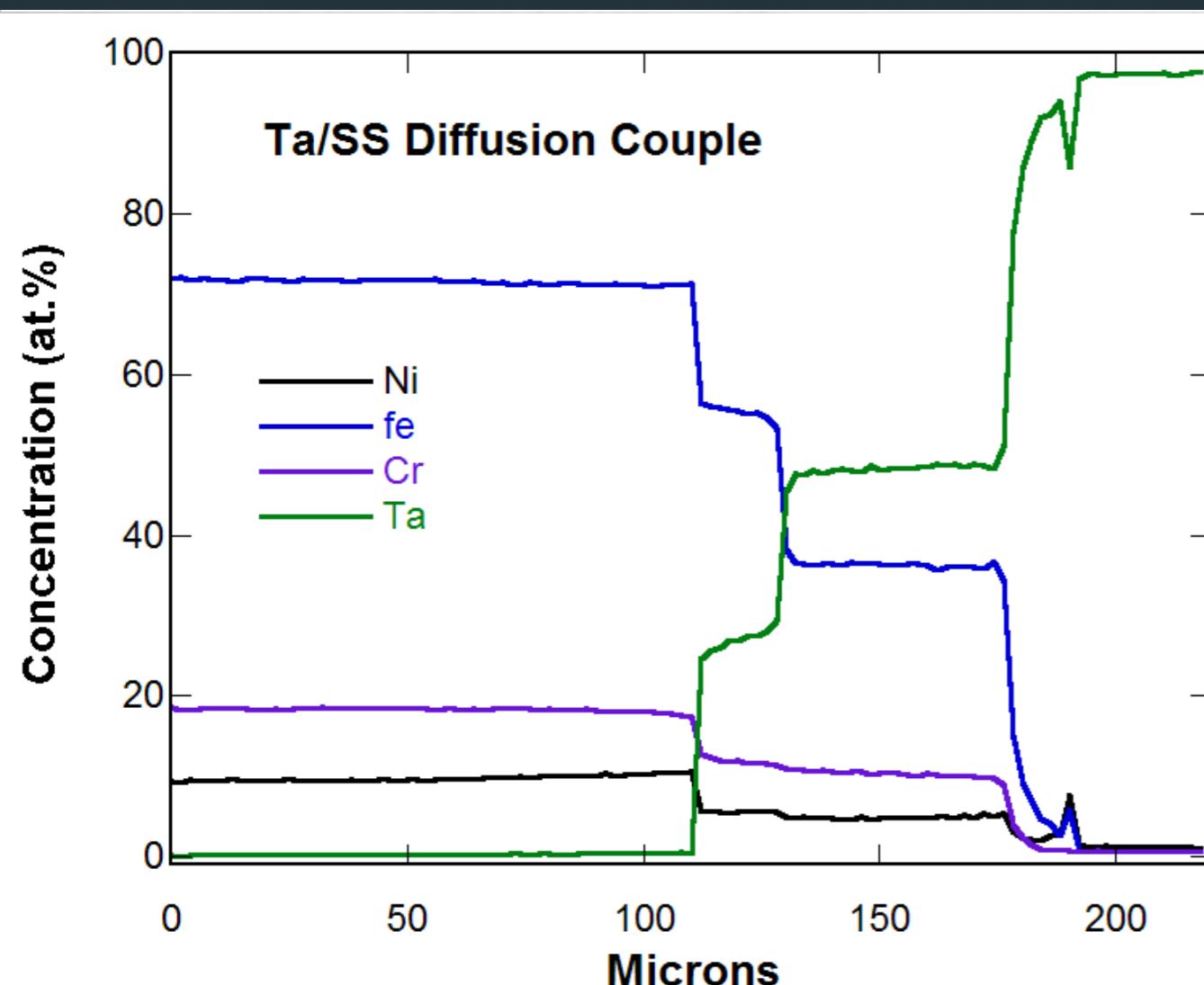


Two Different Diameter Samples,
6mm and
18mm

Top and Bottom Disks, 316L,
4.5 mm thick

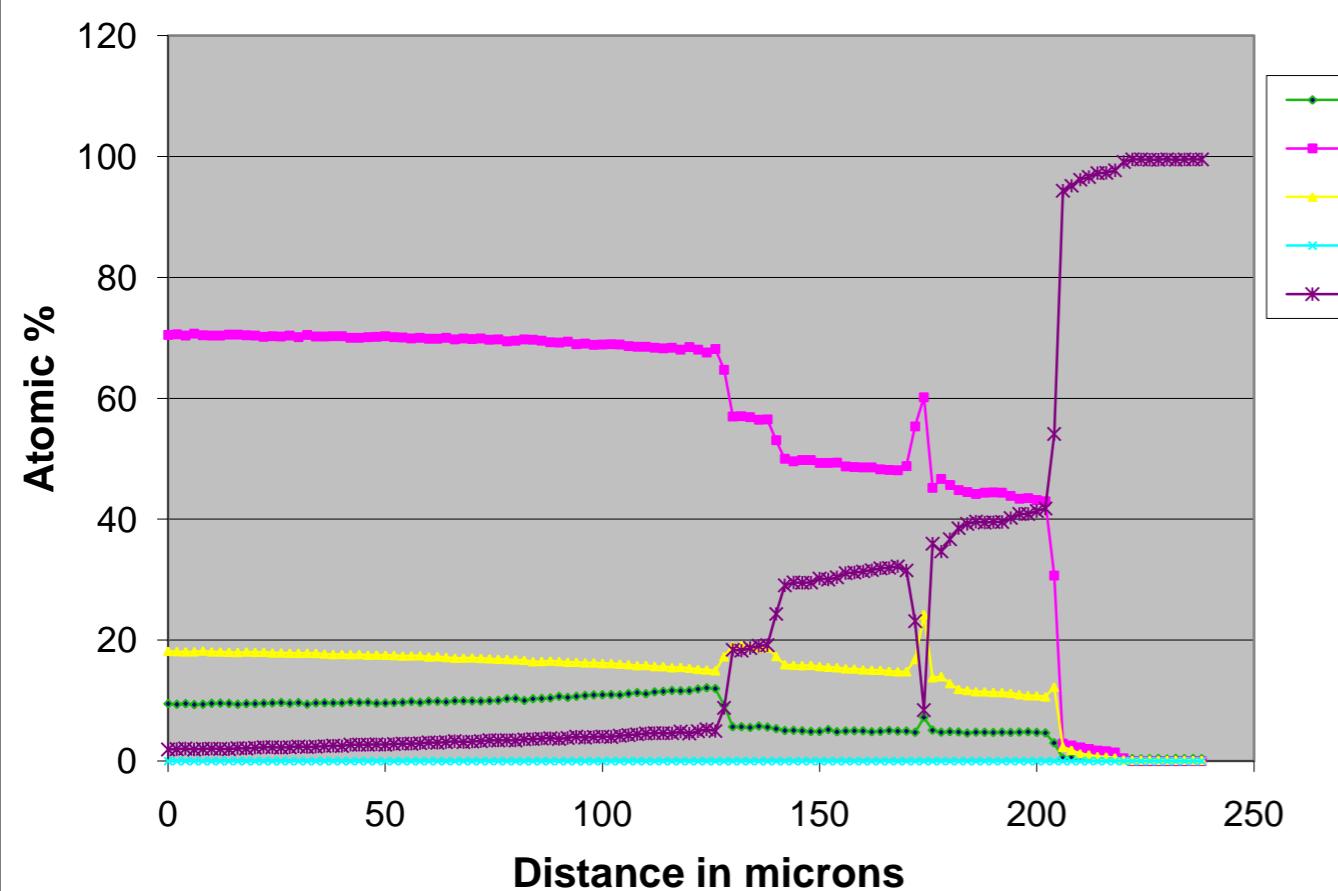
Refractory Layer
Mo, Ta, W, or Nb
1mm thick
Polished to

Composition Profiles across Diffusion Couples: W and Ta

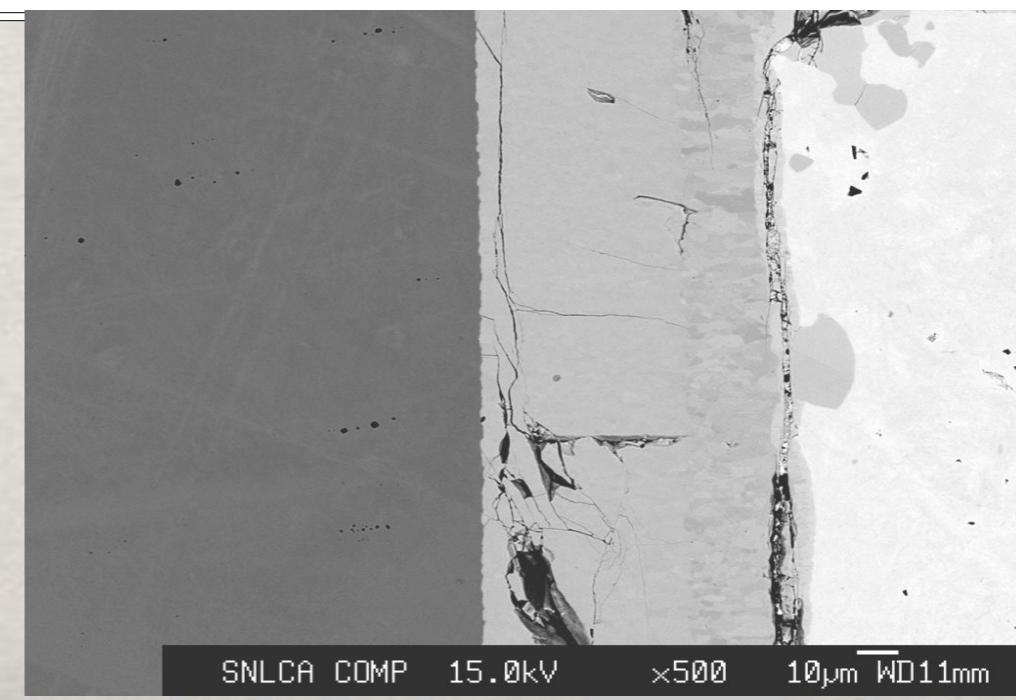
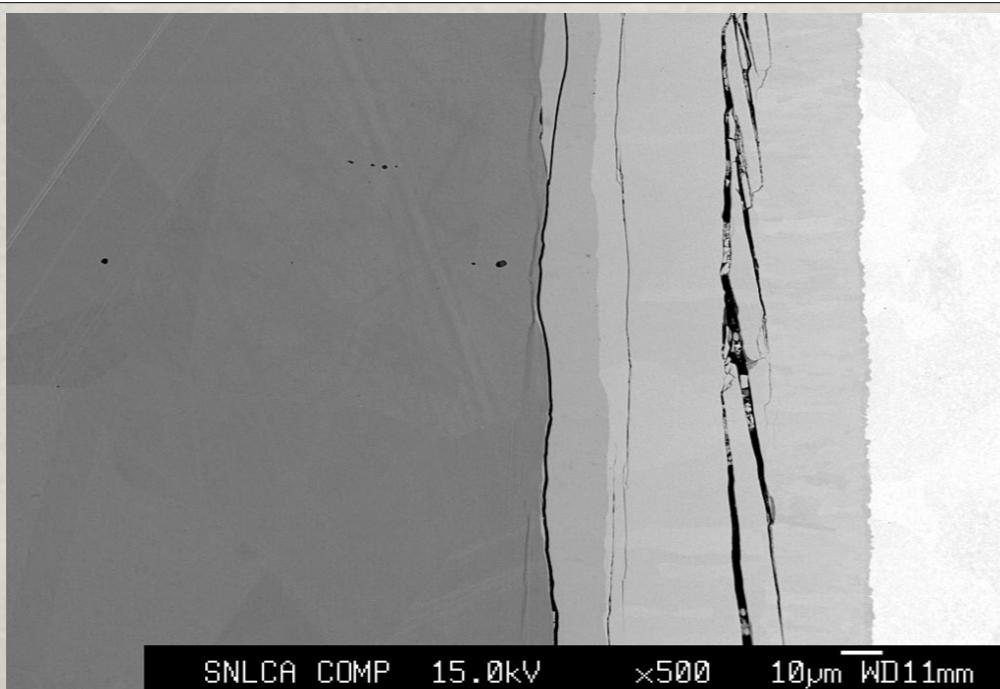
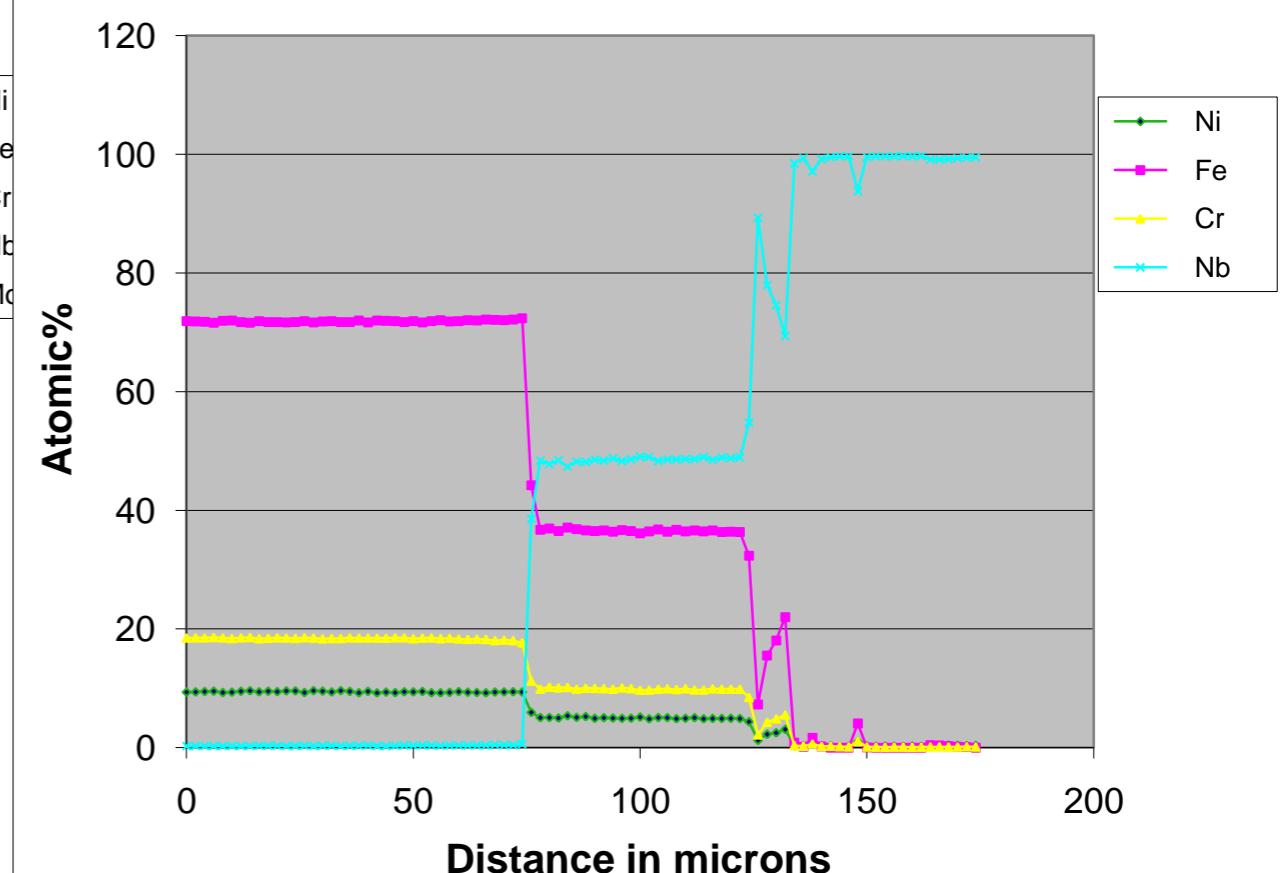


Composition Profiles across Diffusion Couples: Mo and Nb

WDS Mo-316SS Fusion Couple



WDS Nb-316SS Fusion Couple



Nanoindentation Measurement of Hardness and Young's Modulus

Using the Agilent G200 instrument.

- Indentations made in CSM mode (XP head)

- 45 Hz
- 2nm amplitude
- Strain rate =0.05
- Maximum depth 200nm
- Modulus and hardness average from 100-200nm
- Poisson's value of 0.3 used for calculation of modulus.
- Calibration of tip shape using ISO fused silica approach.

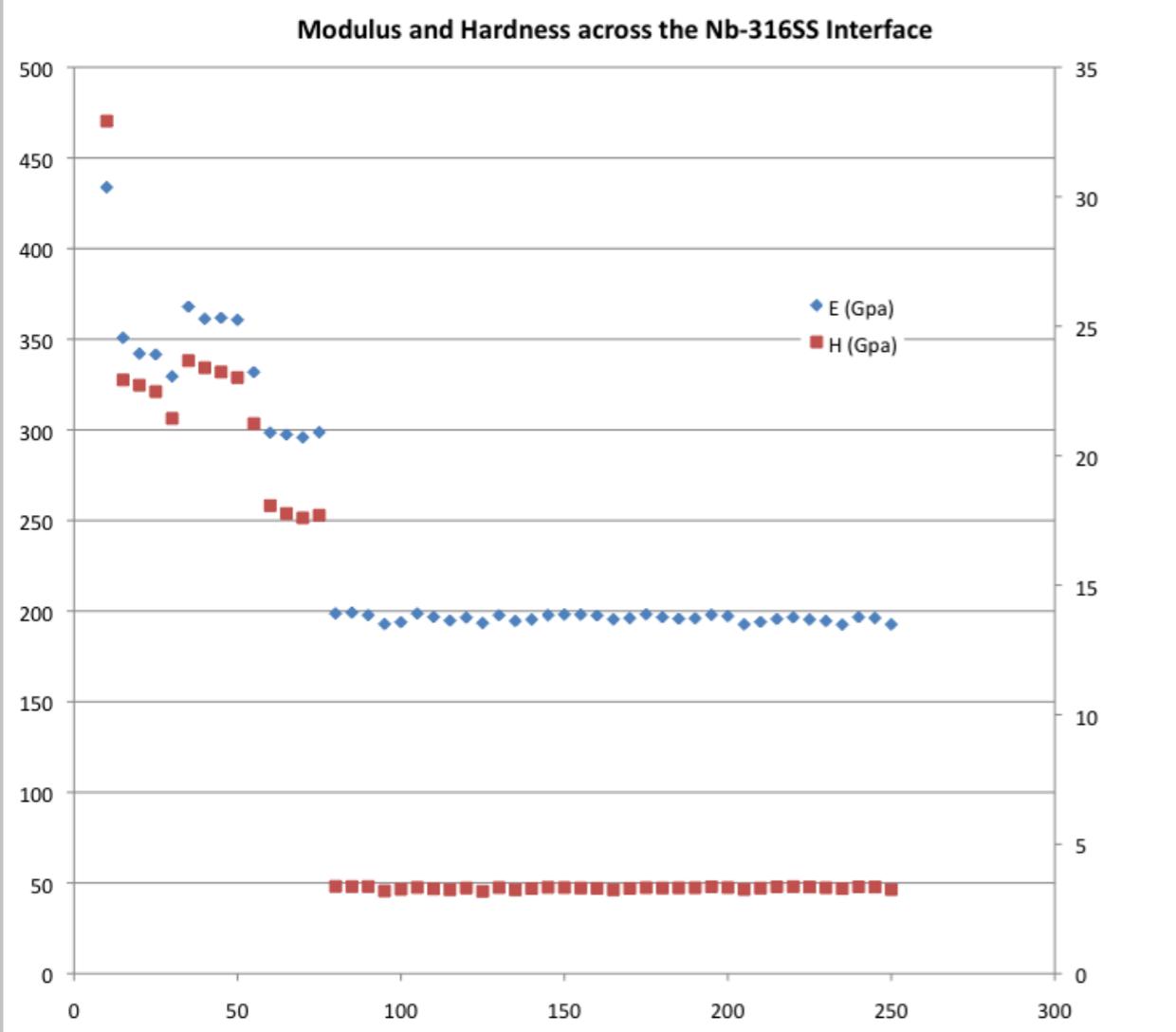
- Depth and spacing of indentations chosen to maximum spatial resolution

- 200nm depth → ~1.4μm width
- space indentations 5μm apart
- Perform 3 indentations at each distance from interface to provide some sampling statistics
- 250μm of distance measured → 150 indentations
- Depth and spacing of indentations in nickel irradiated region → 500nm, 10μm

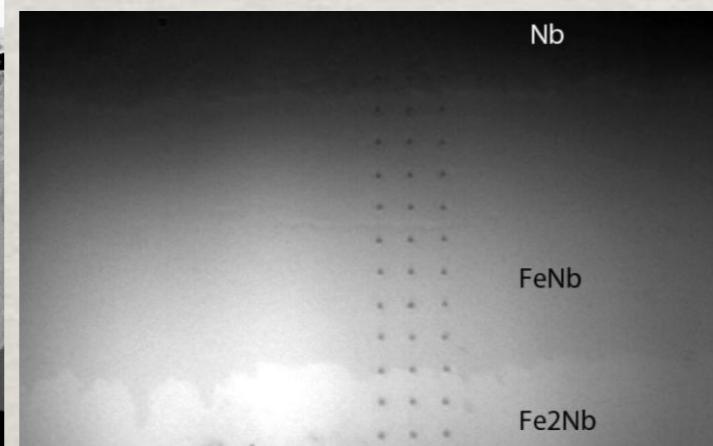
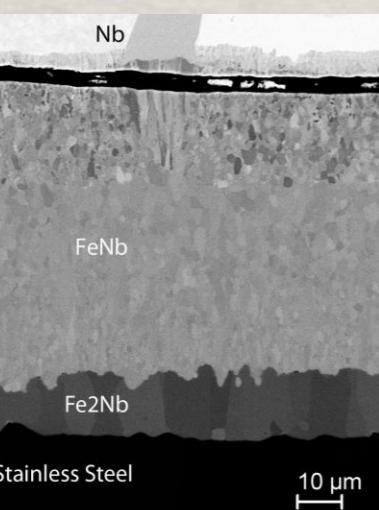
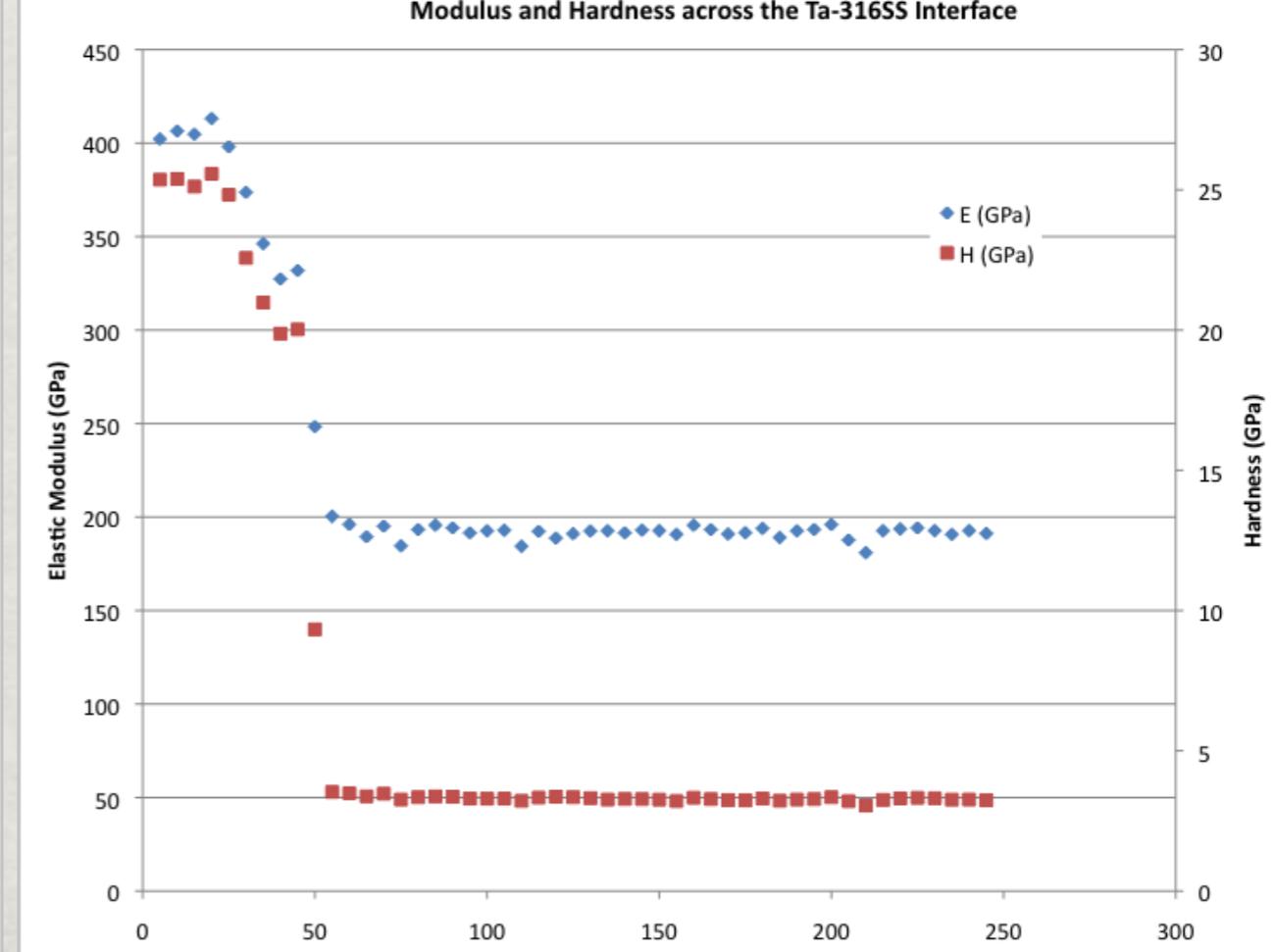


Nanoindentation Across the Nb and Ta Diffusion Couples

Modulus and Hardness across the Nb-316SS Interface



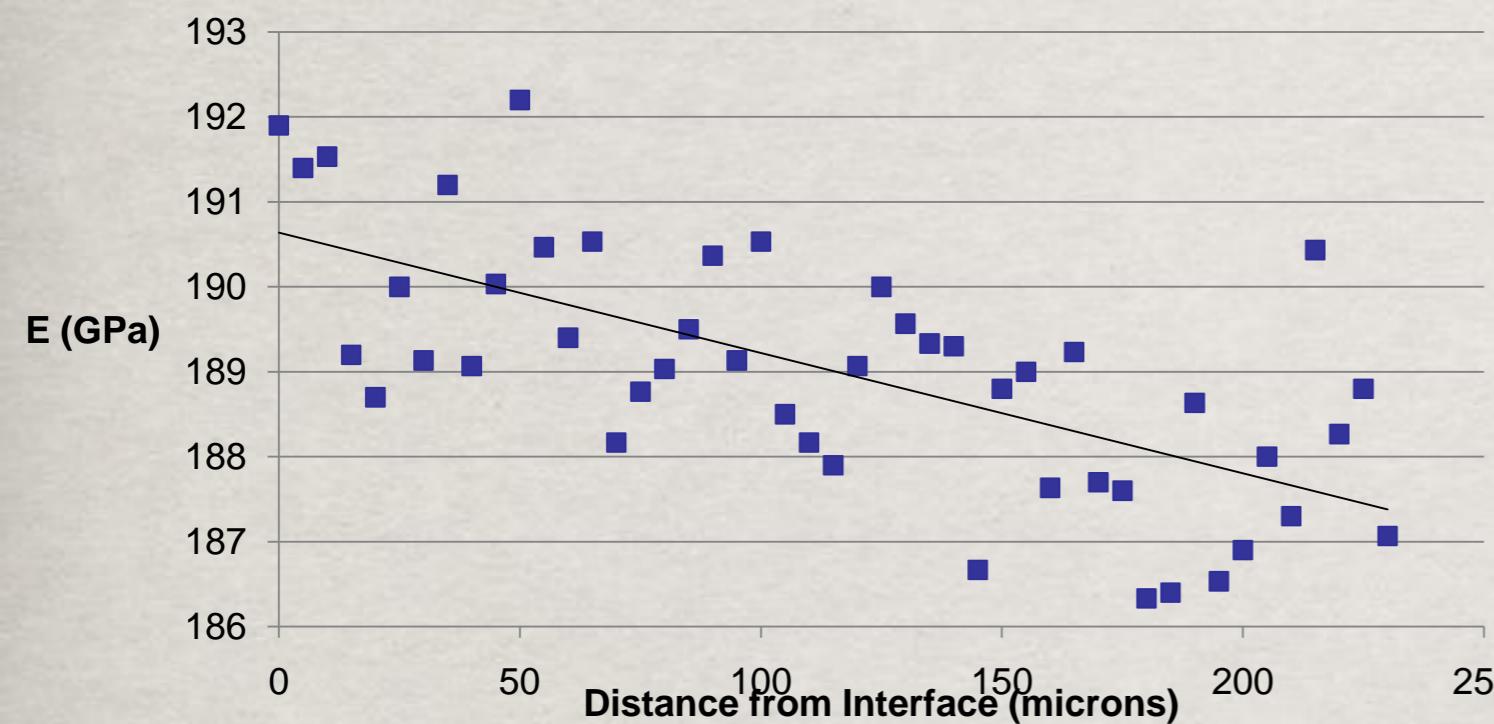
Modulus and Hardness across the Ta-316SS Interface



- Almost no measurable change past the intermetallic/steel interface
- Multiple intermetallics with different moduli and hardness
- Possible variation in modulus/hardness across intermetallics.

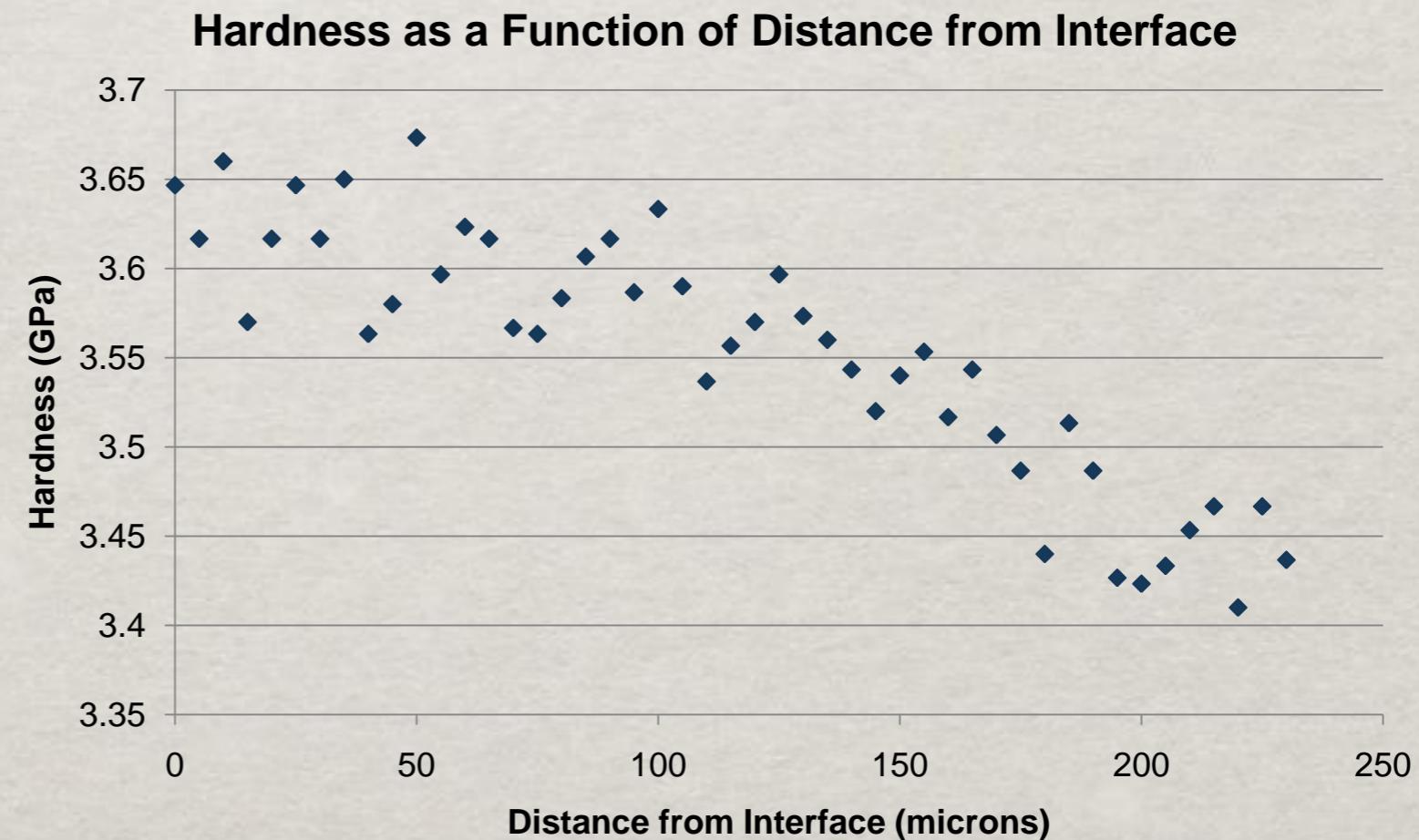
Nanoindentation Across the Nb and Ta Diffusion Couples

Elastic Modulus vs. Distance from Interface



Modulus has very little, if any, measurable change with Mo composition

Hardness (in steel) falls systematically with decreasing Mo concentration.

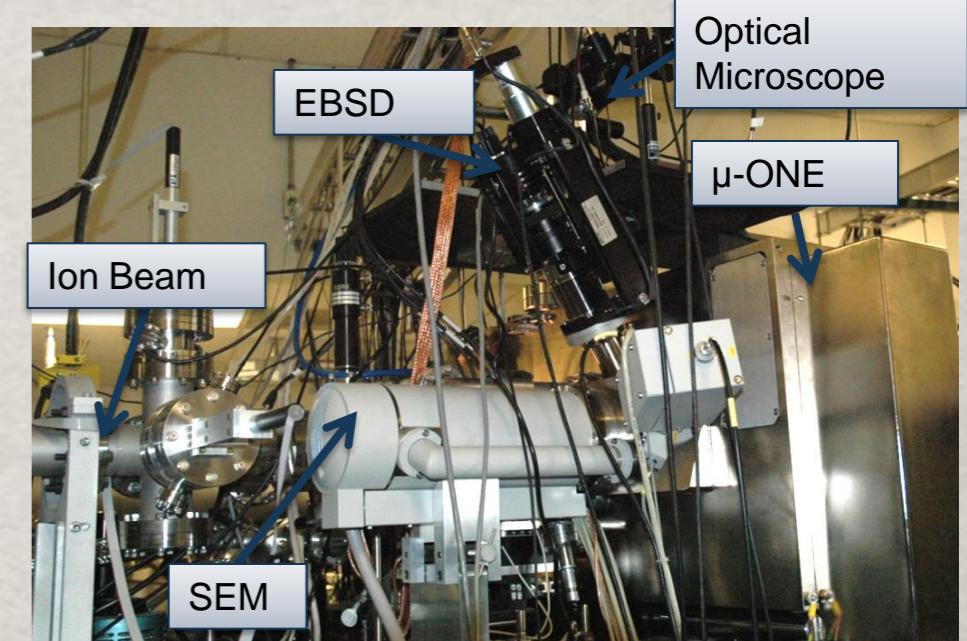


Proton and Heavy Ion (Ni^{4+}) Irradiation of Diffusion Couples

- Proton beam conditions
 - 300keV
 - Estimated ~ 1 dpa
 - Estimated end of range $1.32 \mu\text{m}$
 - “room” temperature exposures
- Nickel ion beam conditions
 - 20 MeV Ni.
 - Damage rate ~ 0.003 dpa/sec
 - Estimated end of range $3.47 \mu\text{m}$
 - Exposures on Mo-316SS: 1, 10, and 30 dpa
 - “room” temperature exposures



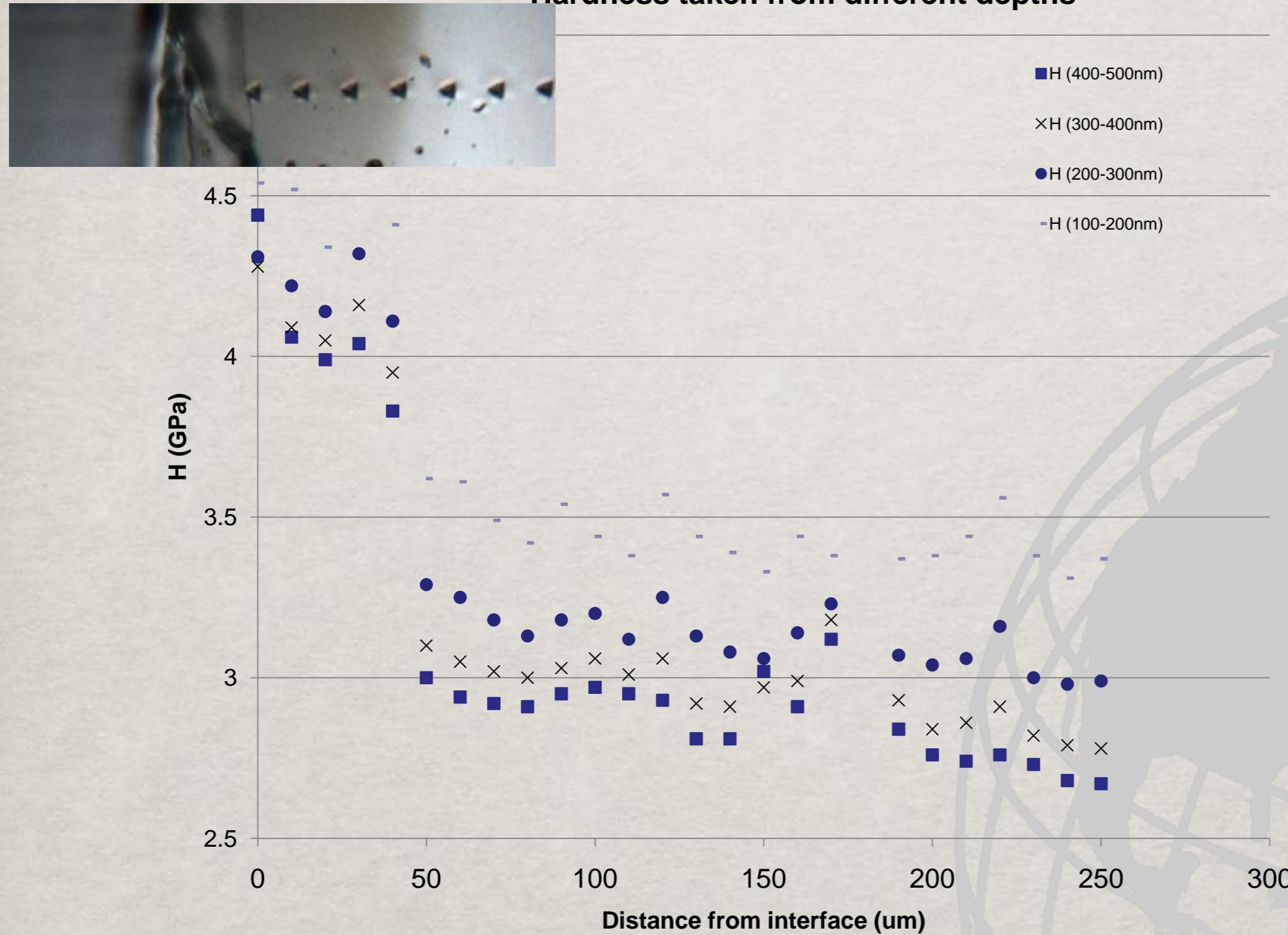
Ion Beam Lab at Sandia



Micro-ONE ion beam line

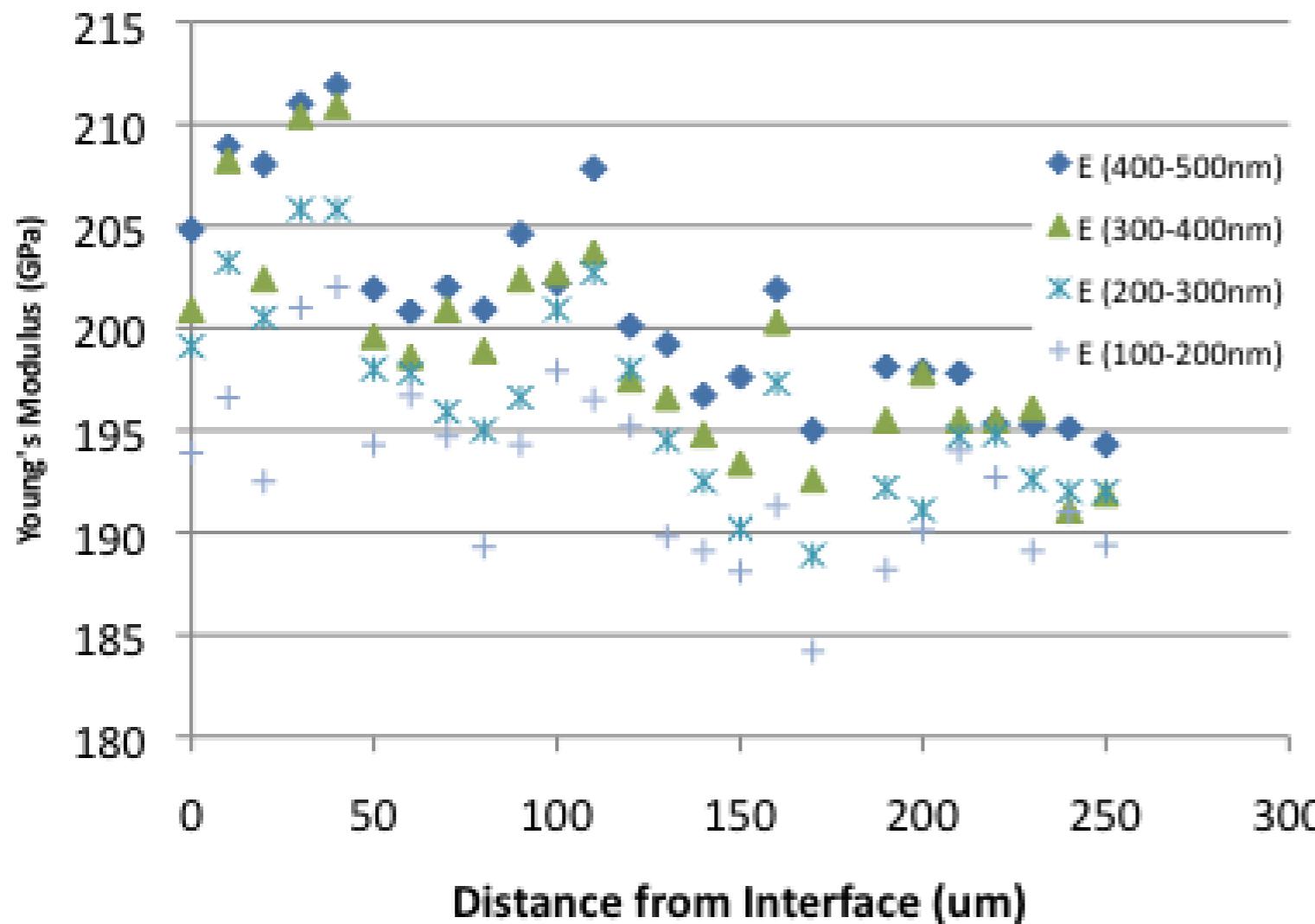
Hardness in Nickel Irradiation Zone

Hardness taken from different depths

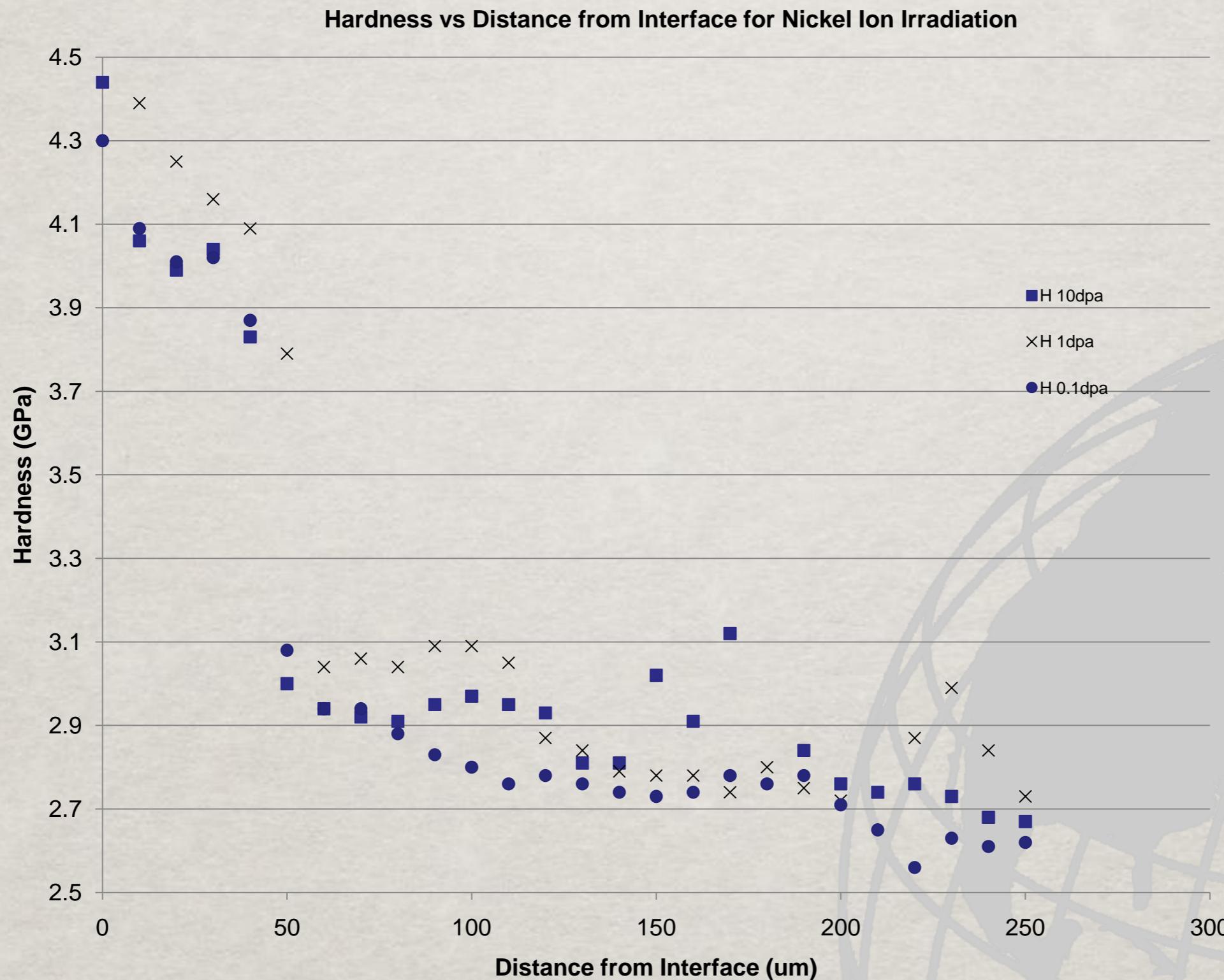


Large improvement for fatigue and corrosion failure

Modulus vs Distance from Interface for 10dpa Irradiation



Hardness profiles for different levels of Ni ion exposure



Developing the diffusion multiple (couple) approach for developing reactor steels

- Successfully produced diffusion couples between 316SS and refractory metals: Nb, Mo, Ta, W
 - Clear formation of intermetallics
 - Only Mo has substantial interdiffusion
 - Solid solution strengthening effect observed in nanoindentation hardness (for Mo)
- Effects of irradiation on interface can be seen
 - Room temperature nickel and proton exposures
 - Indentation hardness clearly tracks with irradiated zone
 - What is the best depth from which to take the data?
- Future work:
 - High temperature irradiation to look at changes in strength and RIS
 - Application to HT9 alloy
 - Extension to full diffusion multiples.