Size Scale Effect on the Mechanical Properties of Irradiated Metals

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Sandia National Laboratories

Naval Post-Graduate School

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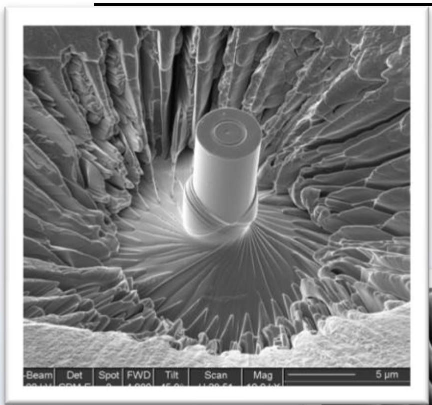
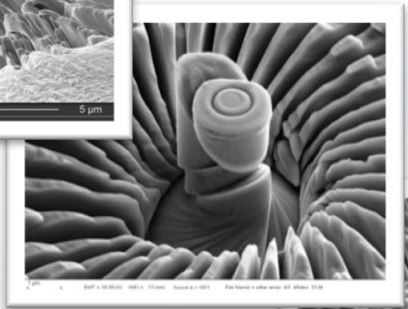
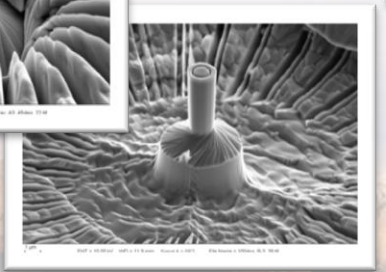
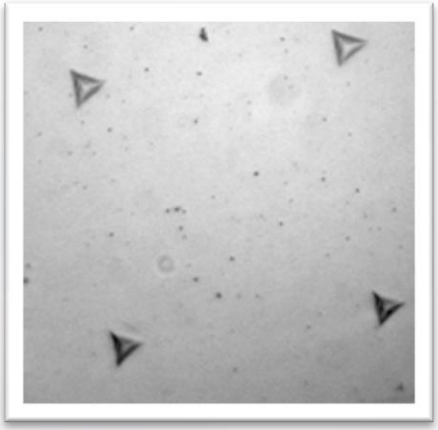
Micropillar Compression of Irradiated Single Crystal Cu

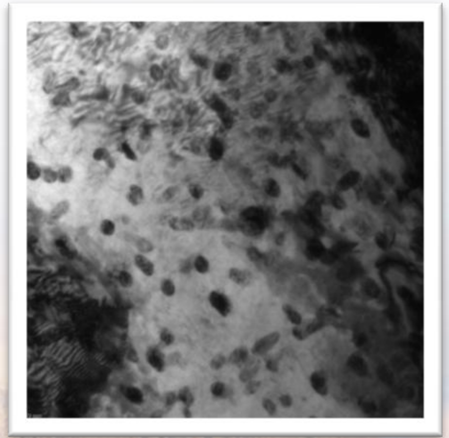






Nanoindentation of Irradiated 316L Stainless Steel





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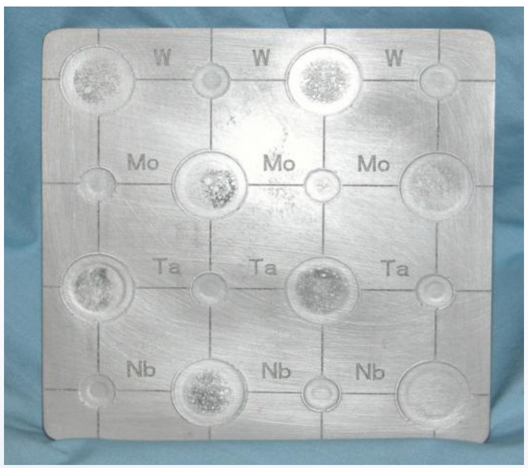
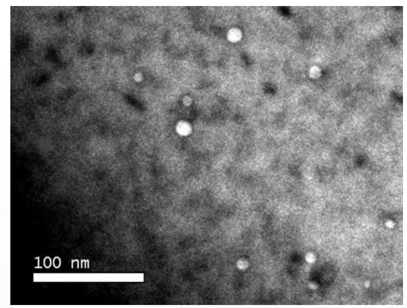
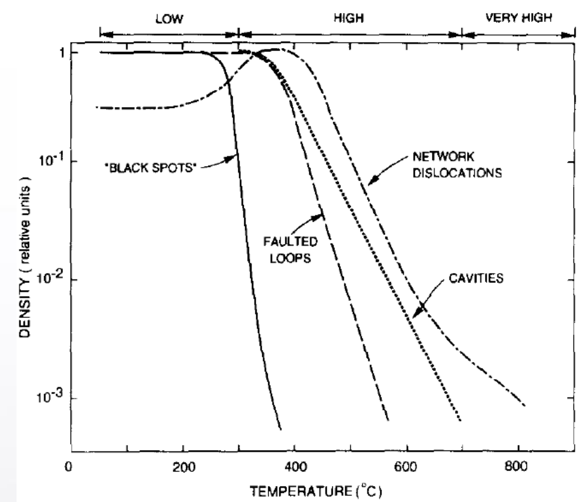
Sandia's Approach to Rapid Material Validation for Advanced Materials Necessary for New Reactors

- Advanced Materials are Needed
- Several Theories exist for the desired microstructure
- New materials have been made
- Current Neutron fluxes require decades for testing





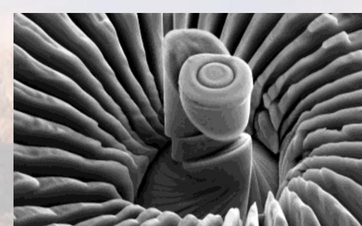


Microstructural Characterization (XTEM)

Mechanical Properties (small-scale testing)

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

Validating Comparison to Neutron Irradiation Experiments + Investigation into new materials



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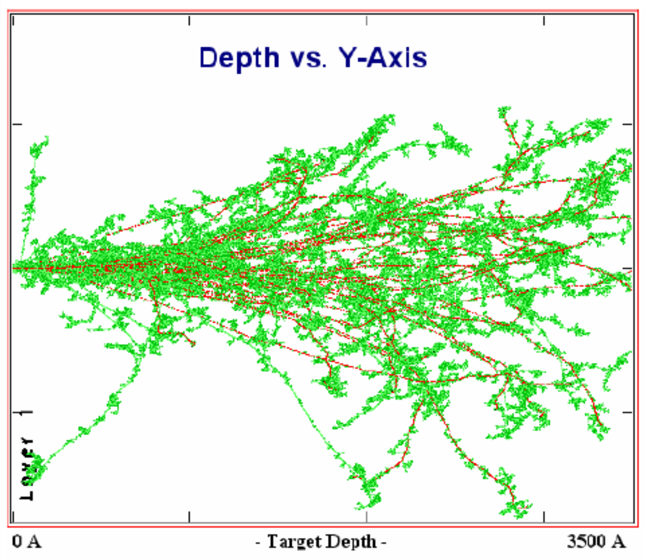
Length Scale Limitations due to Ion Irradiation

Advantages

- High total damage in short periods of time
- Relatively accessible

Disadvantages

- Unknown effect of damage rate
- Limited to small volumes
- Heterogeneous microstructure



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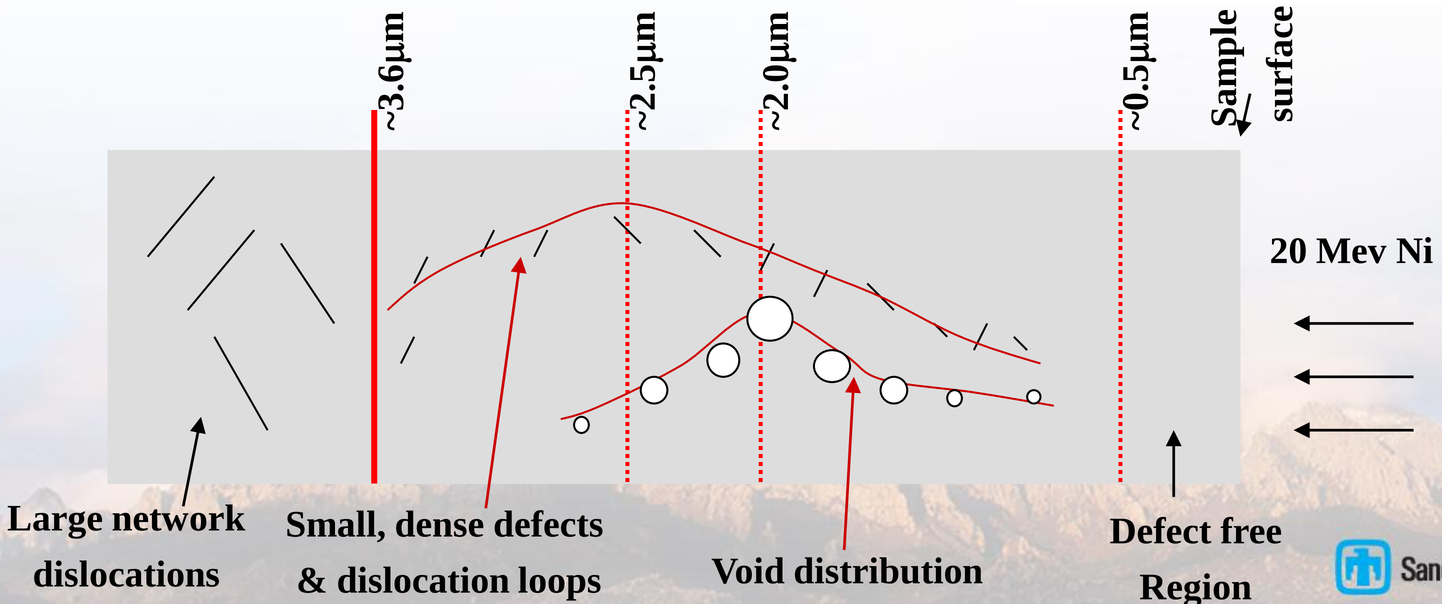

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Micropillar Compression Experiments

Sample Preparation:

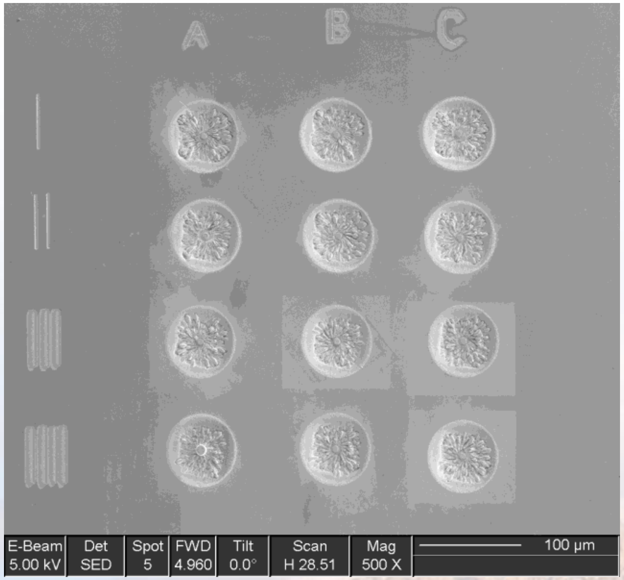
- Copper single crystals (FCC)
- Different crystallographic orientations: (100), (110), and (111)
- Self-ion Implants at 30 MeV to
- 0 (control), 50 dpa, and 100 dpa.

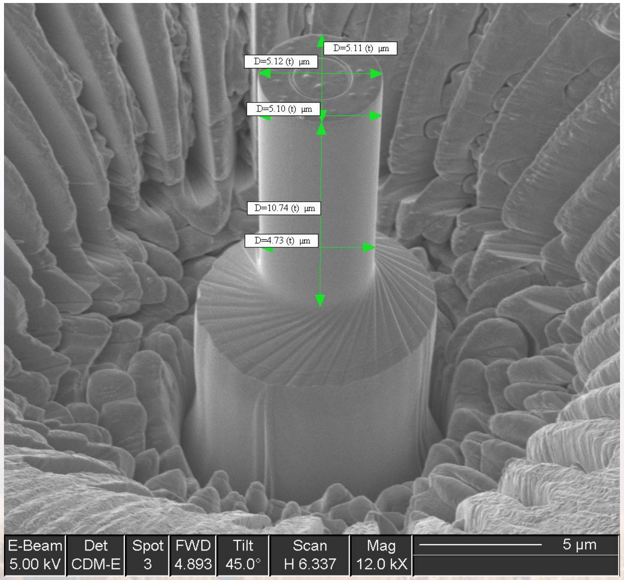
Pillar Manufacturing:

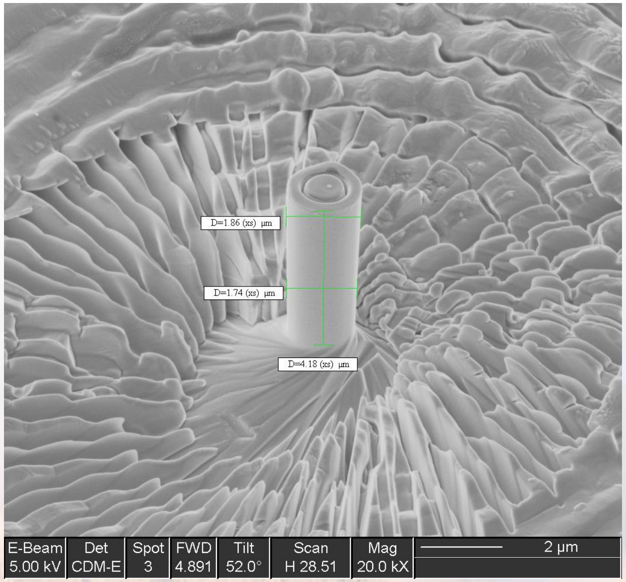
- We employ Uchic's FIB lathe machining process for straight-walled cylinders.
- Array of at least 9 nominally identical pillars tested per condition to assess statistical variability.
- Height varies from 4 µm to 10 µm

Compression Testing:

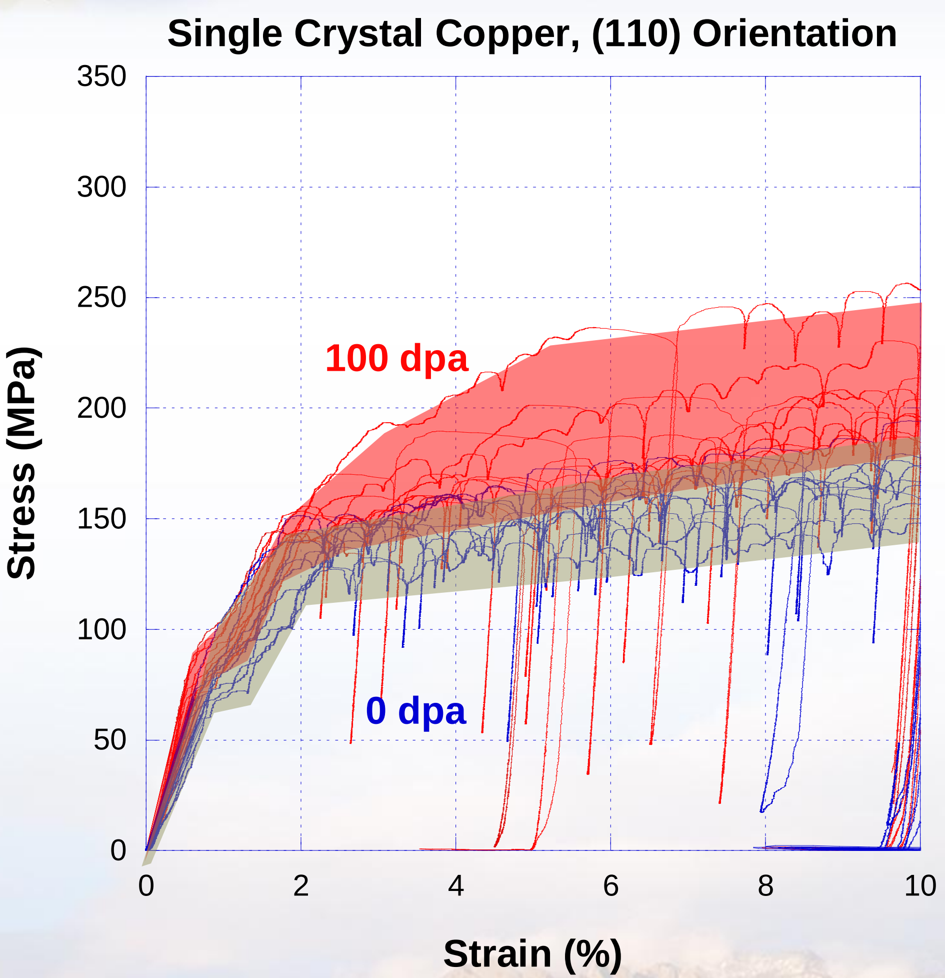
- Hysitron Performech Nanoindenter permits <1 nm and <1 µN resolution.
- 25 µm flat ended cone indenter in feedback displacement control, rather than typical force control.
- Pillars compressed 10% strain at a strain rate of $0.025\ s^{-1}$.

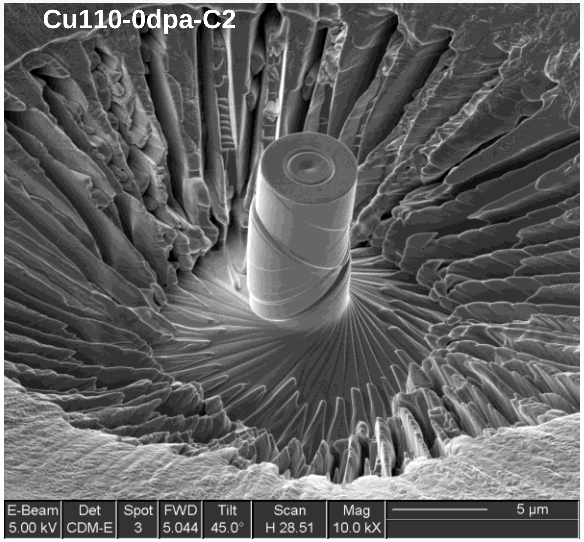


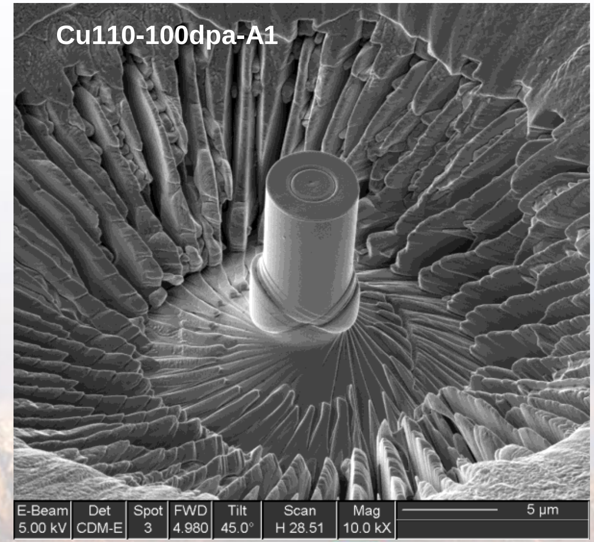




Large Micropillar Compression







Minimal difference between the control and irradiated 10 µm-tall pillars. Slip occurred in the bottom fraction of the pillars.

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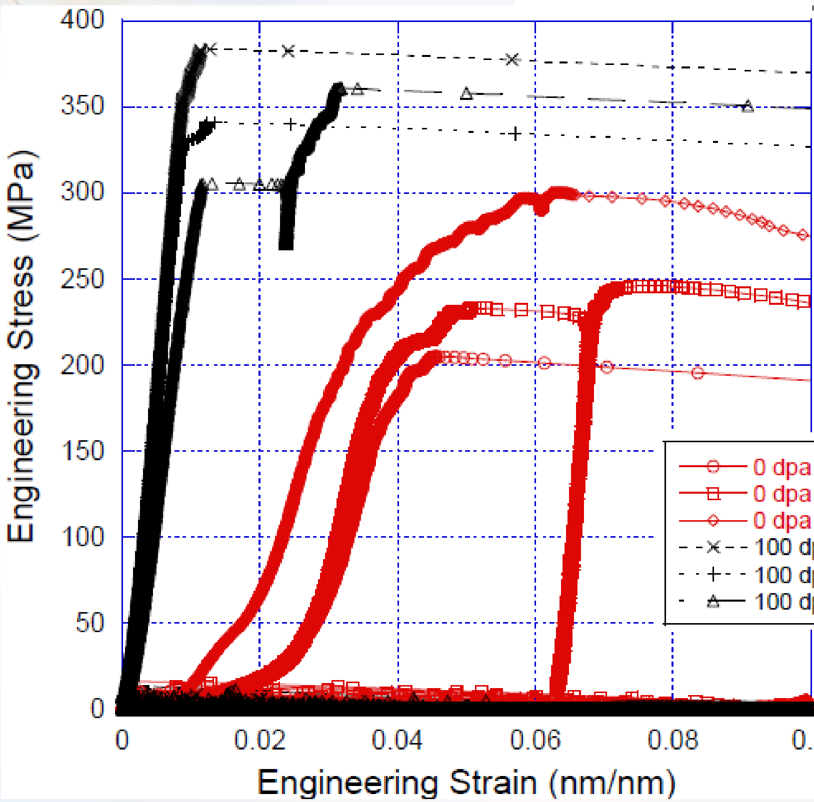
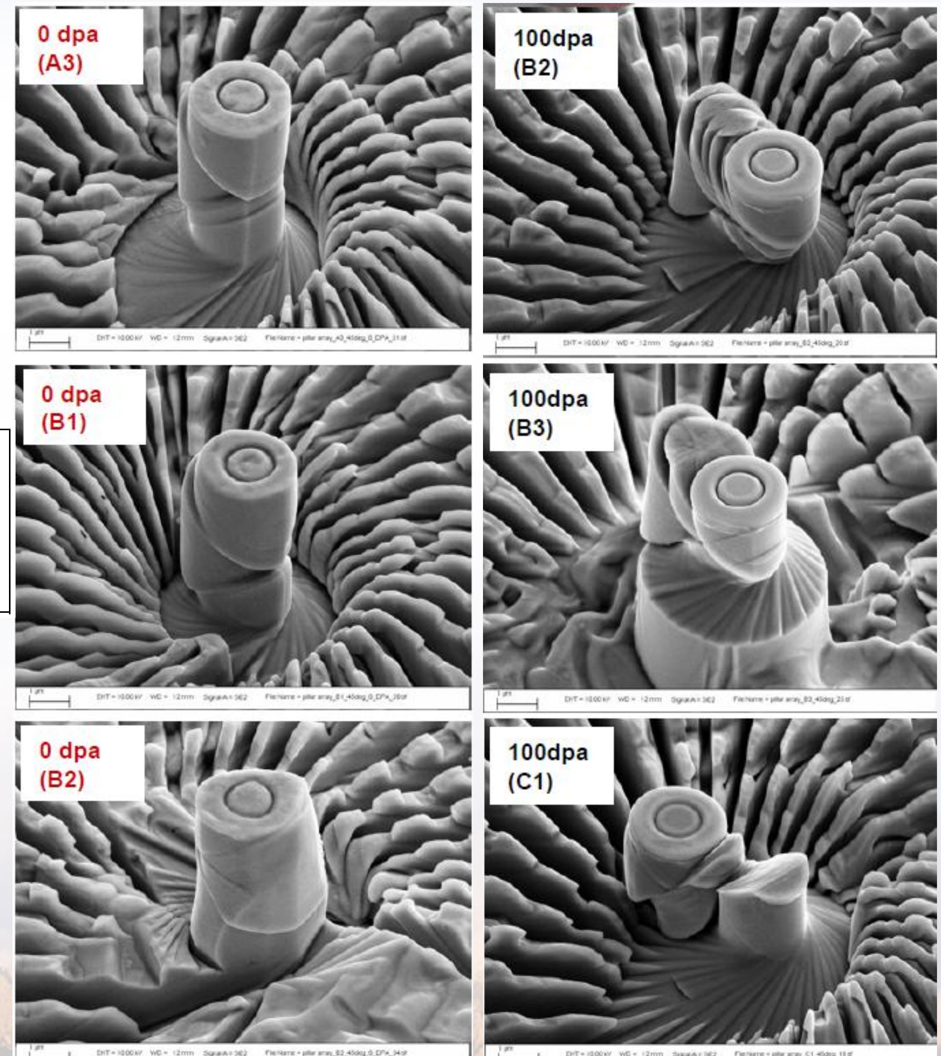
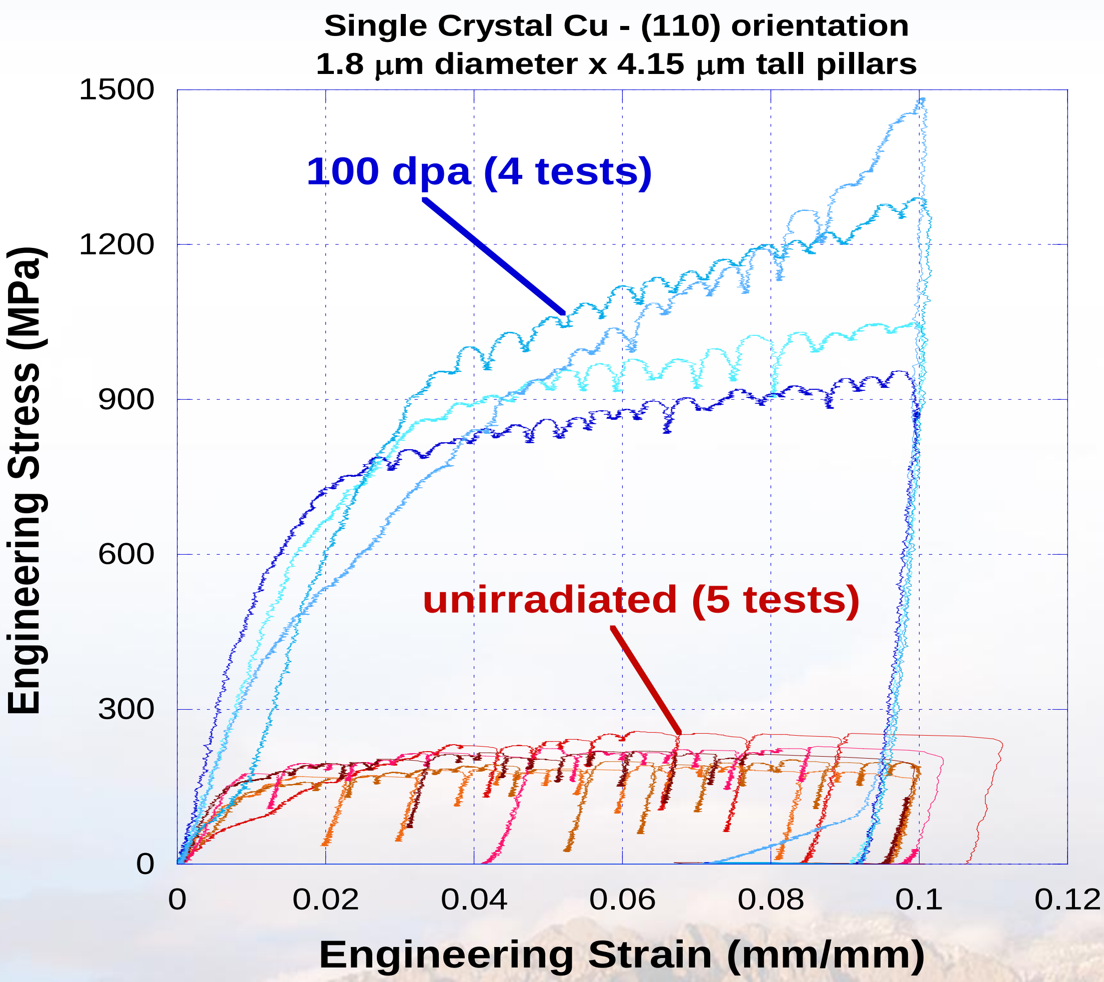
Intermediate Micropillar Compression

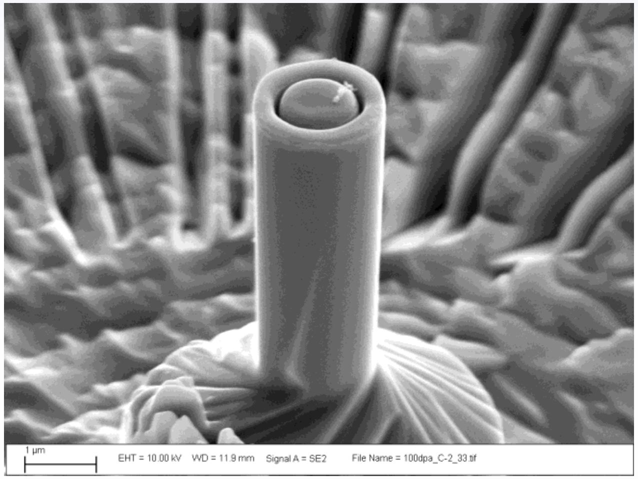


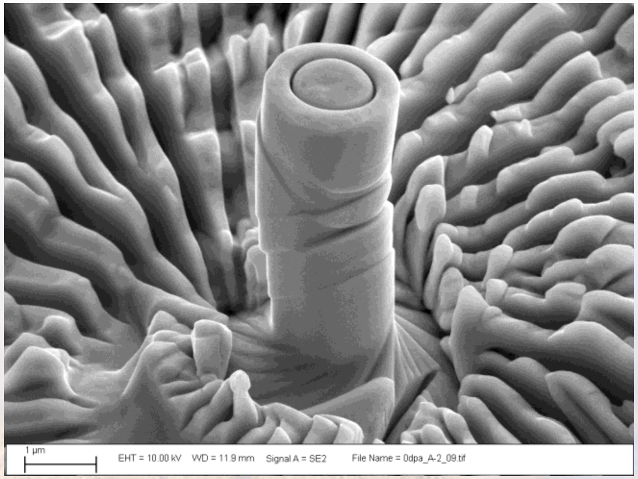


5 µm-tall pillars show greater distinction with catastrophic failure

Small Micropillar Compression







Initial tests indicate that the 4 μm-tall pillars are 5 times stronger and show no signs of slip band formation

Testing of Irradiated Stainless Steels

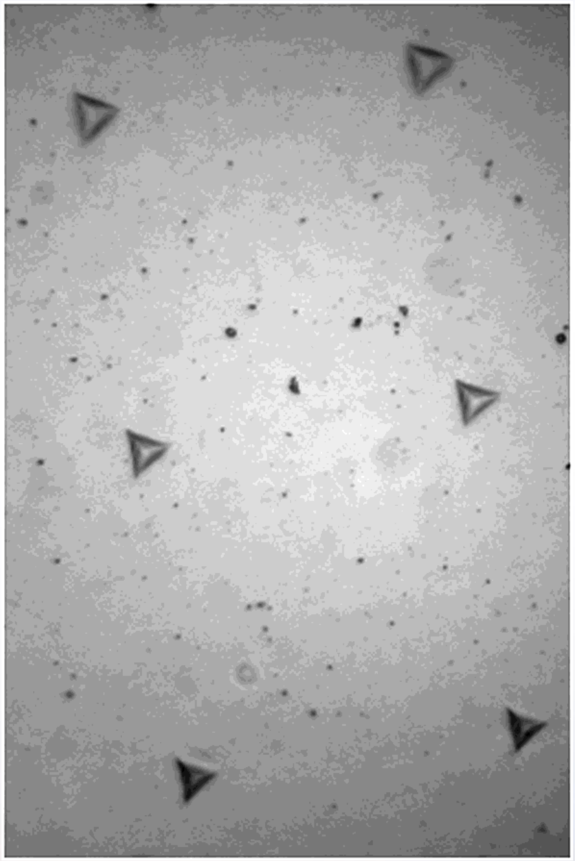
- Micropillar is difficult for many polycrystalline materials
  - Due to the dependence of FIB milling rate on orientation

To validate the approach:

1. Metals previously tested by Neutron Irradiation must be tested
2. The effect of temperature and various ion characteristics must be considered

Thus, we irradiated

- 420, 409, and 316L SS
- Approximately 10 dpa, 40 dpa, and 100 dpa
- Temperatures of 400 ºC, 500 ºC, and 600 ºC



Three steel compositions were irradiated under various conditions. Nanoindentation was selected as the optimal small scale testing method.

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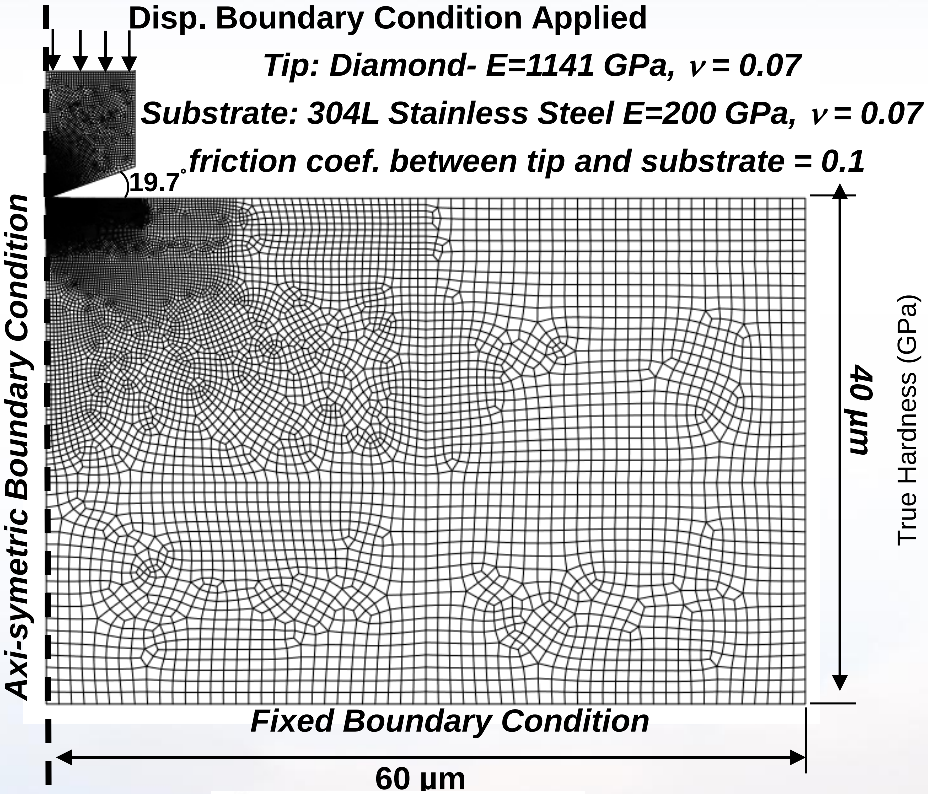
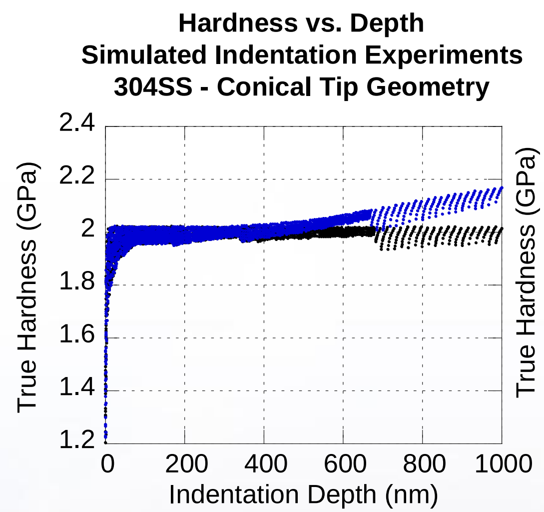
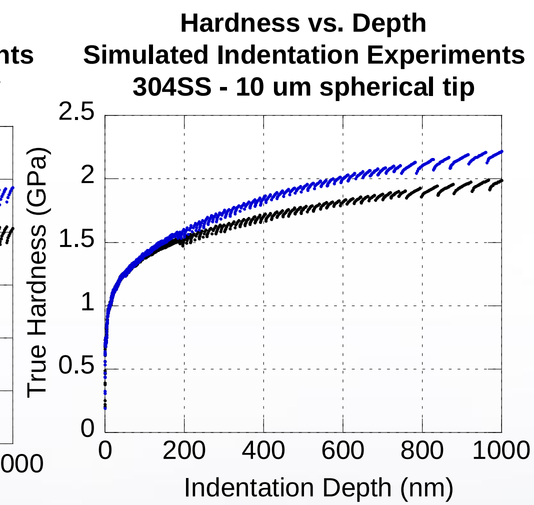
Finite Element Simulations for Spherical Indentation

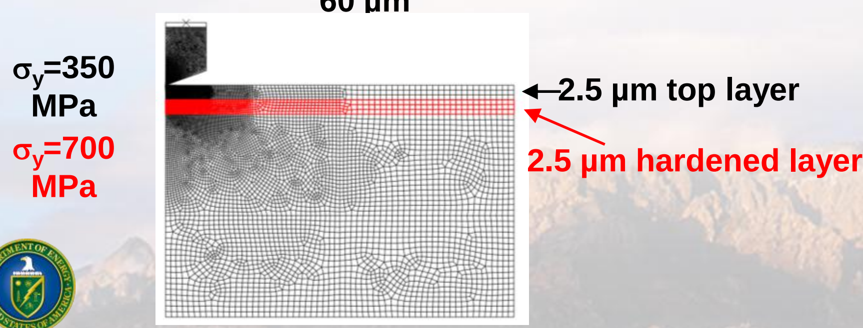






Without hardened subsurface layer

With hardened subsurface layer



Deviations due to ion irradiation are expected from both spherical and conical indentations

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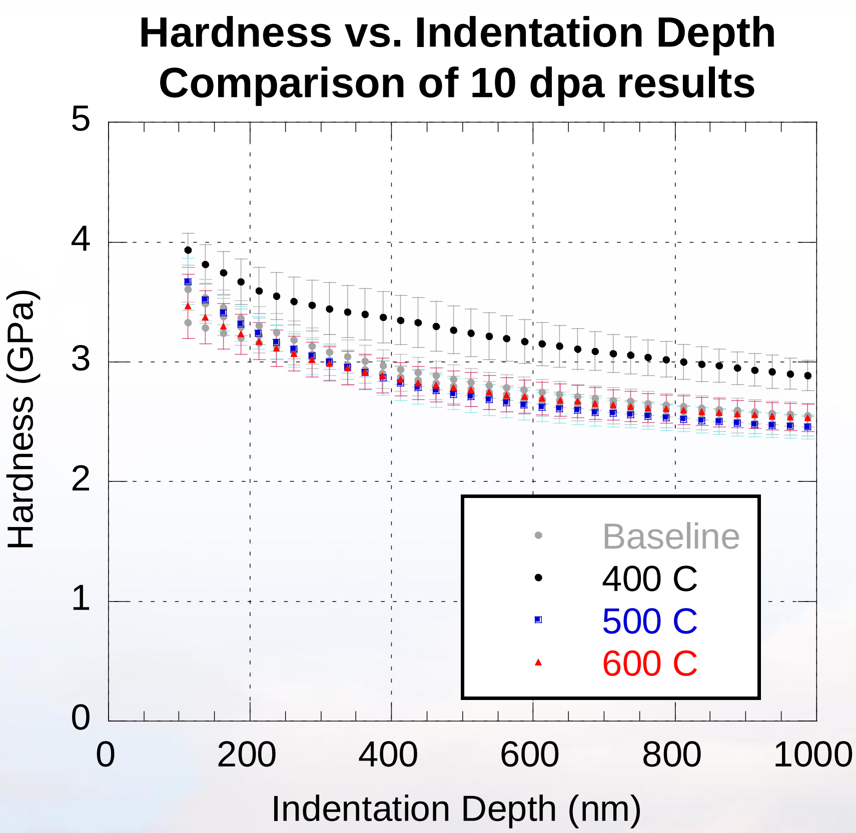
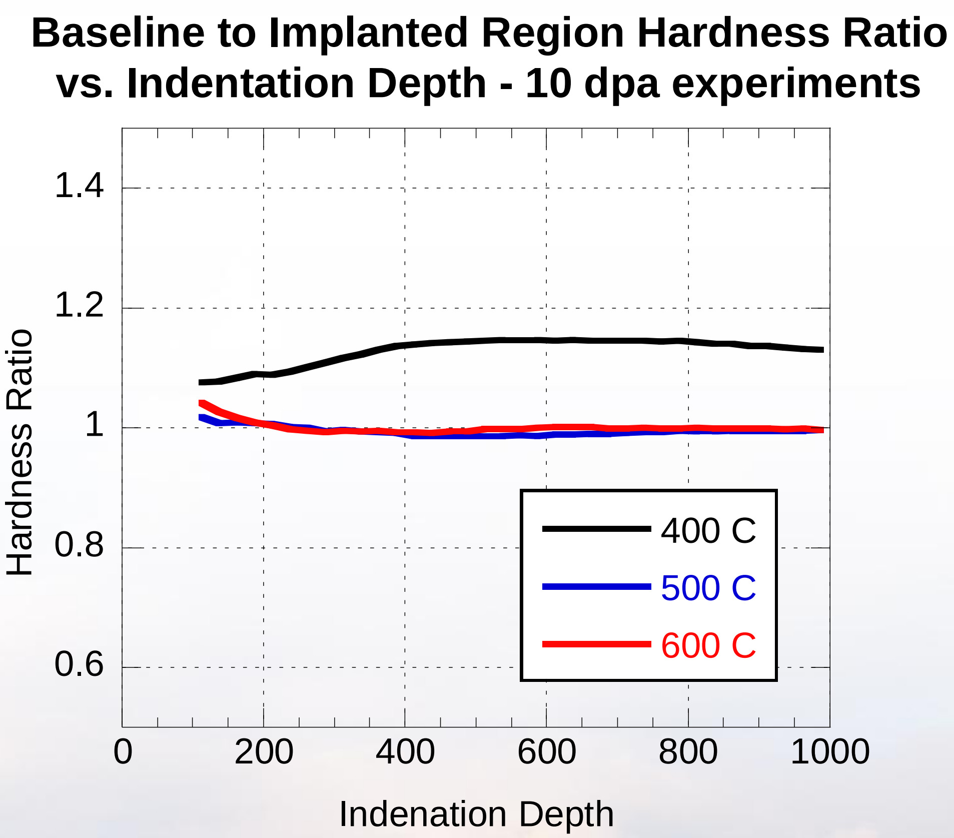
Berkovich Indentation of 10 dpa Irradiated Samples

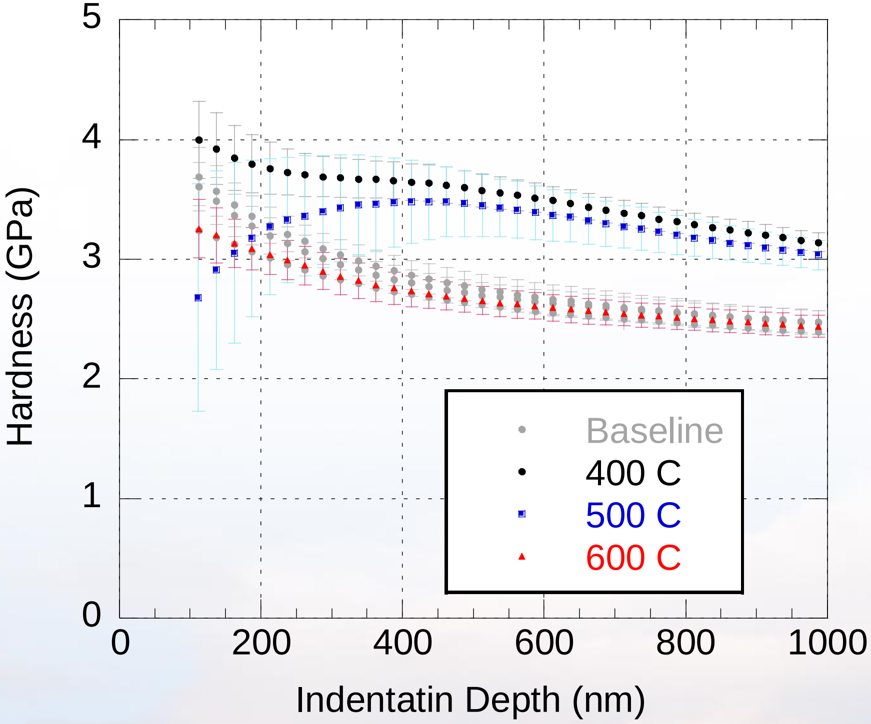




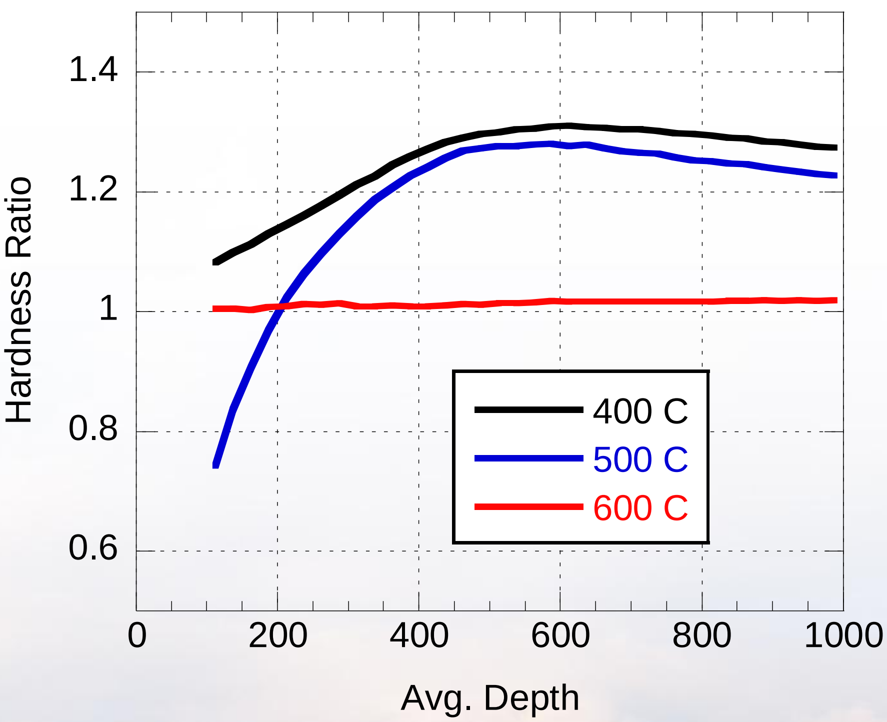
At 10 dpa, only the 400 ºC sample is significantly harder than the control microstructure.

Berkovich Indentation of 40 dpa Irradiated Samples

Hardness vs. Indentation Depth Comparison of 40 dpa Results



Baseline to Implanted Region Hardness Ratio vs. Indentation Depth - 40 dpa experiments



At 40 dpa, both the 400 ºC and 500 ºC sample are significantly harder than the control microstructure.

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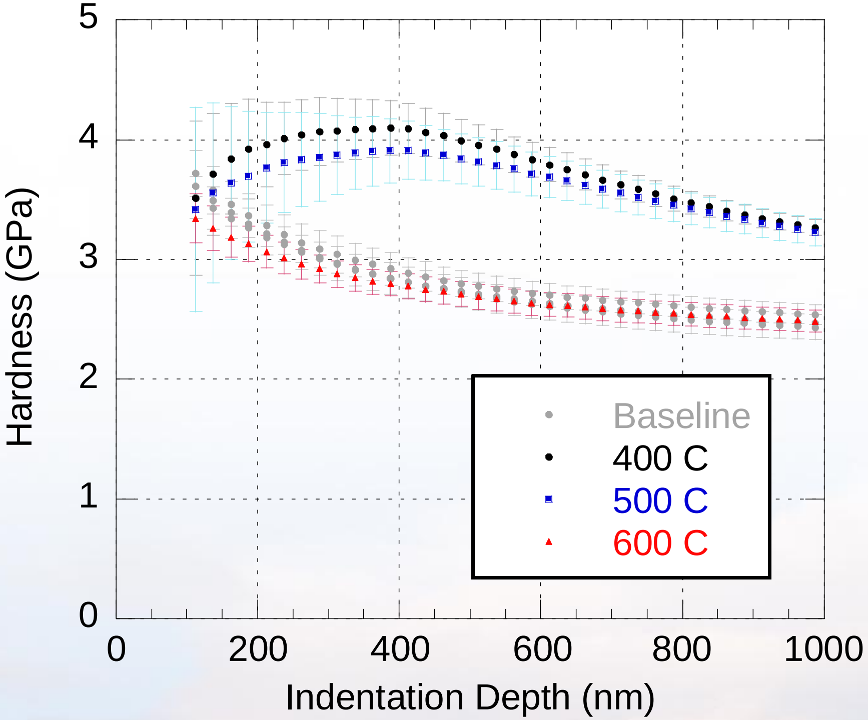
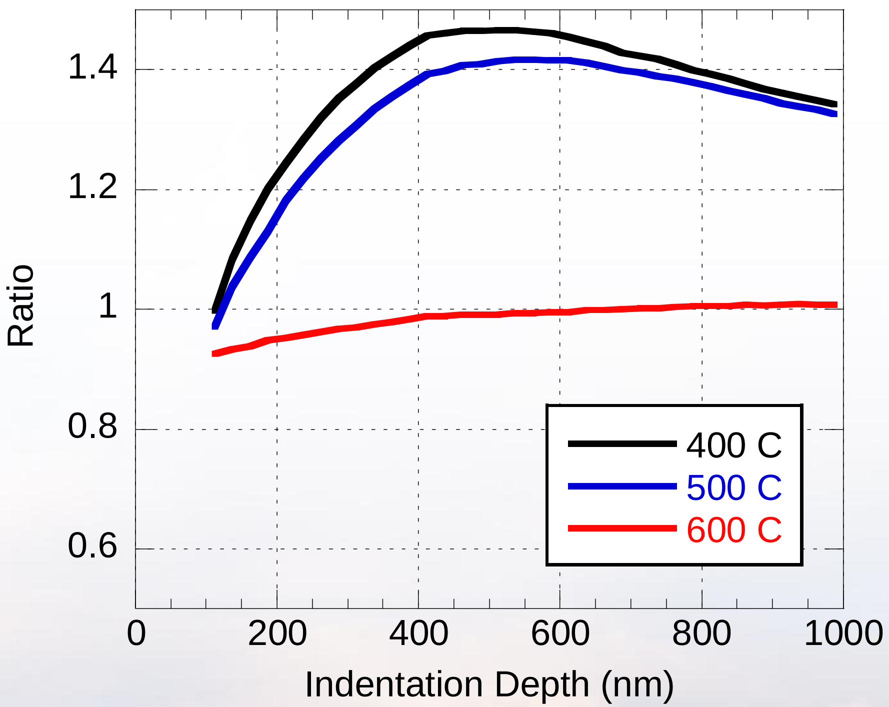
Berkovich Indentation of 100 dpa Irradiated Samples

Hardness vs. Indentation Depth Comparison of 100 dpa measurements



Baseline to Implanted Region Hardness Ratio vs. Indentation Depth - 100 dpa experiments



At 100 dpa, the hardness difference between 400 ºC and 500 ºC sample and the control microstructure has increased.

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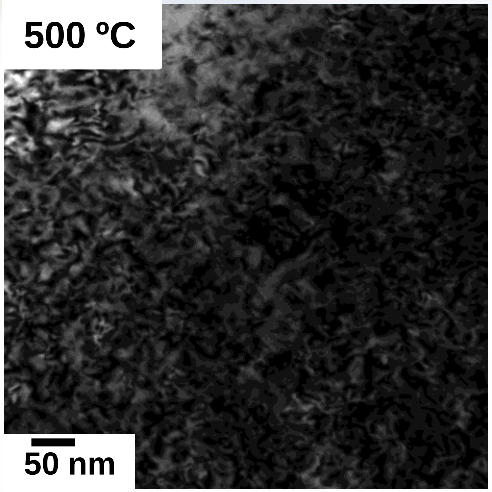
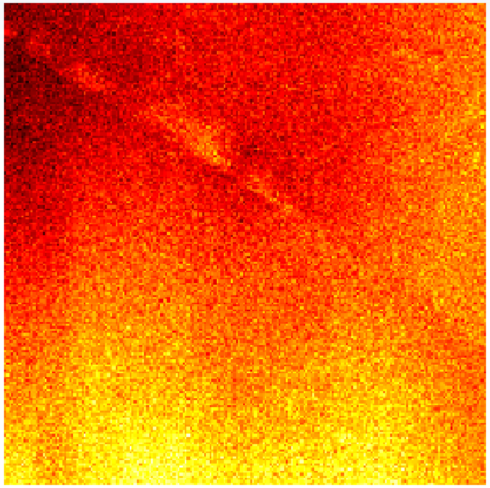
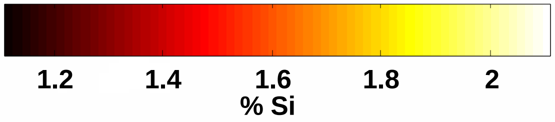
Microstructural Evolution between 500 ºC and 600 ºC

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

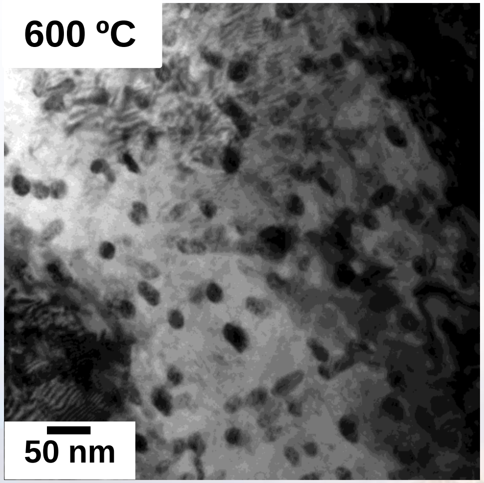


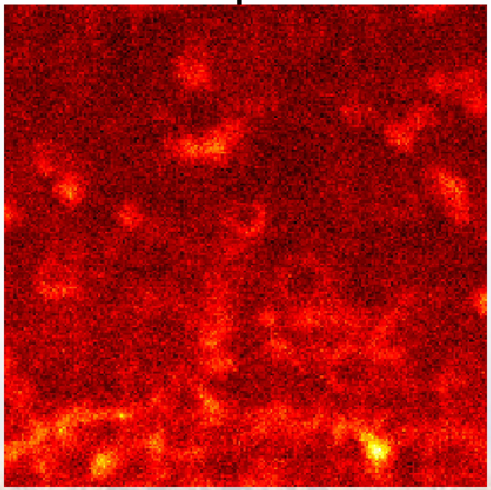


2 µm

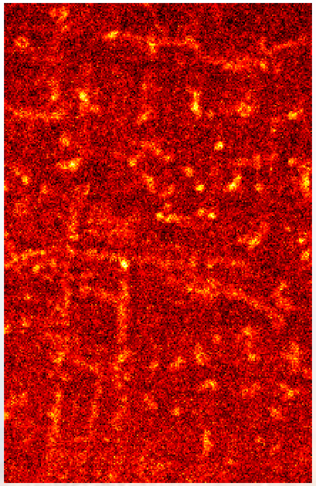


- Large number of small defects present in the irradiated region
- No significant segregation of either the Ni or Si constituents



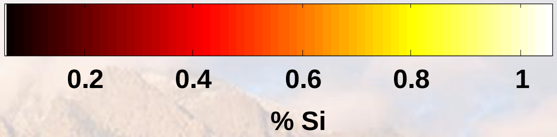


500 nm



1µm x 2µm

- Voids are formed and are self-ordered
- Significant segregation of either the Ni or Si constituents



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

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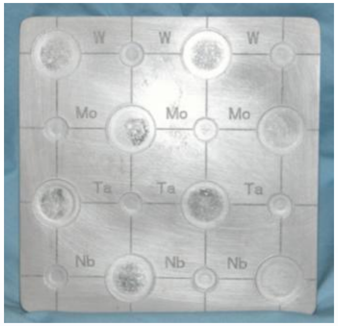
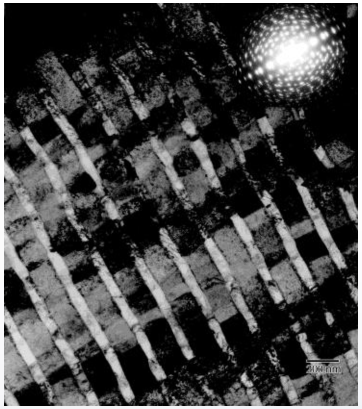
Conclusions

- Developed a combinatorial approach to rapidly test the radiation damage for nuclear energy applications
  - Ion Implanted to high dpa using heavy ion irradiation
  - A variety of small-scale mechanical property testing
    - Berkovich and spherical indentations
    - Micropillar compressions

Length scale effects are critical to the success of any technique to characterize new materials for future nuclear reactors





Future Work

- Detailed microstructural analysis of the irradiated and deformed regions
- Employ a gradient FEM based on microstructure
- Implantation followed by thermal and mechanical characterization of other advanced materials: Diffusion couples and Engineered interfaces

Acknowledgements

- All staff and technologists of the IBL at SNL
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