New research capabilities: He-3 Ion Beam Analysis System and Indentation of Tritiated Samples

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October 13, 2004
Albuquerque, NM

Thanks to: J. E. Mikkalson, S. Van Deusen, E. Staab, and T. D. Kraus

Sandia National Laboratories

Two new capabilities for ^{3}He studies

- **Multi-element ion beam analysis system**
  - -- Simultaneous, non-destructive profiling of H, D, T, ^{3}He, O, and surface C.
  - -- Unique system based on heavy ion Elastic Recoil Detection in building 884.
  - -- Status: assembly underway

- **Nanoindentation of tritiated films**
  - -- Mechanical property measurements for tritiated thin films.
  - -- Containment procedures allow use of indenter in 884.
  - -- Status: operational

New IBA system to study ^{3}He release

- **Problem: profile light elements in Erbium Tritide films**

  -- Need to simultaneously analyze H, D, T, ^{3}He, O, and surface C.

  -- Existing HDT analysis chamber cannot measure ^{3}He or measure O on a Si substrate.

  -- New system detector configuration determined using extensive ion beam simulations.

- **Applications:**

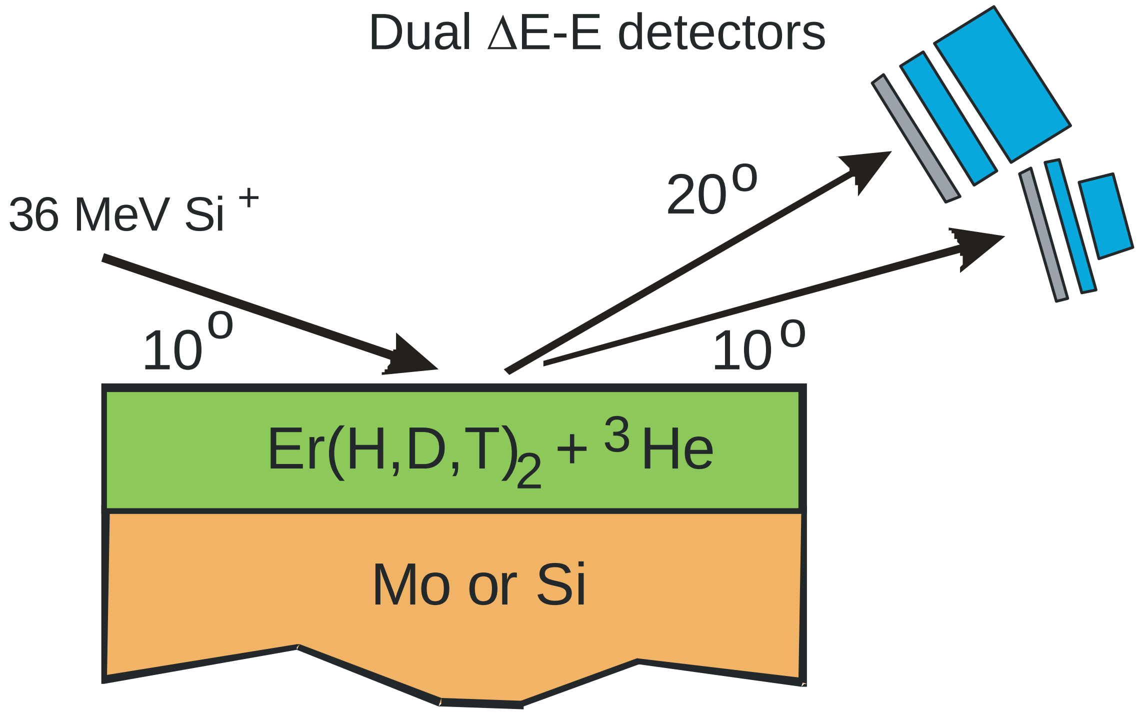
  -- Profile ^{3}He and T to study "denuded" zone and early ^{3}He release.

  -- Study profile changes as films age. (technique is non-destructive).

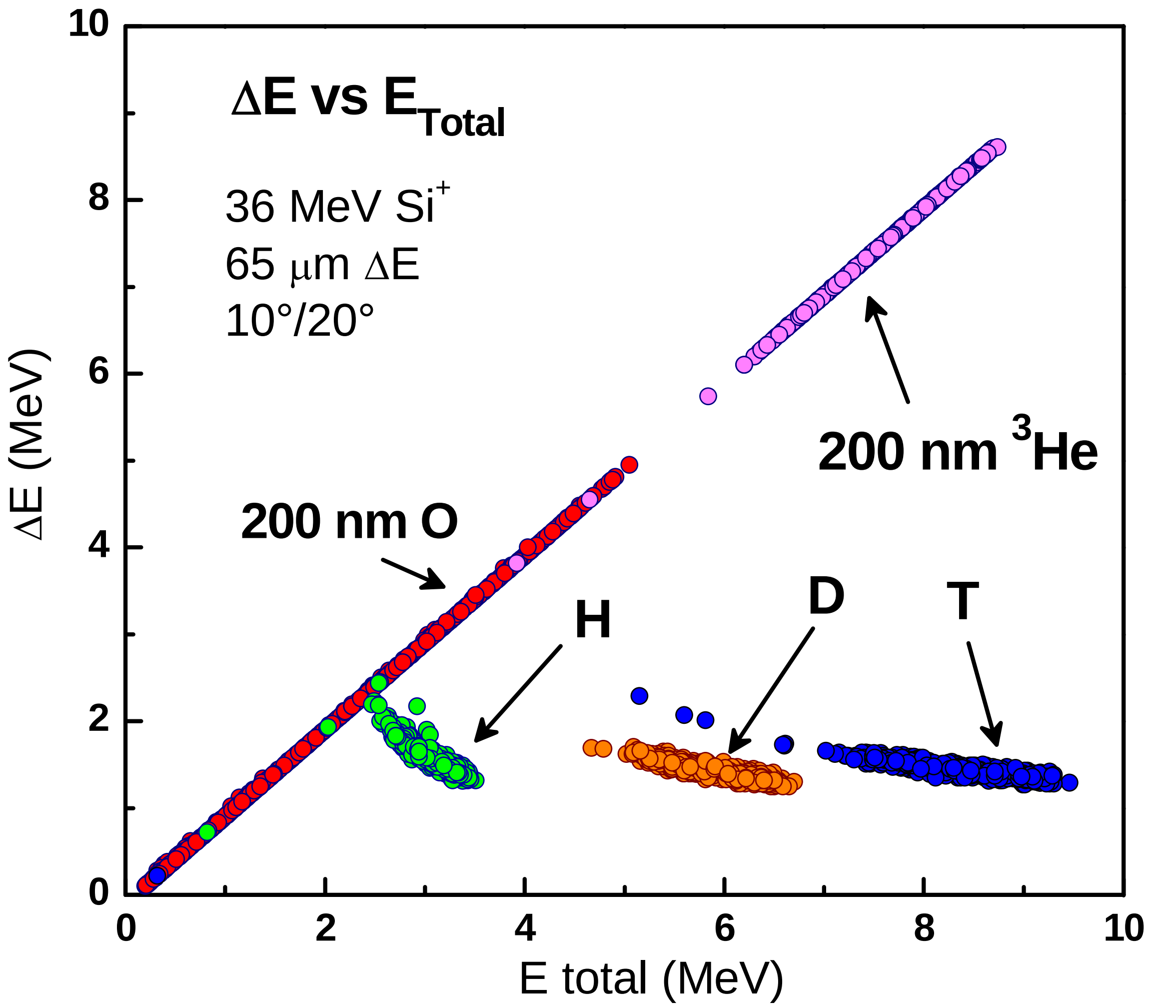
  -- Correlate film composition with fabrication parameters and mechanical properties.

Analysis method

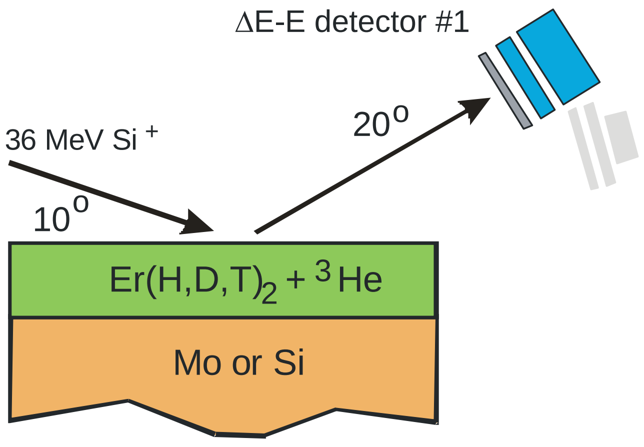
- **Heavy ion elastic recoil detection (ERD) with 36 MeV Si^+ analysis beam**
- **Thick ΔE-E detector at 20º to profile H, D, T, ^3He**
- **Thin ΔE-E detector at 10º to profile O, C**
- **Each detector pair has a thin foil to block the Si analysis beam.**



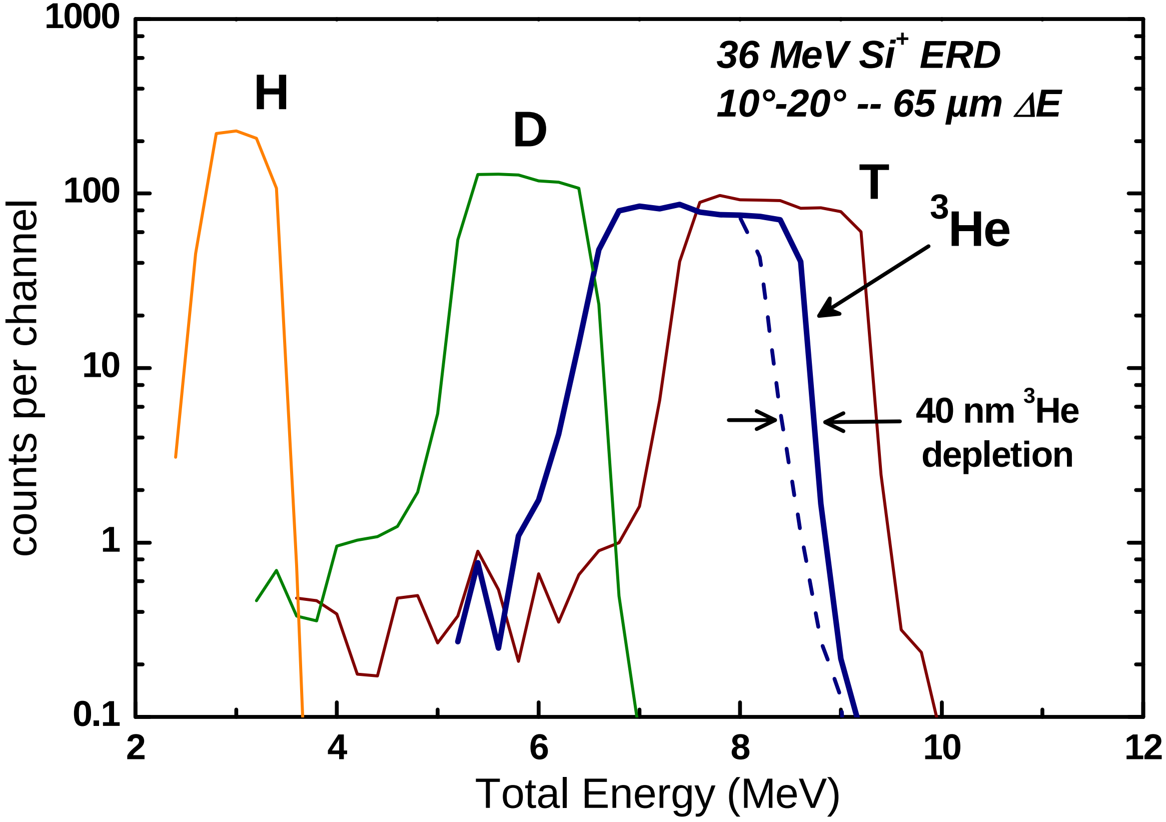
ΔE-E detector at 10° in, 20° out



- Separates H, D, T and ^{3}He from O

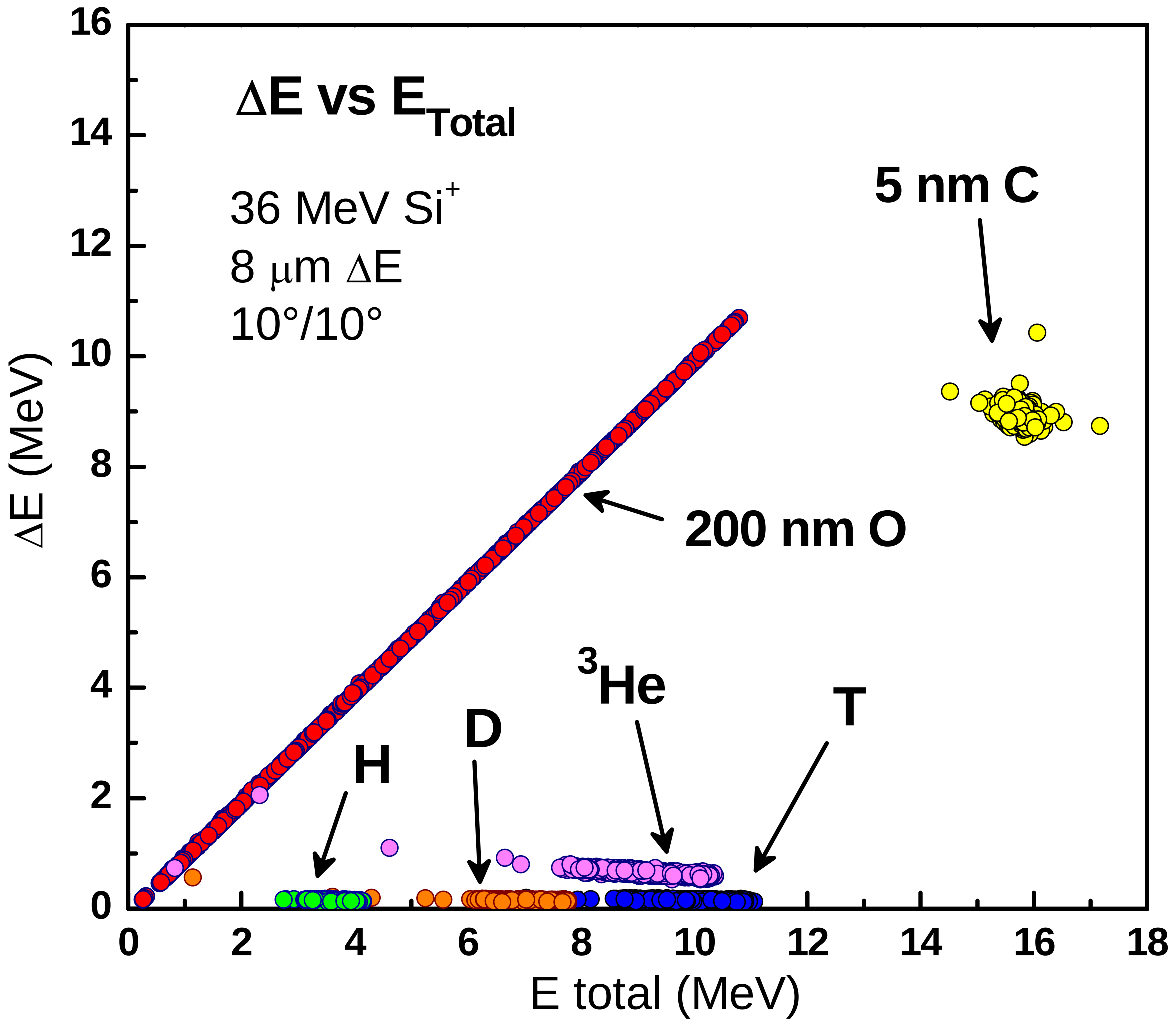


Energy spectra - ΔE-E detector #1

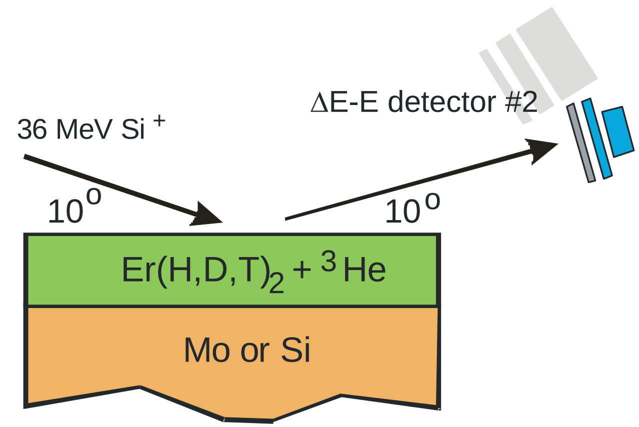


- **Sample: 200 nm $Er(H,D,T)_2$ with 10% ^3He and 5% O**
- **36 MeV Cl^+ beam incident at 10°, exit at 20°, 1.3 msr**
- **65 µm ΔE**
- **$1x10^5$ ion histories**

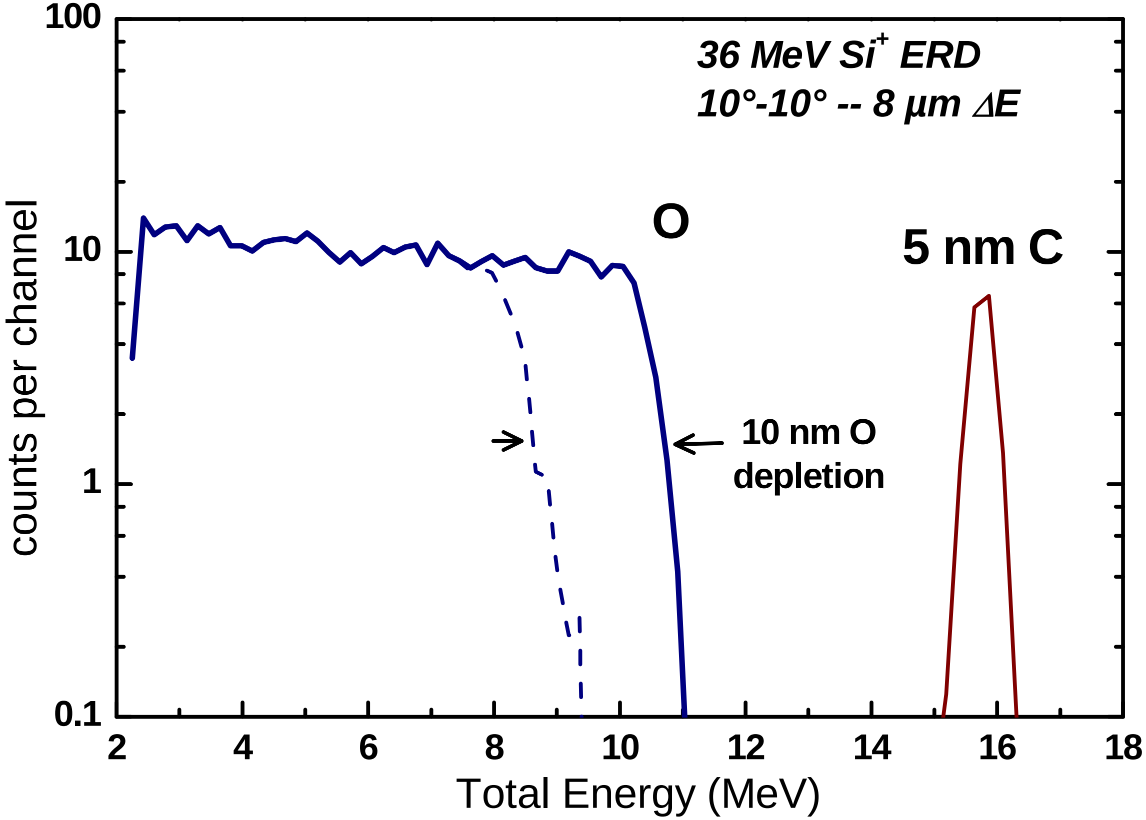
ΔE-E detector at 10° in, 10° out



- **Separates O and C from lighter elements.**

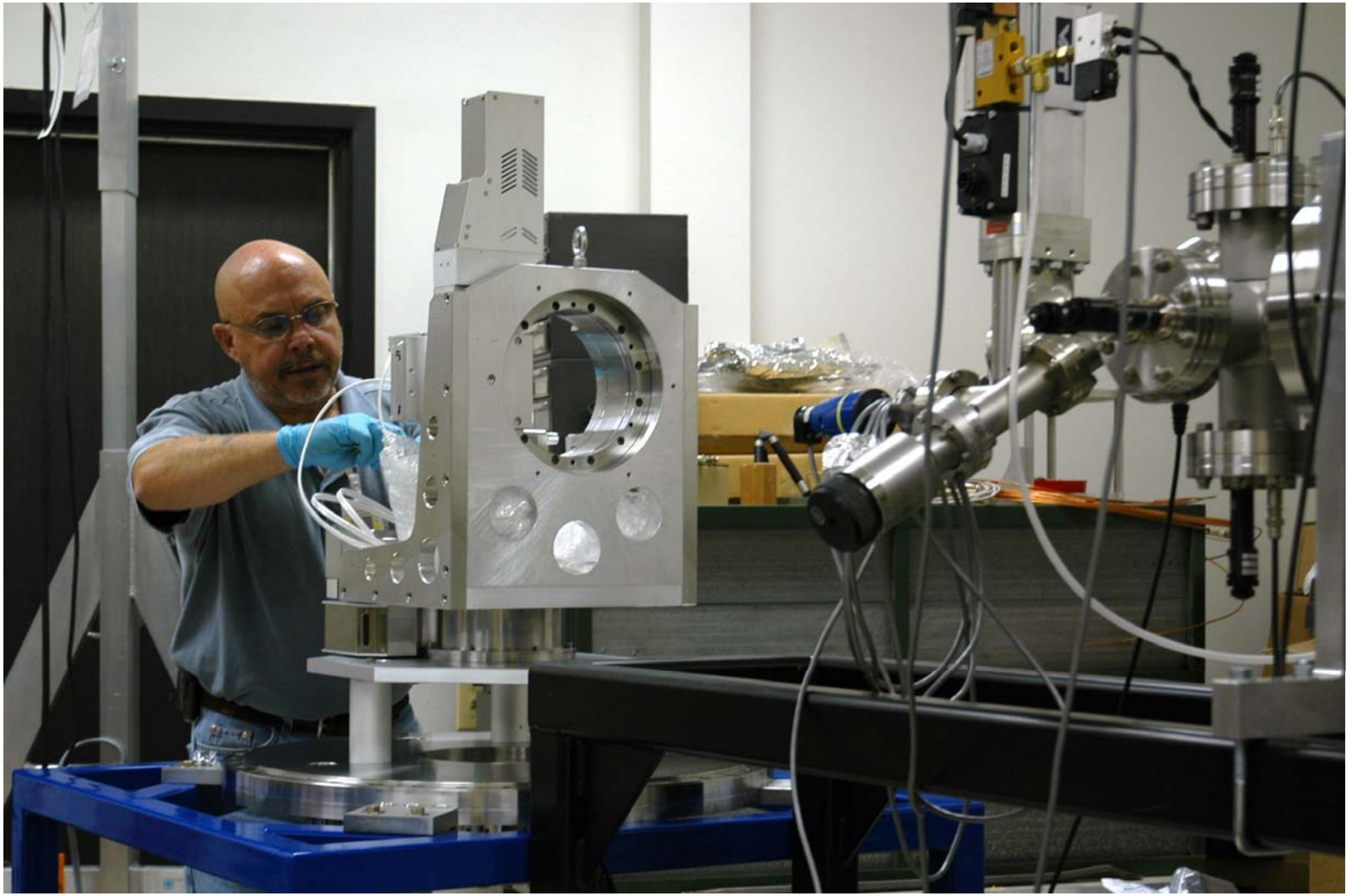


Energy spectra - ΔE-E detector #2

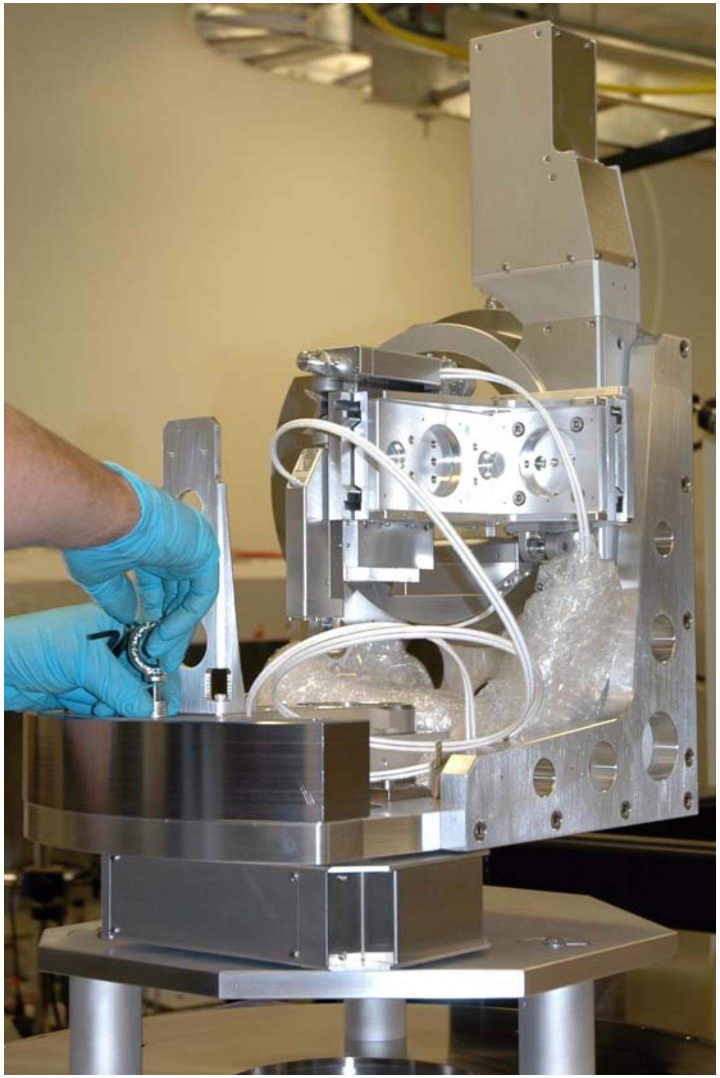


- **Sample: 200 nm Er(H,D,T)$_2$ with 10% ^{3}He and 5% O, plus 5 nm surface C**
- **36 MeV Cl$^+$ beam incident at 10°, exit at 10°, 1.3 msr**
- **8 µm ΔE only**
- **1x10^5 ion histories**

He-3 ion beam analysis system



Goniometer and main chamber



Sample goniometer

- X-Y-Z axes
- 3 rotation axes
- Sample loading and heating in separate chamber

Mechanical properties of tritiated films

- **Problem: Target film mechanical properties are important but largely unknown**

  -- Need to nondestructively measure mechanical properties of tritiated films as they age.

  -- Nanoscale mechanical properties are central to understanding early He release and film failure.

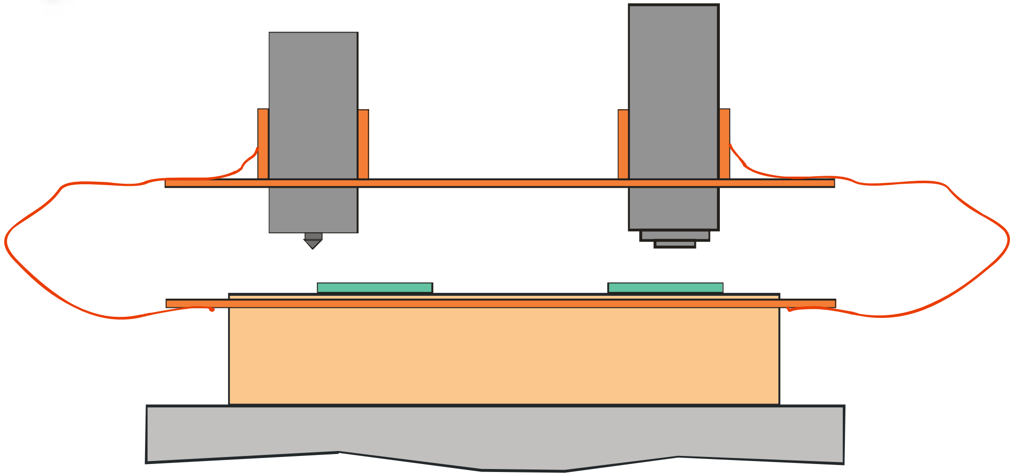
  -- Separating thin film properties from the substrate is an additional complication requiring detailed modeling.

- **Applications:**

  -- Track changes in mechanical strength as ^3He increases and composition changes.

  -- Study effects of alloying additions and impurities on strength.

Nanoindentation of tritiated films

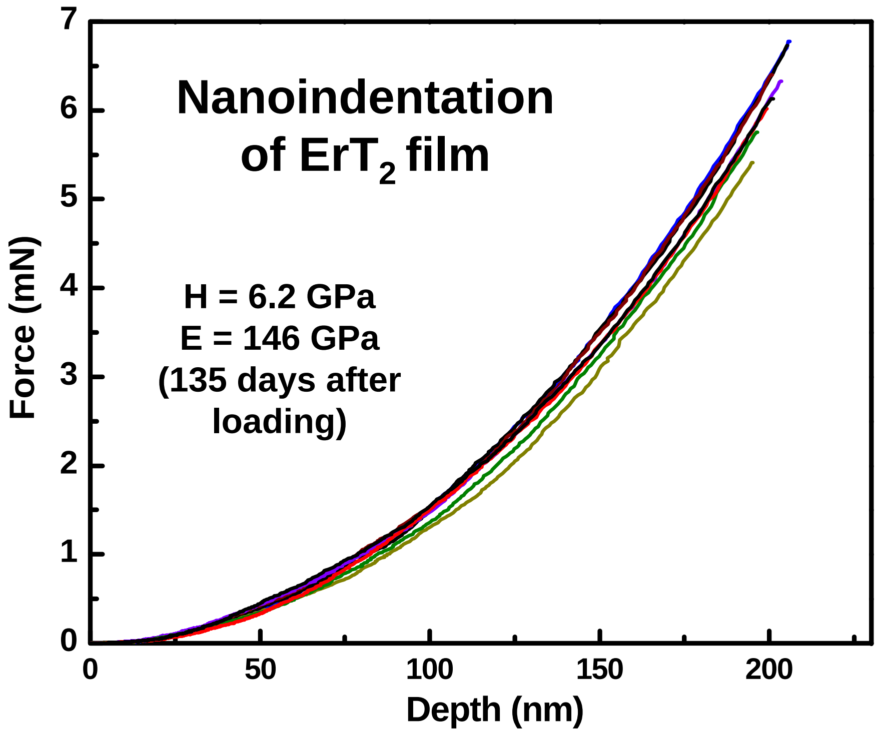


Sample containment fixturing

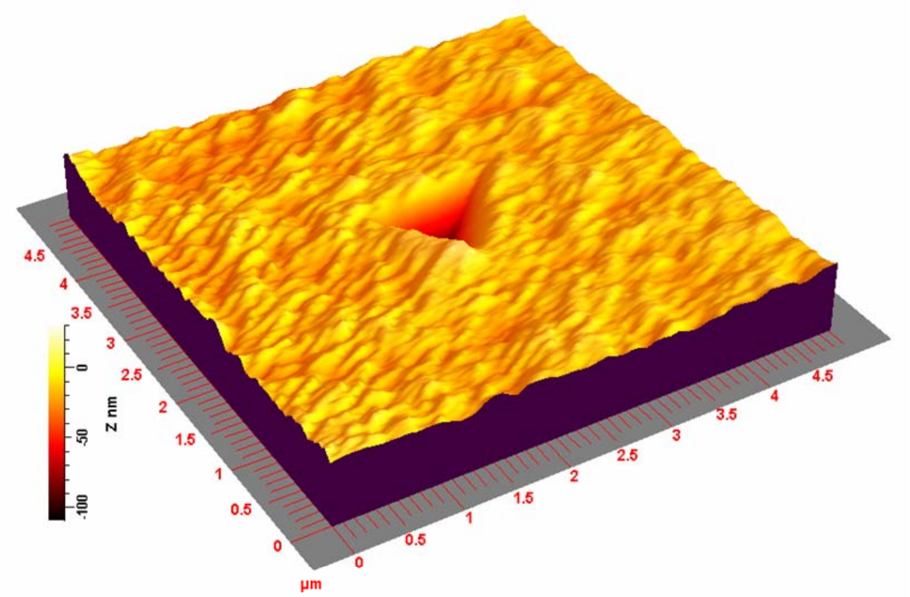
- Samples, indenter tip and optical microscope enclosed in a bag.
- Samples loaded and unloaded in 870.
- Tip and sample hardware dedicated to tritium usage.

Nanoindentation of Erbium Tritide

Finite-Element modeling **of force and stiffness determines film hardness and Young's modulus.**



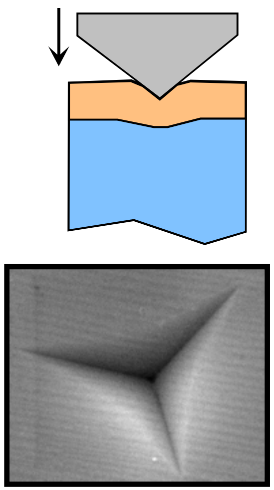
In situ **scan profiling of the residual indent may determine the coefficient of work hardening. (under development)**

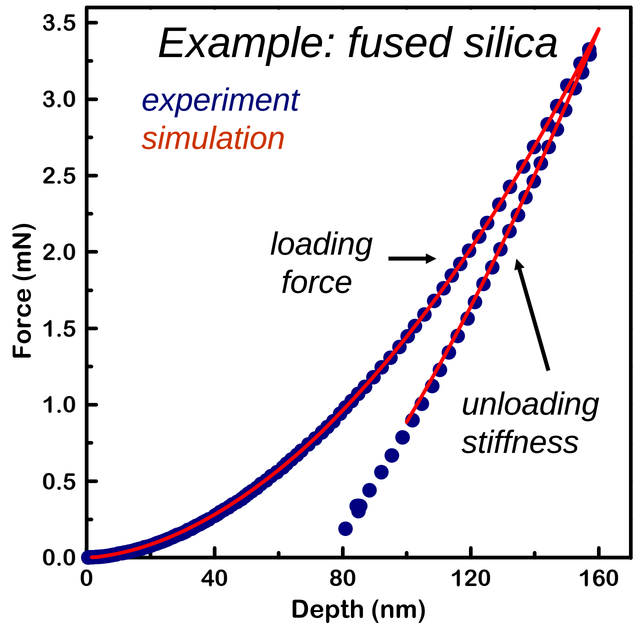


Residual indent profile and pileup imaged using the indenter tip and the new nano-positioning stage

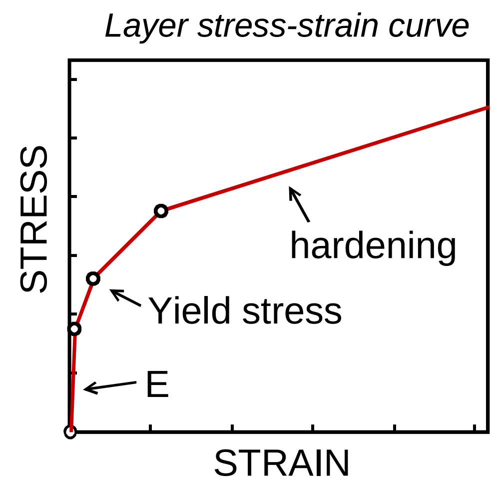
Modeling of nanoindentation

EXPERIMENT





SIMULATION

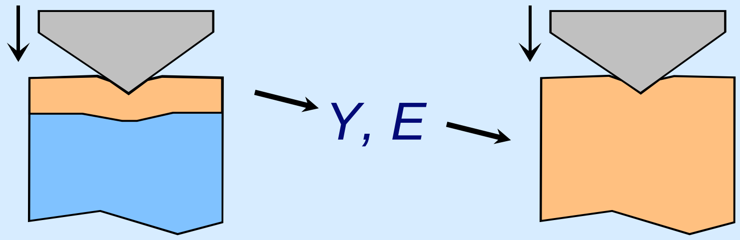


- Experiment: triangular tip pressed into specimen – force required depends on the mechanical properties of both layer and substrate.
- Simulation: finite element modeling – vary yield and elasticity for just the layer until a good fit to experiment is obtained.

Finite-element simulations of nanoindentation

- Simulations use Abaqus/Standard 6.3 on a 450 MHz Sun workstation.
  - *2D: 30-60 mins.*
  - *3D: up to a few days.*
- Properties of the indenter and underlying layers and substrate are fixed at known values.
- Y and E for the layer are varied until a good fit to experiment is obtained.
  - *Tip yielding, stress, friction are all modeled.*
- Two primary simplifications:
  - *2-dimensional axisymmetric meshes*
  - *isotropic elastic-plastic materials with Mises yield criteria*

- Hardness of the layer material is determined by an additional simulation of a "bulk" sample of just the layer material:



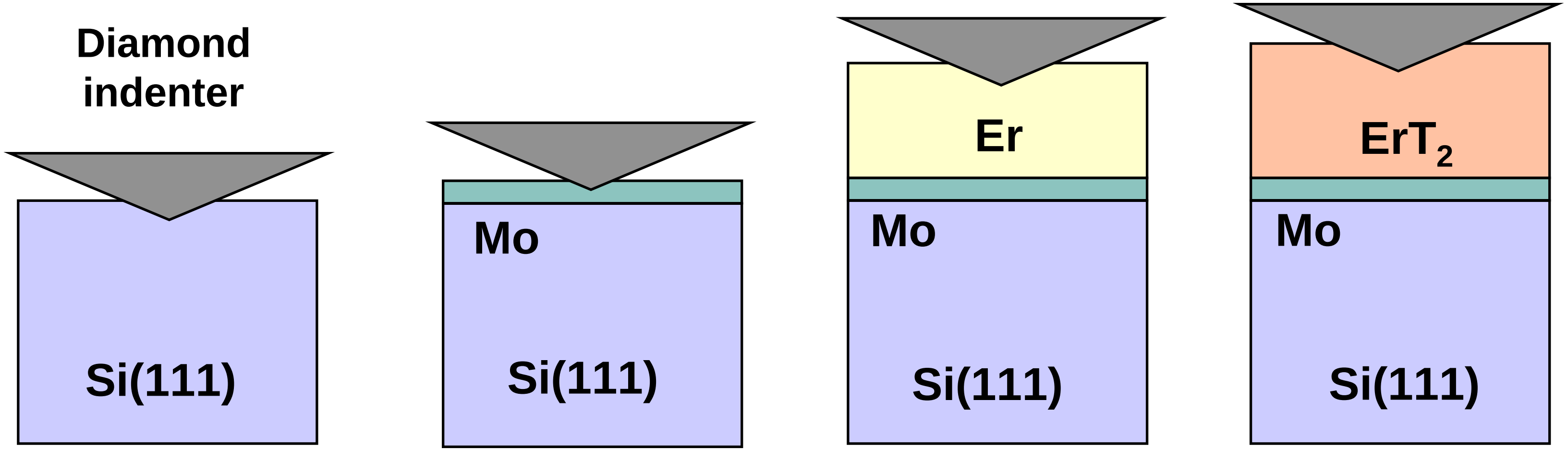
Samples: ErD_2 and ErT_2 layers on Mo/Si

(1) Measured Si(111) substrate

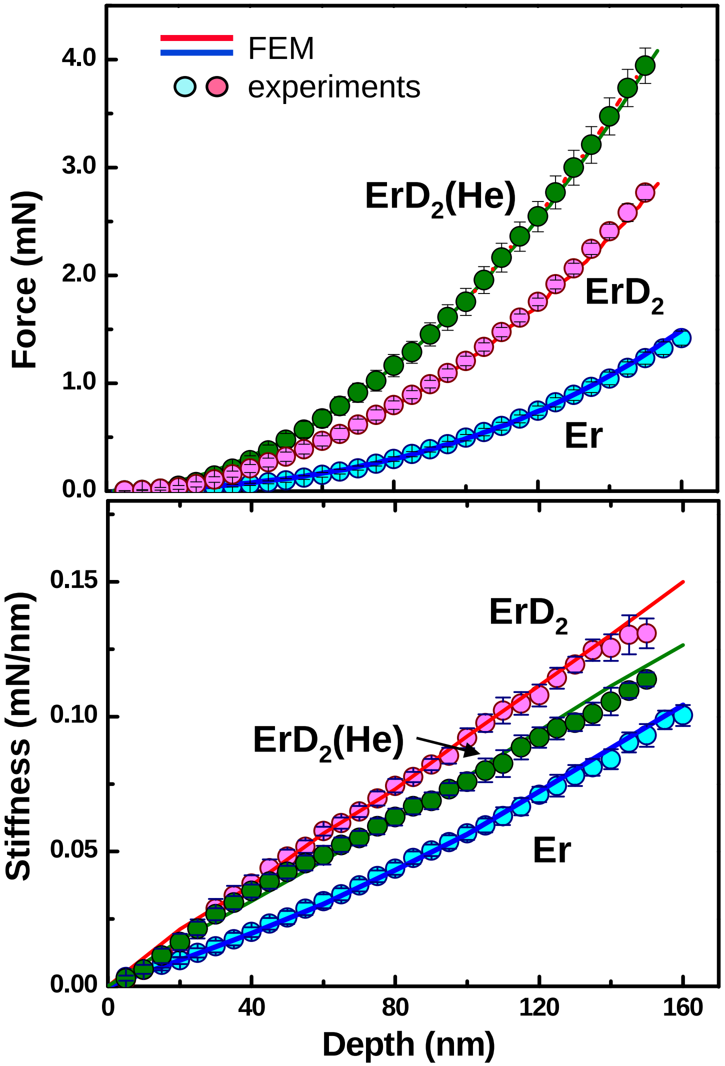
(2) Deposit 95 nm Mo - measured properties

(3) Deposit 500 nm Er - measured properties

(4) Load with D or T - measured properties



Er, ErD_2 and ErD_2 implanted with 5% ^4He



Finite-element analysis

Er layer on Mo/Si(111):

Y: 0.15 ± 0.04 GPa
E: 77 ± 12 GPa
H: 1.9 ± 0.4 GPa

ErD_2 layer on Mo/Si(111):

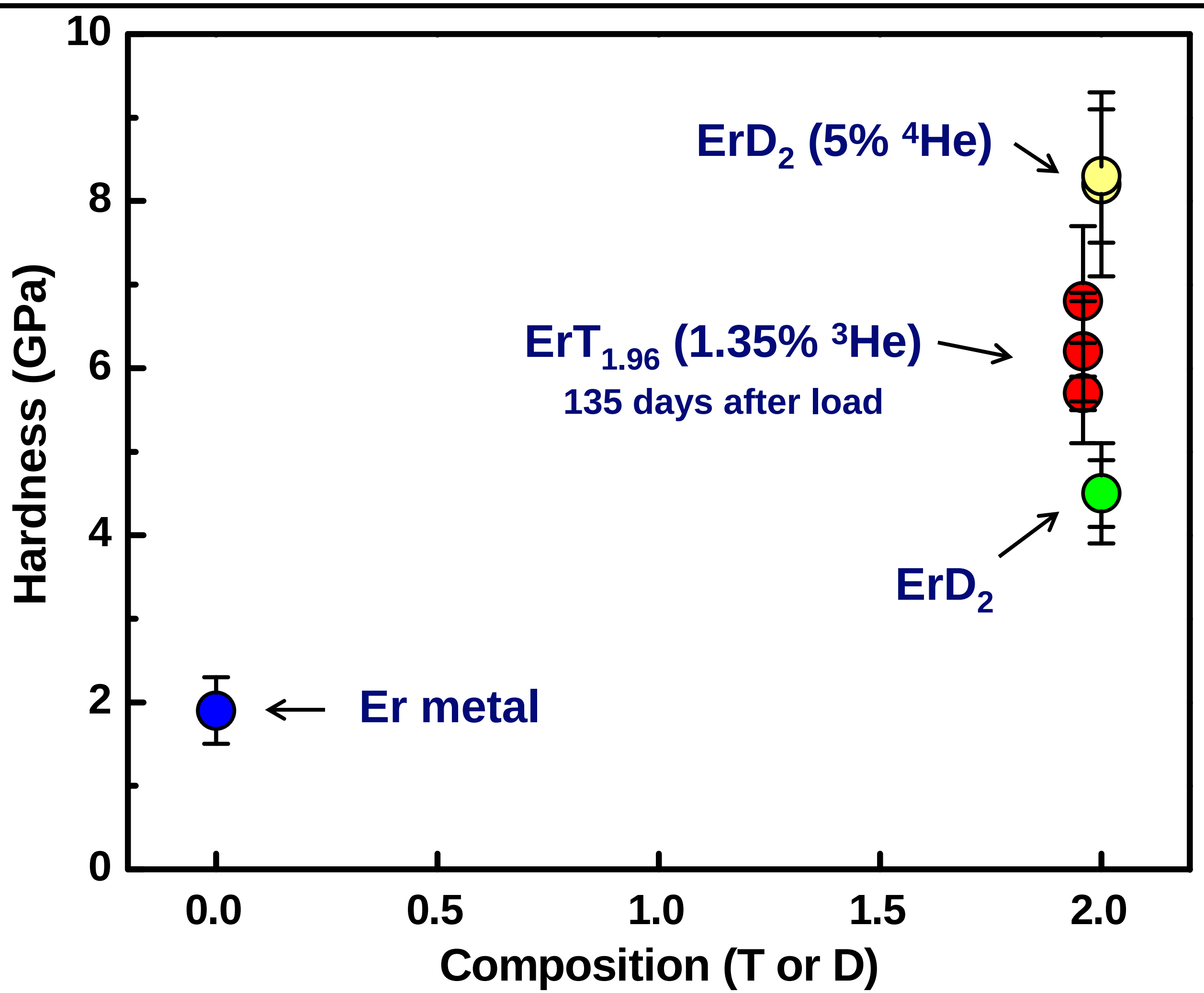
Y: 1.53 ± 0.19 GPa
E: 155 ± 9 GPa
H: 4.5 ± 0.4 GPa

ErD_2 layer implanted with He

Y: 3.32 ± 0.41 GPa
E: 142 ± 10 GPa
H: 8.3 ± 0.8 GPa

- **Er layer is 2-3x harder and ~2x stiffer after loading with D**
- **Implanting 5% He further increases hardness ~2x**

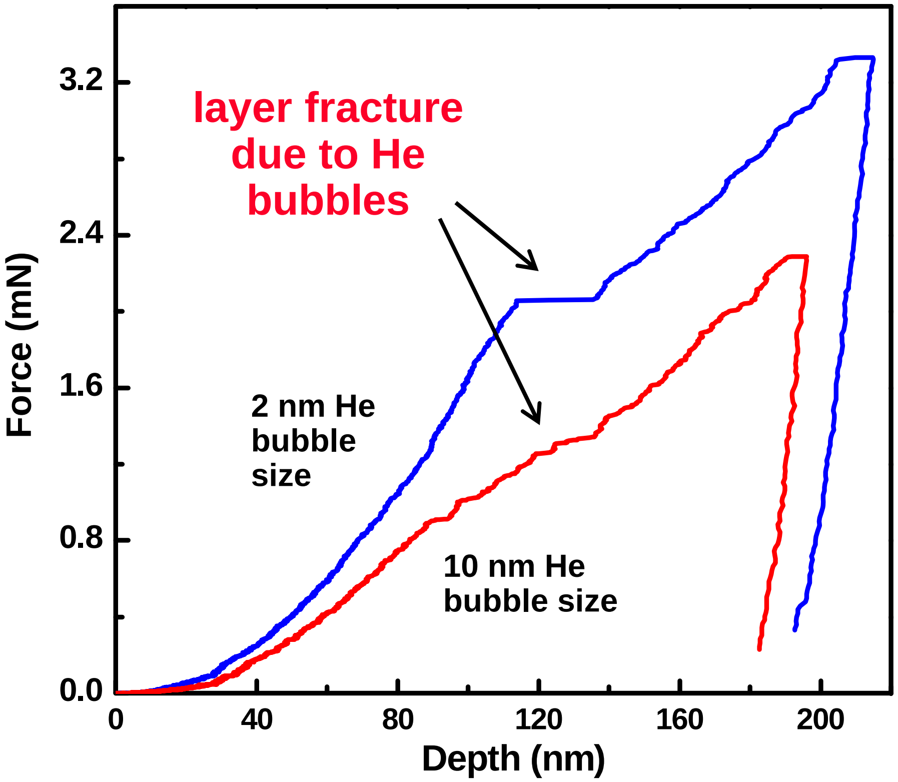
Hardness vs. composition and He content



He bubbles in Ni

Nanoindentation of He-implanted Ni

- **He bubbles strengthen the material, but are susceptible to failure in shear.**



Summary

- **<u>Multi-element ion beam analysis system</u>**

  -- Simultaneous, non-destructive profiling of H, D, T, ^{3}He, O, and surface C.

- **<u>Nanoindentation of tritiated films</u>**

  -- Mechanical property measurements for tritiated thin films.