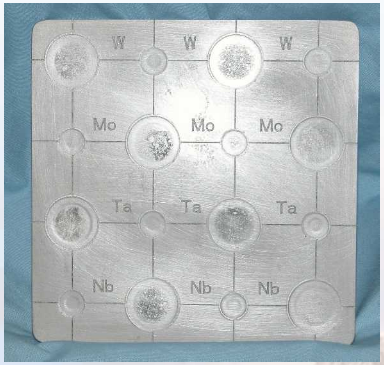
Combinatorial Approach to Determining Radiation Tolerant Materials

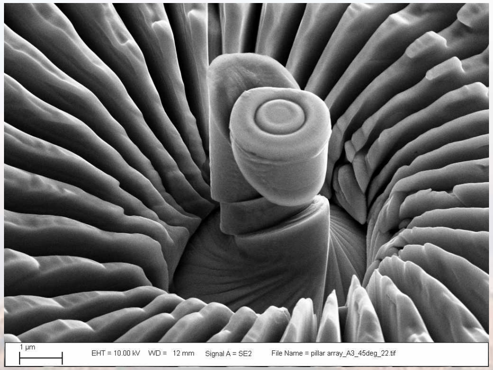
SAND2010-6471C

K. Hattar, L.N. Brewer, B.L. Boyce, and J.D. Puskar

Sandia National Laboratories & Naval Postgraduate Research Institute

August 24, 2010





Outline

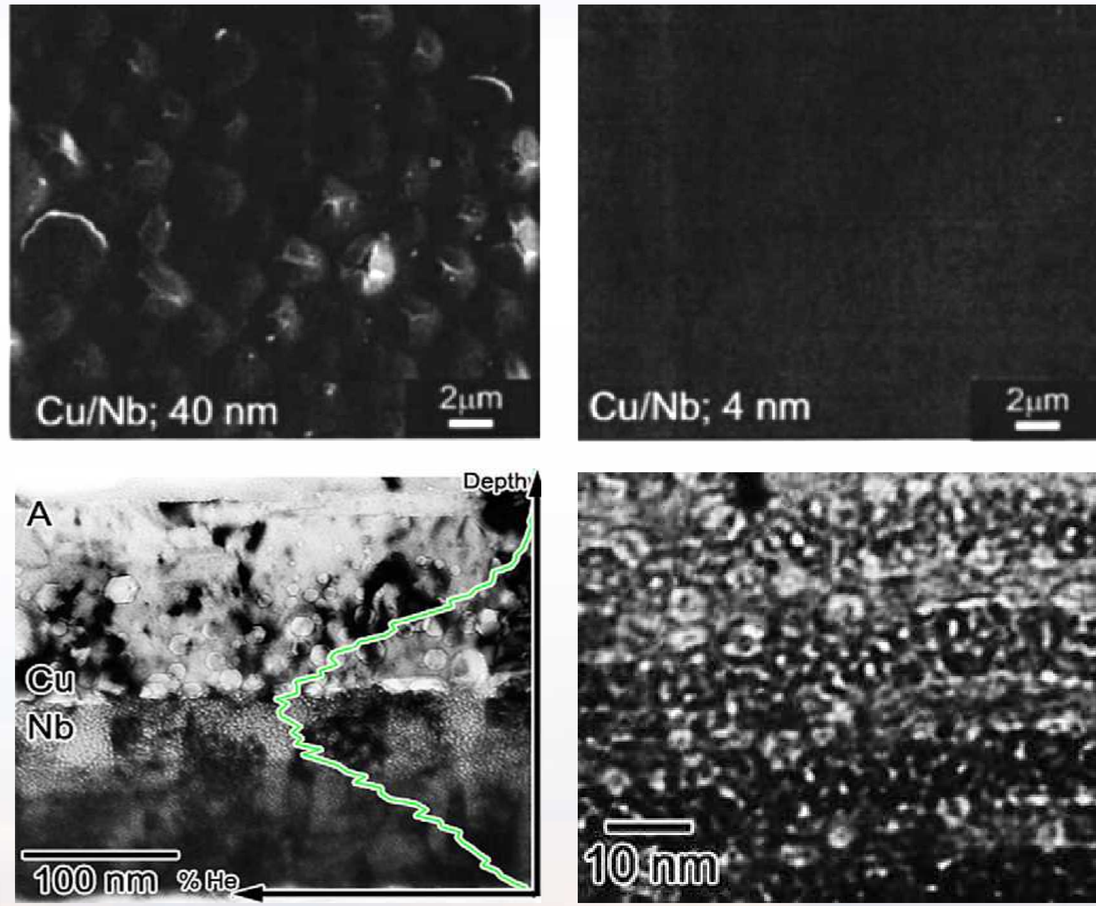
- **Need for new materials and testing**
- **Current and future capabilities and studies at the new IBL**
  - Mico-ONE beam line
  - → Combinatorial Characterization of Cladding materials
  - In-situ Irradiation TEM
  - → Fundamental studies of radiation Damage and corrosion
  - Nanoimplantor
  - → Determination of localized radiation effects
- **Discussion of future directions**

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND2009-2801P

Sandia National Laboratories

Validation of Advanced Materials

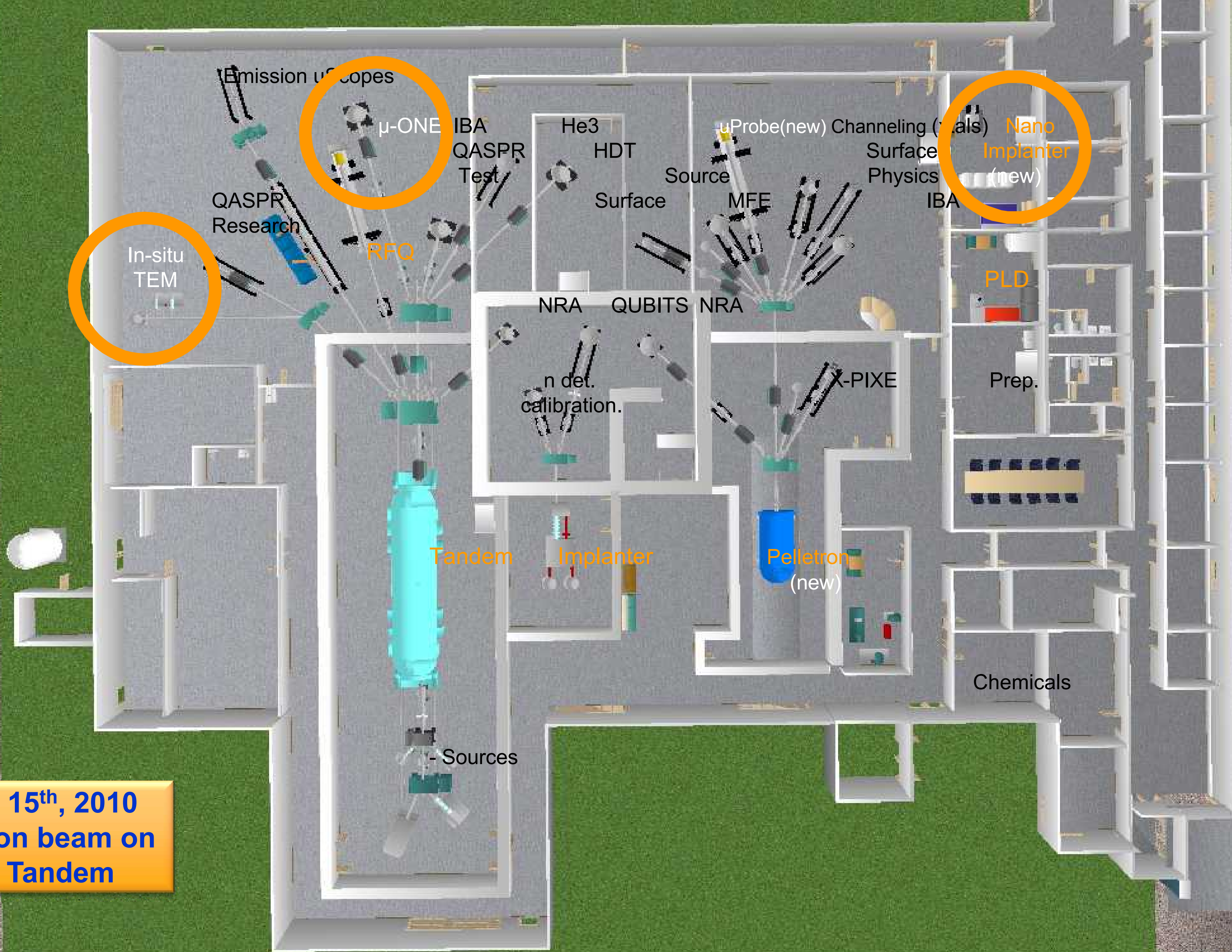
- Many materials systems are being considered.
  - Advanced steels
  - Nanostructured materials
- Interface engineering is providing a potential solution
- For example, copper-niobium nanolamellars (immiscible system with a weak interface) provide a plethora of interfaces that readily distribute He bubble and associated damage in films irradiated at 10^{17} cm^2 of 33 keV $^4He^+$ at 763 K



Hattar et al. Scripta Mat. 2007

Advanced materials and the need for rapid testing often requires new experimental testing techniques

New Ion Beam Laboratory



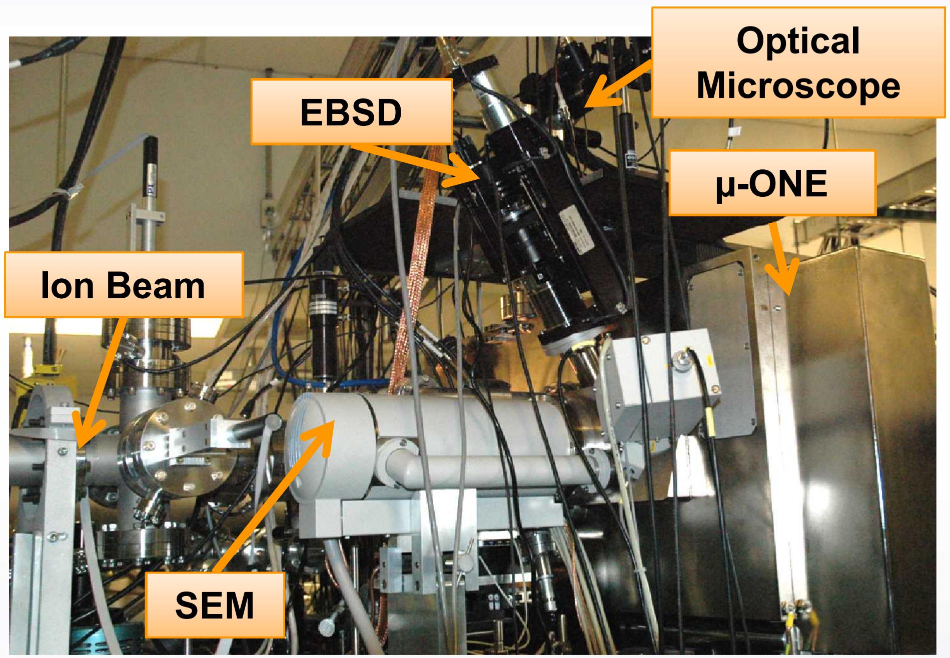
June 15th, 2010
First ion beam on the Tandem

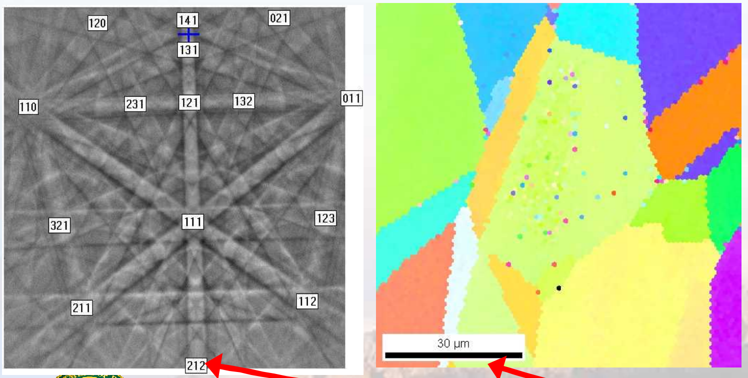
Micro-ONE Capabilities

Micro-ONE = Micrometer resolution Optical, Nuclear, and Electron Microscope

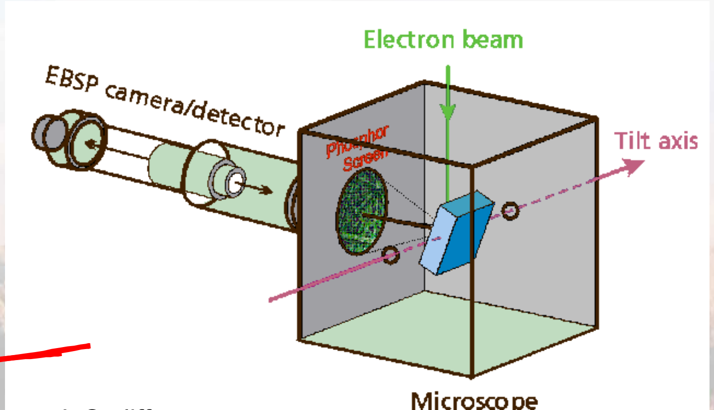
Micro-ONE capabilites

- Beam size: < 1µm (~ 0.5 µm)
- Current: single ions to 10,000 ions/s
- Ions: H, He, heavy ions
- Energy: (q+1)*6 MeV for heavy ions
- Scan size: ~ 100x100 μm^2
- Stage position with 50 nm resolution
- Fast blanking capabilities
- Navigation based on GDS II files
- IBIC and TRIBIC capabilities
- EBSD mapping





First EBSD Pattern and Map obtained with this system

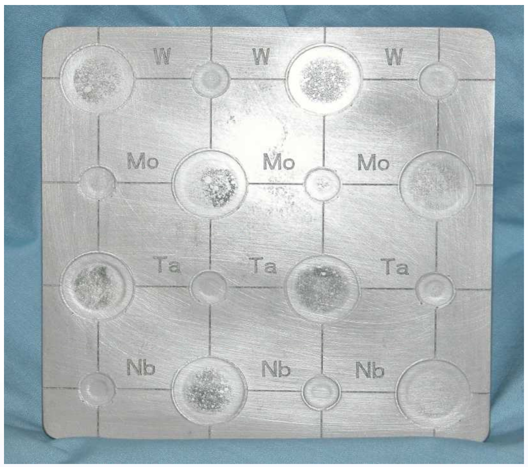


J. Sutliff

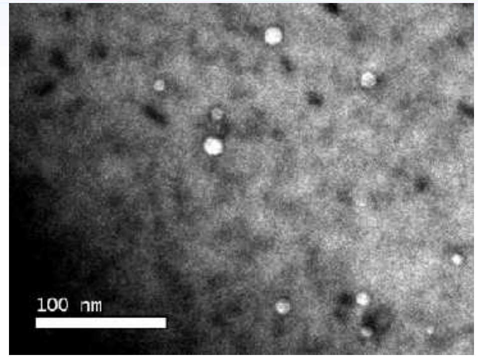
Will allows parallel imaging of changes in microstructure: grain size, phase transformations.

Sandia National Laboratories

Sandia's Approach to Rapid Material Validation

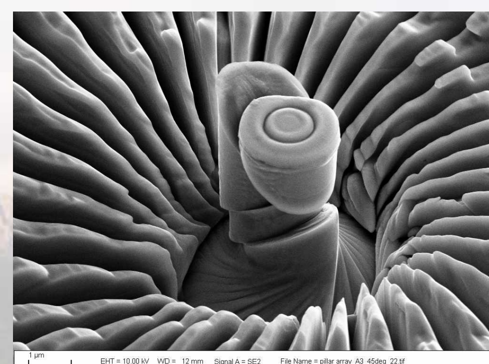


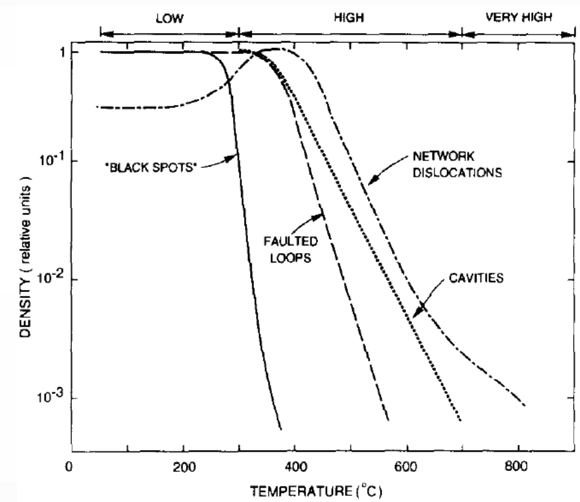
Local Composition
(Diffusion Couples)
+
Local Microstructural
Control (Ion Irradiation)



Microstructural
Characterization
(XTEM)

Mechanical Properties
(small-scale testing)



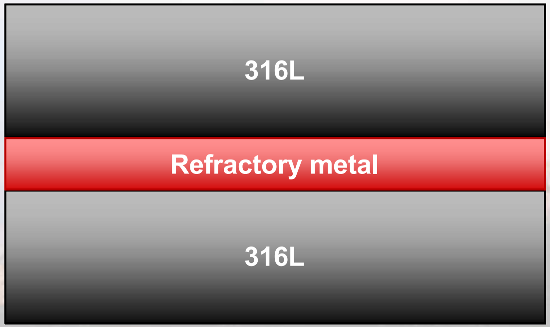


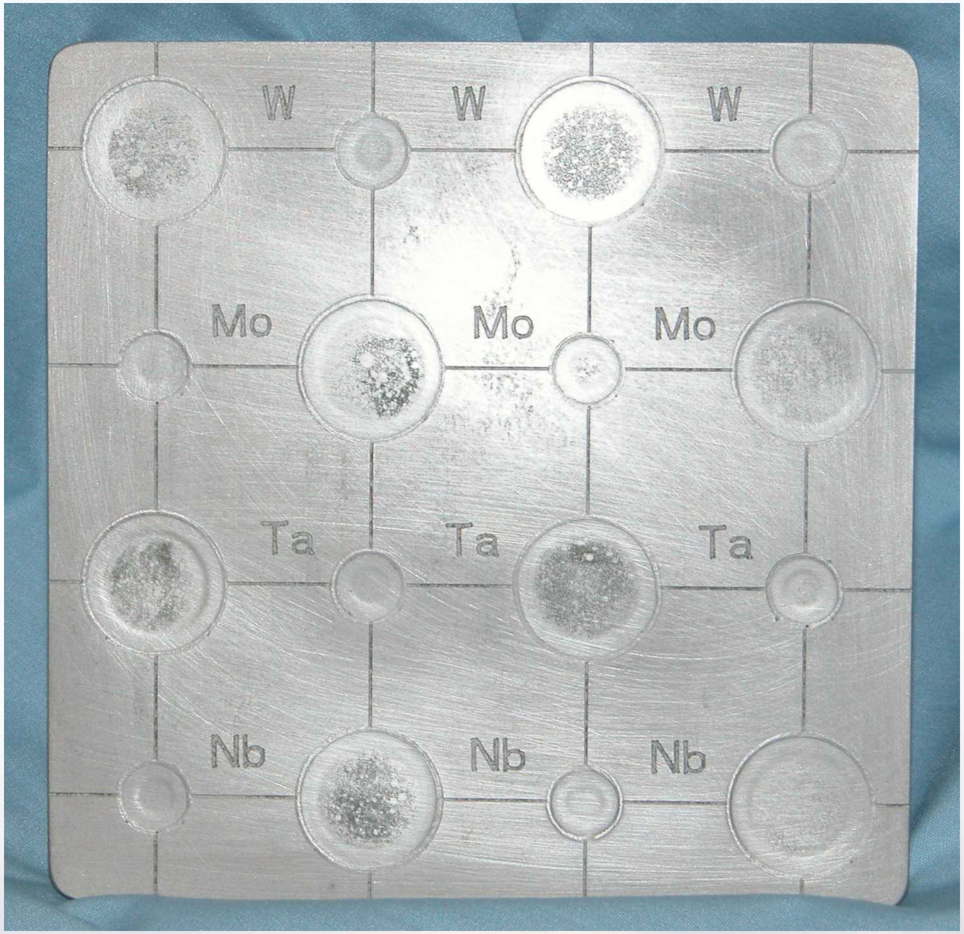
Validating Comparison
to Neutron Irradiation
Experiments +
Investigation into new
materials

Sandia National Laboratories

Diffusion Couple Production

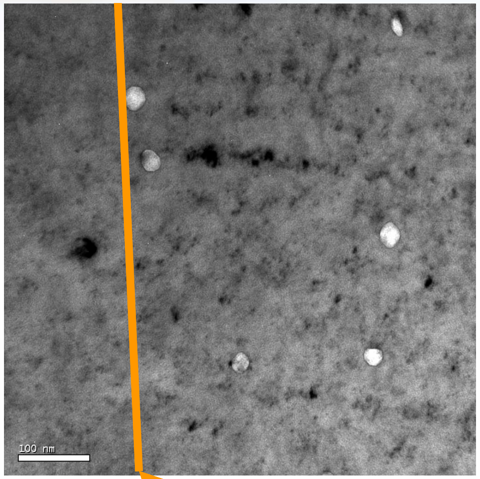
- Initial work was started on single crytal Cu, 316L, 409, and 420
- Two sets binary couples are being produced based on
  - 316L and HT9 steels
- Coupled with:
  - Refractory metals-Ta, W, Nb, Mo
- Coupled by HIP
- Vacuum annealed for 400 Hrs. at 1100 °C
- EDM cut for ion irradiation, microstructural analysis, small-scale mechanical property testing.



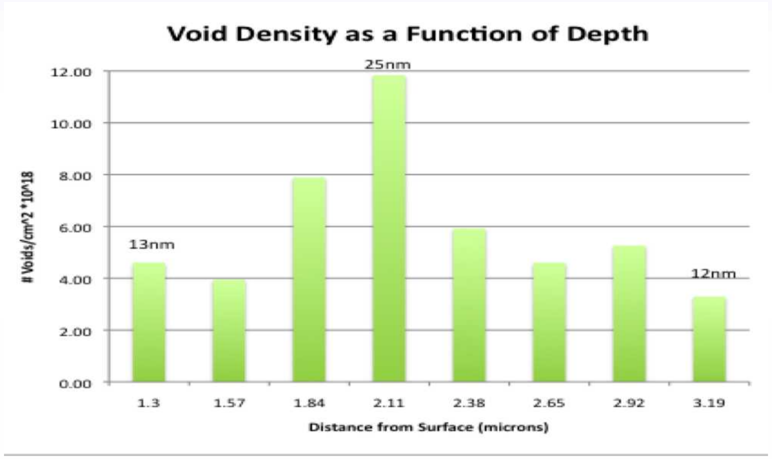


Diffusion Multiples provide a method to rapidly test many alloy compositions

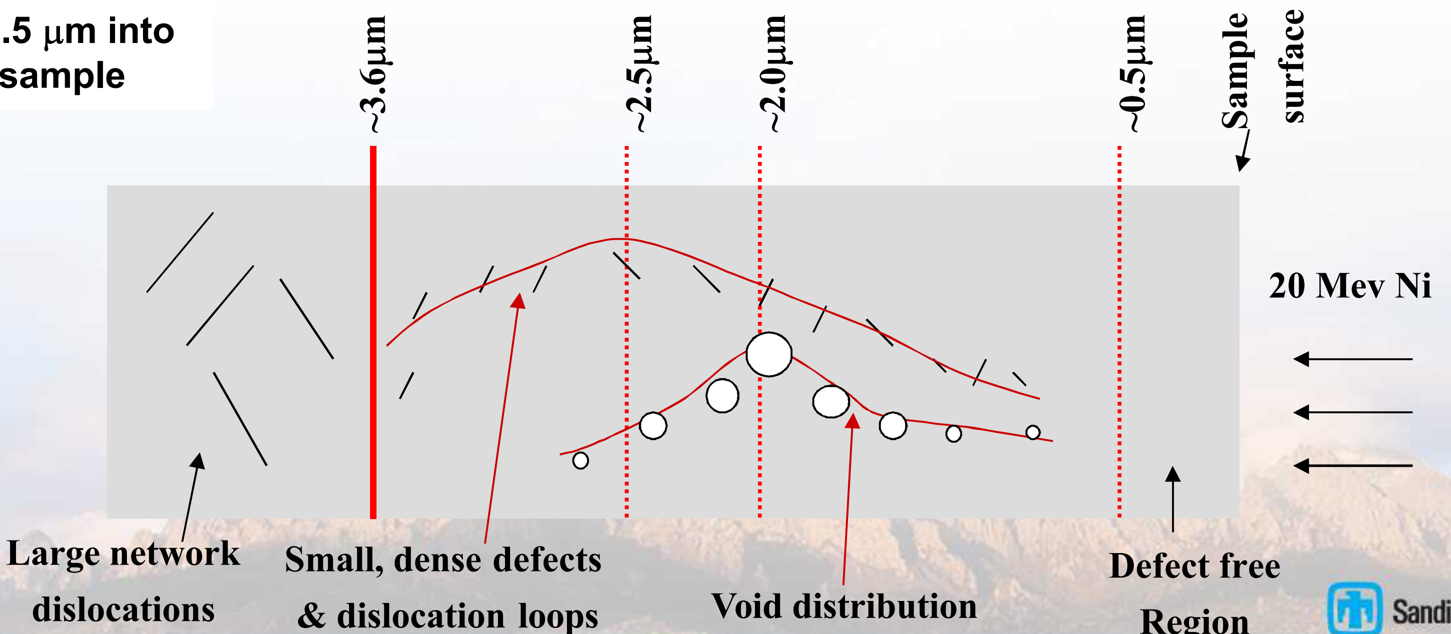
Microstructural Analysis



~ 2.5 μm into sample

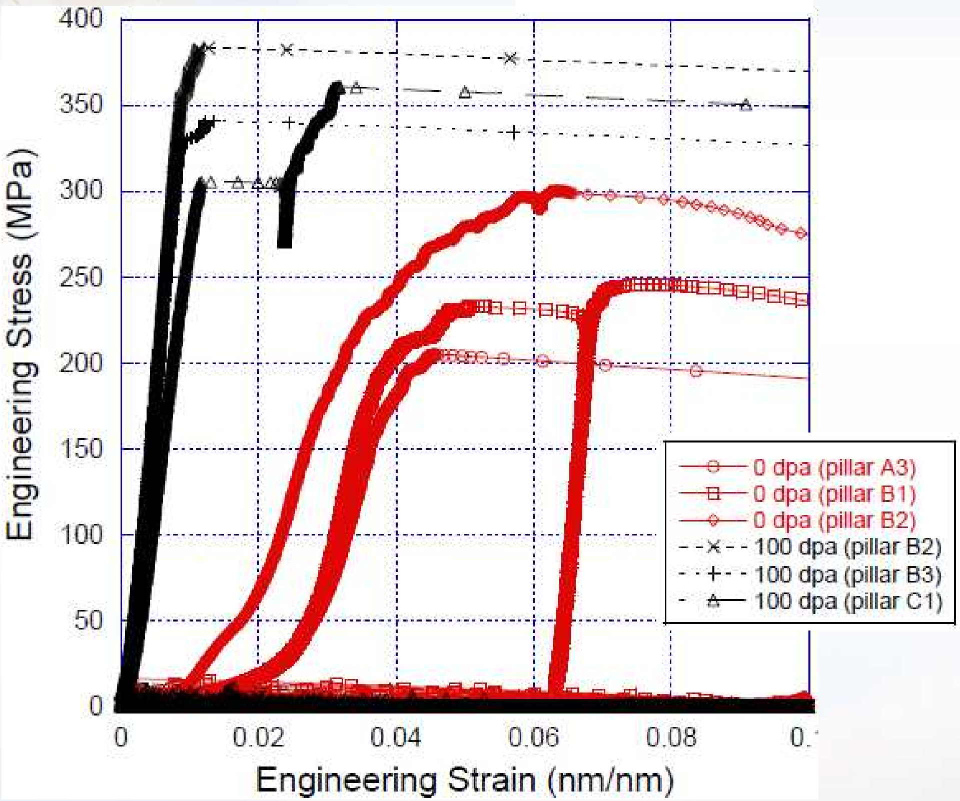


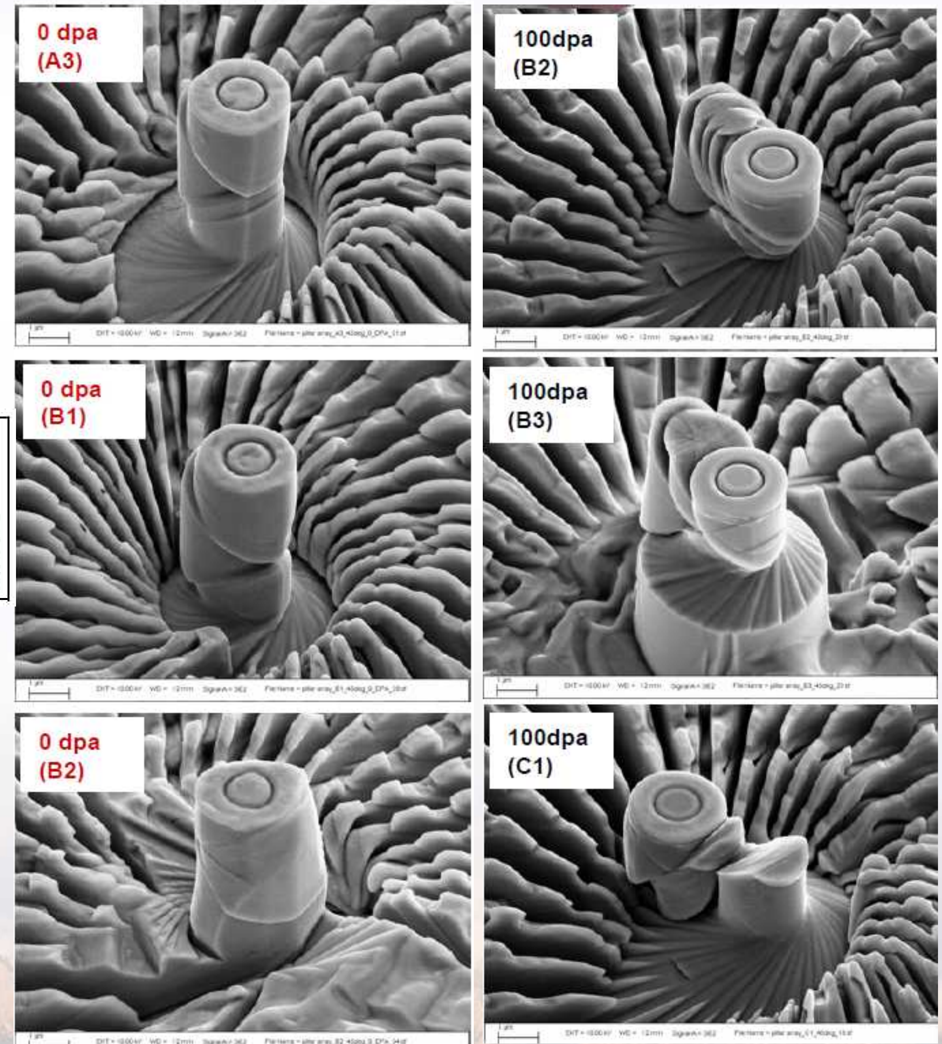
316 SS samples were irradiated at 400°, 500° and 600°C by 20 MeV Ni ions to a maximum peak dpa of ~100 at about 3.5 μm. The FIB was used for TEM sample preparation.



Sandia National Laboratories

Irradiated Micropillars

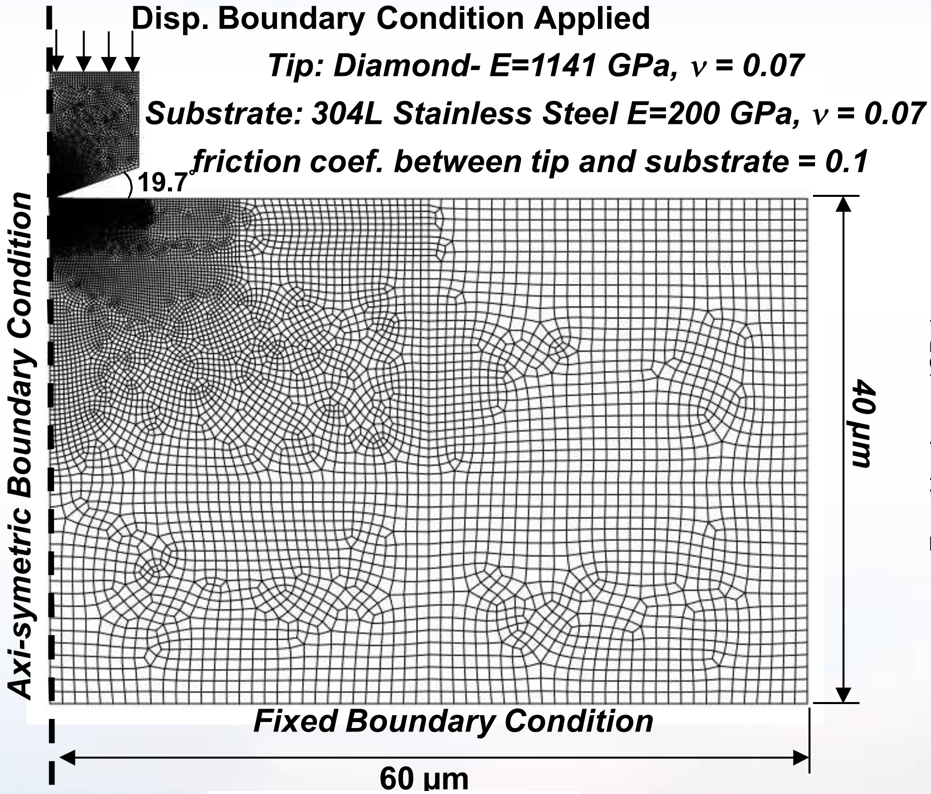


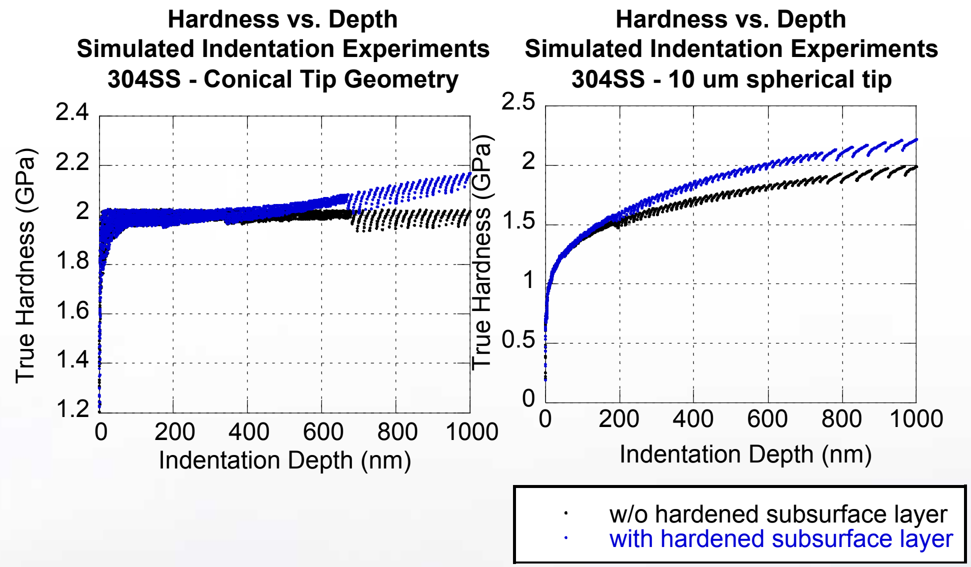


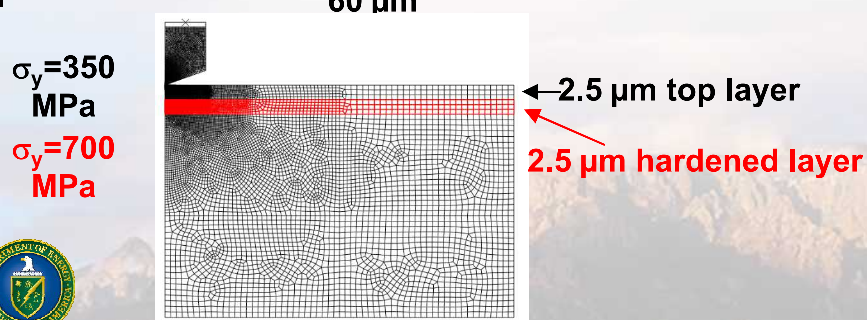
Irradiated and FIB prepared micropilars combine two Ion Beam Techniques

Sandia National Laboratories

Finite Element Simulations for Spherical Indentation



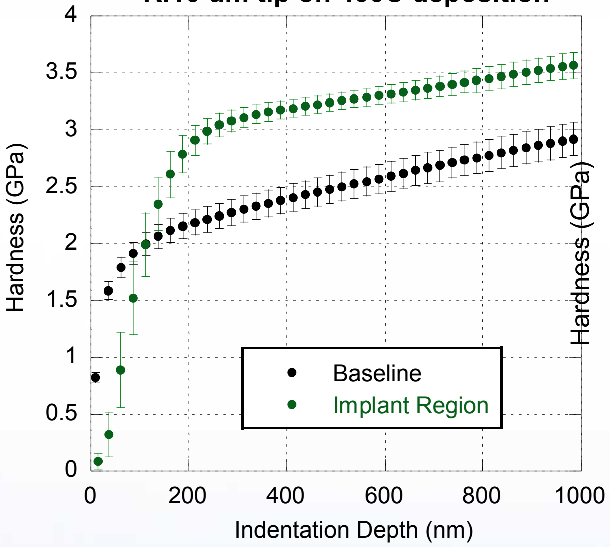




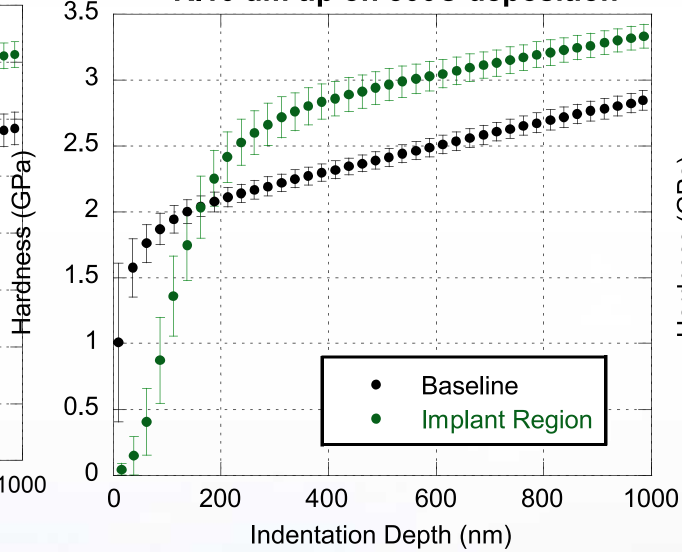
Greater deviation in properties is expected from spherical indentation in comparison to conical tip indenters

Sandia National Laboratories

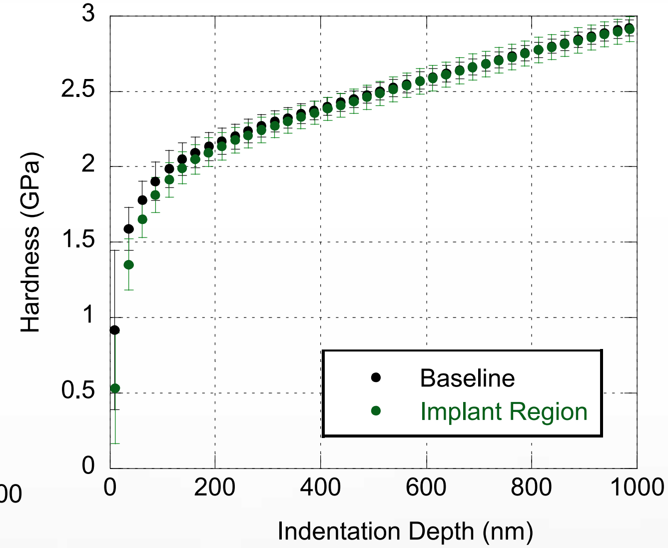
Spherical Indentation of Irradiated Samples



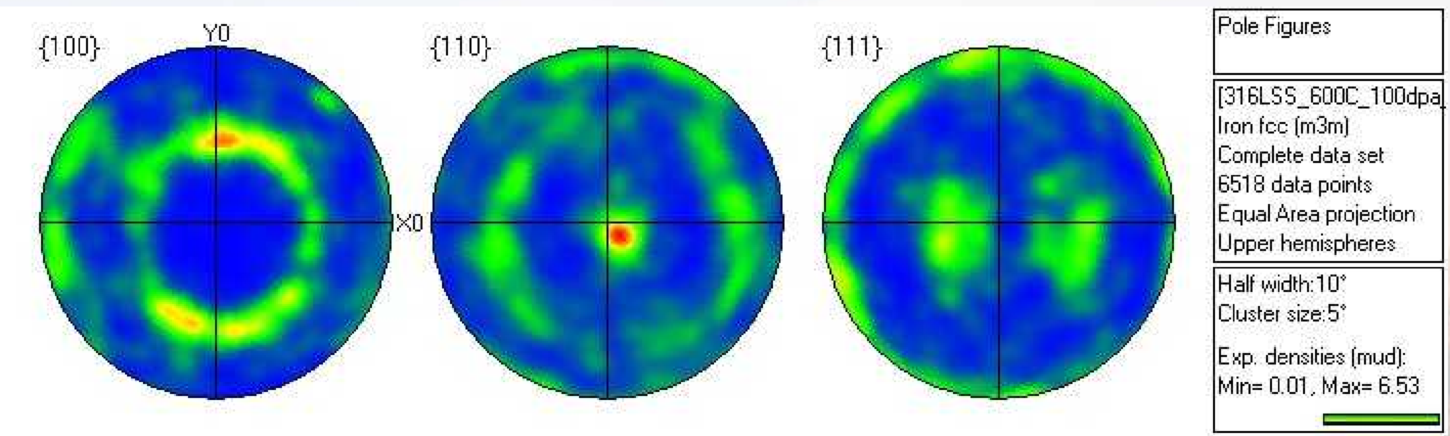
Hardness vs. Indentation Depth
Ion-Implant experiment on Stainless Steel
R.10 um tip on 400C deposition



Hardness vs. Indentation Depth
Ion-Implant experiment on Stainless Steel
R.10 um tip on 500C deposition



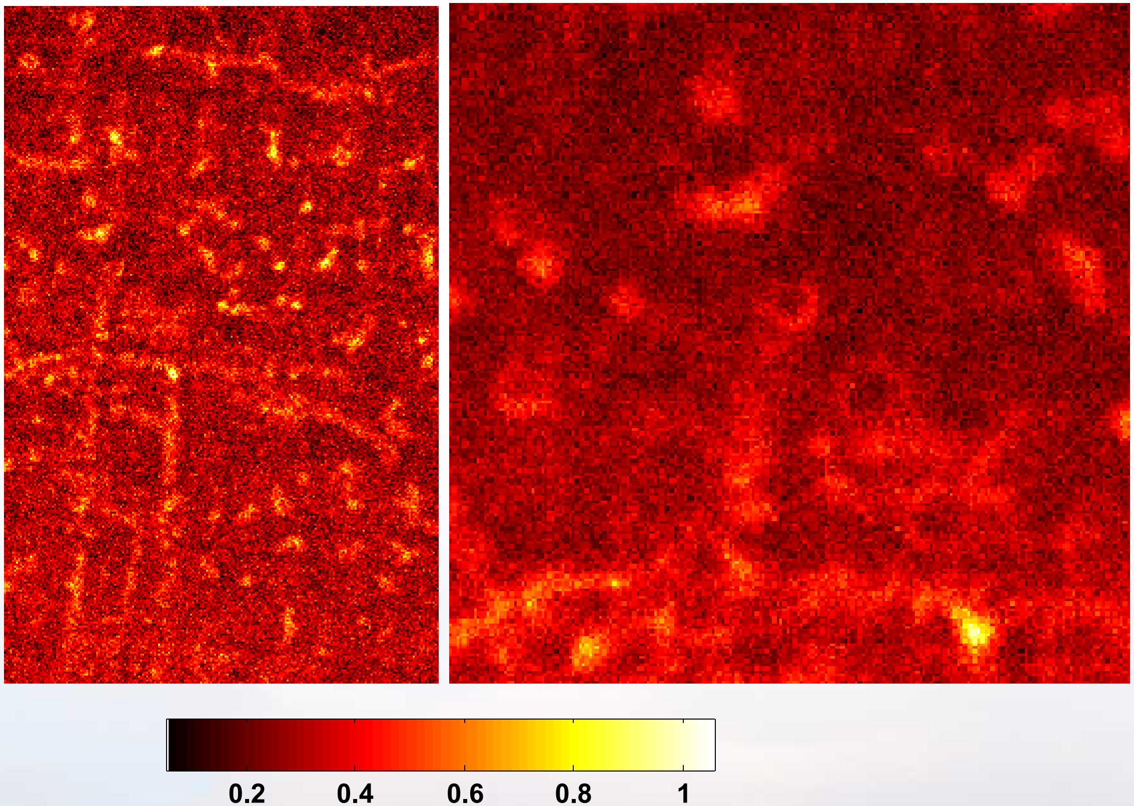
Hardness vs. Indentation Depth
Ion-Implant experiment on Stainless Steel
R.10 um tip on 600C deposition



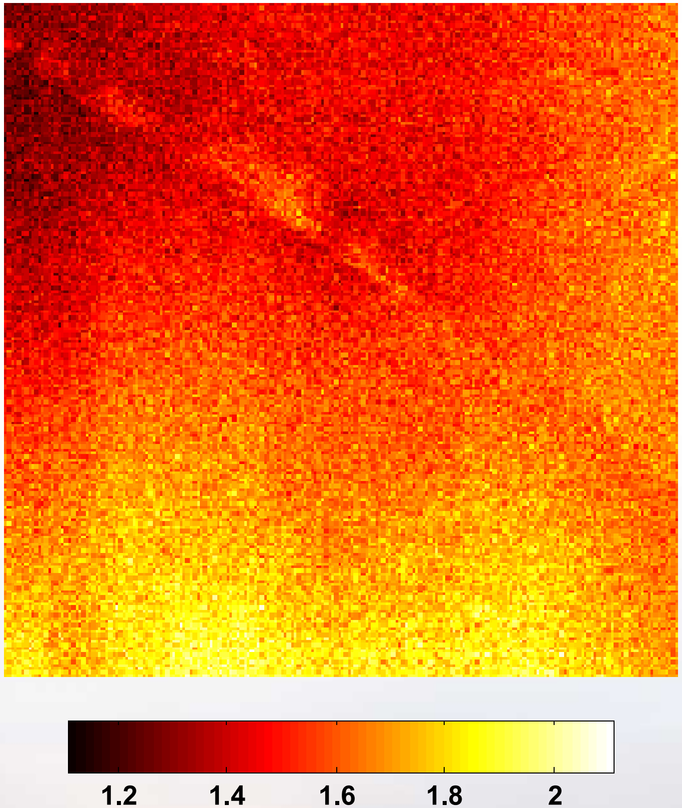
Further work is needed to characterize the change in hardness between 500 and 600 C

Radiation Induced Segregation

316L Stainless Steel: 100dpa,
20MeV Nickel Ions, 600°C



316L Stainless Steel: 100dpa,
20MeV Nickel Ions, 500°C

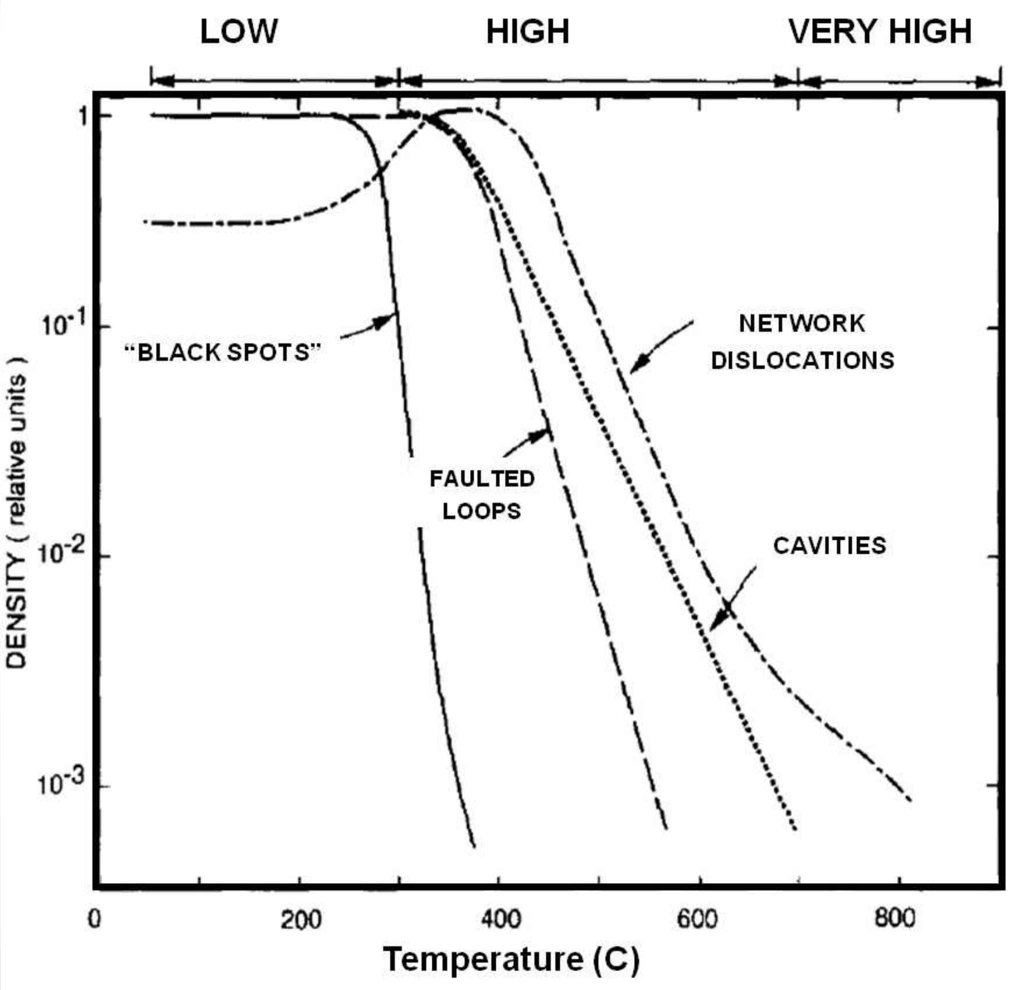


2 µm field of view

Ni and Si rich regions appear to self-organize and sometimes surround voids at 600 C, but not 500 C

Sandia National Laboratories

Ion Irradiation for Neutron Radiation Simulation



- **Low temperature: (50°C-300°C)**
  - **from the onset of the vacancy motion to vacancy cluster thermally unstable**
- **High temperature: (300°C-700°C)**
  - **from the onset of thermally-induced vacancy emission from vacancy clusters to the interstitial clusters thermally unstable**
- **Very high-temperature: (>700°C)**
  - **all irradiation-induced defect clusters except gas-pressured bubble are thermally unstable**

Note: actual temperature boundaries of the three regimes will be somewhat dependent on the damage rate.

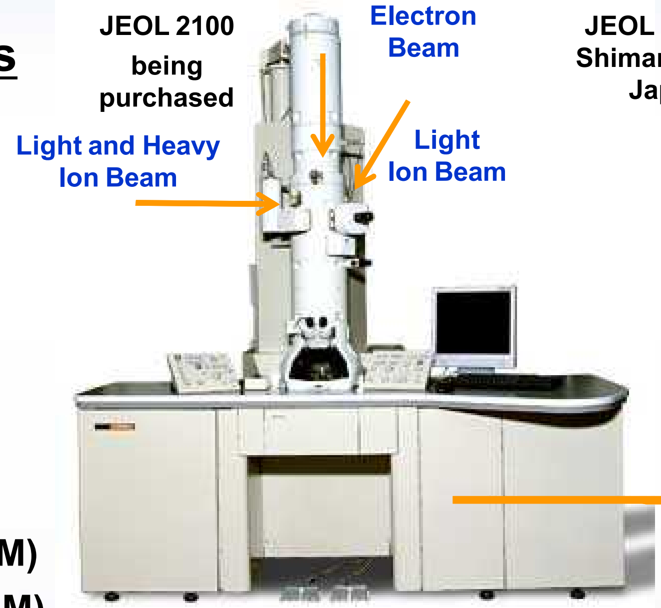
Ion damage shows similarity to neutron damage

In situ Ion Irradiation TEM

Proposed Capabilities

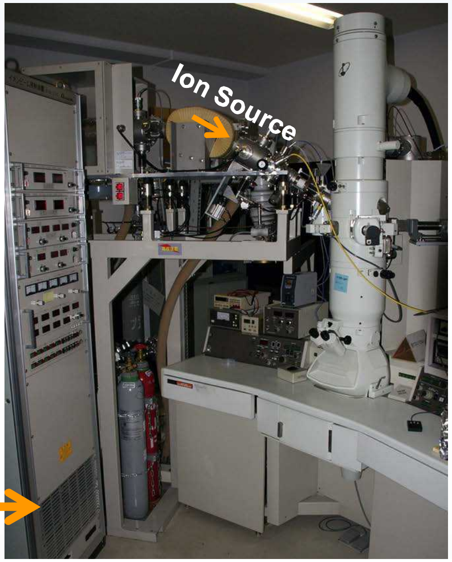
- 200 kV LaB_6 TEM
- Ion beams considered:
  - 32 MeV Fe^{4+}
  - 250 keV D^{2+}
  - 250 keV He^{+}
  - 300 keV e^{-}
- All beams will hit same location
- 30 to 60 degree considered
- nanosecond time resolution (DTEM)
- procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IL
- *In situ* heating and cooling stage
- *In situ* electrical measurement stage
- *In situ* straining stage
- *In situ* vapor phase stage
- *In situ* liquid mixing stage

JEOL 2100 being purchased

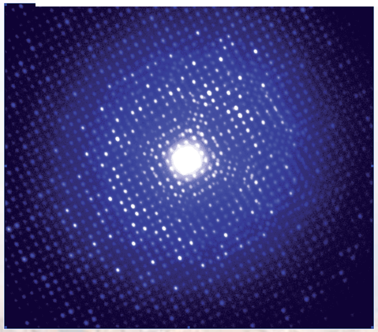


Delivery December 2010

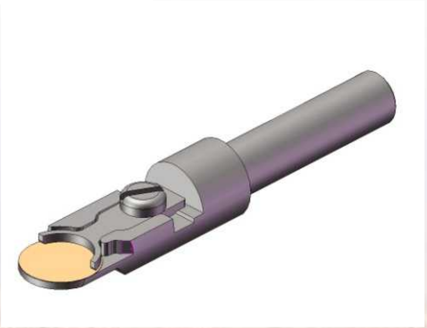
JEOL 2010 at Shimane Univ. Japan



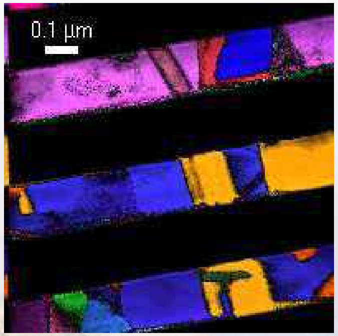
1 in the US (ANL)
11 world-wide



TVIPS



Hummingbird



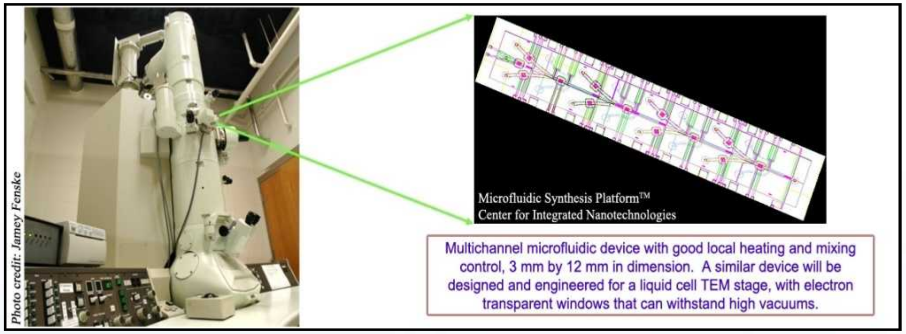
Nanomegas

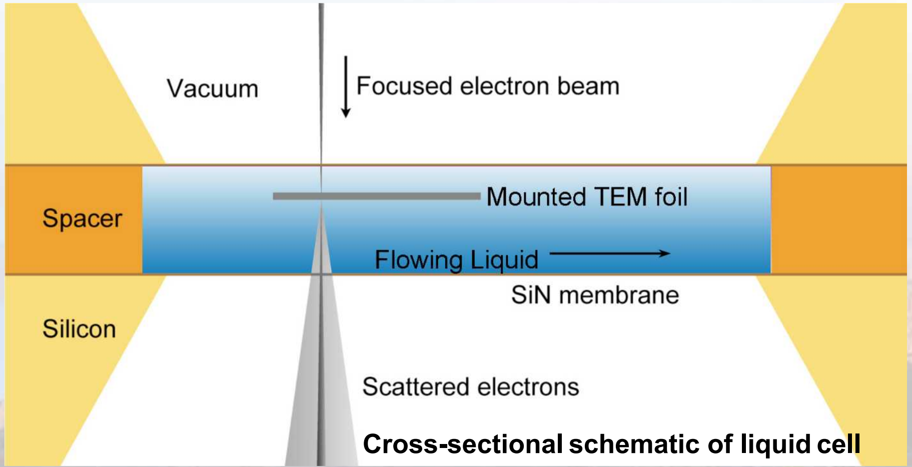
We are at the beginning stages of planning. Many potential additions for an *in situ* triple beam facility are being considered

In Situ TEM Fluid Flow Stages

Microfluidic Stage

- Mixing of two or more channels
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable

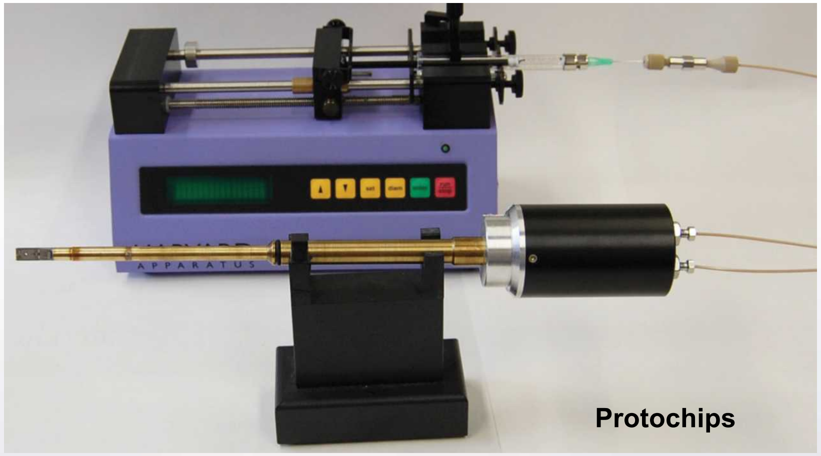




Cross-sectional schematic of liquid cell

Vapor-Phase Stage

- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools



- Initial use for corrosion studies of dry nuclear waste storage

Stages alone provide new research opportunities and even more when combined with ion beams

Investigation into Dry Storage Validation

Accelerated Simulations

- Currently there are 121 storage sites (at both active and decommissioned reactors), and 2,000 MT/yr of new waste
- During storage, degradation of materials could lead to loss of material integrity
- Trend to high burn-up fuels results in increased heat and the presence of additional actinides
- An *in situ* TEM study of the microstructure as a function of:
  - Thermal Cycles
  - Ion irradiation dose
  - Corrosive environments

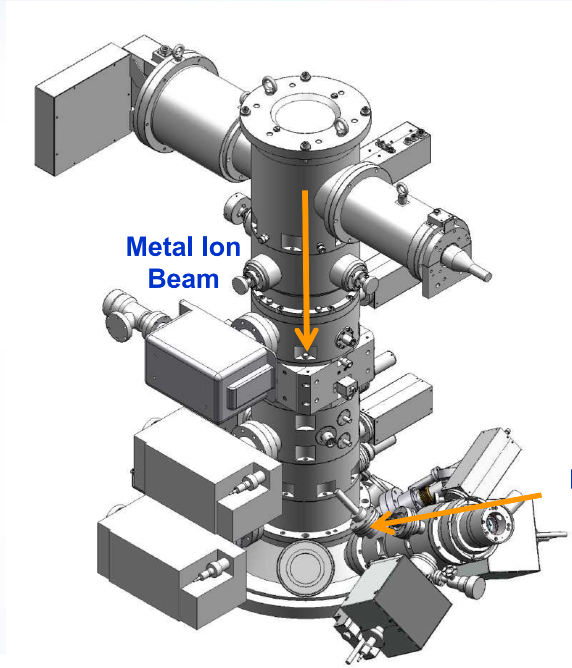
Connecticut Yankee dry storage site

Sandia's Approach:

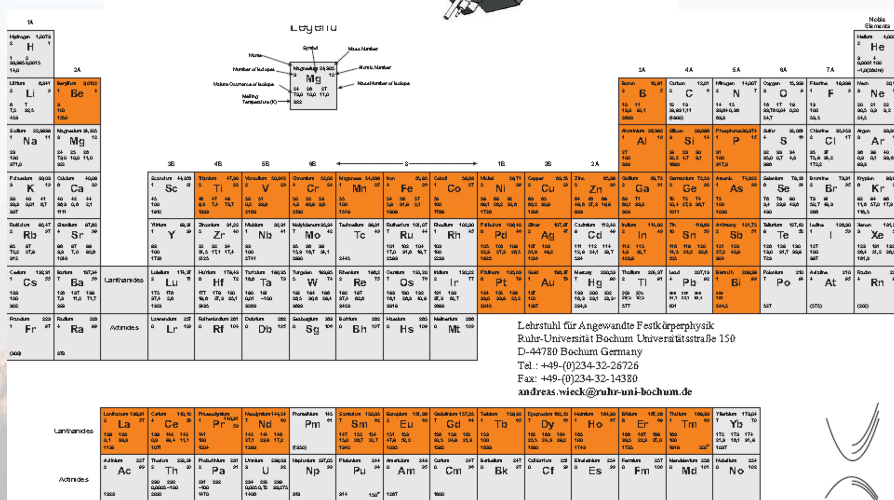
Prepare cladding, storage vessel and fuels samples with in-service microstructure & damage → Expose to environment experienced in storage, under ambient and accelerated conditions → Observe key mechanisms at external cladding surface and fuel-cladding interface → Validate observations with mesoscale experiments

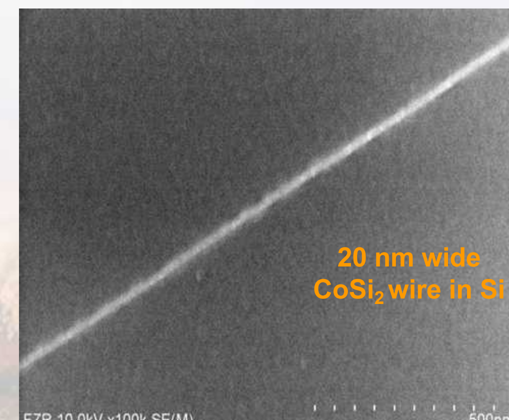
Nanoimplantor

- Multiple liquid metal ion source (LMIS)
- *E* x *B* filter
- Ion column with a maximum accelerating voltage of 100 kV
- 10 nm spot size
- High-resolution SEM
- 4 manipulators with 10 nm resolution and low current measurement capabilities



These capabilities greatly exceed the capabilities of commercially available Ga^+-based dual-beam FIB systems in terms of irradiation doses, ion species, and nanofabrication.





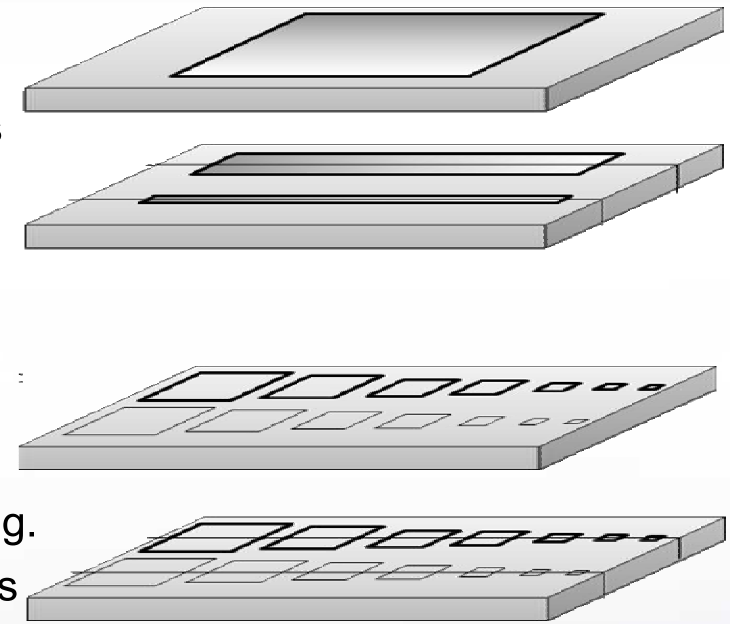
Local Control of Structural Defects

- **Local Control of Grain Boundaries**

•Control of ion species, energies, and beam size permit local rearrangements of interfaces and grain boundaries.

• The altered region surrounding various grain boundary types are controlled via the size and duration of self-ion implantations.

• Length scales associated with solutes will be investigated by tailored heavy ion implantation.



- **Deformation of Confined Volumes**

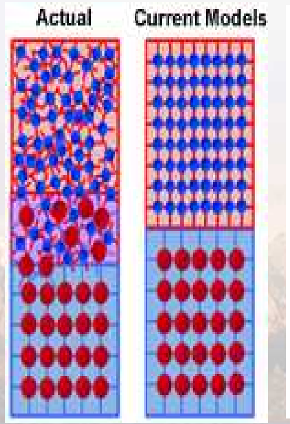
•Diffusion couple Ion beam will be used to confine the area of thin films down to a few nanometers prior to mechanical testing.

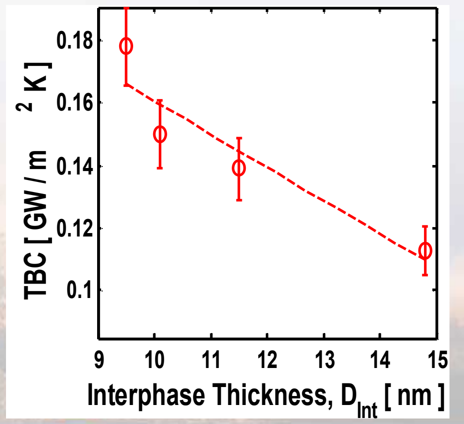
• This confinement can encapsulate identified defect structures in the film including: grain boundaries and particles.

• This will provide an alternative method to FIB milling for the production nanostructues

- **Controlling Interfacial Structure and Properties**

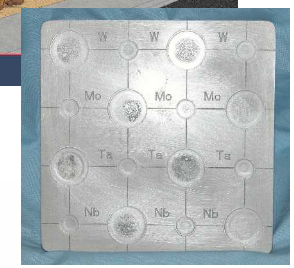
•Determination of thermal transport across interfaces as a function of local disorder



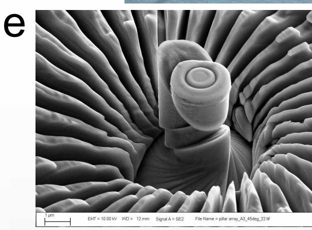


Conclusions

- **Sandia has a new Ion Beam Lab that is becoming operational**
  - Provide capabilities for insightful experiments into radiation damage
  - Provide for parallel SEM and EBSD to ion irradiation
  - *In situ* TEM irradiation with a variety of TEM stages
  - 10 nm implantation of various metals at energies up to 100 keV



- **Developed a combinatorial approach to rapidly test the radiation damage for NE applications**
  - Diffusion couple with multiple irradiation zones of various dpa per sample
  - A variety of small-scale mechanical property testing in each zone
  - *In situ* TEM characterization in a variety of irradiation environments

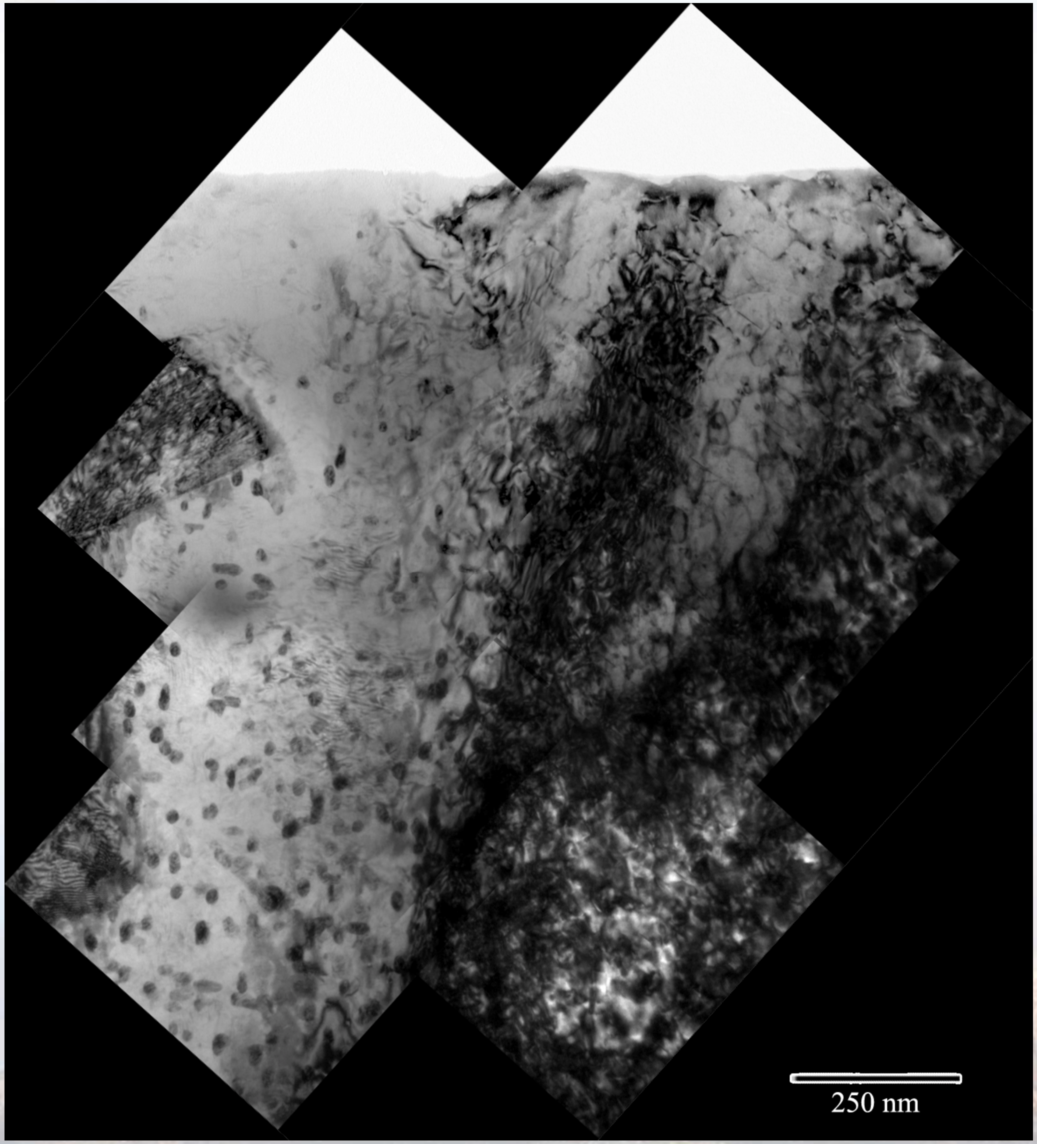


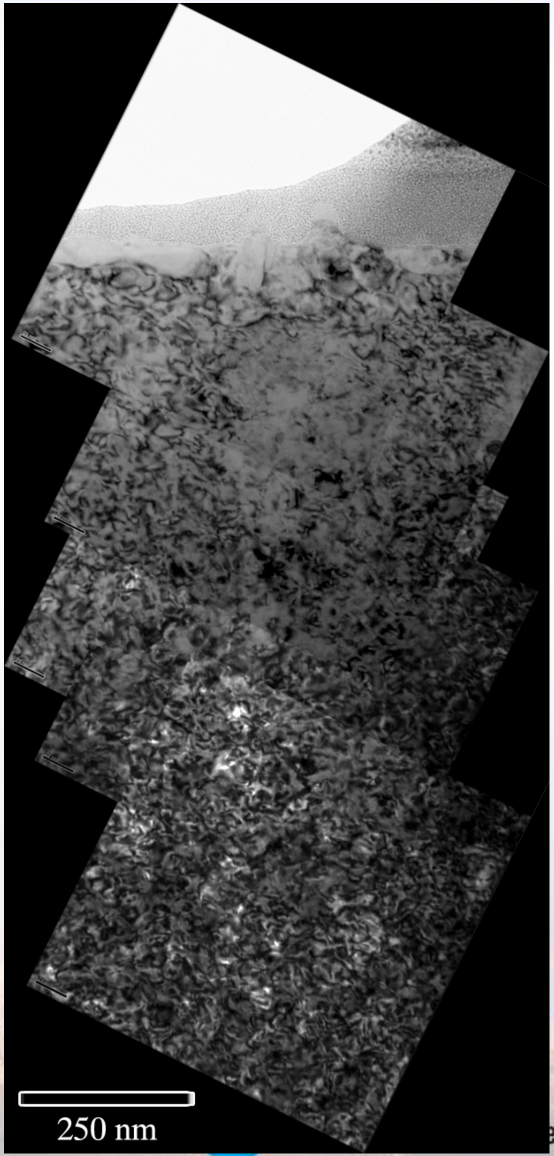
Sandia is developing tools for a variety of ion irradiation experiments and small-scale mechanical testing. This combination is promising for rapid characterization of new materials for future nuclear reactors

Acknowledgements

- All staff and technologists of the IBL at SNL
- P. Lu, P. Kotula, J.R. Michael, and S. Foiles at SNL
- A. Misra, R. Hoagland, and M. Demkowicz at LANL
- And many other collaborators

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND2009-2801P


250 nm

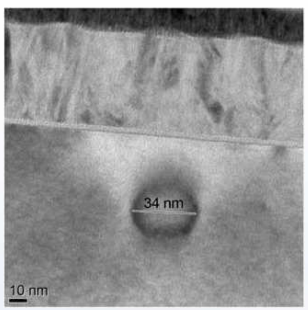

250 nm

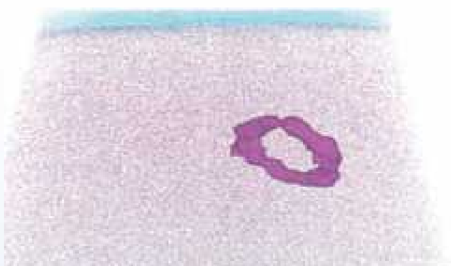
Additional Applications of Future Capabilities

- **FIB beyond Ga^{+} ion beam**

Collaboration with D. Gianola at Univ. of Penn.

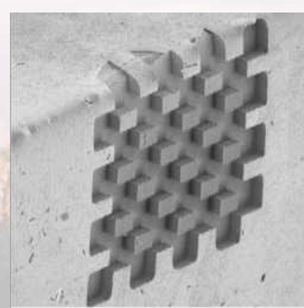
- **Nanostructured Sub-surface Composition Control**





Thompson et al. Science **317** 1370

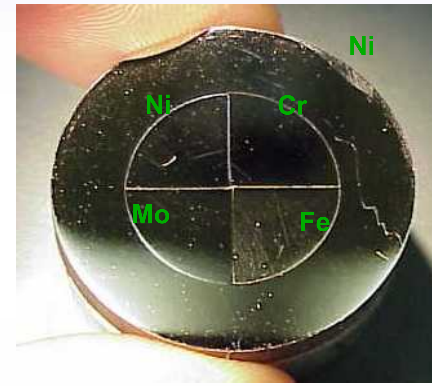
- **Completely written structure**



Gomez-Mrilla et al. J. Micromec. MicroEng. **15** 706

- **Combinatorial Irradiation Studies**

Collaboration with J.-C. Zhao at OSU



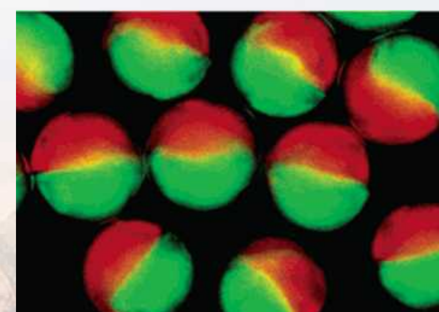
- **Study of the long term stability of glass for nuclear waste**

Collaboration with A. L. Billings at SRNL

- **Janus Particle Fabrication**

Collaboration with J. C. Conrad at U. of Houston

Shepherd et al. Langmuier 2006

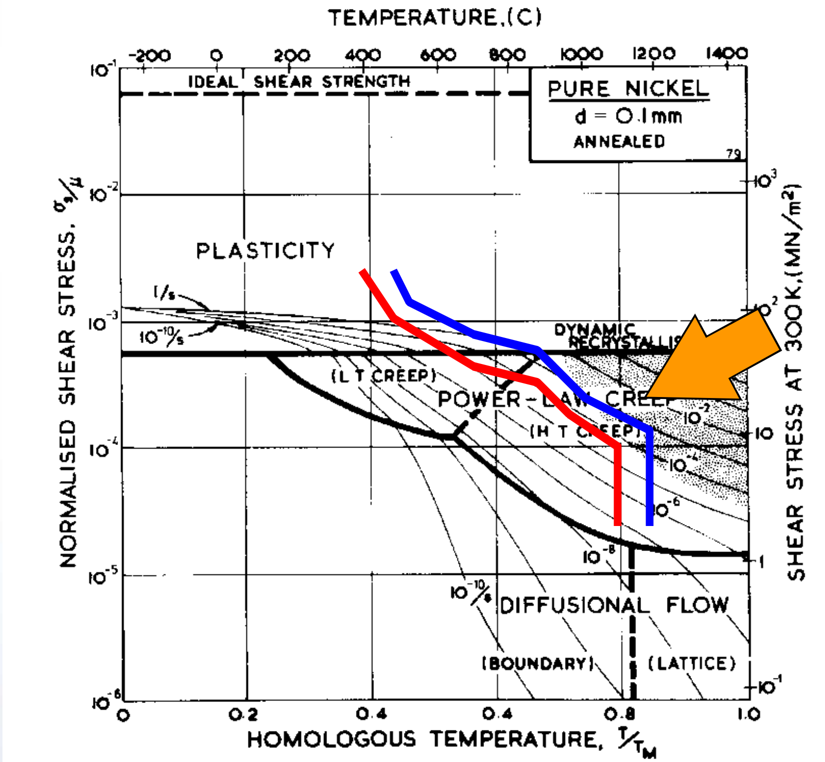


- **And many more…**

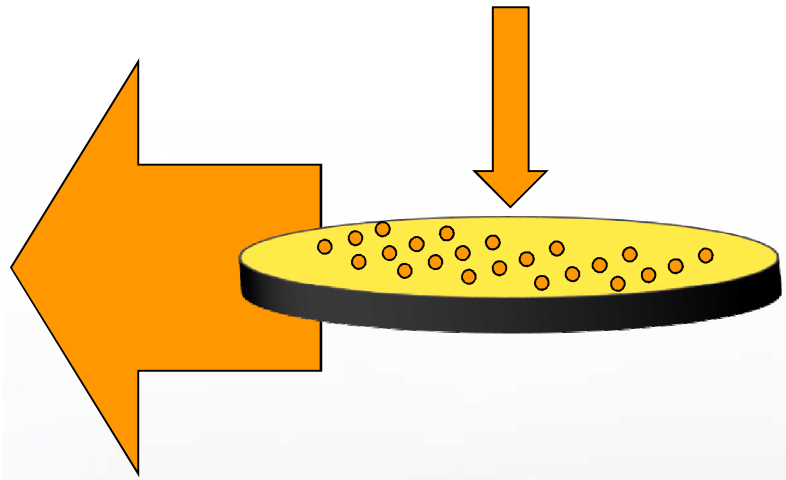
Sandia National Laboratories

Capabilities of Spherical Indentation

Deformation Mechanism Map (Frost & Ashby 1982)



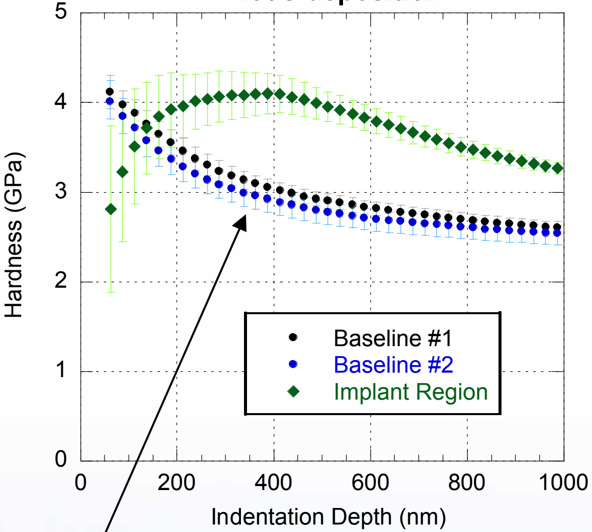
Spherical indentations at temperature in an array



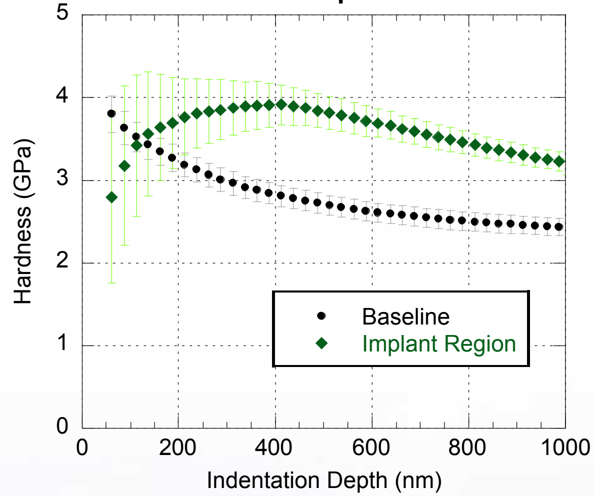
- Spherical indentation will allow plotting of mechanism transition lines
  - Can measure multiple areas to include irradiation effects
- Good complement to micropillar work—averages over many grains

Berkovich Indentation of Irradiated Samples

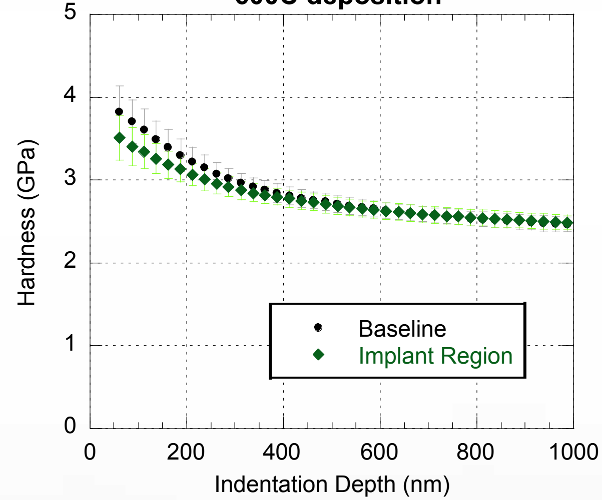
Hardness vs. Indentation Depth
Ion-Implant experiment on Stainless Steel
400C deposition



Hardness vs. Indentation Depth
Ion-Implant experiment on Stainless Steel
500C deposition



Hardness vs. Indentation Depth
Ion-Implant experiment on Stainless Steel
600C deposition



This trend in hardness is typical for the classic indentation size effect

Experiment	Tests	H (GPa)	E (GPa)
400C baseline #1	16	2.46± 0.07	203 ± 10
400C baseline #2	16	2.40± 0.08	202 ± 7
400C implant region	16	3.07± 0.07	197± 7
500C baseline	14	2.30 ± 0.11	195 ± 6
500C implant region	15	3.05 ± 0.10	198 ± 11
600C baseline	16	2.33 ± 0.09	191 ± 8
600C implant region	16	2.36 ± 0.09	190 ± 9