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Sandia National Laboratories

In-situ Irradiation Induced Microstructural Evolution and Micro-Mechanical Properties of TPBAR 316 Stainless Steel Cladding

PRESENTED BY

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CAARI
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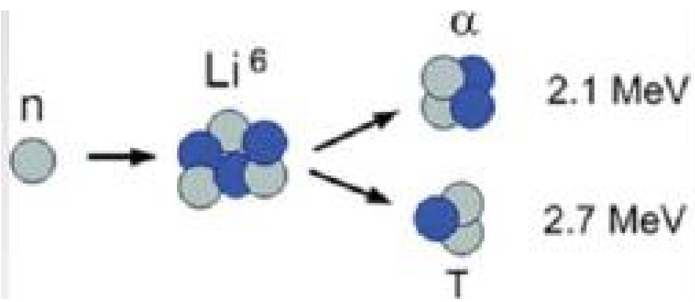
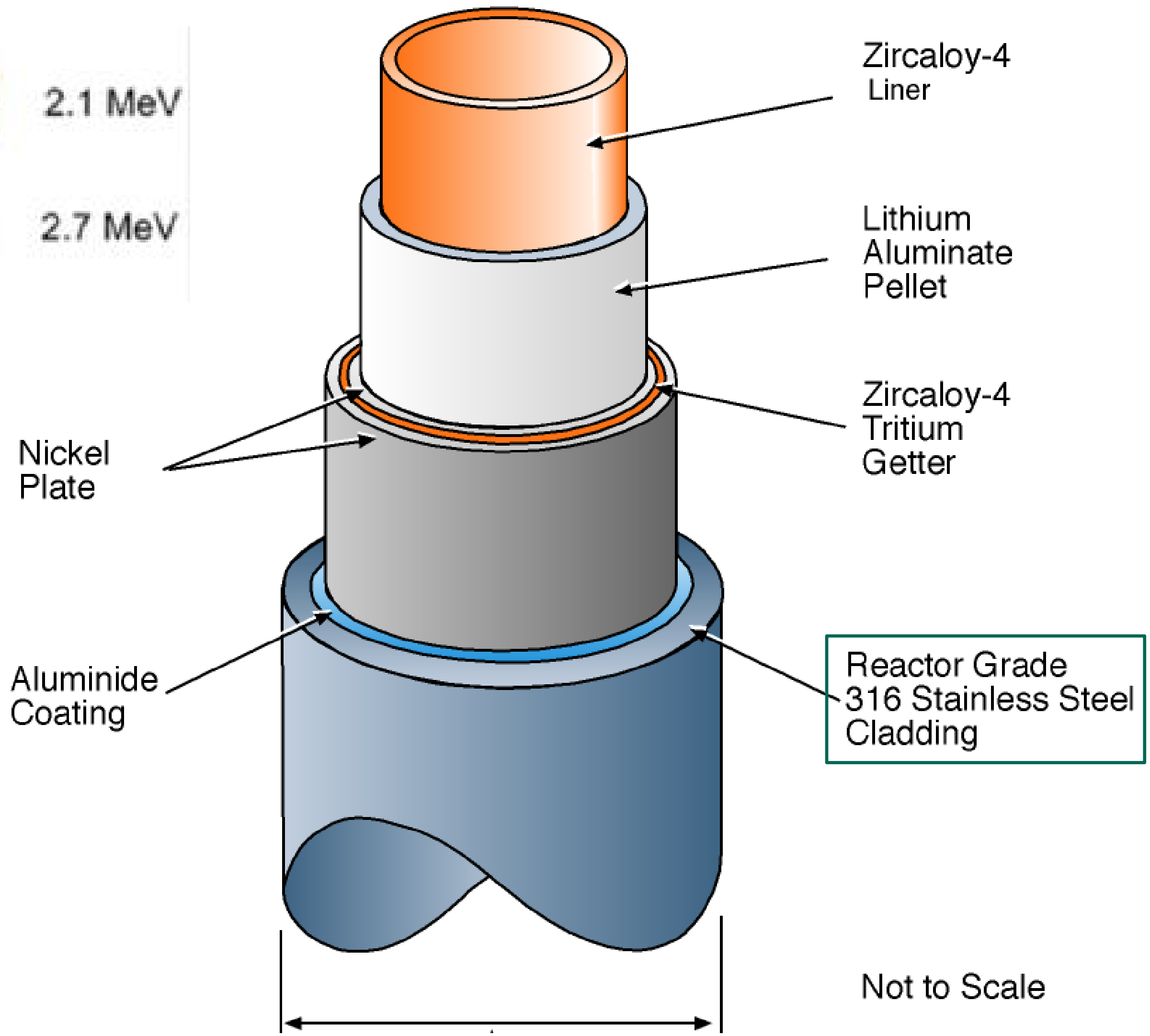
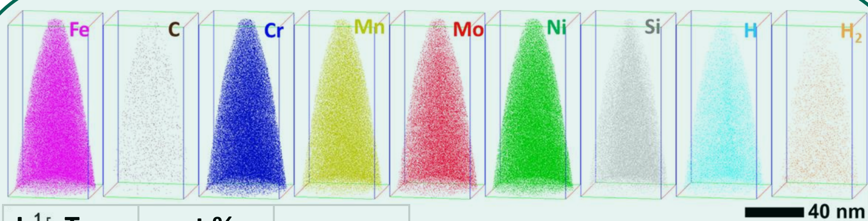
Tritium Producing Burnable Absorber Rod



- Displacement Damage
- Helium Implantation
- Tritium Implantation
- Elevated Temperatures
- Mechanical stresses



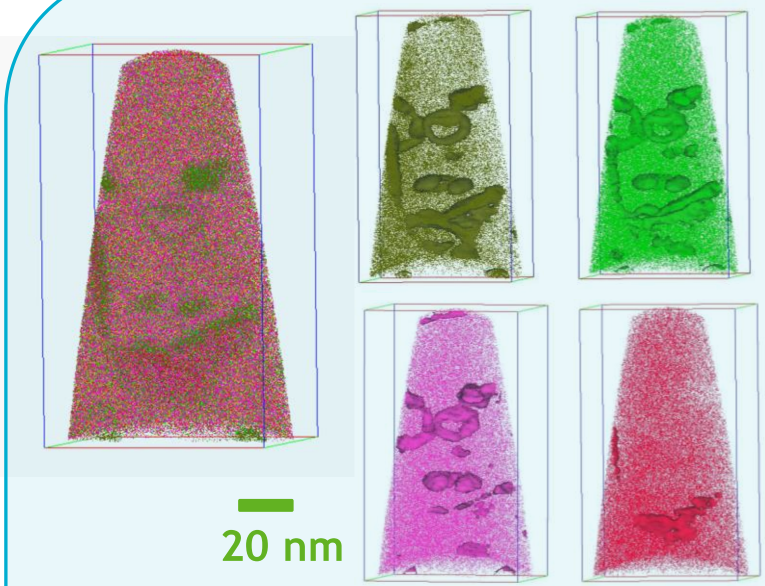
316 SS used as cladding material, but undergoes microstructural changes under neutron irradiation



Ion Type	at %	error
Fe	65.407	0.054
Cr	18.124	0.024
Ni	11.430	0.018
Mn	1.656	0.007
Si	1.271	0.006
Mo	1.208	0.006
H	0.812	0.005
C	0.066	0.001
P	0.028	0.001

APT shows uniform distribution of all solute elements and impurity element distribution.

Pre-Irradiation
Uniform elemental distribution



- 5 dpa
- 300 C

Si 4 at%
Ni 17 at%
Cr 13 at%
Mo 4 at%

Post-Irradiation
Ni-Si-Cr precipitates

1. **What affect does this precipitation have on mechanical properties?**
2. **Can we mimic this process with ion beams?**

Ion irradiation used to mimic neutron damage

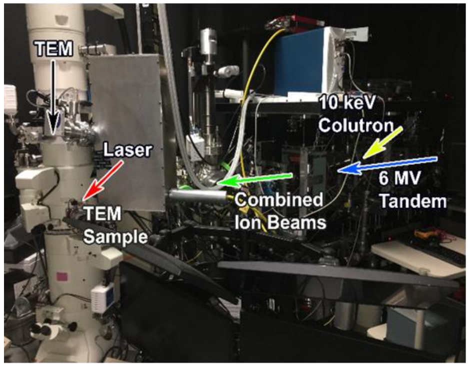
Why utilize ion beams?

- Higher damage rates
- No activation
- Faster and lower cost

But,

- Only study knock-on damage
- Need higher temperature

I3TEM at SNL



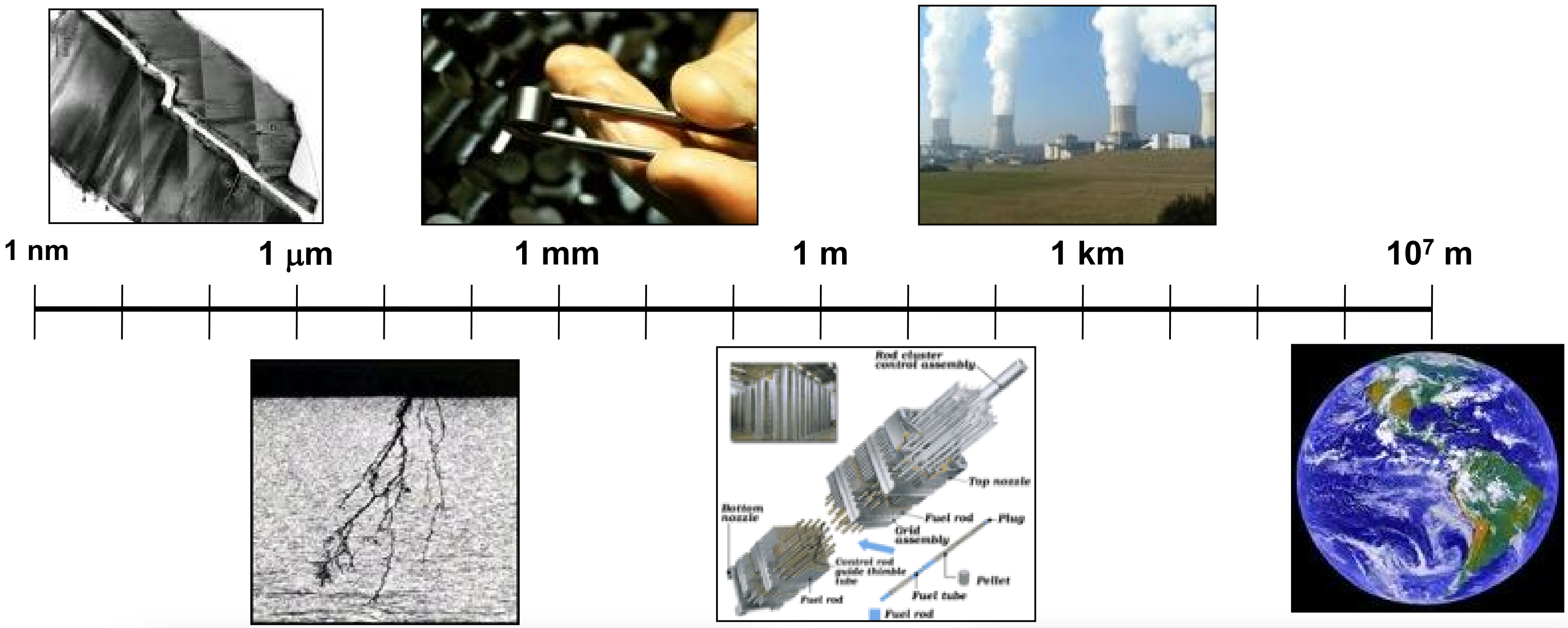
6 MV Tandem Accelerator

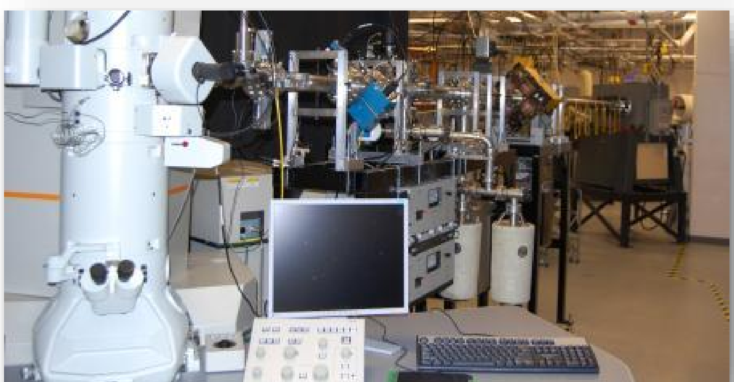
Sandia National Labs is well-suited to study ion irradiation induced effects on materials

In situ **Ion Irradiation TEM (I^{3}TEM)**

Ion Beam Lab (IBL)

Investigating the nm Scale to Understand the km Scale

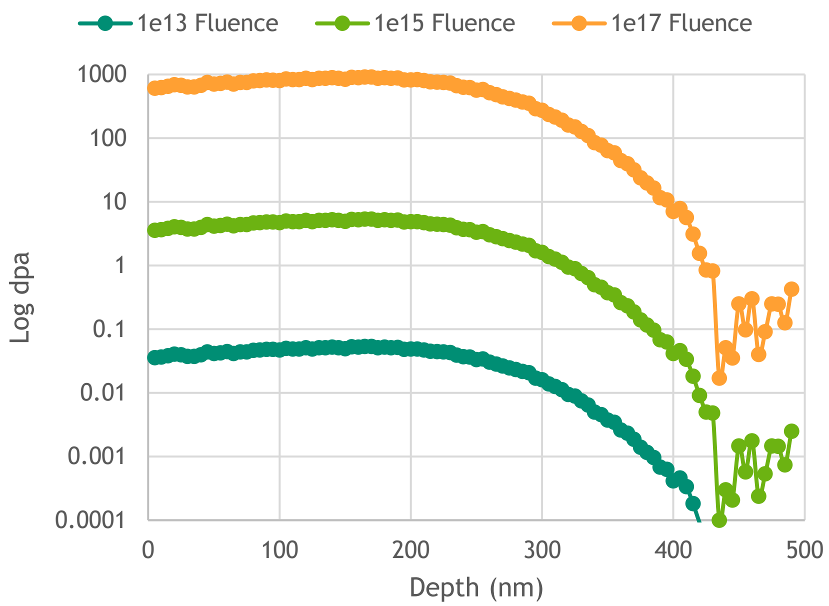


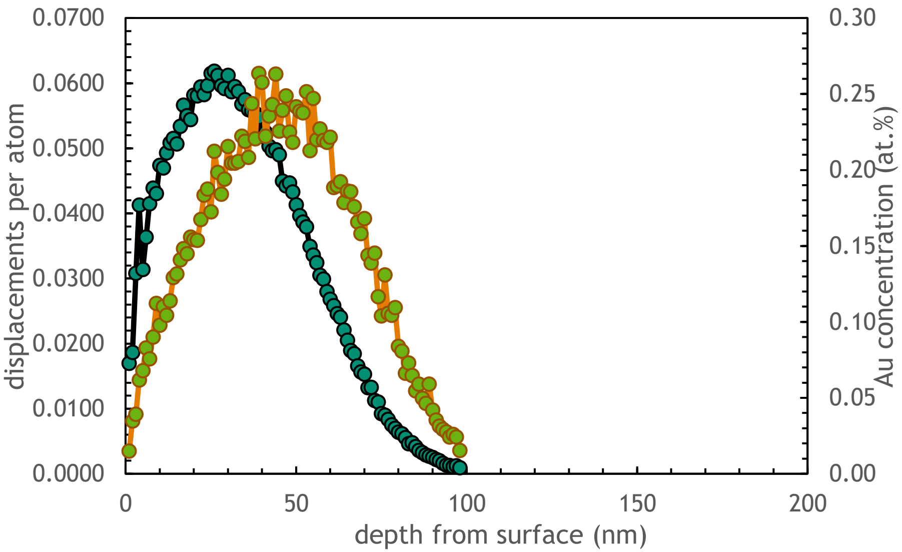


In situ Ion Irradiation TEM (I^3TEM)

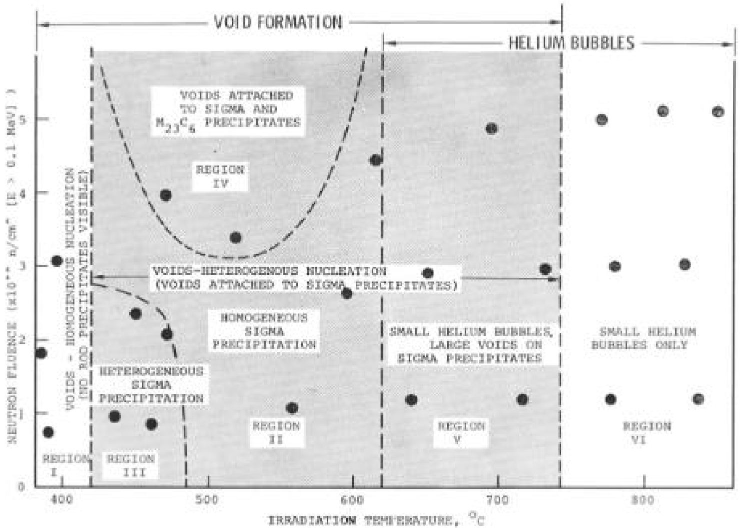
Ion Beam Lab (IBL)

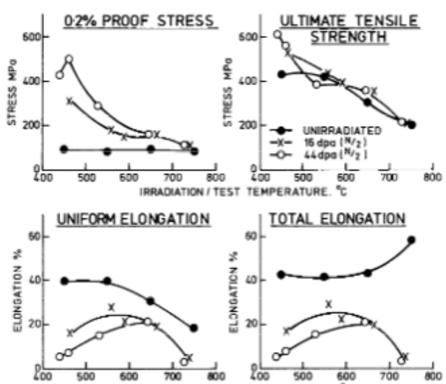
Can we accurately mimic the neutron damage with high dose, elevated temperature ion damage?

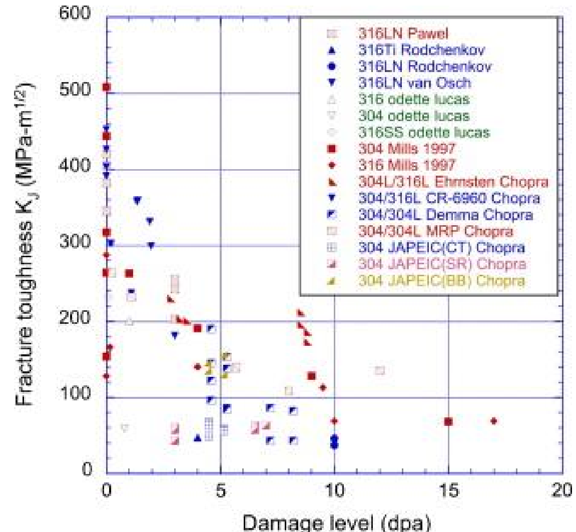


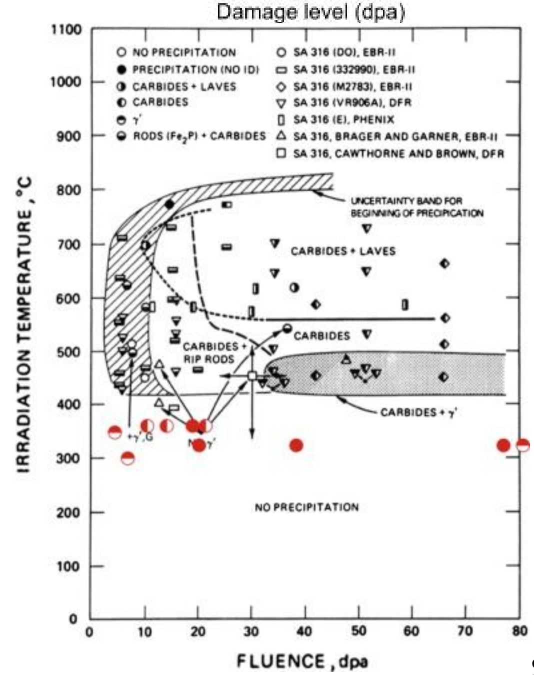


But how does irradiation affect the material properties?









Act synergistically to cause embrittlement, decrease in fracture toughness, hardening

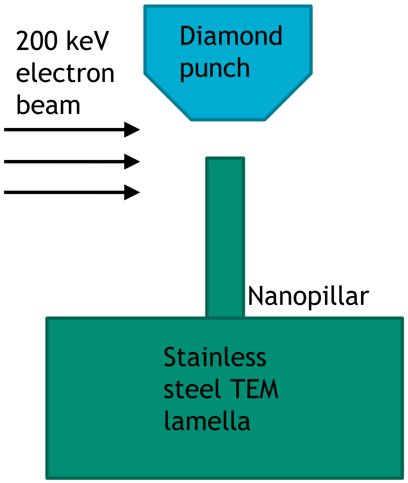
Void swelling, precipitates, helium bubbles as a function of irradiation dose and temperature

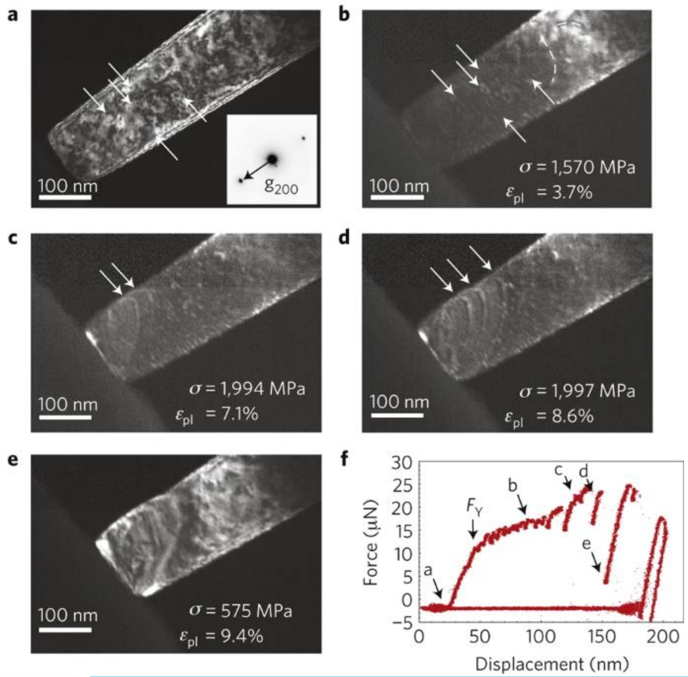
S.J. Zinkle, G. Was, Acta Materialia, 2013.

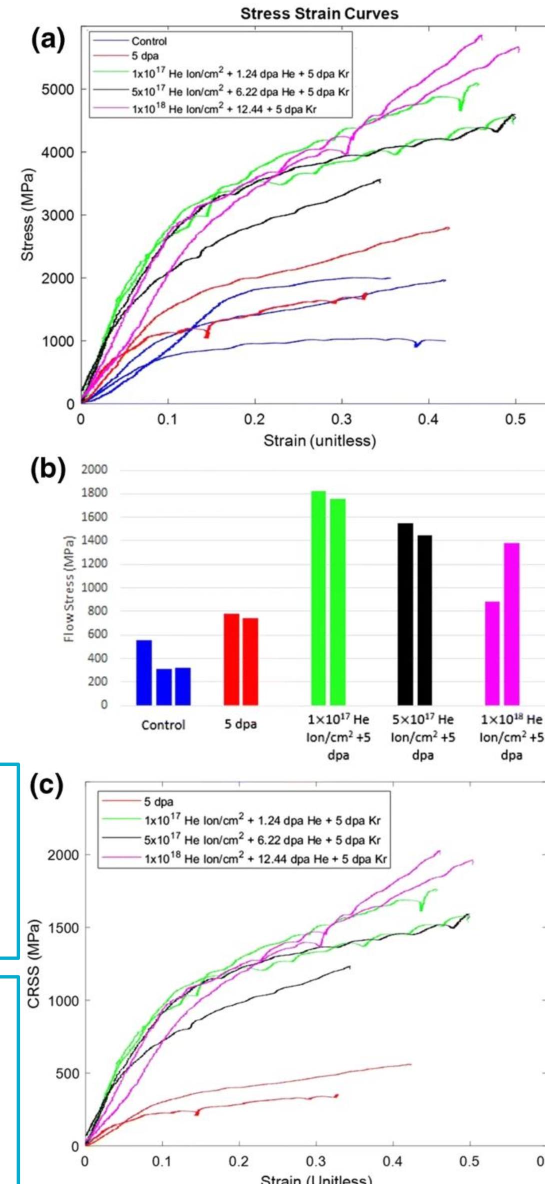
H.R. Brager, JNM, 1973.

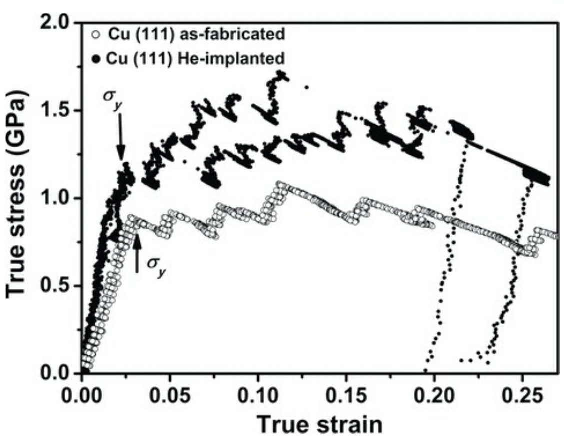
D.R. Harries, JNM, 1979.

Small-scale mechanical testing useful for irradiated samples and visualizing deformation in real-time









Prior work on Cu nanopillars, and 304 SS - but only single crystals

Nanopillar compression well established for analyzing irradiated materials and visualizing deformation mechanisms

R.M. Schoell, JOM. 2020
D. Keiner, Nature Mater. 2011
Q. Guo, Small, 2012.

In situ Mechanical Testing

Qualitative "Bulk" Mechanical Testing

Minimal control over displacement and no "out-of-box" force information

- Successful in studies in observing dislocation-GB interactions/mechanisms
- Ideally both grains have kinematic BF 2-beam conditions: challenging in ST holder

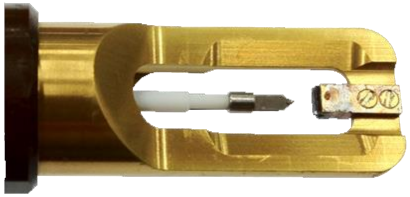
Quantitative Mechanical Testing

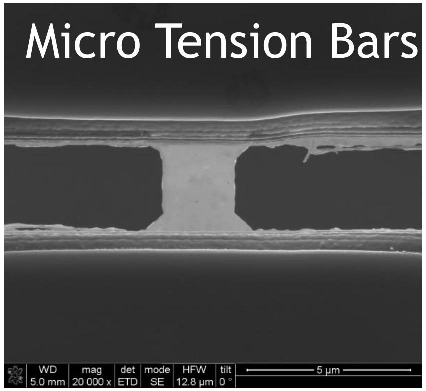
Minimal control over displacement and no "out-of-box" force information

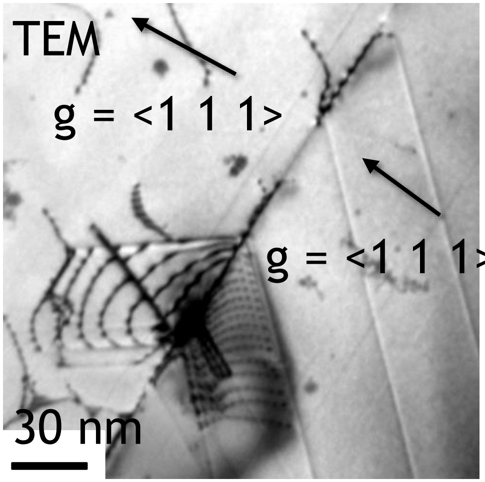
- Sub nanometer displacement resolution
- Quantitative force information with μN resolution

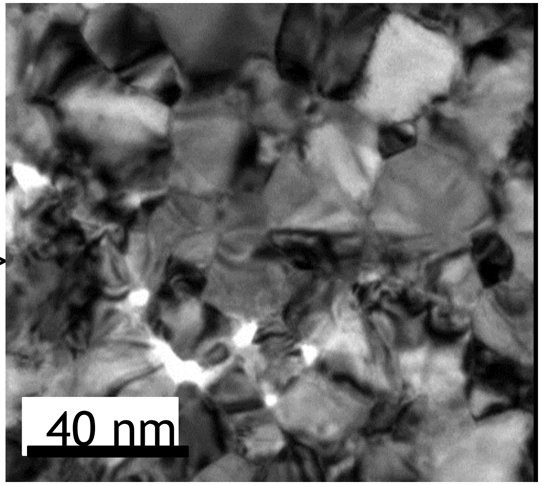
Traditional Gatan Heating and Straining Holder

Hysitron PI-95 Holder







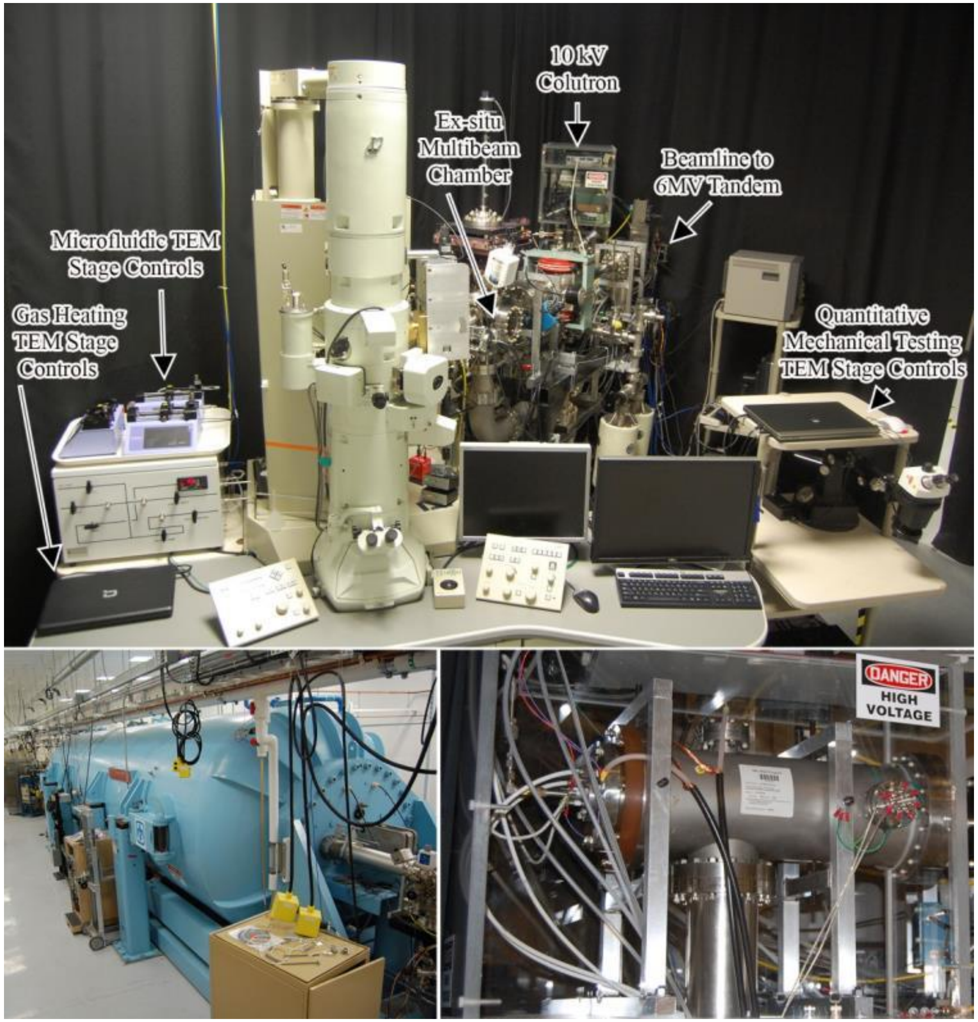


1) Indentation
2) Tension
3) Fatigue
4) Creep
5) Compression
6) Bend

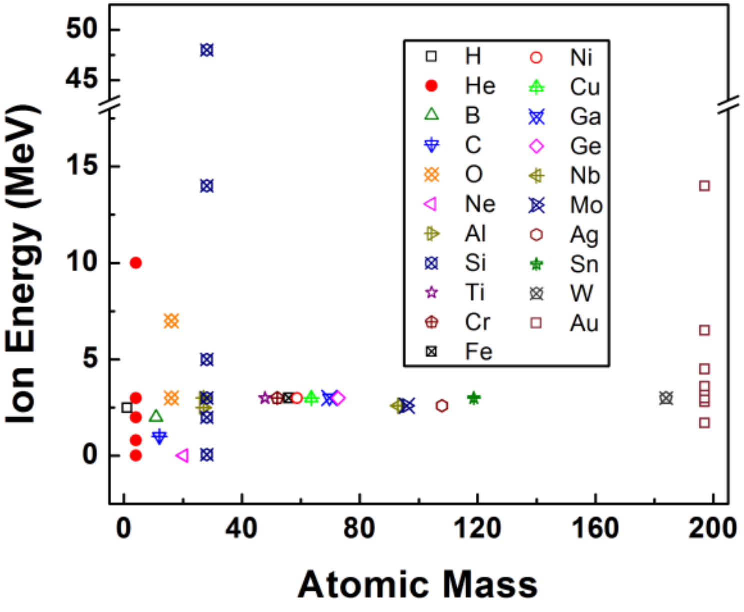
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

10 KV COLUTRON - 200 KV TEM - 6 MV TANDEM

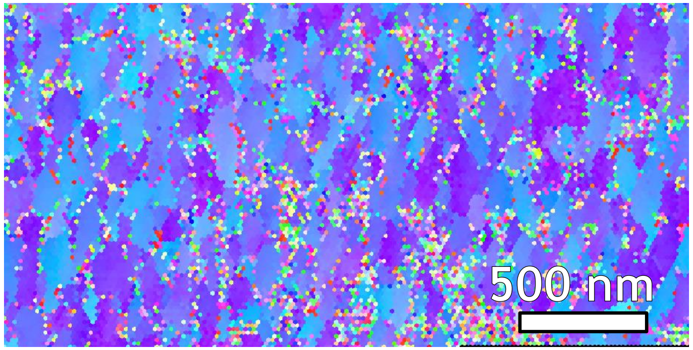
Collaborator: D.L. Buller



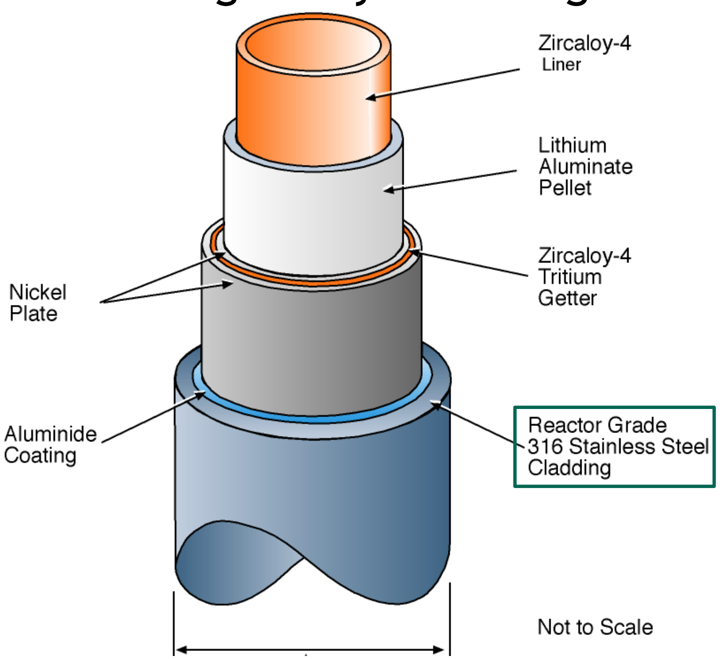
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

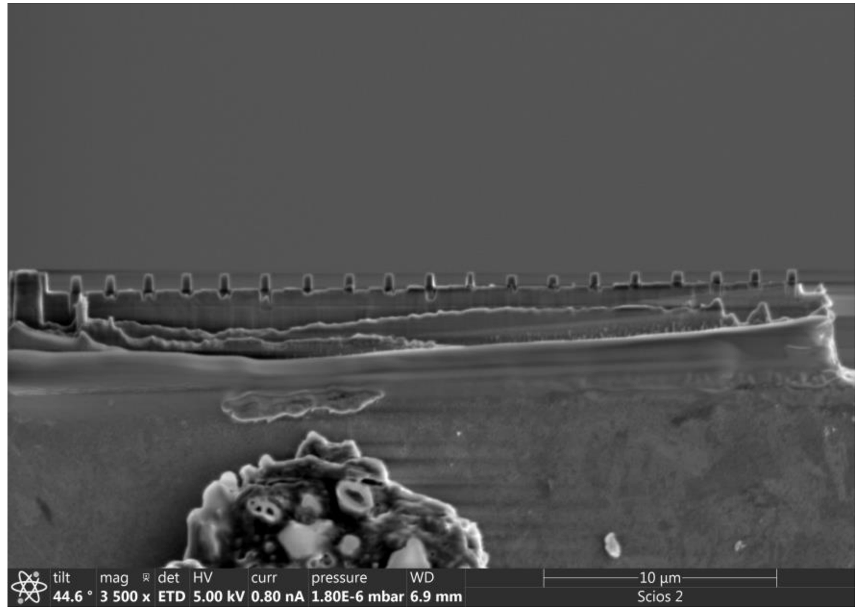


316 stainless steel cladding investigated for its nanomechanical properties



316 bulk piece is a rolled rod. EBSD of as-rolled rod showing heavy texturing

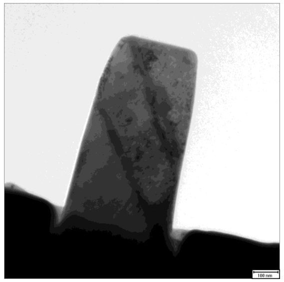




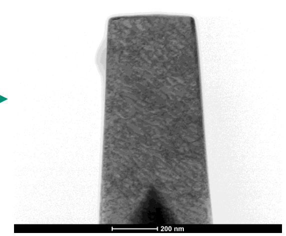
Due to the rolling process, ~100 nm grains form with low angle grain boundaries

Nanopillars have a range of microstructures:

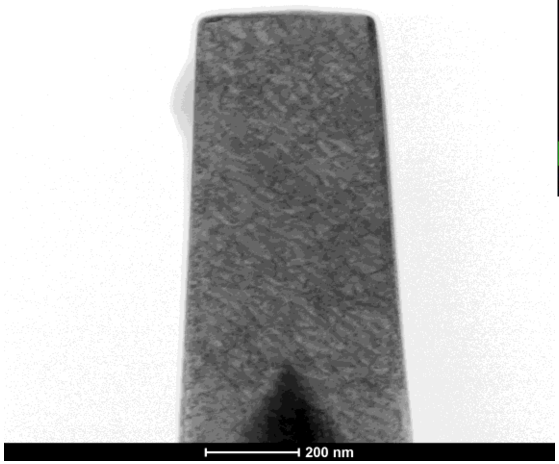
Contain an interface

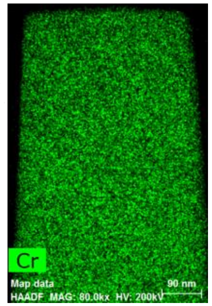


Single crystal

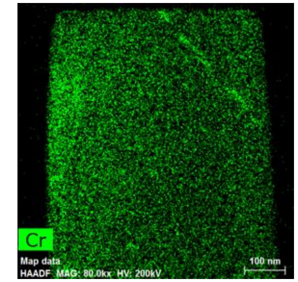


Some pillars are single crystal, contain an interface, and/or contain precipitates

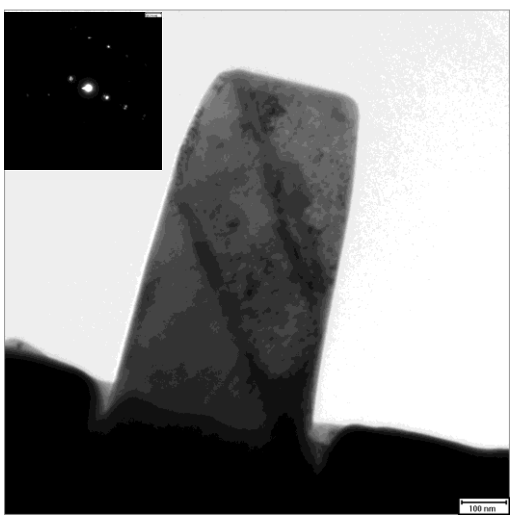


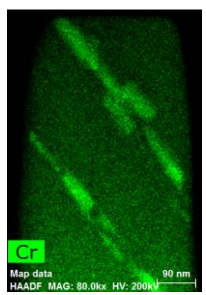


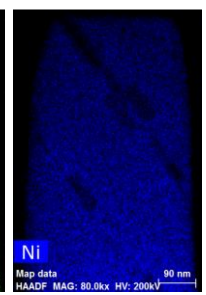
Single crystal, no precipitates

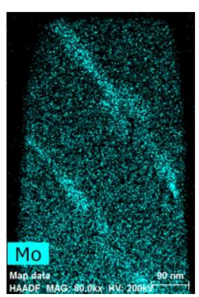


FIB-induced interfaces, precipitates

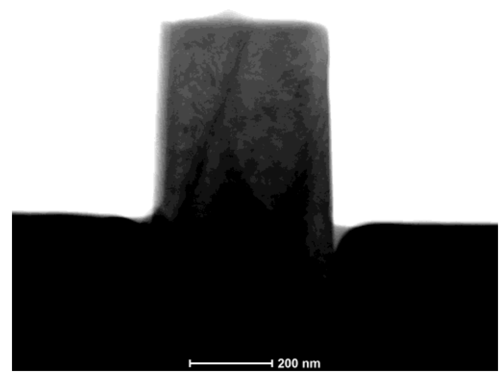


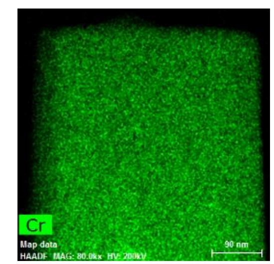






Interfaces, with precipitates

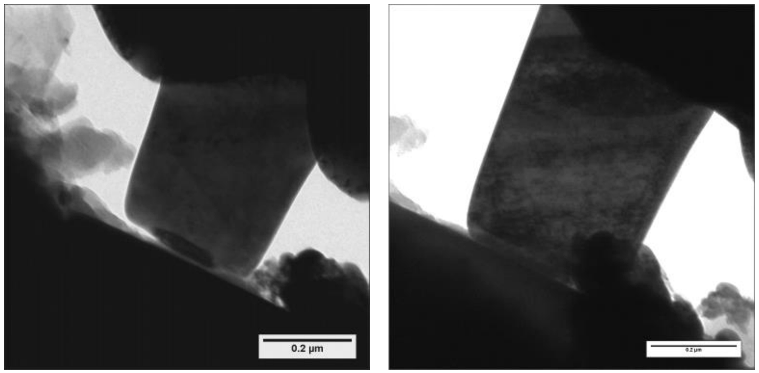




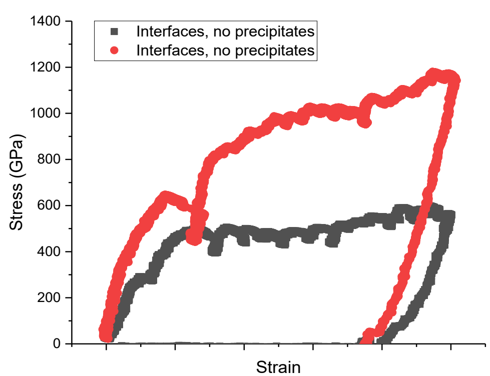
Interface, no precipitates

As-fabricated pillars have properties that depend on the microstructure of the pillar

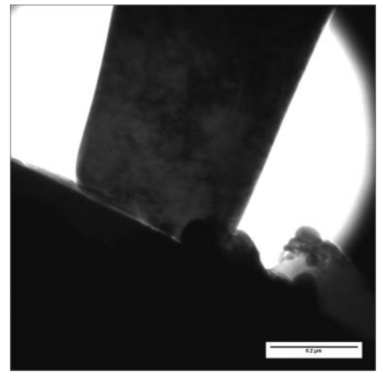
Interfaces, no precipitates



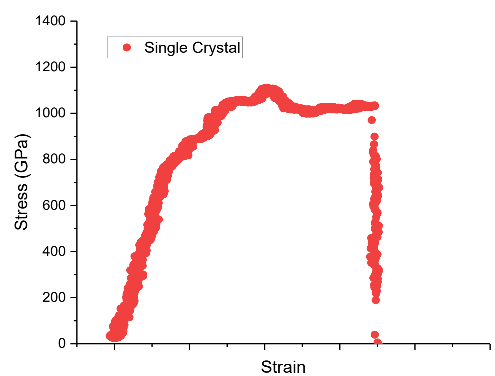
Average Flow Stress:
519 MPa



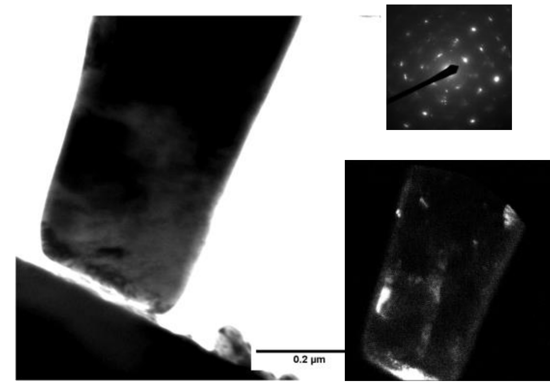
Single Crystal



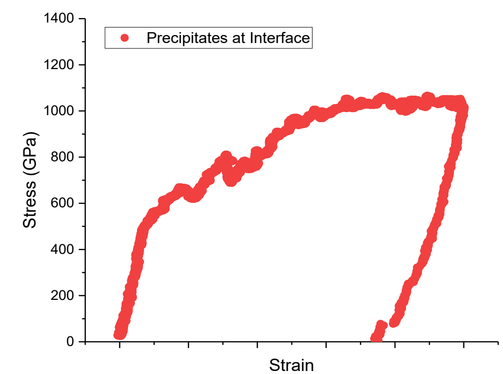
Average Flow Stress:
862 MPa



Interface & precipitates



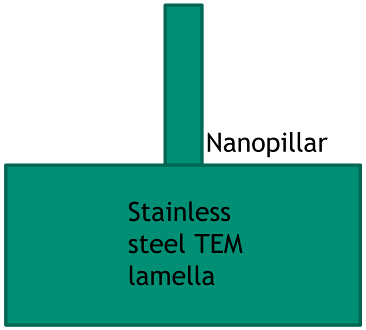
Average Flow Stress:
618 MPa



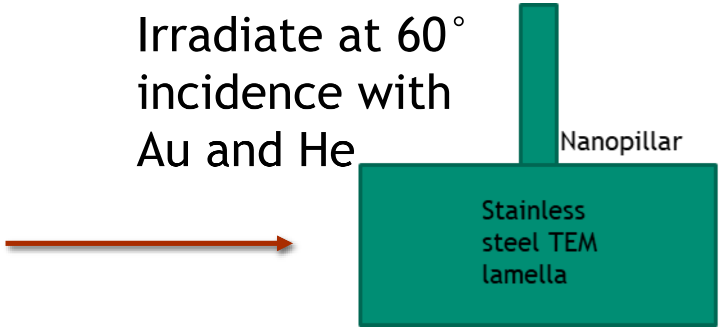
Thanks to R.M. Schoell for assistance

Irradiated pillars – irradiated post fabrication with Au and He ions

Fabricate nanopillars in FIB

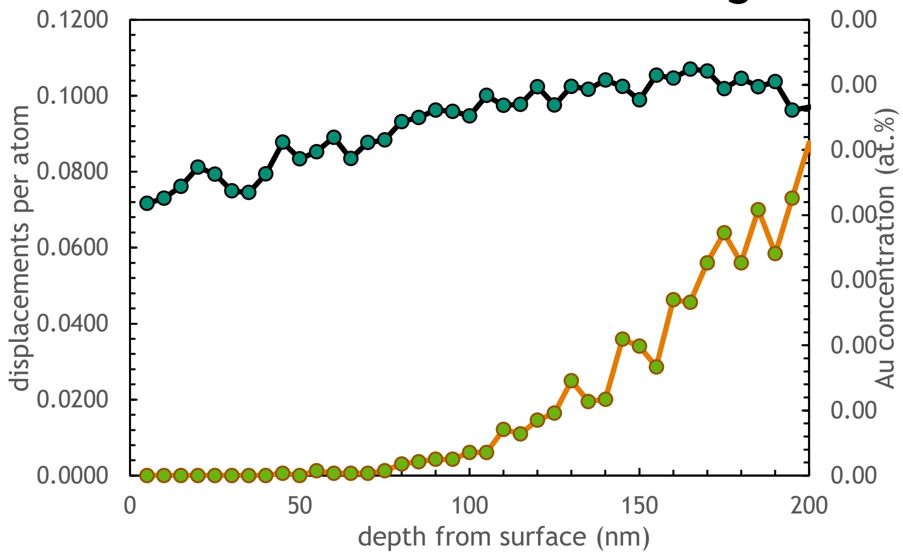


Irradiate at 60° incidence with Au and He



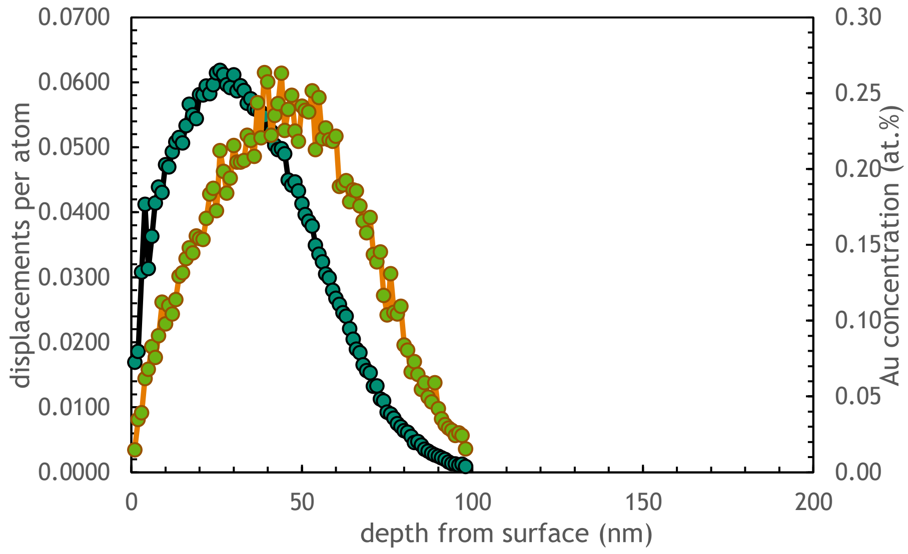
2.8 MeV Au ion damage profile – maximum damage within pillar, limited Au implantation – 5 dpa

Emulate neutron knock-on damage

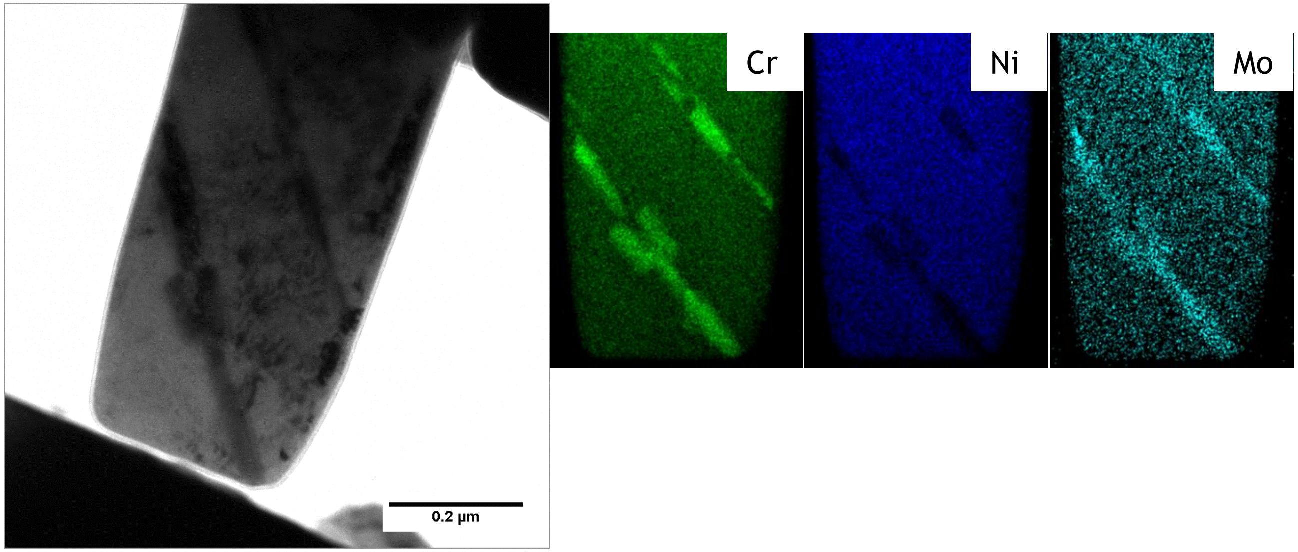


He ion implantation profile – peak He concentration within pillar

Emulate He production – 0.25 He %

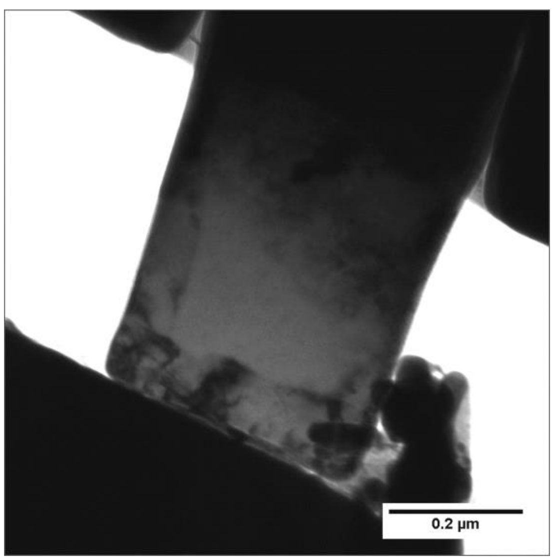


Irradiated pillars show similar behavior

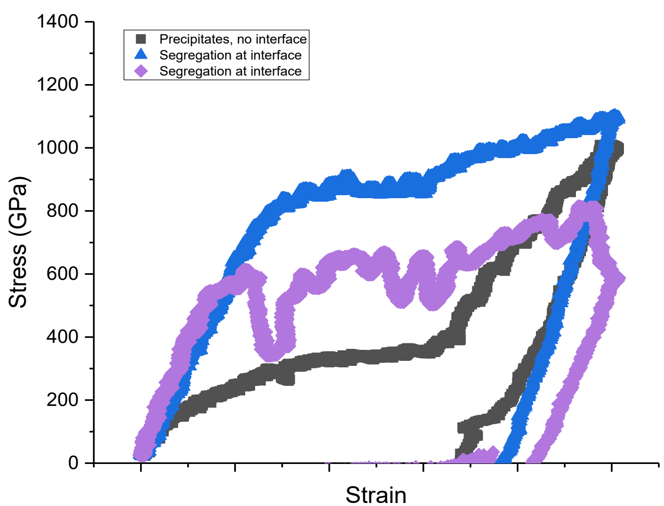


Interfaces and precipitates

Average Flow Stress: 710 MPa

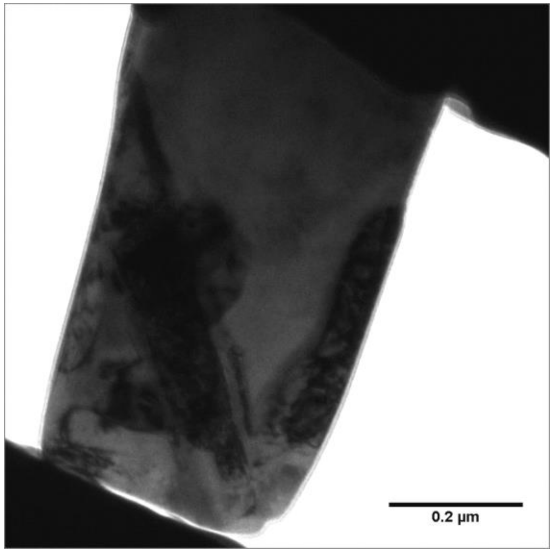


Precipitates, not at interface

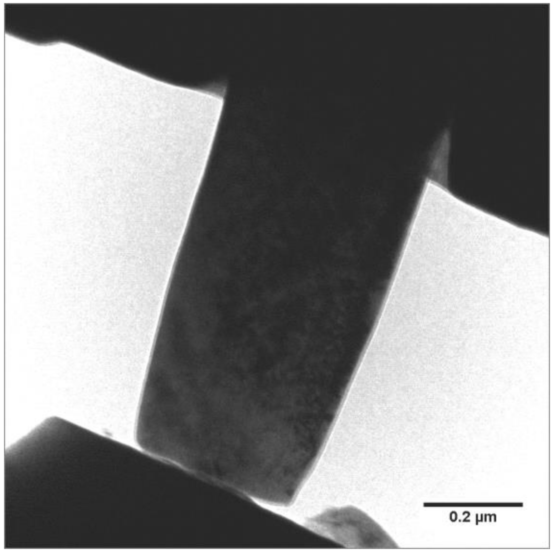


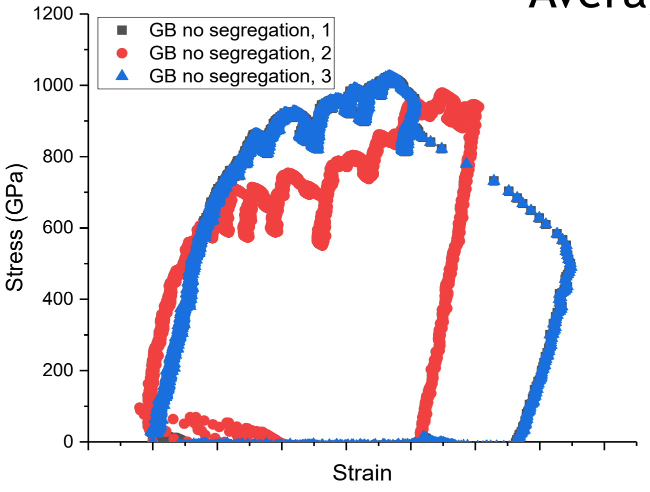
Single Crystal and pillars without segregation

Interfaces, no segregation

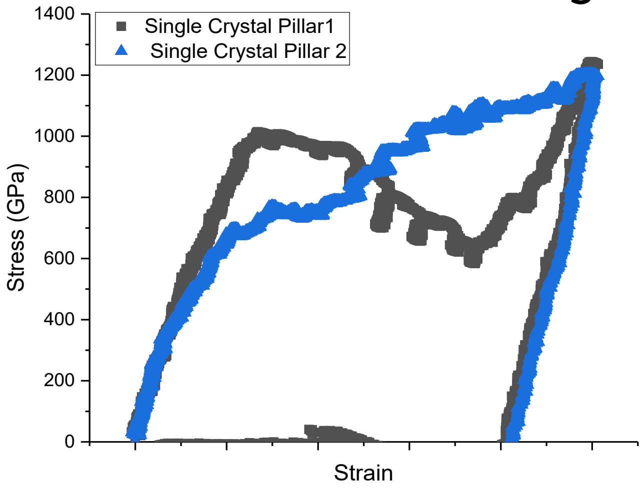


Single crystal





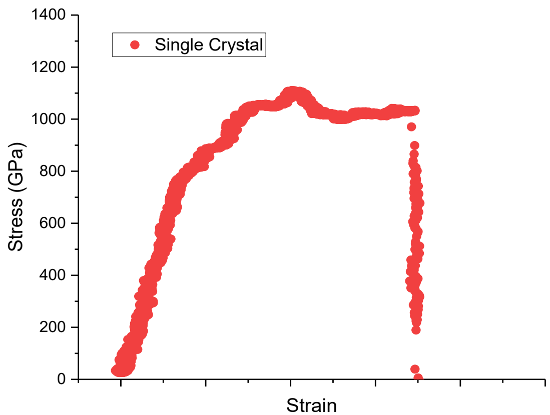
Average Flow Stress:
773 MPa

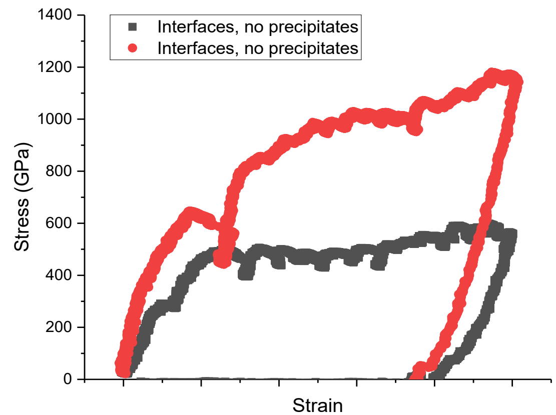


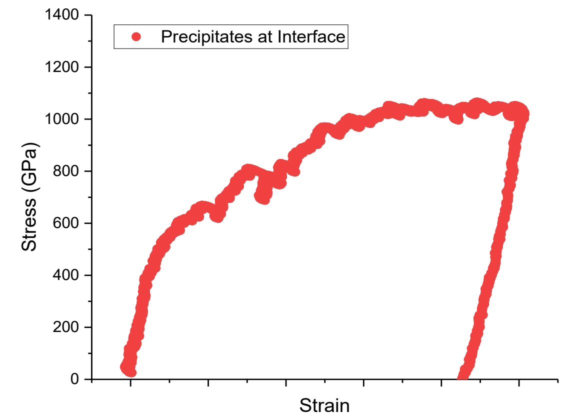
Average Flow Stress:
890 MPa

Irradiated pillars show hardening overall, but lack statistics

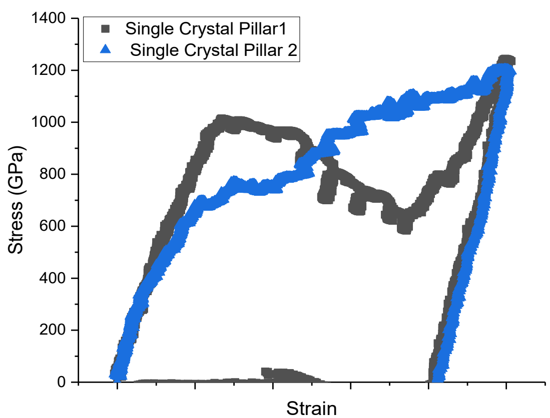
Unirradiated

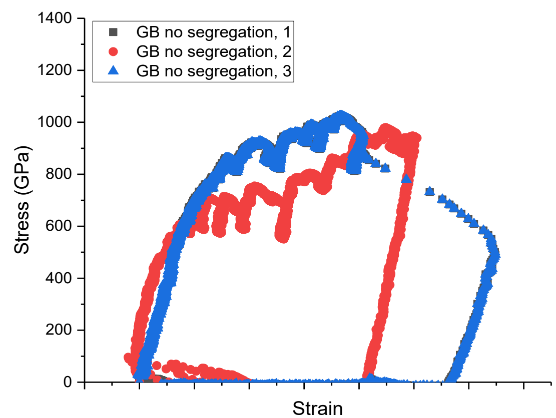


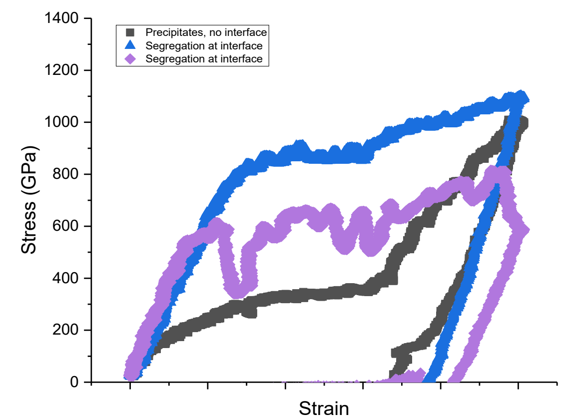




Irradiated





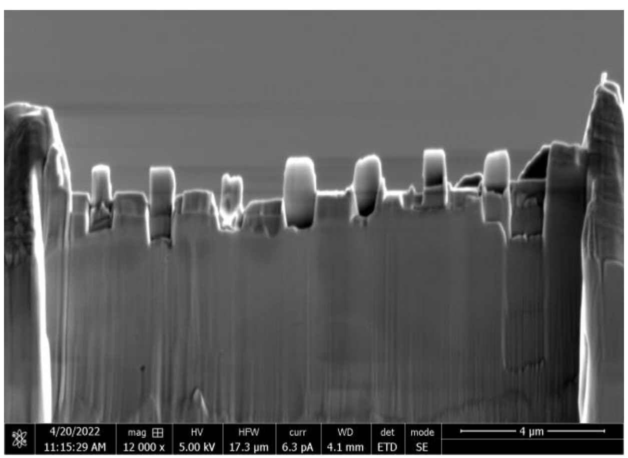


Change in flow stress: 28 MPa | 256 MPa | 92 MPa

In-situ TEM ion irradiation and mechanical testing offers a nanoscale probe into the effect of irradiation-induced defects

Goal: comparison to neutron irradiated pillars to examine if He+Au irradiation can accurately emulate the effect on the nanomechanical properties

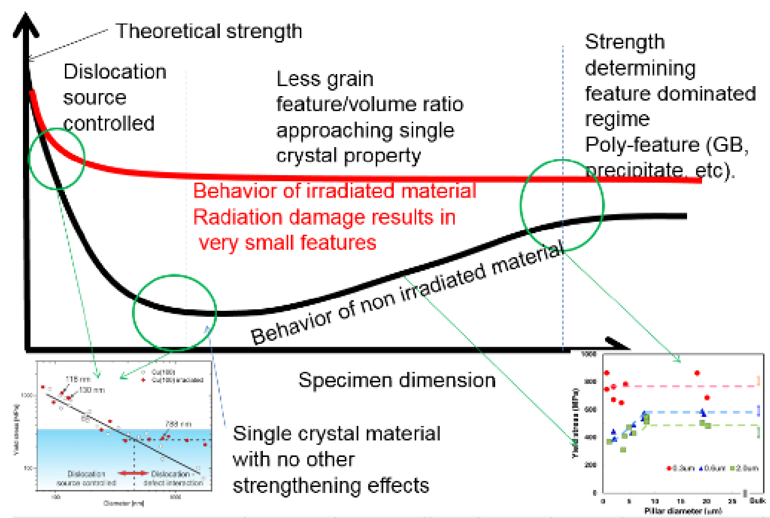
Neutron irradiated 316 pillars were fabricated, but did not survive shipping



Set of pillars from neutron irradiated cladding C13-2-3-CLAD26

We observe hardening of the pillars following irradiation but the original microstructure shows more impact on the properties than irradiation

Also, irradiation-induced defects are on the same scale as the pillar itself, size effects matter



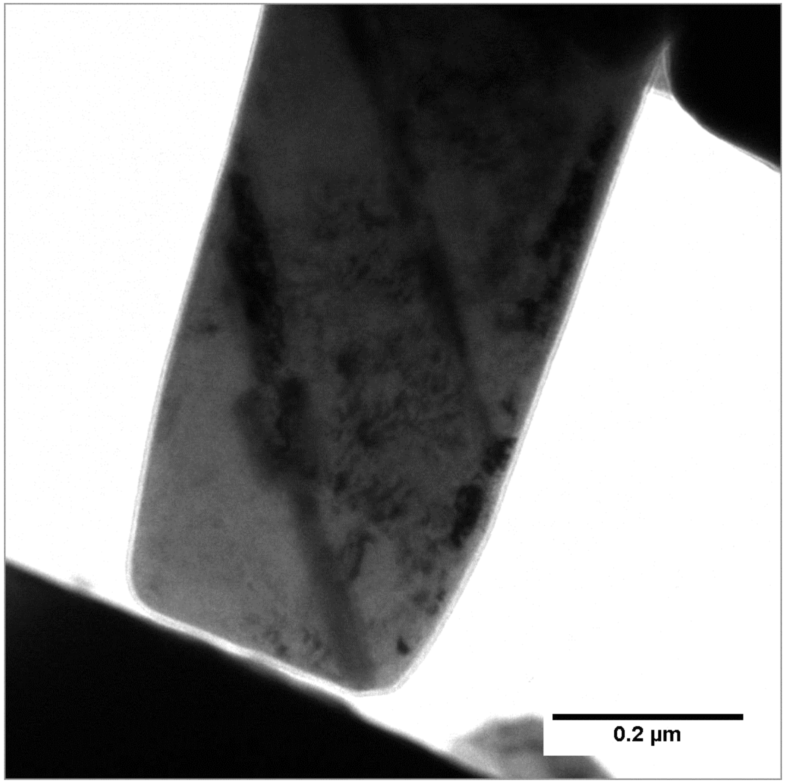
P. Hosemann, Developing Ultra-small Scale Mechanical Testing Methods and Microstructural Investigation Procedures for Irradiated Materials

Successful examinations of high dose heavy ion bombardment coupled with in-situ testing to probe neutron-ion surrogacy

High dose Au bombardment coupled with APT analysis to investigate microstructure

Can we emulate the neutron irradiation-induced change in mechanical properties with ion irradiation?

In-situ TEM nanopillar compression of 316 SS with and without dual beam He+Au ion irradiation



Advanced the understanding of the nanomechanics of 316 stainless microstructures under irradiation a

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