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Ion beam analysis of Tritiated thin films

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Motivation: metal tritide films

- Metal tritide films are essential for applications such as neutron generators.
  - Problems:
  - T decays to ^{3}He, forming bubbles and stressing the film.
  - O contamination influences hydriding efficiency and film stability.

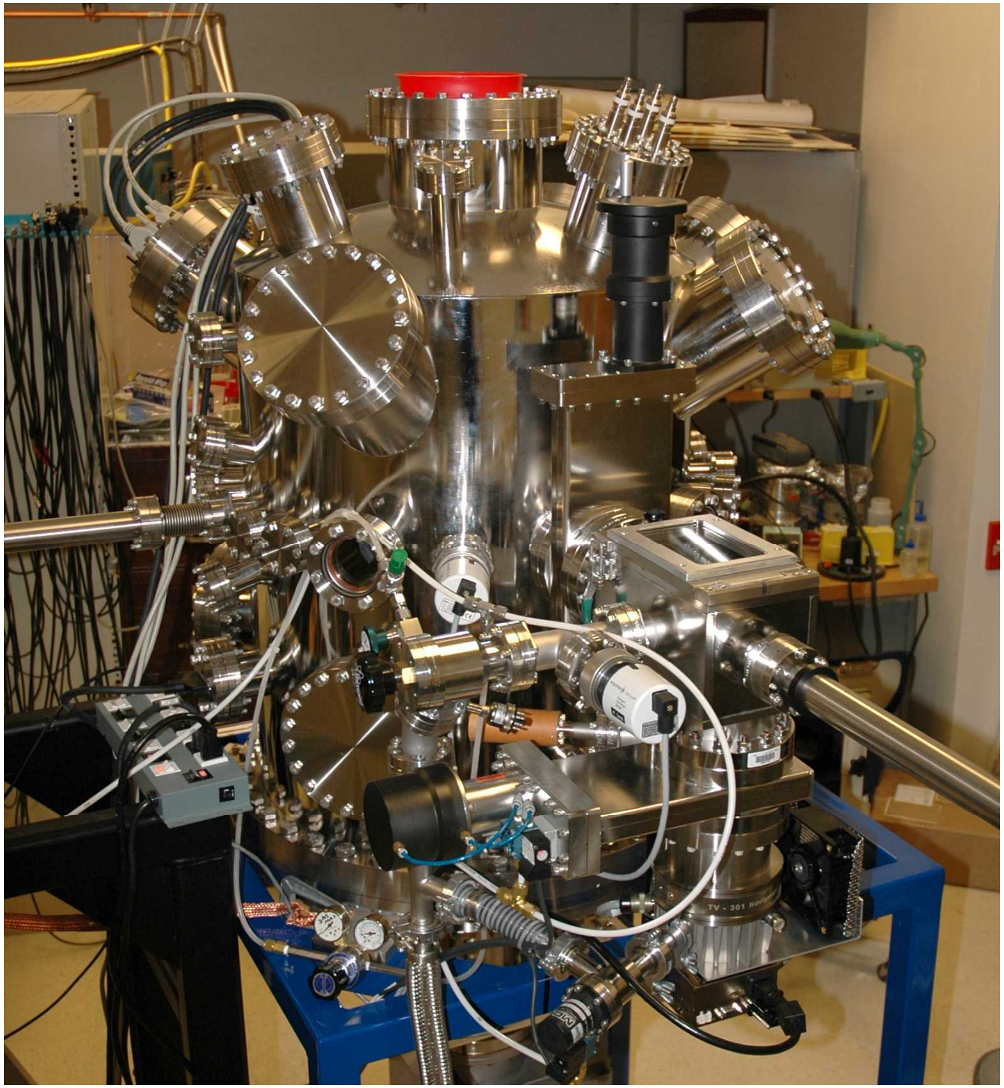
- Tritide film composition and other properties are needed for understanding and controlling hydriding and aging problems.

- Several types of films are being studied:
  - ErT_2 films with 100% T, studied as the films age
  - ErD_2 films hydrided under various conditions

- Here we present:
  - Three examples of high energy, heavy ion ERD profiling of ^{3}He, T, D, H, O and C in these types of samples.

High energy ERD system

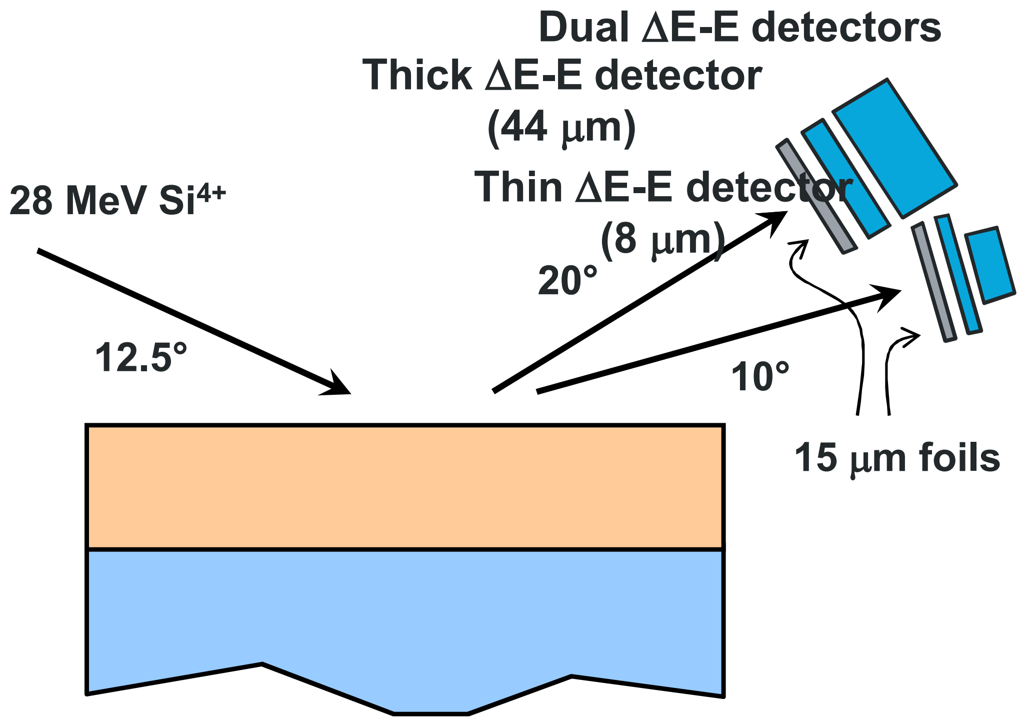
- **Problem:**
  - Need to analyze T, ^{3}He, D, H, O, and C.
  - Our existing ERD analysis chamber used for T could not measure ^{3}He or O on Si.
  - New system designed based on high energy, heavy ion beams and ΔE-E detectors.



- **Analysis chamber:**
  - beam line on 7 MV Tandem
  - 6-axis custom goniometer
  - all-metal ion-pumped chamber
  - load lock with heating stage

Analysis method

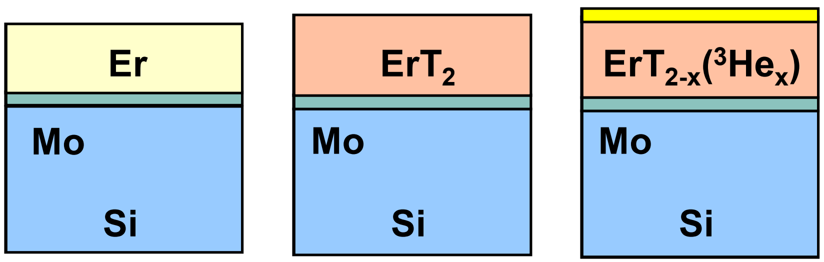
- Heavy ion elastic recoil detection (ERD) with 28 MeV Si^{+4} analysis beam incident at 12.5°
- Thick ΔE-E detector to profile H, D, T, ^{3}He
- Thin ΔE-E detector to profile O, C
- Each detector pair has a 15 μm foil to block the Si analysis beam and curved slits to optimize resolution.



Example: ErT_2 layers on Mo/Si

Sample preparation

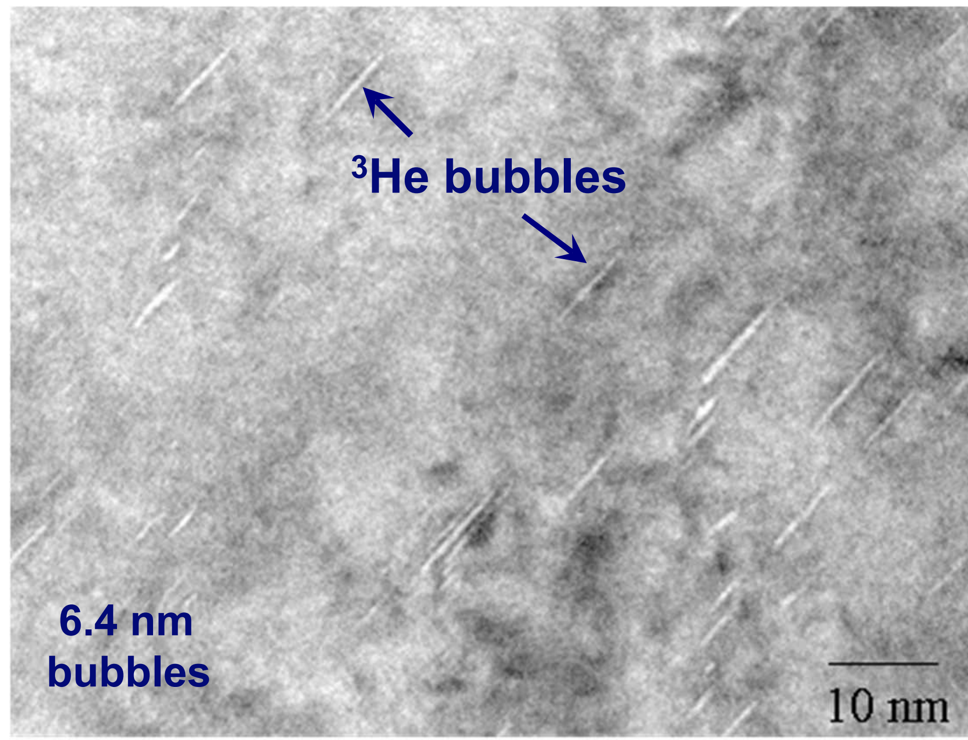
500 nm Er 95 nm Mo Silicon	Hydrided with 100% T at LANL	Aged in vacuum



Oxide forms during hydriding and upon air exposure.

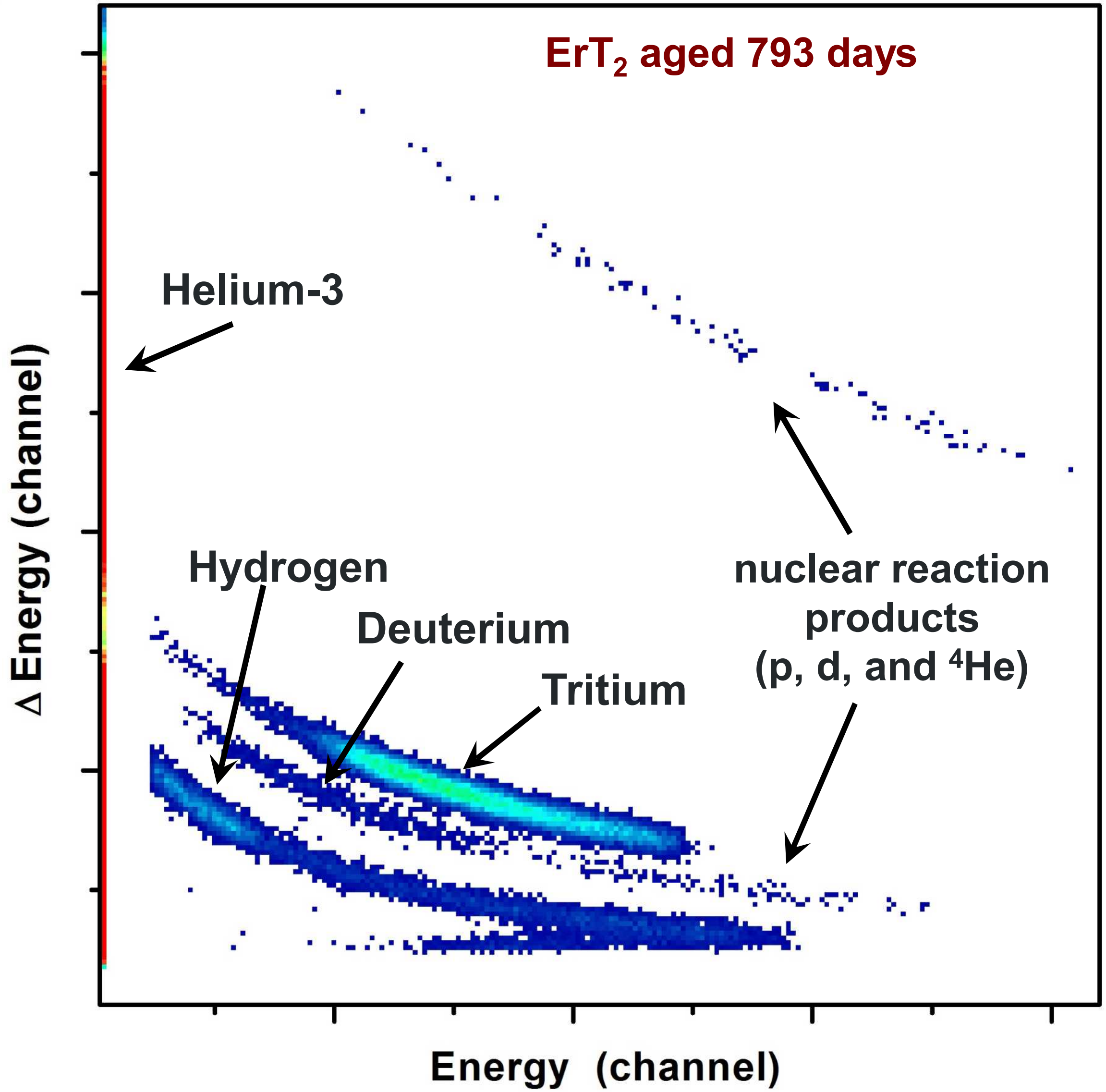
Tritium decays into ^3He, forming platelet-like bubbles on (111) planes.

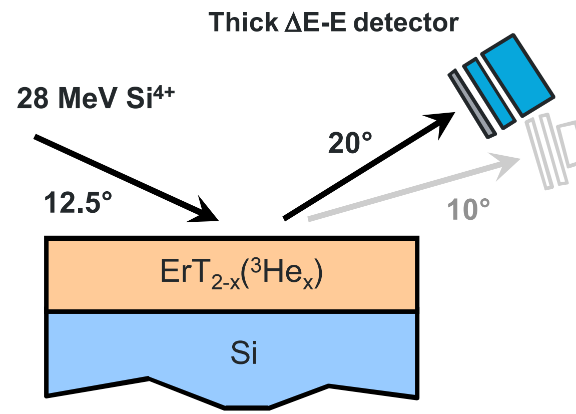
TEM cross-section bright-field, ~{110} zone 62 days after hydriding.



TEM with Gillian Bond, New Mexico Institute of Mining and Technology

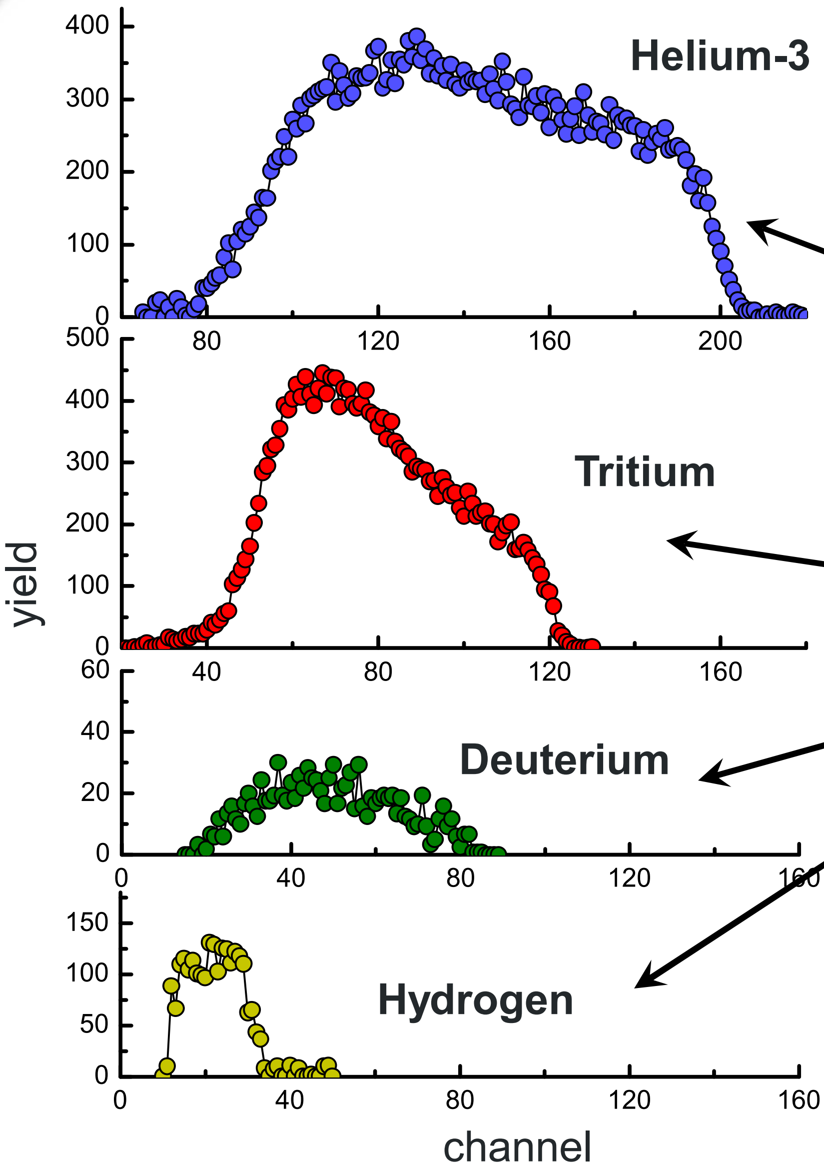
Coincidence map for thick ΔE-E detector

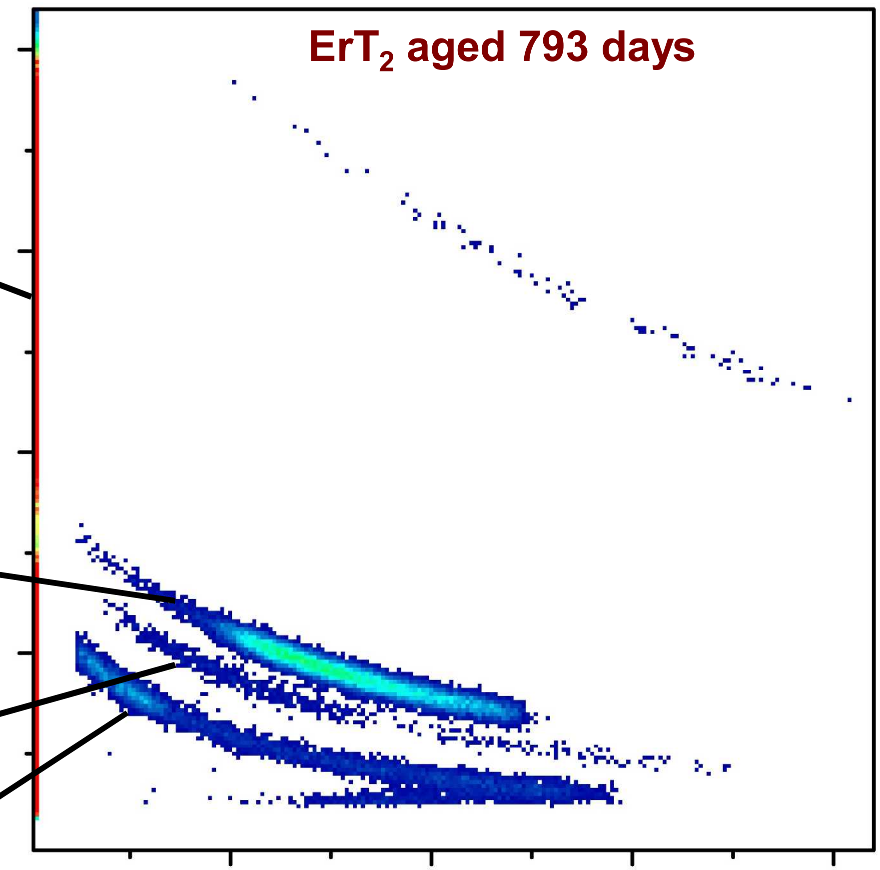




12.5° in, 20° out
28 MeV Si^{+4} beam
15 μm foil
44 μm ΔE detector

Spectra for **thick** ΔE-E detector

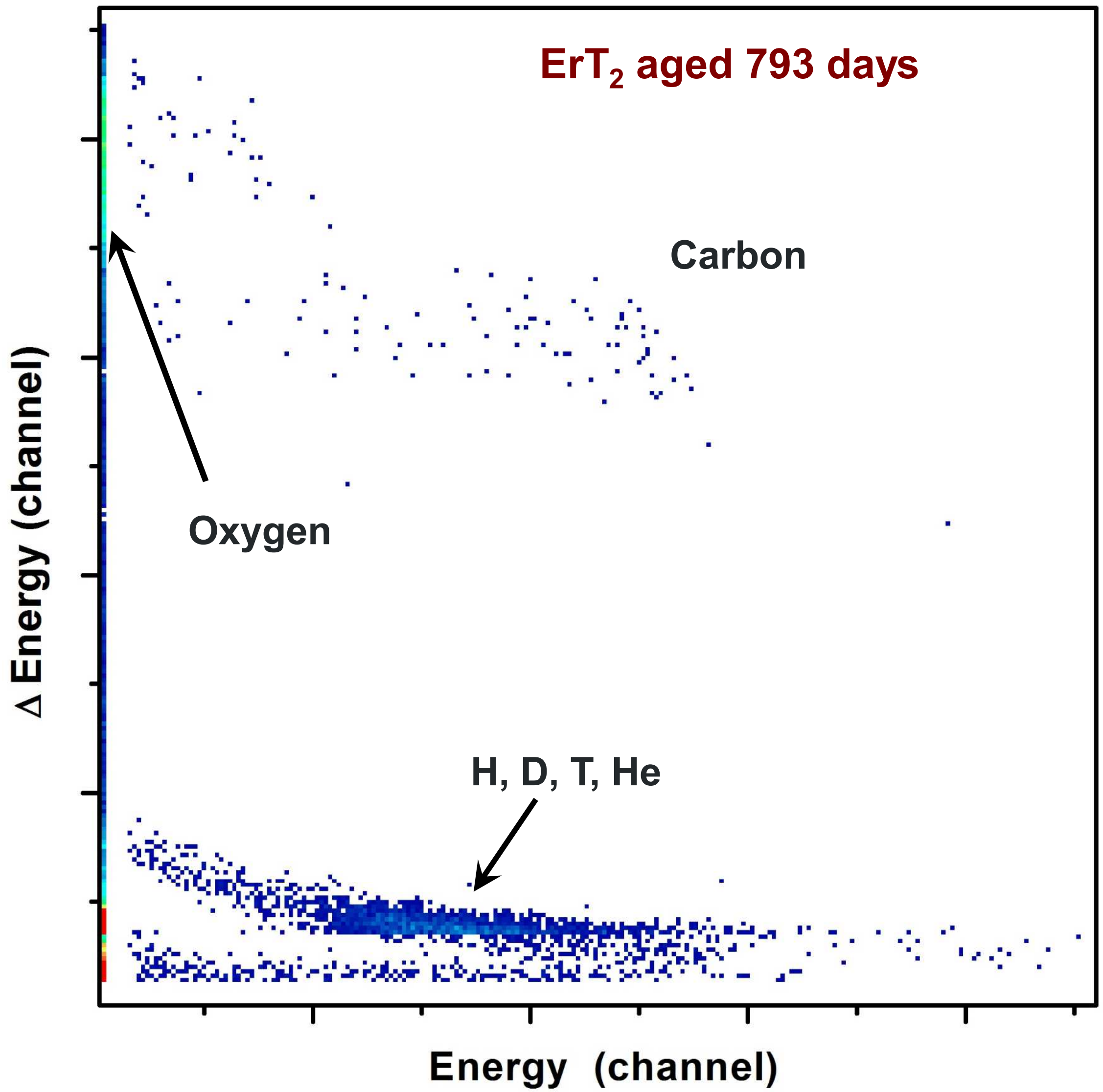


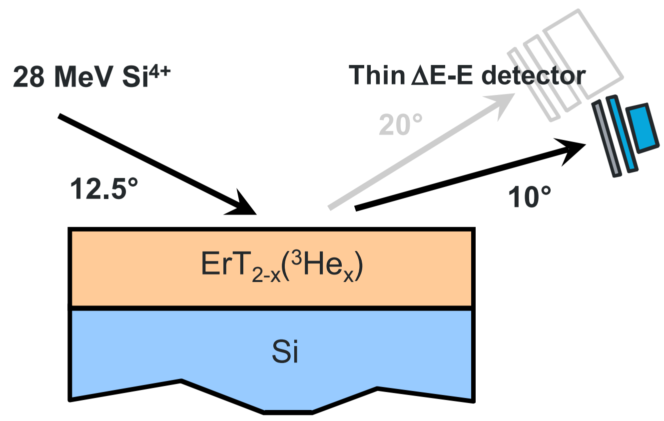


12.5° in, 20° out
15 μm foil

28 MeV Si^{+4} beam
44 μm ΔE detector

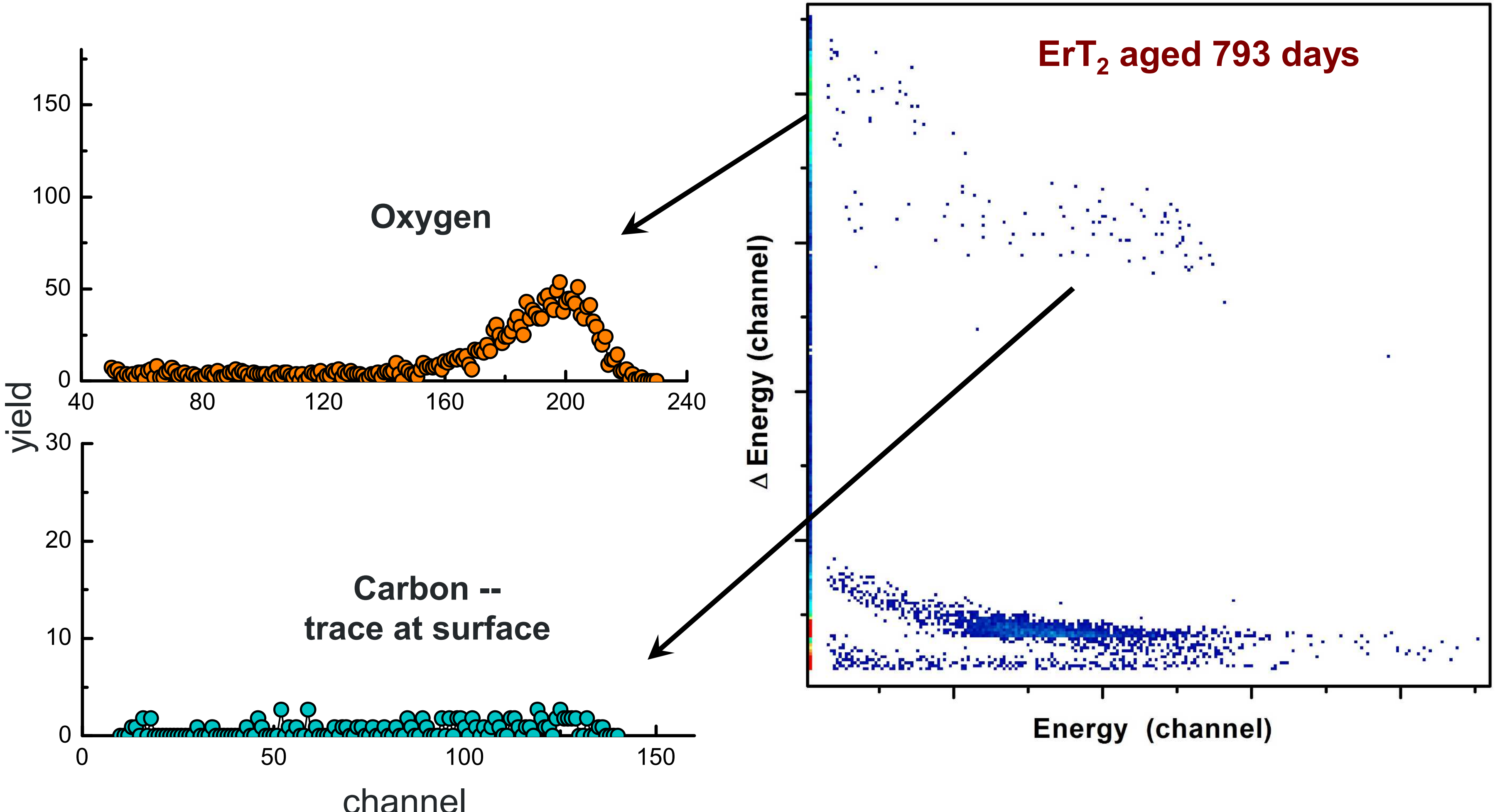
Coincidence map for thin ΔE-E detector





12.5° in, 10° out
28 MeV Si^{+4} beam
15 μm foil
8 μm ΔE detector

Spectra for thin ΔE-E detector

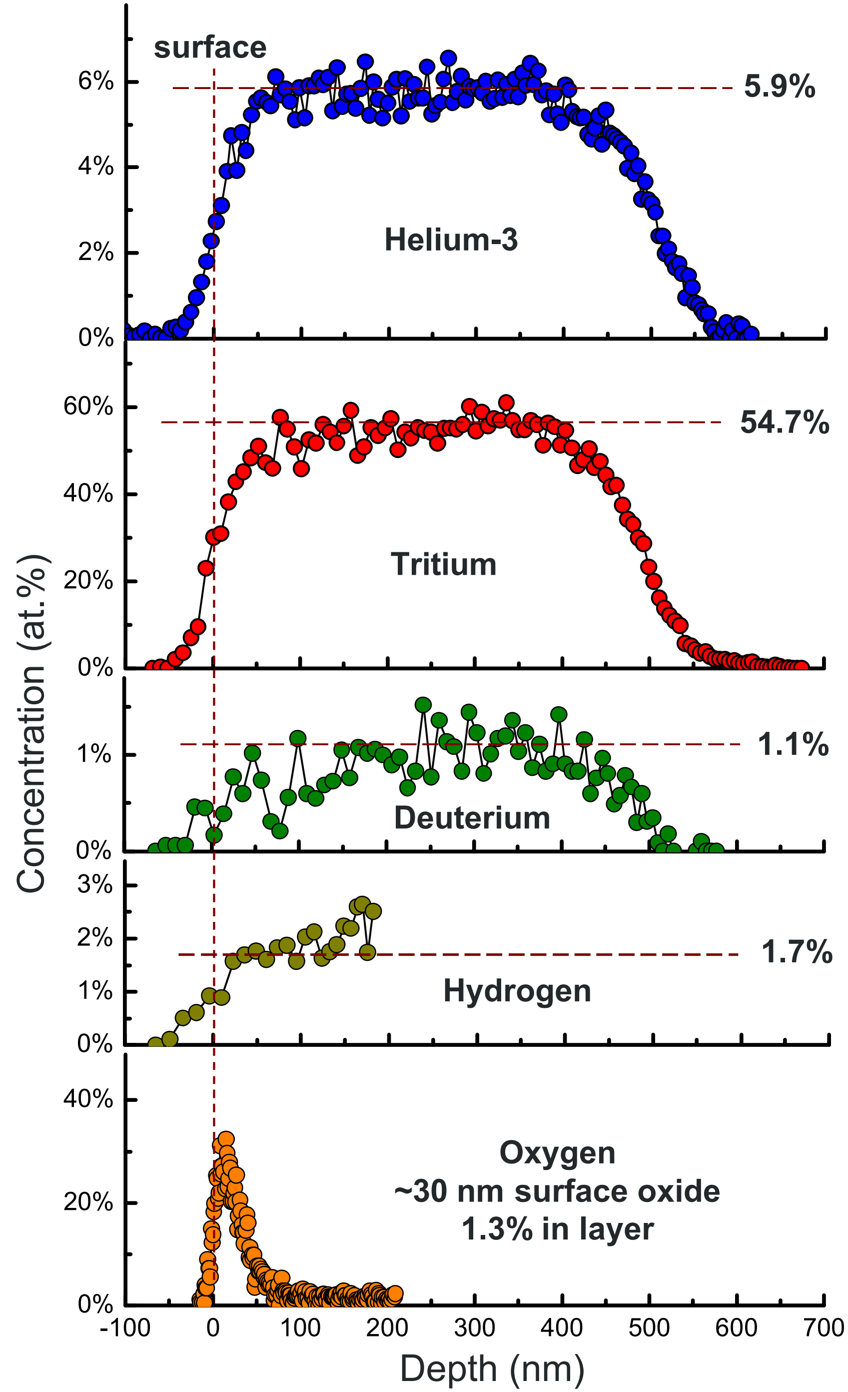


12.5° in, 10° out
15 μm foil

28 MeV Si^{+4} beam
8 μm ΔE detector

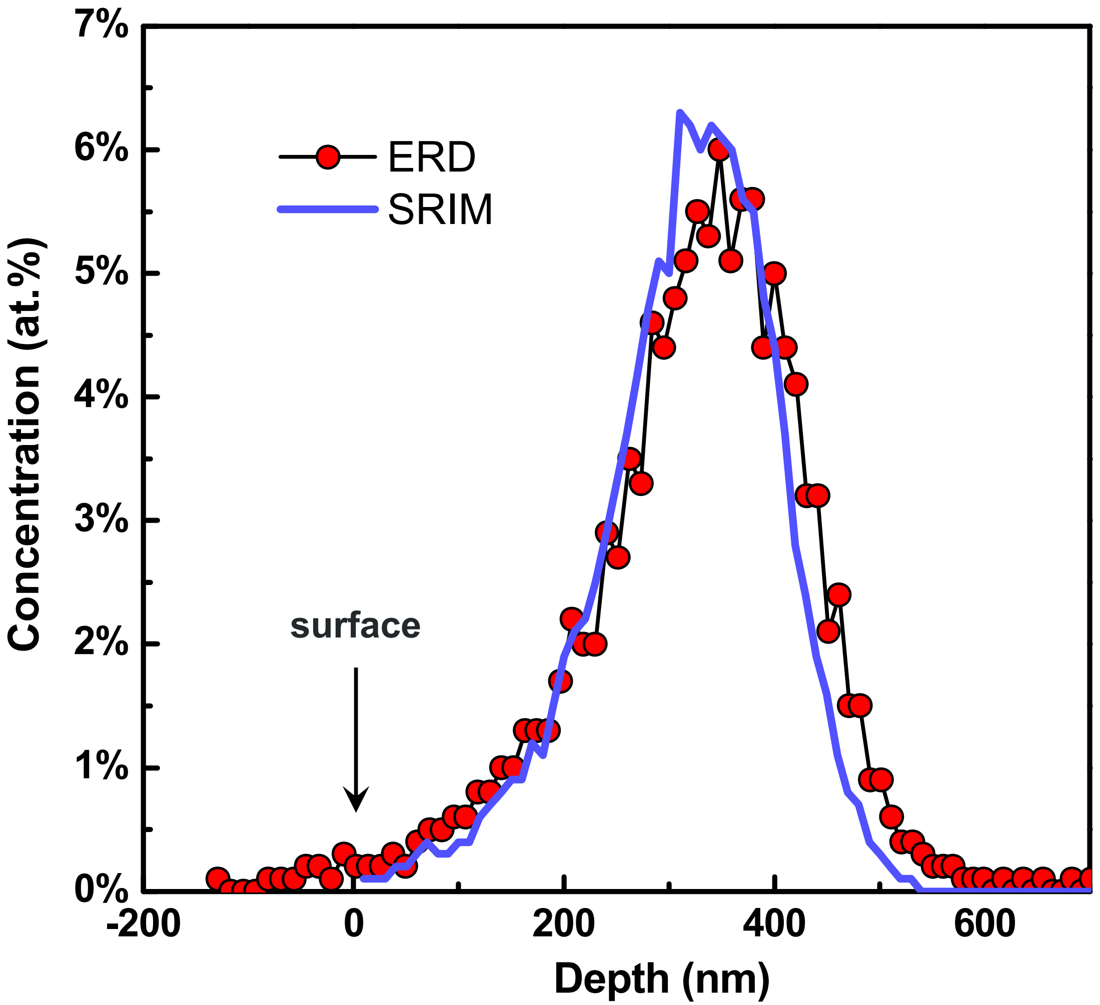
Profiles using SIMNRA

- -- Spectral data scaled in depth and atomic concentration using SIMNRA simulations.
- -- Broadening due to straggling and roughness has not been removed.
- -- Depth scales calculated using the density of ErH_2.
- -- Indicated concentrations are in the bulk of the layer. Near surface concentrations are lower due to Oxygen.
- -- T cross-section is non-Rutherford and was deduced by fitting T spectra obtained at 24–30 MeV, calibrated with lower energy He ERD. ^{3}He cross-section may also need calibration.



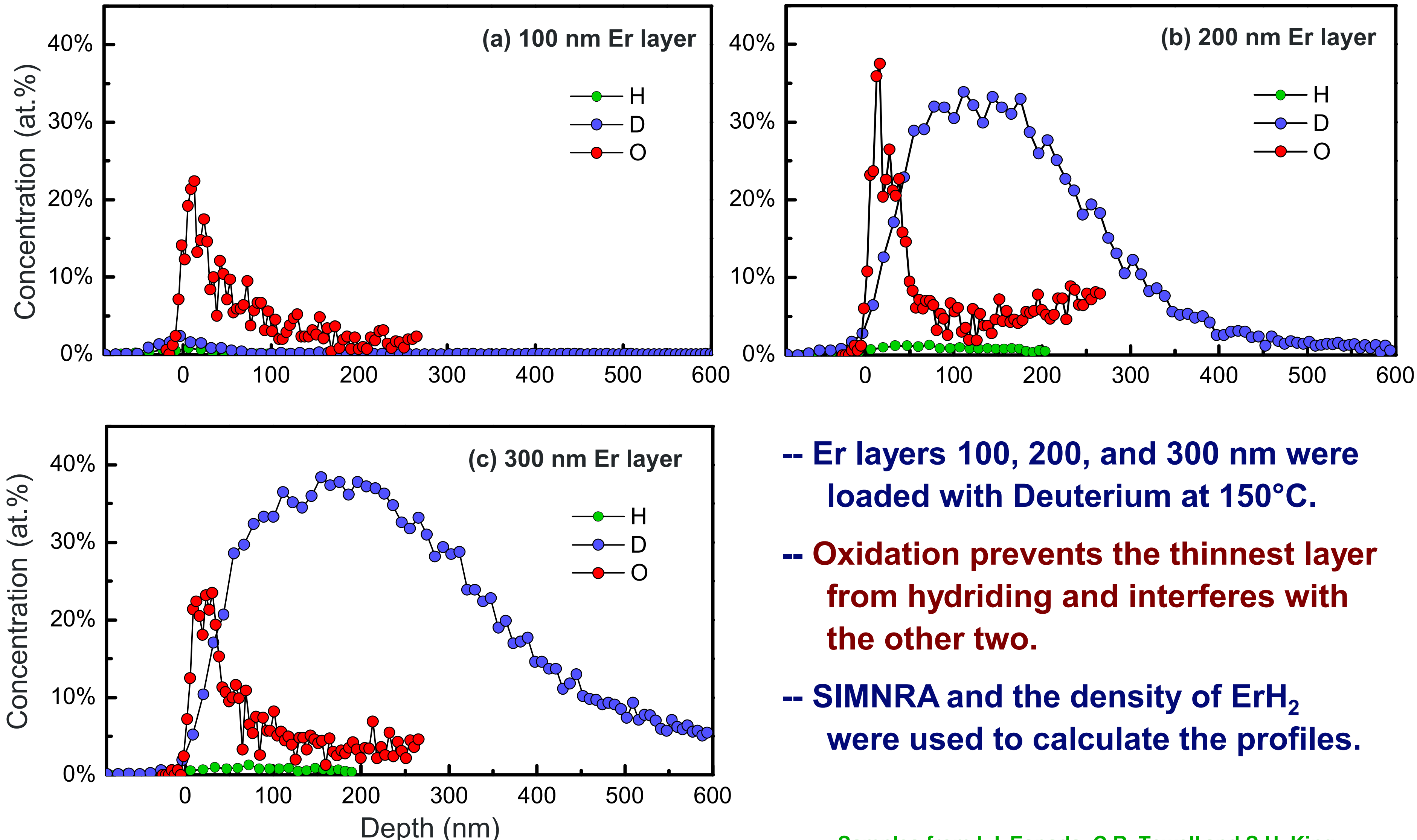
Example: ^{3}He-implanted Si

1.6 x 10^{16} ^{3}He/cm^{2} implanted at 40 keV into Si.



- -- Spectral data scaled in depth and atomic concentration using SIMNRA simulations.
- -- Broadening due to straggling and roughness has not been removed.
- -- Depth scale calculated using the density of Si.
- -- SRIM 2003 was used to estimate the implant profile.

Example: Deuteriding Er layers



-- Er layers 100, 200, and 300 nm were loaded with Deuterium at 150°C.

-- Oxidation prevents the thinnest layer from hydriding and interferes with the other two.

-- SIMNRA and the density of ErH_2 were used to calculate the profiles.

Samples from L.I. Espada, C.R. Tewell and S.H. King.

Summary

Model ErT_2 films have been fabricated and are being examined with a variety of techniques as they age.

Other hydride films are being examined to deduce the effect of contamination on the hydriding process.

The new ERD system allows non-destructive profiling of ^{3}He, T, H, D, O, and C with down to 10 nm resolution, limited by surface roughness.