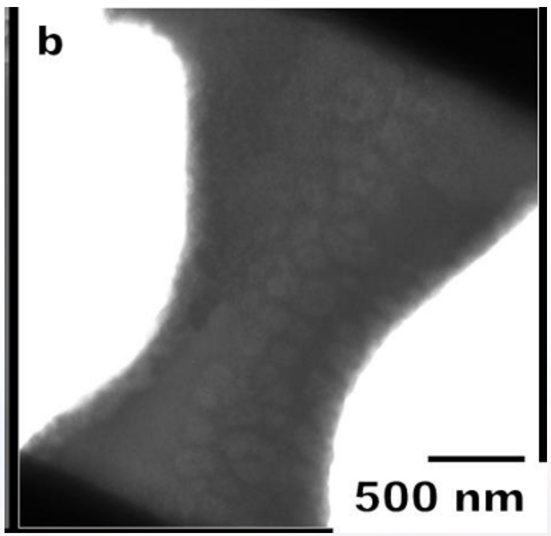
SAND2021-12613C

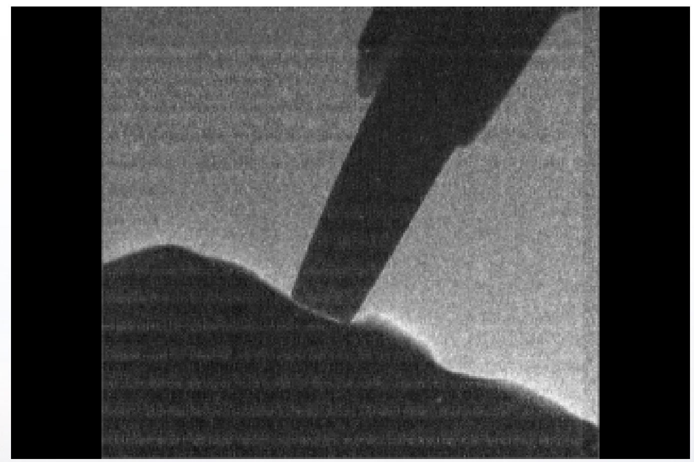
Effects of He on Nanoscale Mechanical Properties of Er

E. Lang, C. Taylor, R. Parrish, N. Madden, P. Price, R. Tandon & K. Hattar

Sandia National Laboratories

October 22, 2021





Utilizing *in situ* TEM mechanical testing to elucidate nanoscale mechanisms dictating mechanical properties of Er

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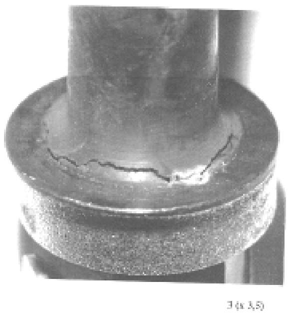
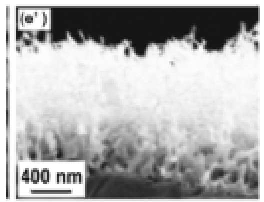
Macroscopic and Microscopic Helium Effects

Reactor Steel Embrittlement

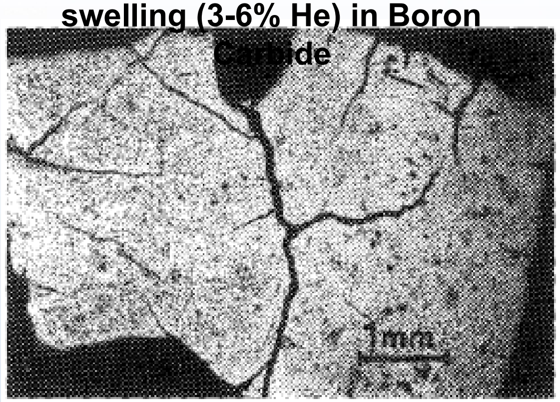


W Fuzz

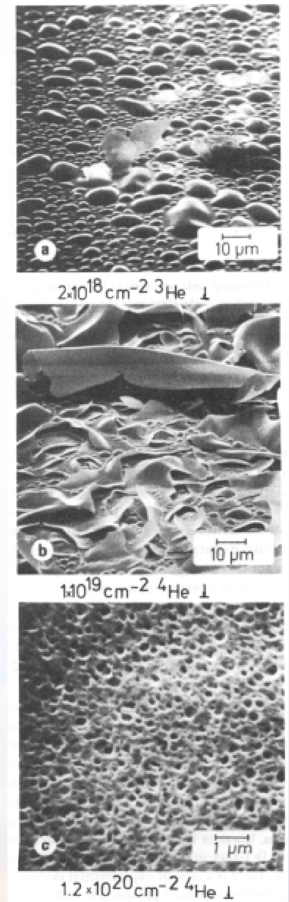


Kajita *et al* Nucl Fusion 49 (2009)

Fast Neutron Irradiation-induced swelling (3-6% He) in Boron Carbide

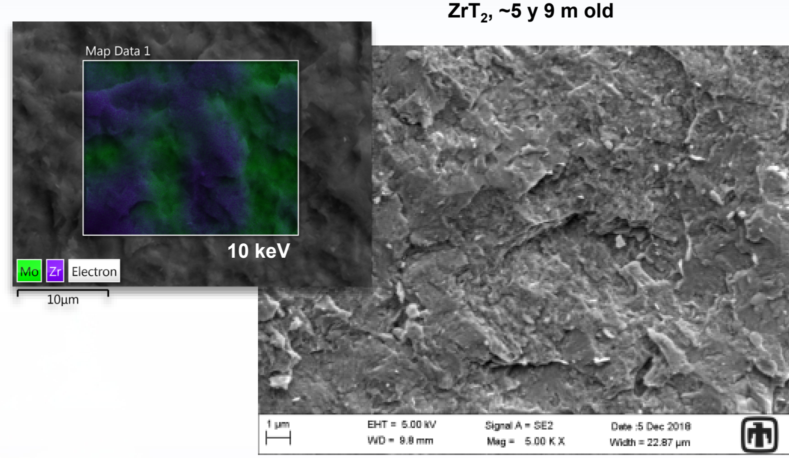


Stoto *et al* J. Appl. Phys. 68 (1990)

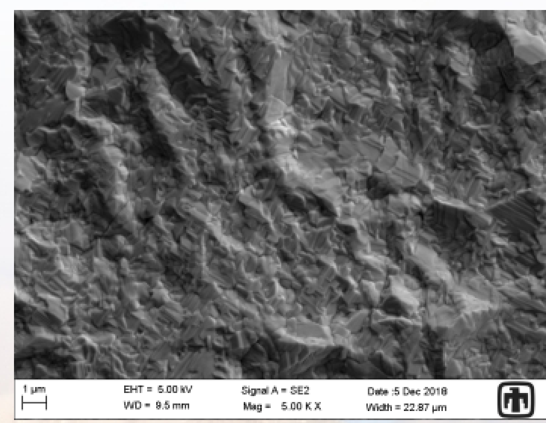


Ullmaier *Nucl Fusion* 24 (1984) 1039

ZrT_2, ~5 y 9 m old

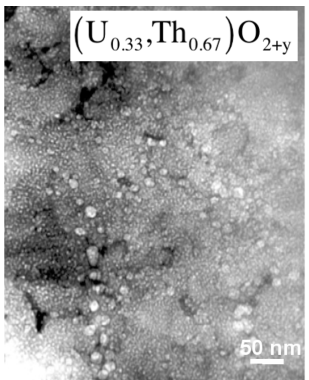


ZrT_2, ~1 y 10m old

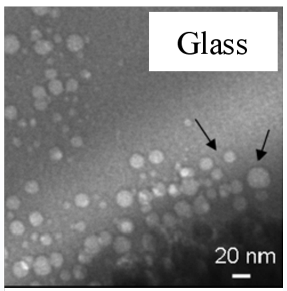


Nanoscale Helium Bubbles

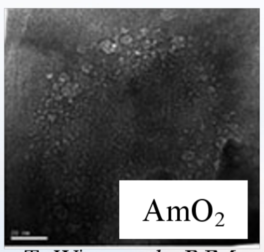
Ceramics



T. Wiss *et al.*, JNM (2014)

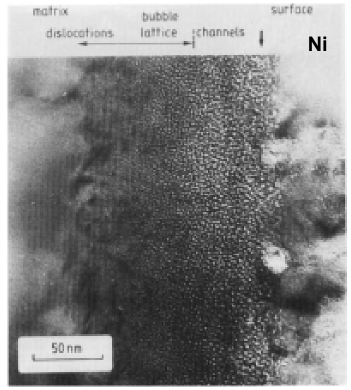


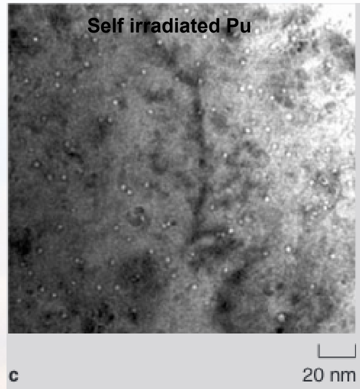
G. Gutierrez *et al.*, JNM (2014)



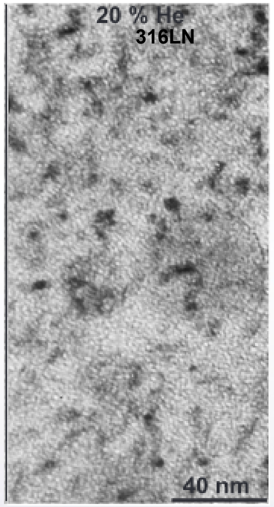
T. Wiss *et al.*, JNM (2015)

Metals



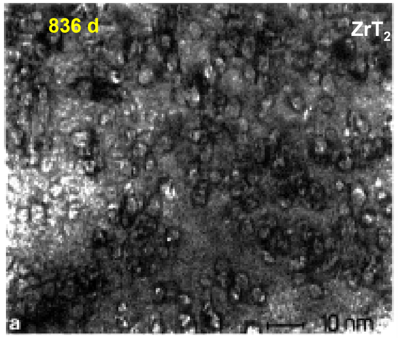


Zocco and Schwartz JOM (2003)

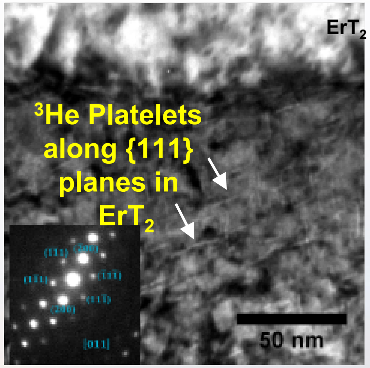


Hunn *et al* JNM 282 (2000) 131-136

Metal Hydrides



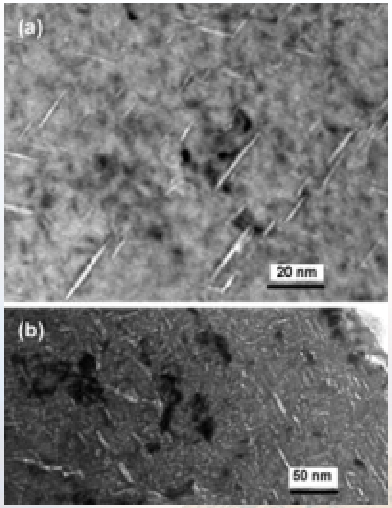
Schober, Trinkaus, Lasser, JNM 141-143 (1986) 453-457

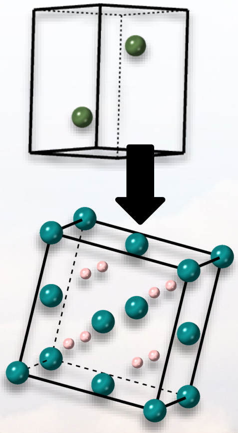


Emulating erbium hydride aging through ion irradiation

- Er undergoes a phase transformation from a hexagonal to a fcc structure under hydriding, forming ErH_2
- Tritium β-decays to ^3He, which models predict to remain in the tetrahedral site. Diffusion may occur through the octahedral site.
- ^3He in ErT_2 tends to form platelet structures along the {111} planes instead of bubbles.
- ErH_2 usually contains some oxide, Er_2O_3.

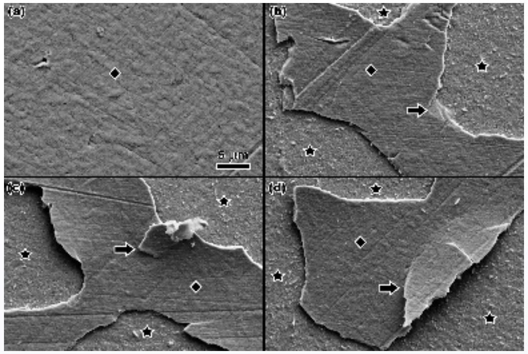
J.A. Knapp, et al. *J. Appl. Phys.*, 105 2009.



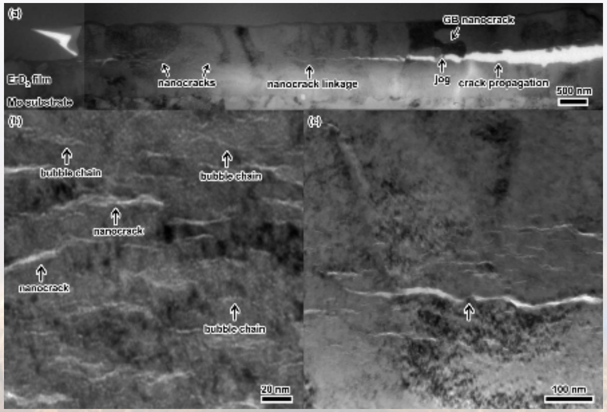


10% volume expansion

He implantation of ErD_2 causes surface flaking through bubble linkage and crack growth

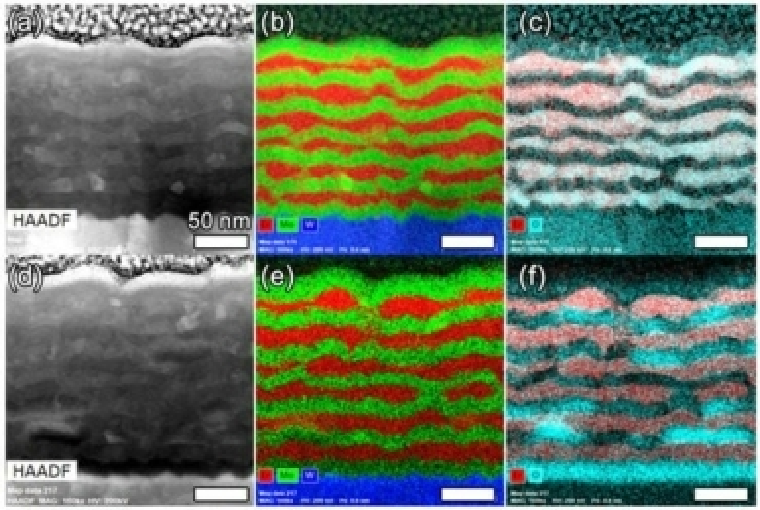


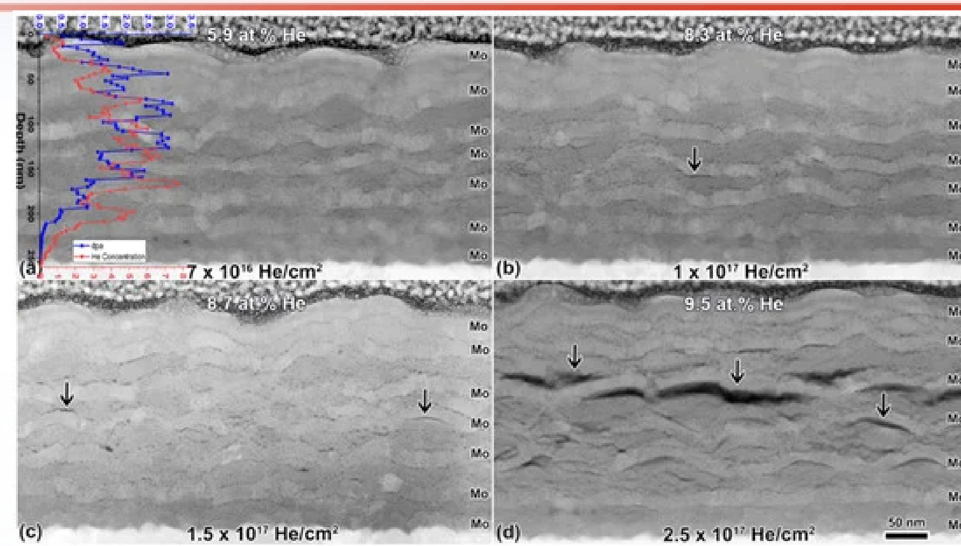
120 keV He, $5x10^{17}$ ions/cm^2



C.A. Taylor, et al. Materialia, Submitted 2021.

Multilayered Er composites to limit He bubble impacts

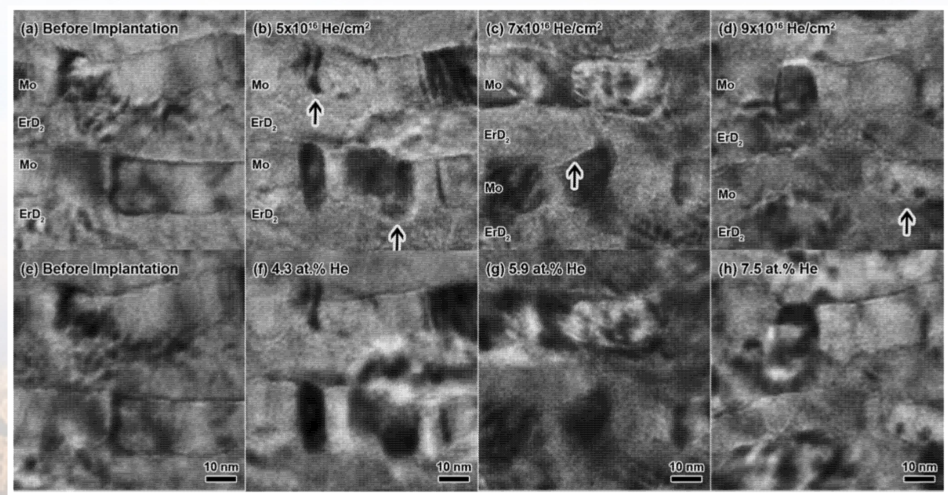




30 keV He *ex situ* implantation at Los Alamos National Lab

- Er/Mo multilayered samples fabricated via e-beam deposition
- Deuterated without formation of intermetallic phases
- He implantation to investigate He bubble nucleation

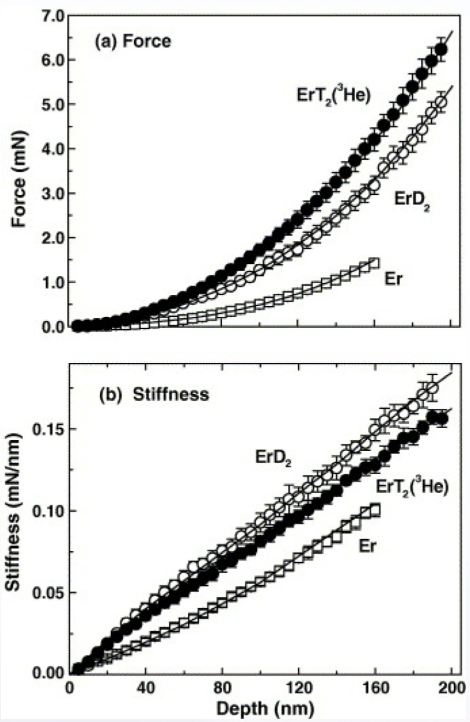
Er/Mo multilayered composites show He bubble accumulation at interfaces



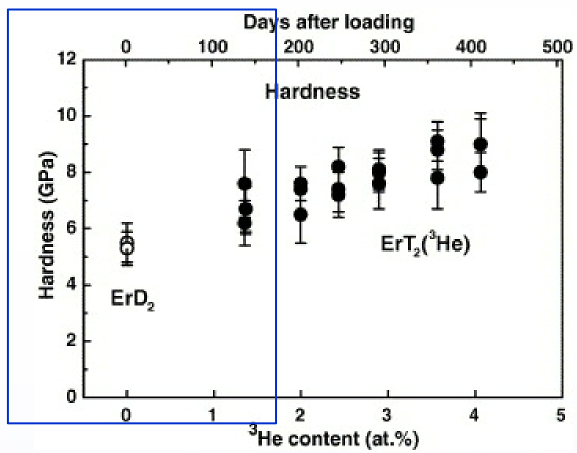
10 keV He *in situ* implantation at SNL

C.A. Taylor, et al. *Materials* 14 2021.

D/T accumulation changes Er mechanical properties

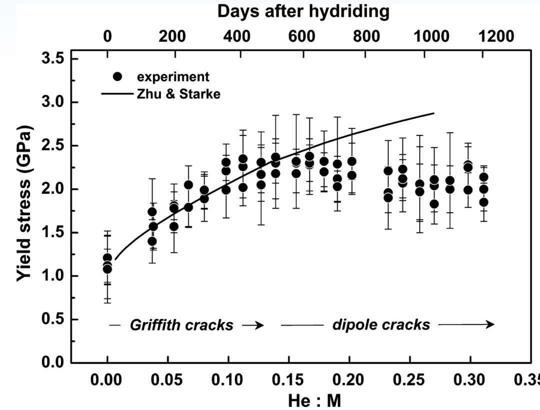


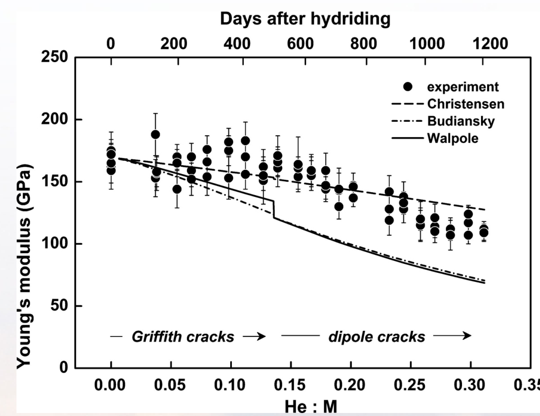
Nanoindentation probes near-surface mechanical properties of thin films to decipher impacts of He on Er properties



ErD_2 shows similar hardness as un-aged ErT_2, yet properties of ErT_2 changes with time

How are the mechanical properties altered via accelerated aging?



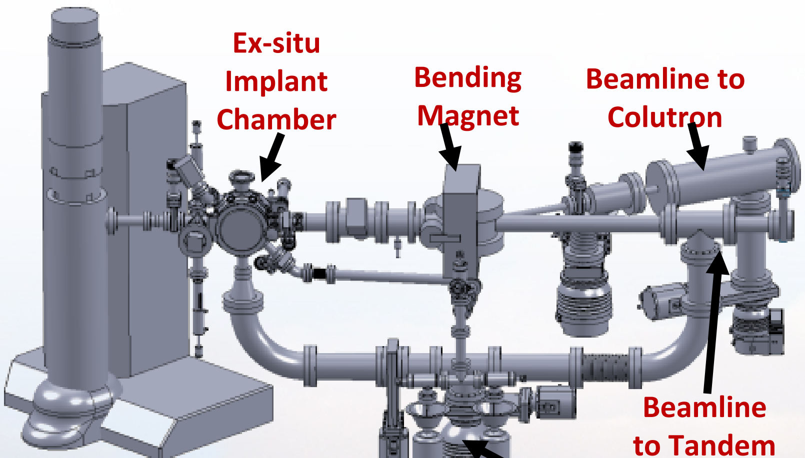


Yield stress and modulus of aged ErT_2

J.A. Knapp, et al. *JNM*, 350 2006.
J.A. Knapp, et al. *J. Appl. Phys.*, 105 2009.

Rapid Evaluation of Helium in Materials using Sandia's I[3]TEM

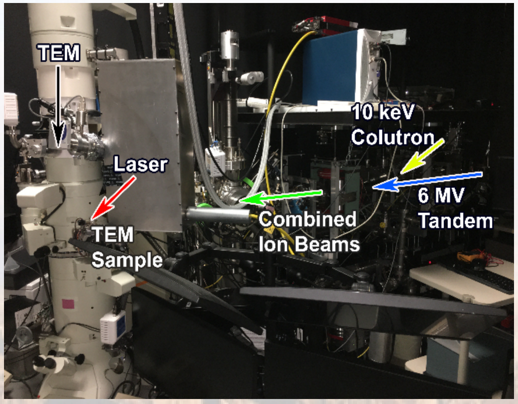
- **In-situ implantation** only takes a few hours – tritium aging takes several months and rad work.
- **In-situ annealing** with the Gatan DT stage is used to quickly assess the stability of bubbles and multilayers.
- **Hysitron PI-95 PicoIndenter** In-situ TEM nanomechanical testing



Quantitative Mechanical Testing

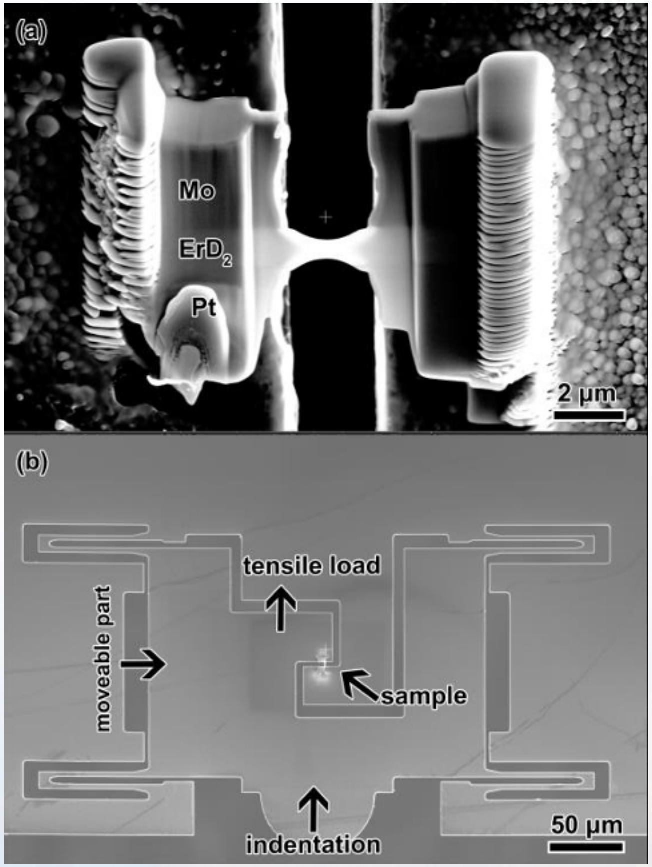
Minimal control over displacement and no "out-of-box" force information

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



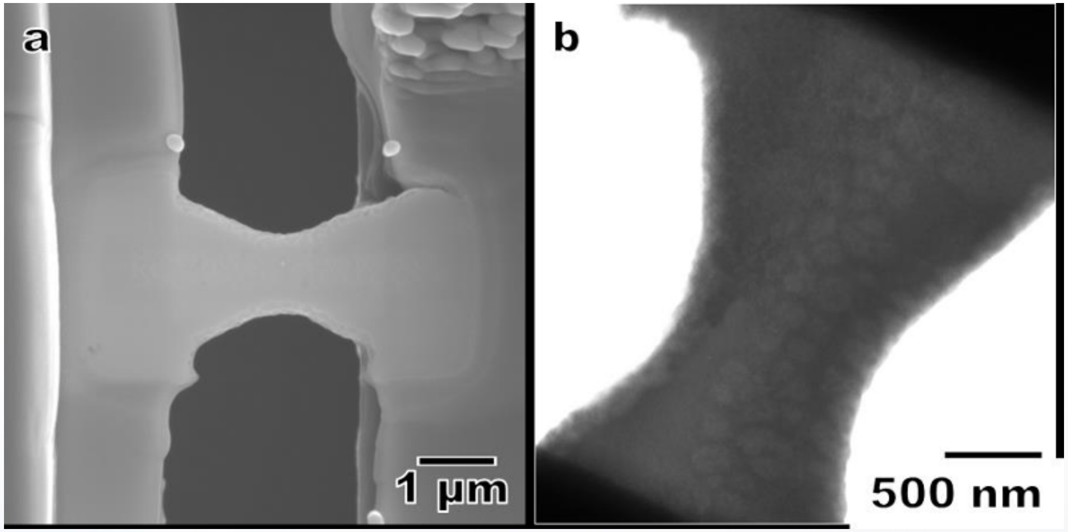
1) Indentation
2) Tension
3) Fatigue
4) Creep
5) Compression
6) Bend

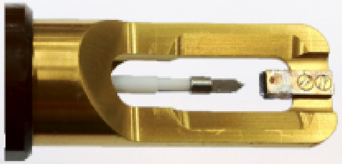
Utilizing in-situ TEM push-to-pull device for tensile testing



Nano-tensile bars for *in situ* TEM tensile testing successfully fabricated via FIB liftout

FIB liftout micro-machining to fabricate tensile bars for TEM testing

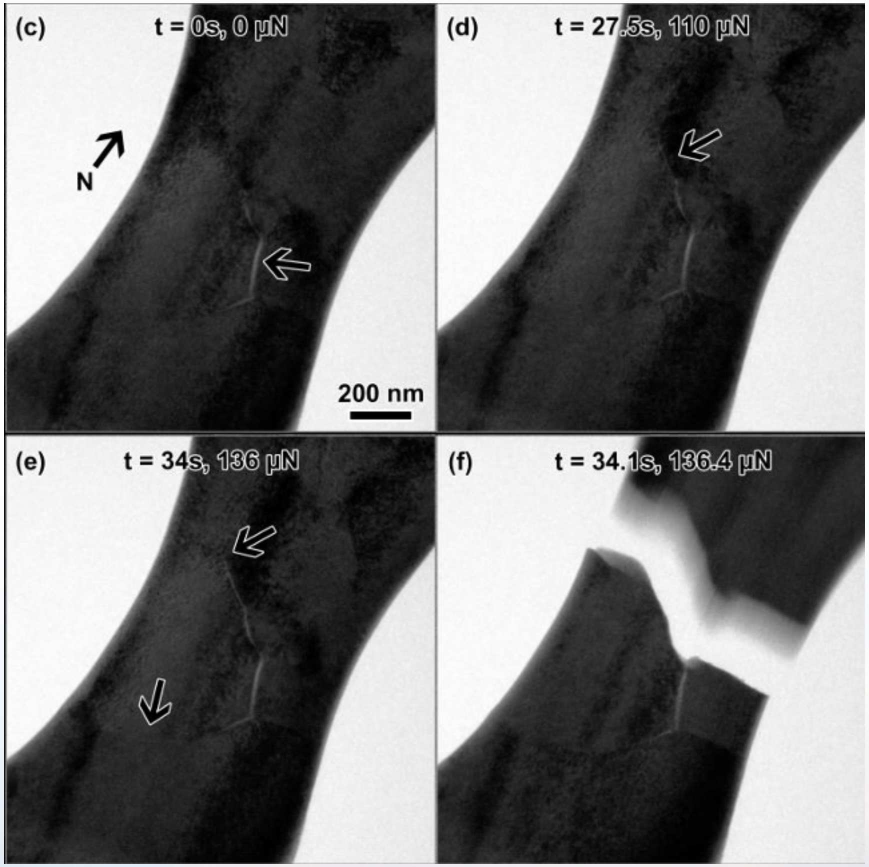




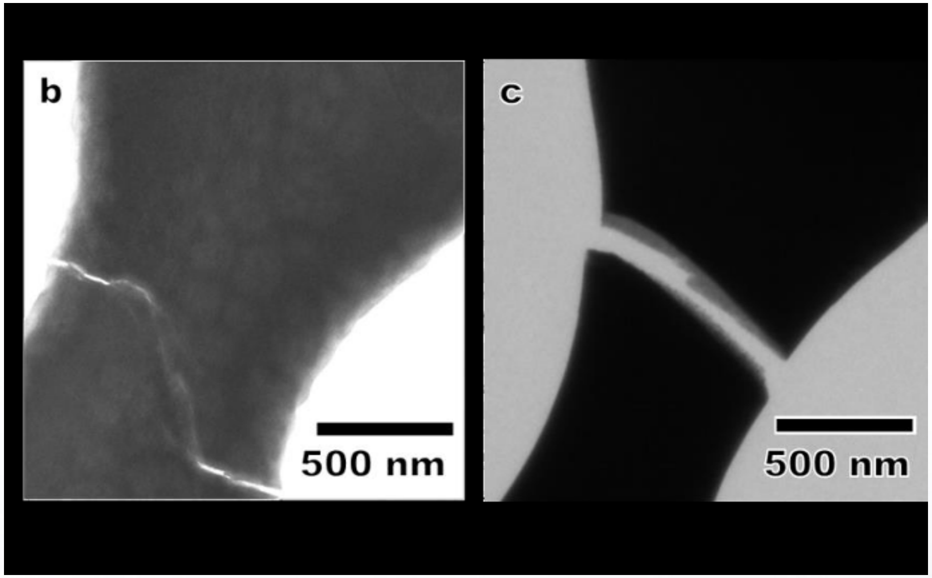
Displacement controlled test, 20 nm/s

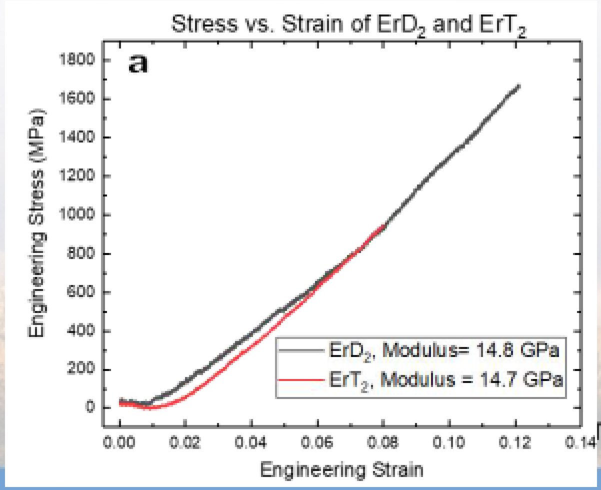
Push-to-pull testing using Hysitron PI-95

In-situ TEM tension tests resulted in brittle failure



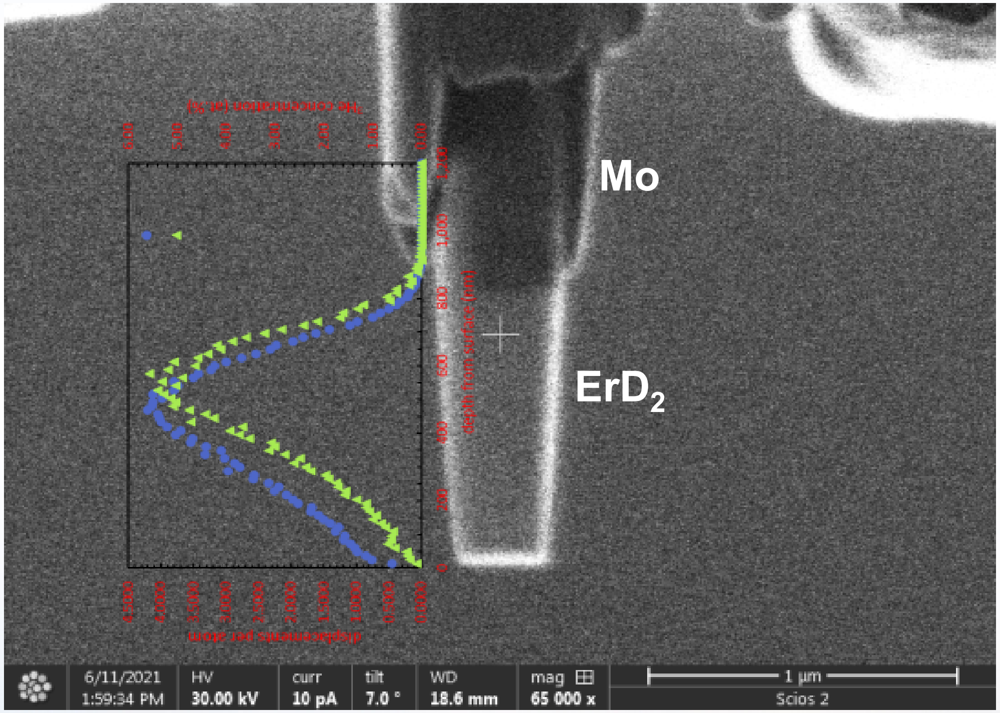
No necking observed, brittle failure





Similar moduli for ErD_2 and ErT_2, though lower stress needed for failure of ErT_2

Nanopillar compression likely more elucidating for brittle material



FIB-milled nanopillars of Er on Mo substrate

He implantation profile shows He peak in center of pillar

Peak He concentration: ~5 at.%

Utilizing *in situ* TEM nanopillar compression of He-implanted ErD_2 thin film

In-situ TEM nanopillar compression tests

Hysitron PI-95 indenter

2 um flat tip

Displacement controlled test

Load: 0-100 nm at 1nm/s

Hold: 10s

Unload: 100-0 nm at 2nm/s

PI-95 Tip

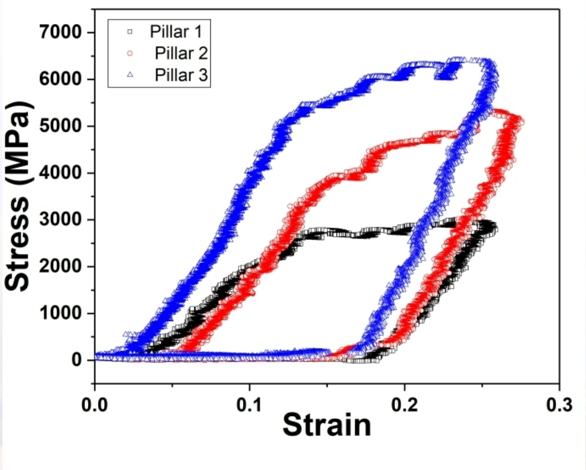
Nanopillar compression of He-implanted ErD2

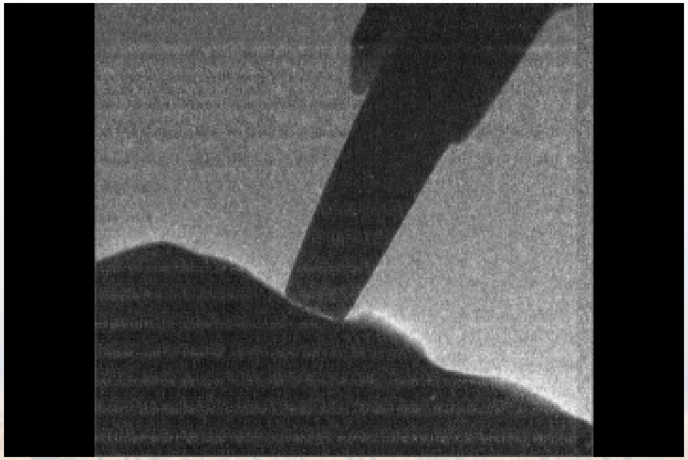
Aged ErT_2:
He concentration of 5 at.%

From nanoindentation:
Yield Strength - ~1.5 GPa
Young's Modulus - ~165 GPa

Pillar	Yield Strength [GPa]	Modulus [GPa]
1	2.68	4.2×10^1
2	4.03	5.5×10^1
3	5.44	7.4×10^1
4	4.98	9.3×10^1

J.A. Knapp, et al. *J. Appl. Phys.*, 105 2009.

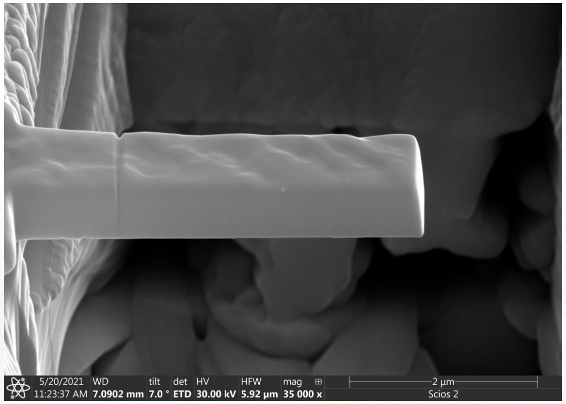




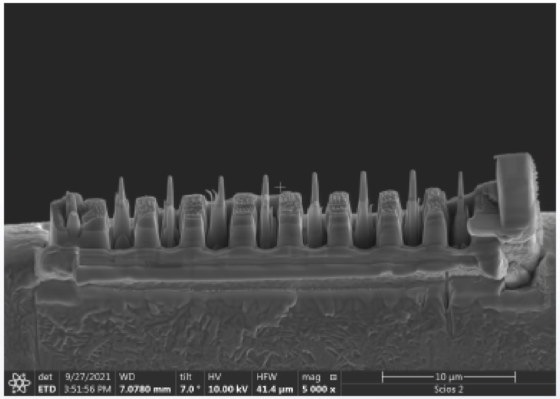
Nanoindentation can be used to test effects of He implantation on acerated-aged ErD_2, scalable to ErT_2

Further testing to decipher impacts of D and He loading

- Nanopillar compression testing on pure Er and ErD_2
- Micro-cantilever testing to scale up testing to microscale
- Nano-tensile tests on more ductile He-implanted Pd
- Verify testing routine before testing ErT_2 to compare to accelerated aged specimens



Micro-cantilevers of He-implanted ErD_2 for in-situ SEM testing



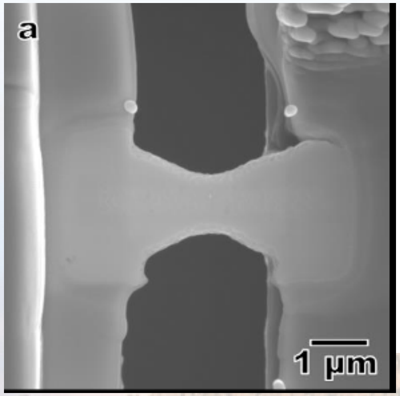
Nanopillars on pure Er metal

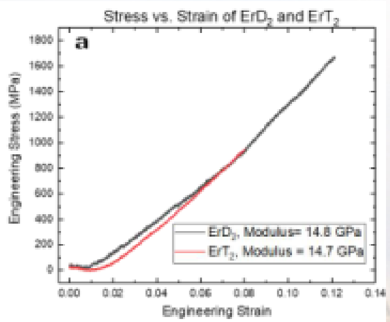
Utilizing *in situ* TEM mechanical testing to qualify the accelerated aging techniques in Er

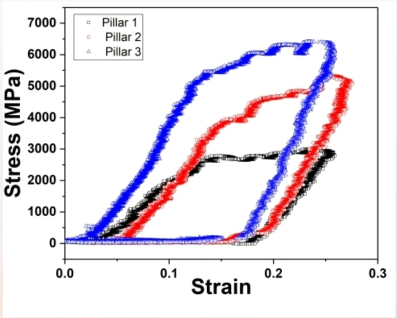
In-situ TEM techniques to elucidate helium effects on metals and metal hydrides

- Tritiated metals have mechanical properties that change as they age
- Can we simulate their aging through ion implantation?
- He implantation of deuterated metals used to simulate aging of tritiated metals
- In-situ TEM tensile and compression tests important for determining mechanical properties of aged and implanted hydrides

Preliminary results show accelerated aging may be useful to qualifying mechanical properties of aged films







Acknowledgements

- Film Deposition: Ron Goeke (SNL)
- STEM/EDS: Paul Kotula (SNL)
- Bulk He Implantation: Yong Wang (LANL)
- In-situ Tensile Test: Riley Parrish (SNL), Patrick Price (SNL), Caitlin Taylor (LANL), Khalid Hattar (SNL)
- In-situ Compression Test: Nathan Madden (SNL), Khalid Hattar (SNL)

Access to the I^{3}TEM and associated facilities is now available through both the Nuclear Science User Facilities (NSUF) and the Center for Integrated Nanotechnologies (CINT).

https://nsuf.inl.gov

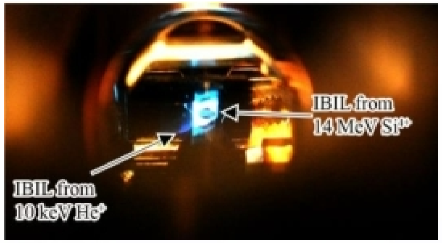
http://cint.lanl.gov

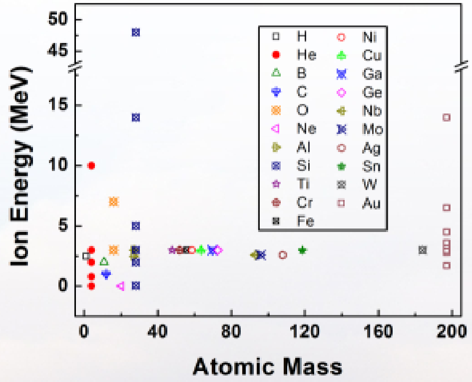
This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

Sandia's Concurrent *In situ* Ion Irradiation TEM (I3TEM) Facility

10 kV Colutron - 200 kV TEM - 6 MV Tandem

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





Heavy Ion Irradiation + Gaseous Implantation

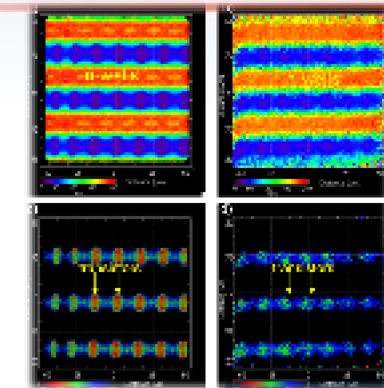
Control ratio of dpa and gas species implantation and characterize coupling effects

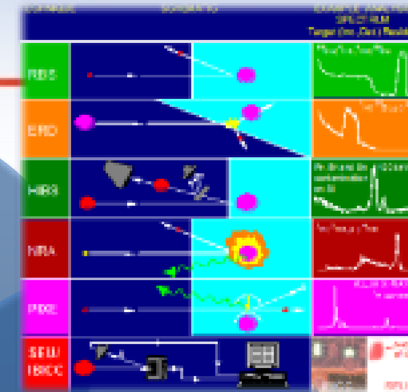
Capabilities

- 200 kV LaB_6 TEM
- Ion beams considered:
  - Range of Sputtered Ions
  - 10 keV D^{2+}
  - 10 keV He^{+}
- All beams hit same location
- Nanosecond time resolution (DTEM)
- Procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL
- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- *In situ* heating
- Tomography stage (2x)
- *In situ* cooling stage
- *In situ* electrical bias stage
- *In situ* straining stage (3x)

Sandia's USER Capabilities

Core Facility - SNL





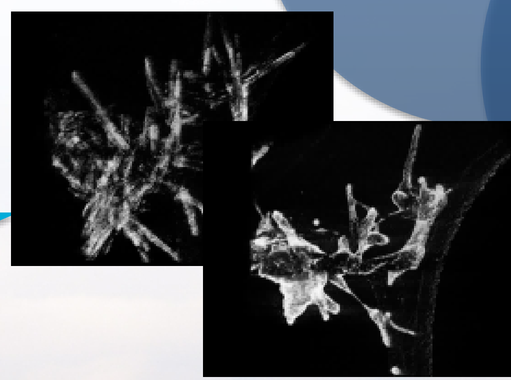
- **Nanophotonics & Optical Nanomaterials**
- **Soft- Biological & Composite Nanomaterials**
- **Quantum Materials**
- **In-situ Characterization and Nanomechanics**

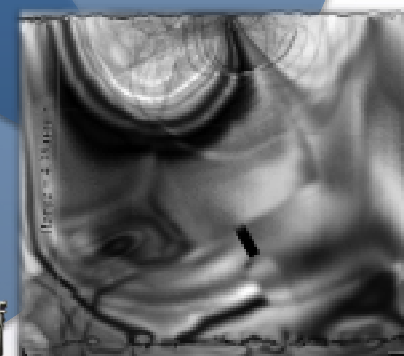
Ion Beam Analysis (IBA)

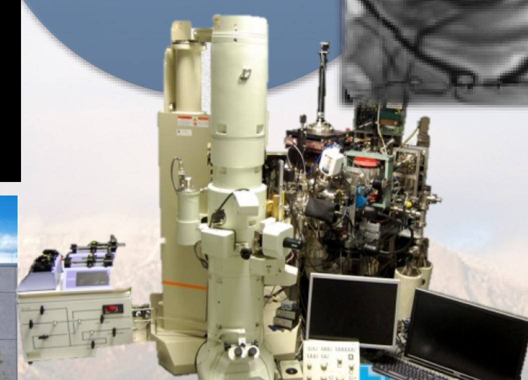
Radiation Effects Microscopy (REM)

Ion Beam Modification (IBM)

In situ Ion Irradiation Transmission Electron Microscopy (I^3TEM)







Gateway Facility - LANL

He-implanted ErD_2 Pillars

Pillar 1

Pillar 2

Pillar 3

Pillar 4