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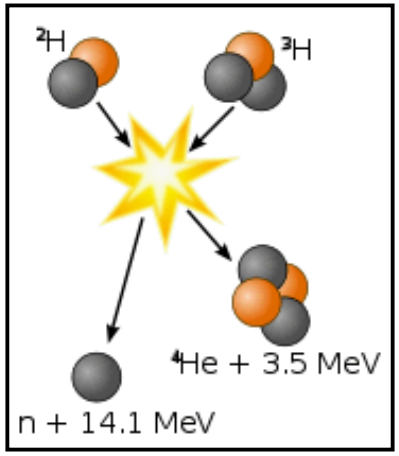


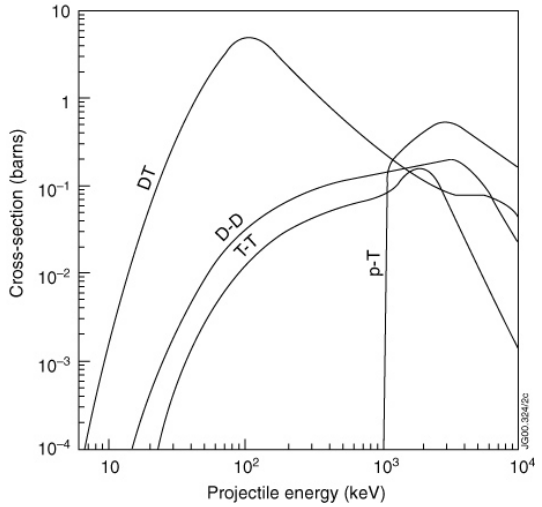


# Overview of Sandia's Analytical Tools for Measuring and Monitoring Tritium and Contaminated Materials

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Sandia National Lab, NM

U.S. DEPARTMENT OF ENERGY | NNSA National Nuclear Security Administration

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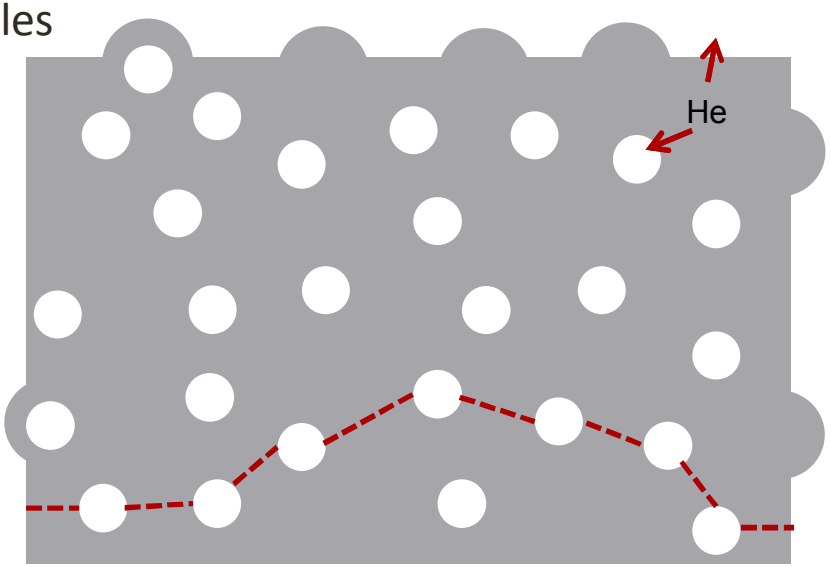
# Outline

- Tritium materials interactions, and tube operations
- Tritium Safety
- Tritium Loading
- Tritium Forensics Tools and examples
- Other Facilities

# Tritium-Material Interactions

- Tritium dissolves in material lattice causing metal embrittlement
- Tritium decays to $^{3}He$
  - Insoluble in most materials
  - Forms high pressure nanometer sized bubbles
- Detrimental Impact on Material Properties
  - Embrittlement
  - Swelling

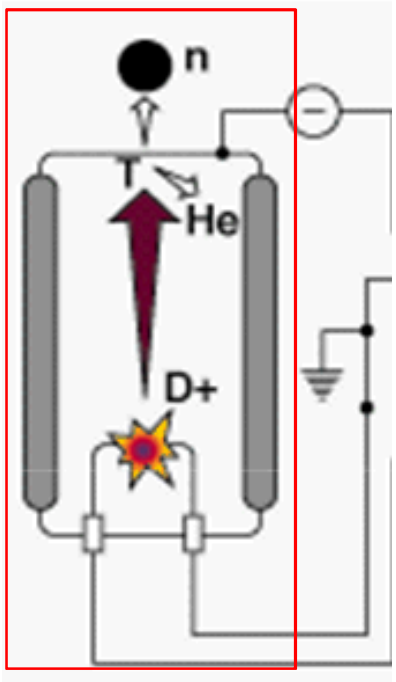
Helium bubbles cause materials to become prone to fracture and **catastrophic release** of helium




Schematic of Helium Bubbles Causing Cracks

# Overview Neutron Generators

- Neutron generators are neutron source devices which contain compact linear accelerators and that produce neutrons by fusing isotopes of hydrogen together. The fusion reactions take place in these devices by accelerating either deuterium, tritium, or a mixture of these two isotopes into a metal hydride target which also contains deuterium, tritium or a mixture of these isotopes.




Tube

# Overview of Sandia/NM Tritium Capabilities

- All tritium operation is performed inside a “tritium envelope” with dedicated single-pass & monitored ventilation
- Equipment can be connected to both
  - Tritium Exhaust (smoke stack release).
  - Tritium Capture System
    - Only within the Tritium Envelope
    - Tritium Capture System (TCS)
    - Tritium Loaders (U-bed to Films)
    - Mass Spectrometers (analysis of produced parts, then to TCS)
    - Other Research Equipment

# Tritium Capture System-Tritium Safety


**80 ft$^3$ accumulation tank**

- **Sub-atmospheric pressure**

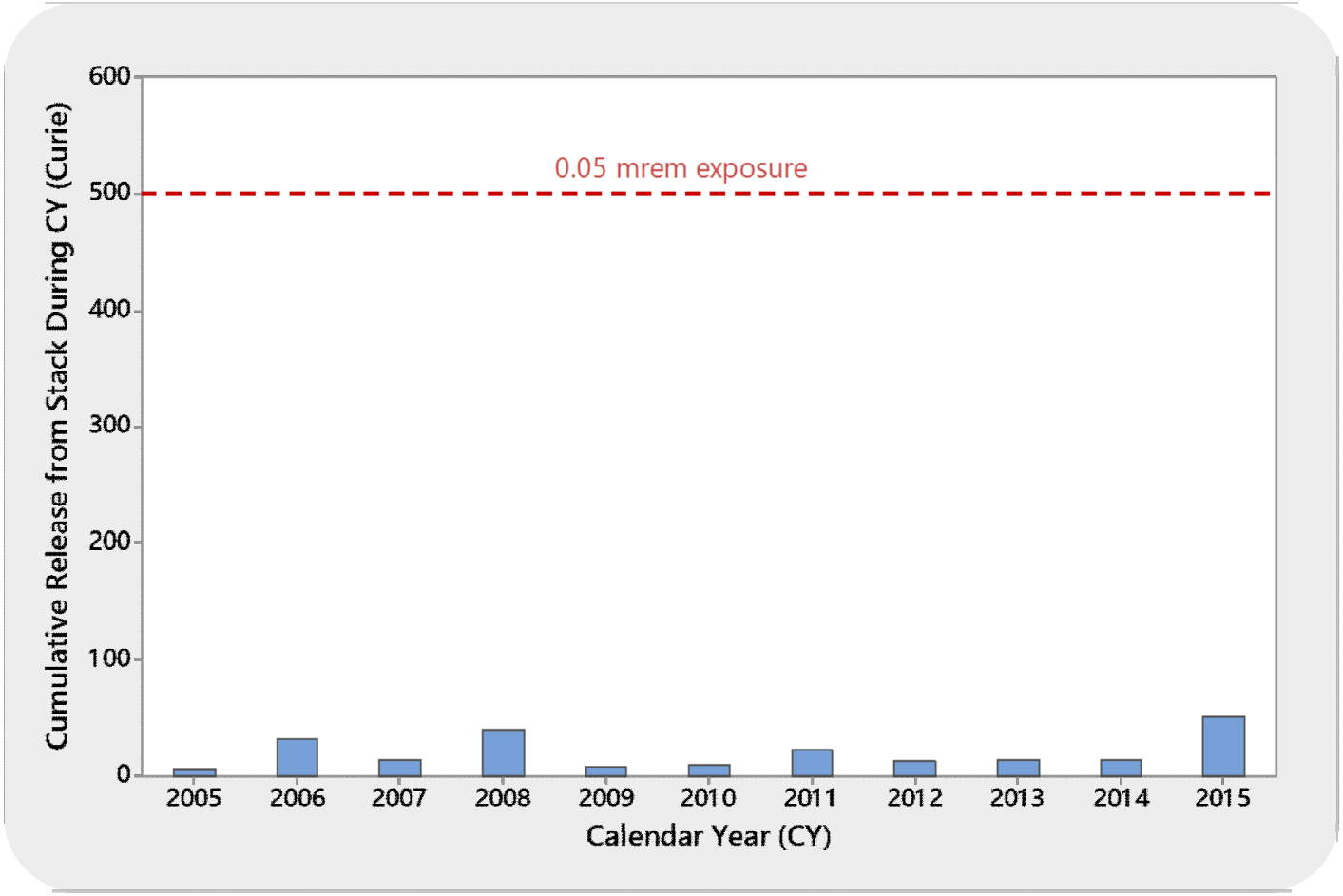
**Valve control panel, sieve beds, catalytic burners**

- **Effluent tritium verified, then sent to stack**

**Calorimeter for sieve bed analysis**

Ensures that effluent gasses have minimal tritium content-tritium safety

# Overview of Sandia/NM Tritium Capabilities



# Tritium Gas Loading

**Tritium Loader – gas manifold built in a glove box, capable of 100% tritium or dilute loading of metal tritides**

# Gas Analysis Capabilities

Used for thin film analysis

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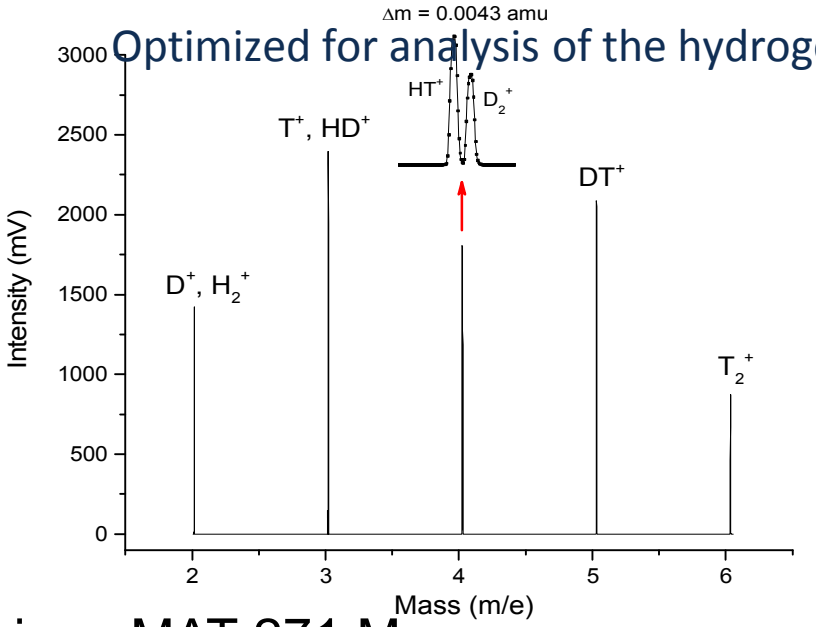
## Mass Spectrometer


Optimized for analysis of the hydrogen isotopes



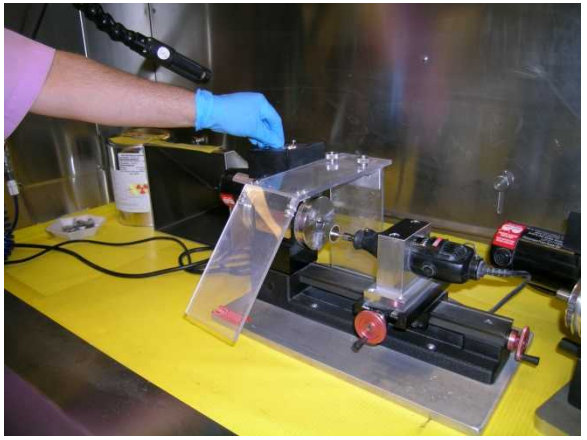

## Finnigan MAT 271 Mass Spectrometers

- Measurement uncertainty < +/- 2%
- 1-150 mass range
- Capable of separating and independently measuring all hydrogen isotope combinations (Resolution > 1500)
- No other technology demonstrates as good a sensitivity/resolution in the low 1-6 mass range.
- Dynamic range of $10^9$
- Very high adjacent mass rejection ratio
- Advanced ion source design which exhibits a low rate for hydrogen isotope exchange

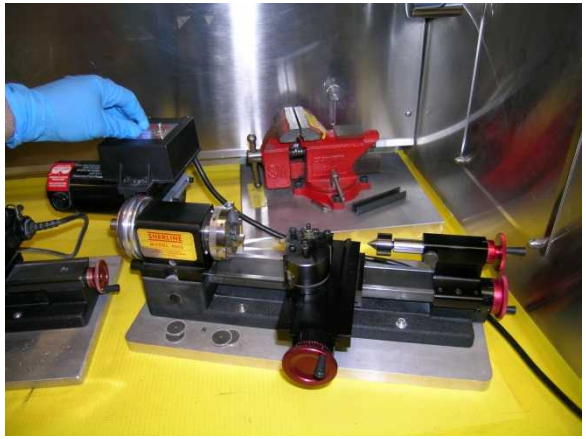
# Tube Disassembly and Inspection

Glove box Lathe is used to cut through the large flange.


Glove box Drill press is used to drill out the targets.



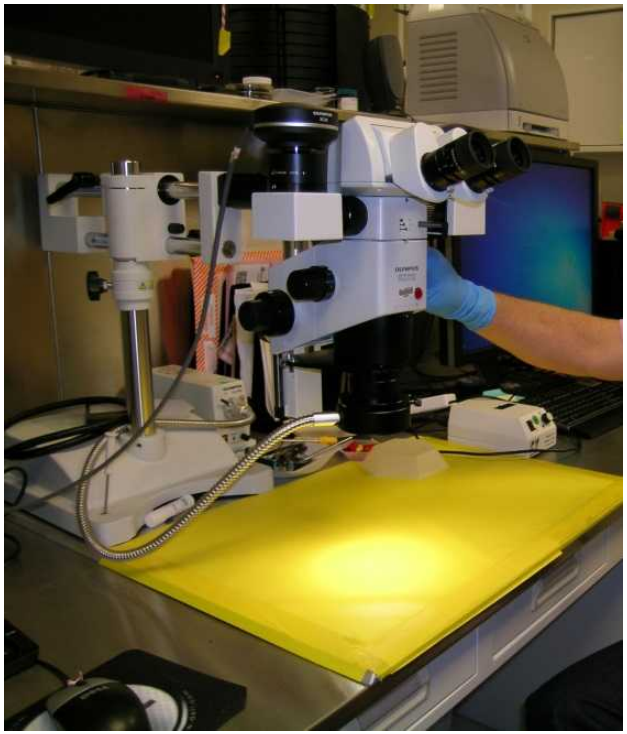
Hood Dremel Lathe used to cut through the frame.



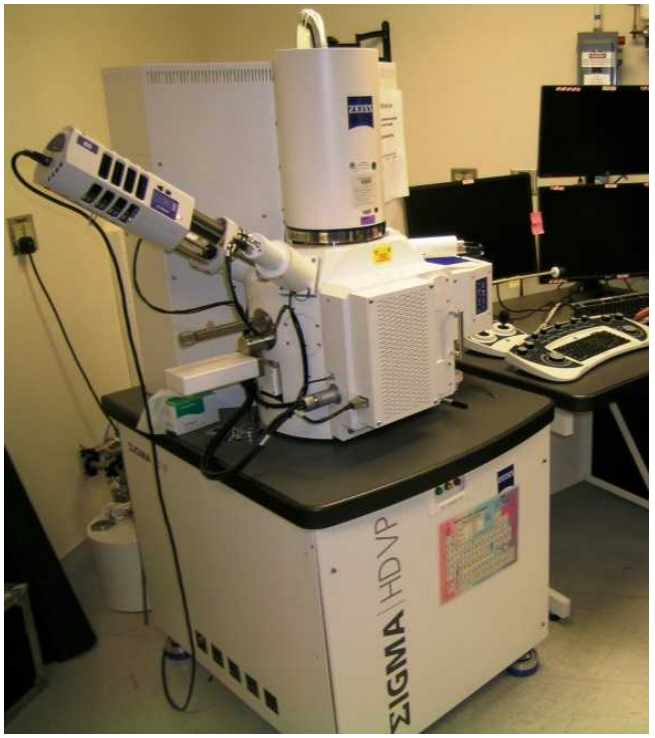
End mill lathe is used cut through the source weld.

Disassembled tubes (rad) are examined
- To ensure products meet specification
- For troubleshooting when tube anomalies are observed in testing

# Forensics and Materials Science



Optical Microscopy
- Magnifications of 3.5 to 1000x
- Ultraviolet light for Dye Penetrant

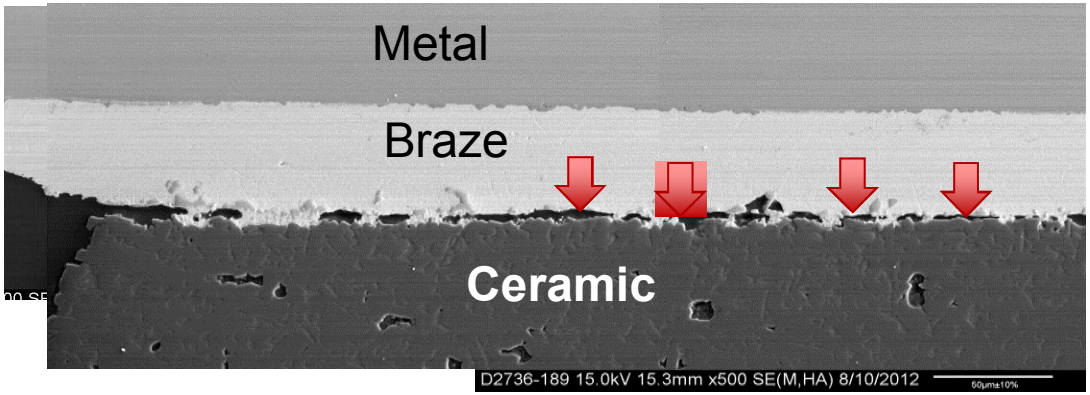



Scanning Electron Microscope (SEM)
- Variable pressure for insulating materials
- Magnifications up to 200,000x

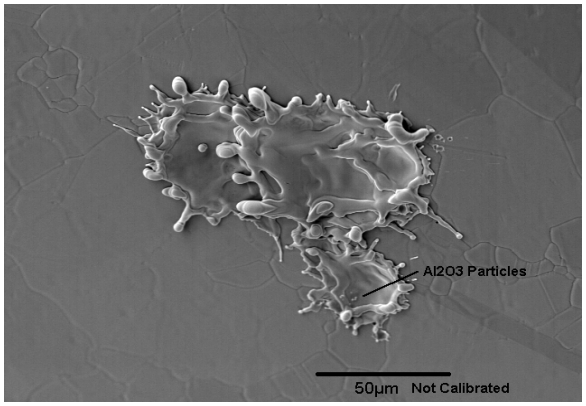
Rad-capable tools used for product inspections, problem solving and knowledge gap work

# Examples: Troubleshooting

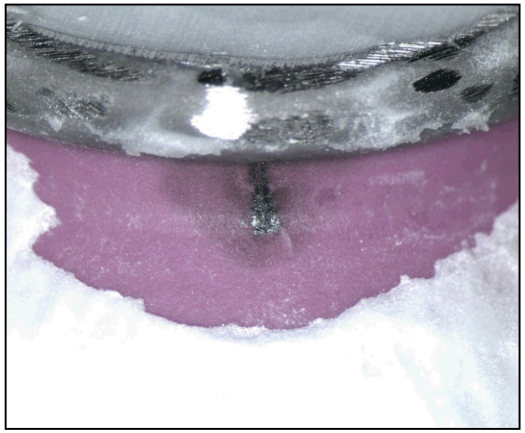
## Leak path identified using dye penetrant




Potential leak path




I-High Voltage Breakdown

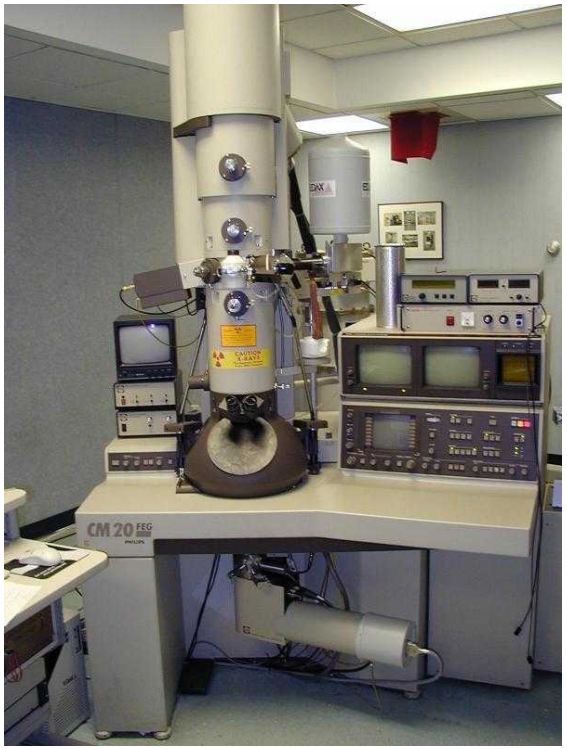


E-High Voltage Breakdown

# Materials Science Tools

X-Ray Diffractometer (XRD)

- Phase identification
- Structure refinement
- Texture analysis




Transmission Electron Microscope (TEM)

- Resolution down to 0.3 nm
- Maximum magnification 750,000X
- Capable of chemical analysis on tritiated materials

# Phase Identification

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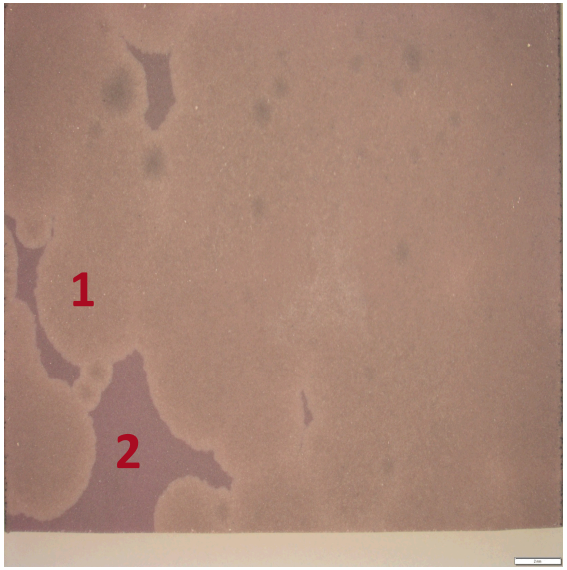
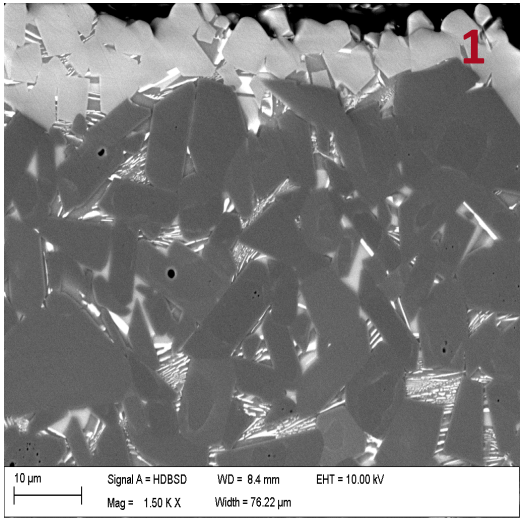
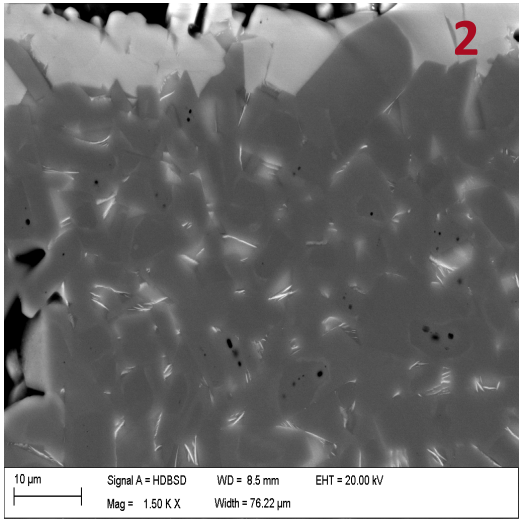
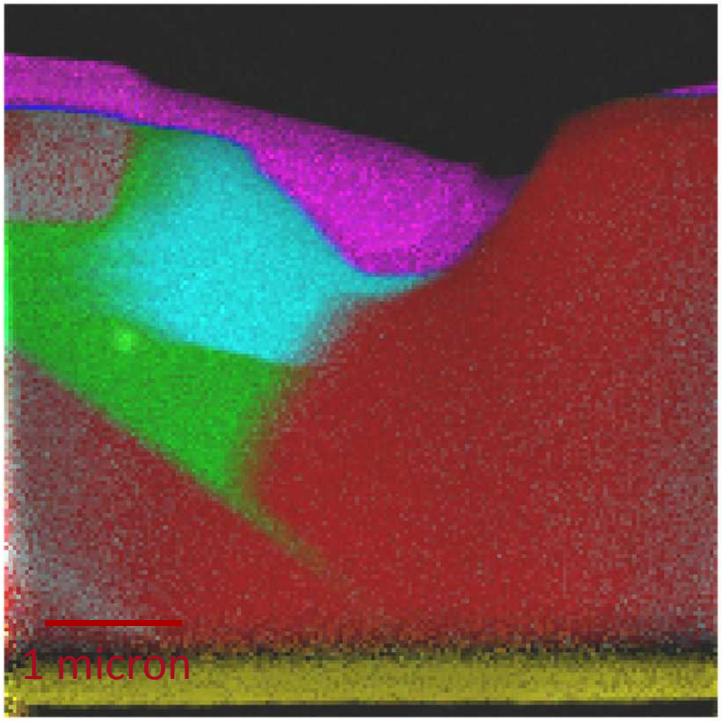
**Optical View**




**SEM View**



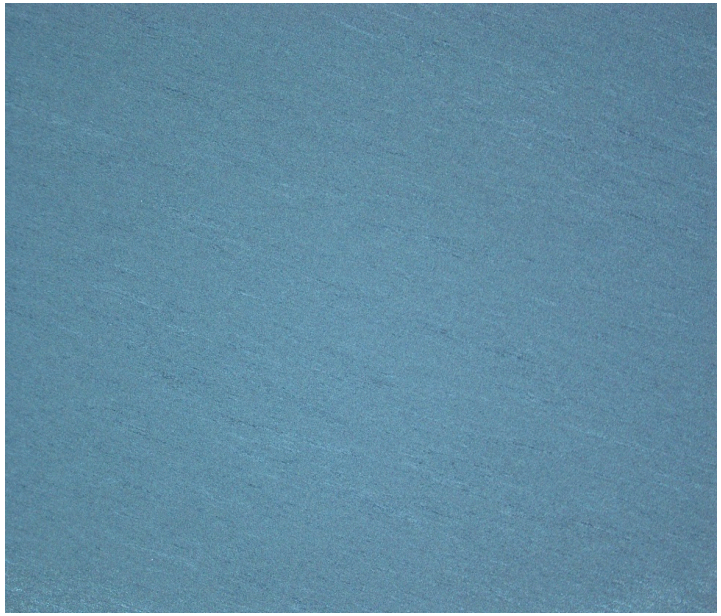

**TEM View**

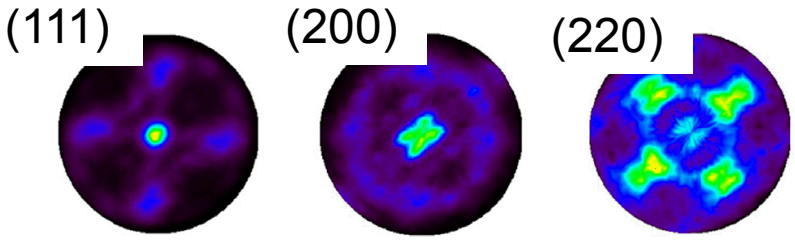
Multi-scale techniques used to identify relevant physical phenomenon

Concentration of Ti-rich Precipitates is different

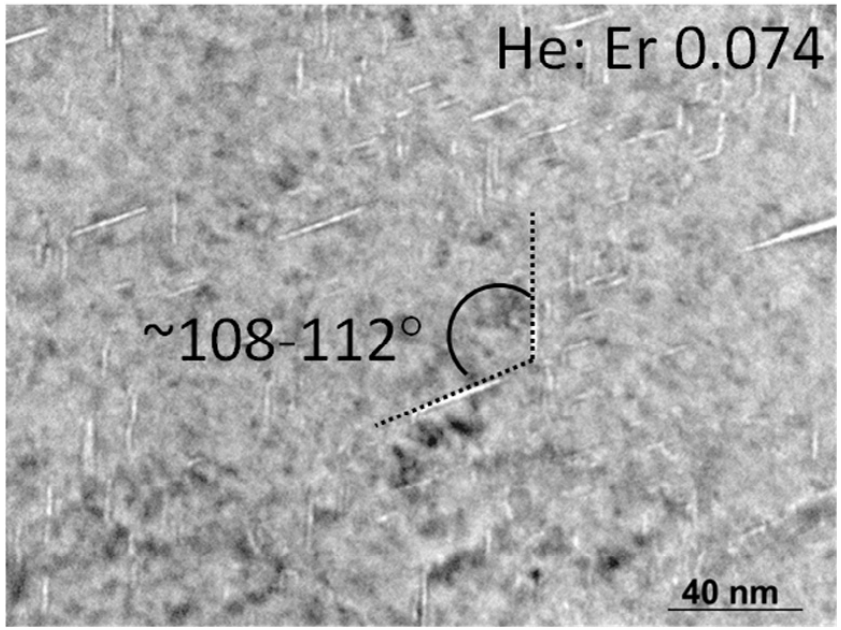
# Aging of Metal tritides

## $ErT_2$ loaded film






Strong (111) texture with secondary (200) texture




Platelet helium bubbles which are aligned along the [111] crystallographic axis.

High resolution TEM imagining necessary to understand bubble evolution and lifetime of materials

# Surface Analysis Capabilities

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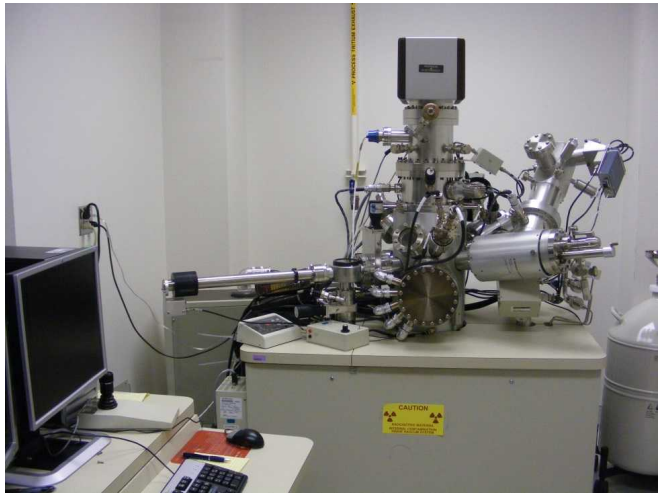
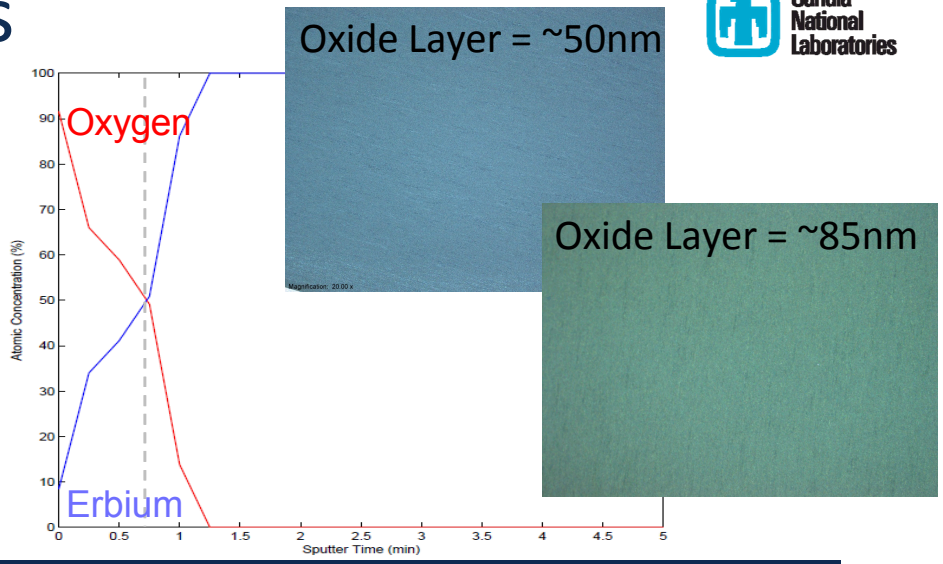
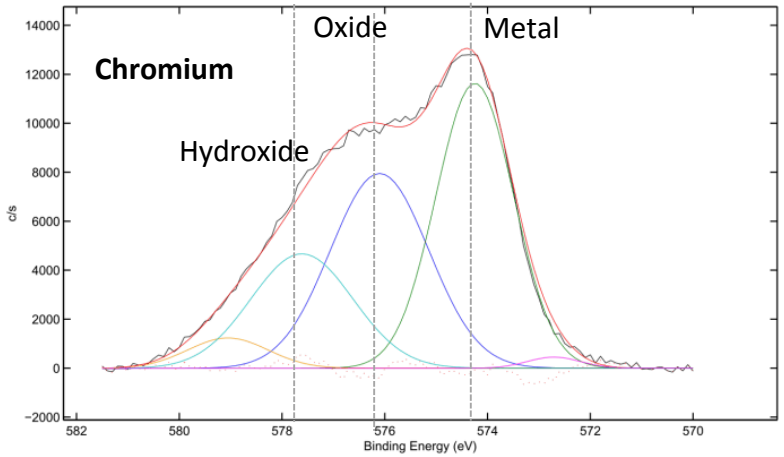
## Auger Electron Spectrometer






Rad-capable tools used for production inspections, design and knowledge gaps

## X-ray Photoelectron Spectrometer


Passivation studies on surfaces of tritium gas bottles

# Quantitative and Trace Metal Analysis

**Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP–OES)**

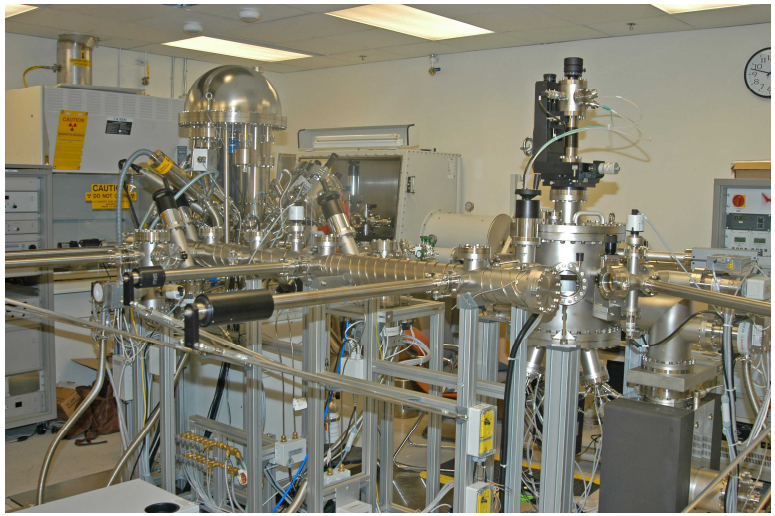
- Utilized to identify and quantify elemental constituents at the ppm level (parts per million, mg/L).

Erbium (targets) and scandium (source witnesses) metal analyses for T/D load ratio determination

**Inductively Coupled Plasma – Mass Spectroscopy (ICP – MS)**

•Simultaneous multi-element analysis of most metals at ultra-trace ppb levels (**parts per billion, µg/L**) in aqueous solutions.

# Film Deposition, Thermal desorption

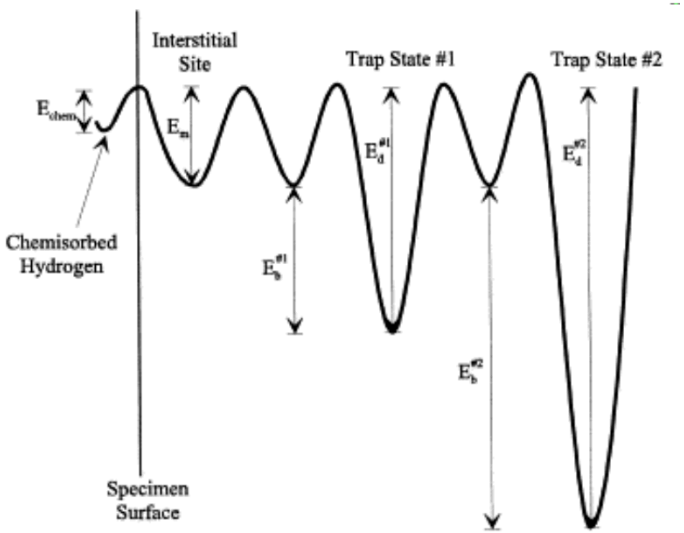


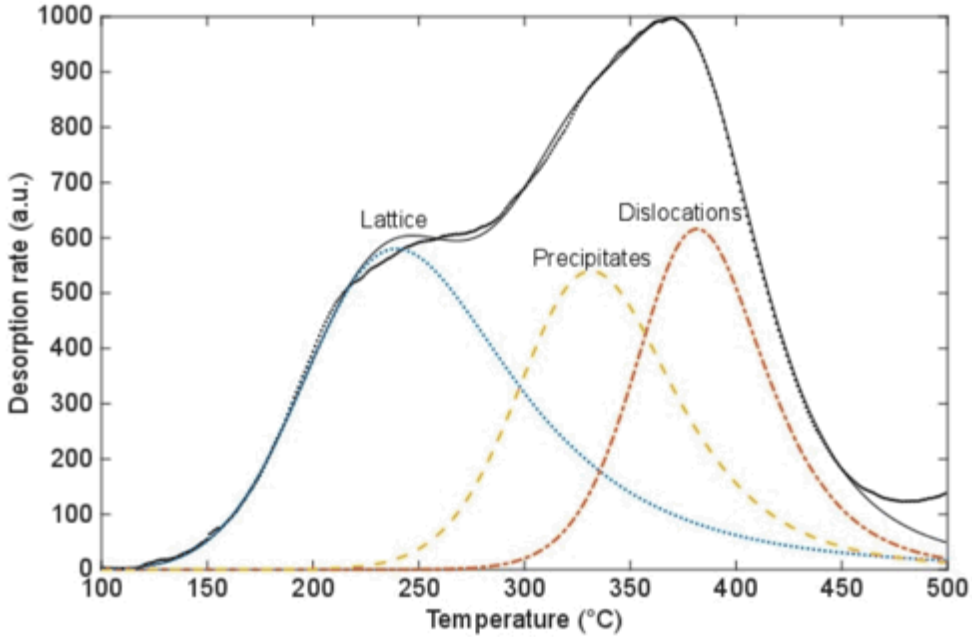
- **Dual Chamber Thin Film Deposition**
  - 2 electron-beam evaporation cells
  - Can deposit 1-3 materials per run
  - Experimental films can be deposited

- **Thermal Desorption System**
  - Angular reflection time-of-flight mass spectrometer
  - Mass resolution ~ 212
  - ~750C upper temperature limit

Tools used to develop new materials, and study evolution of tritium from these materials

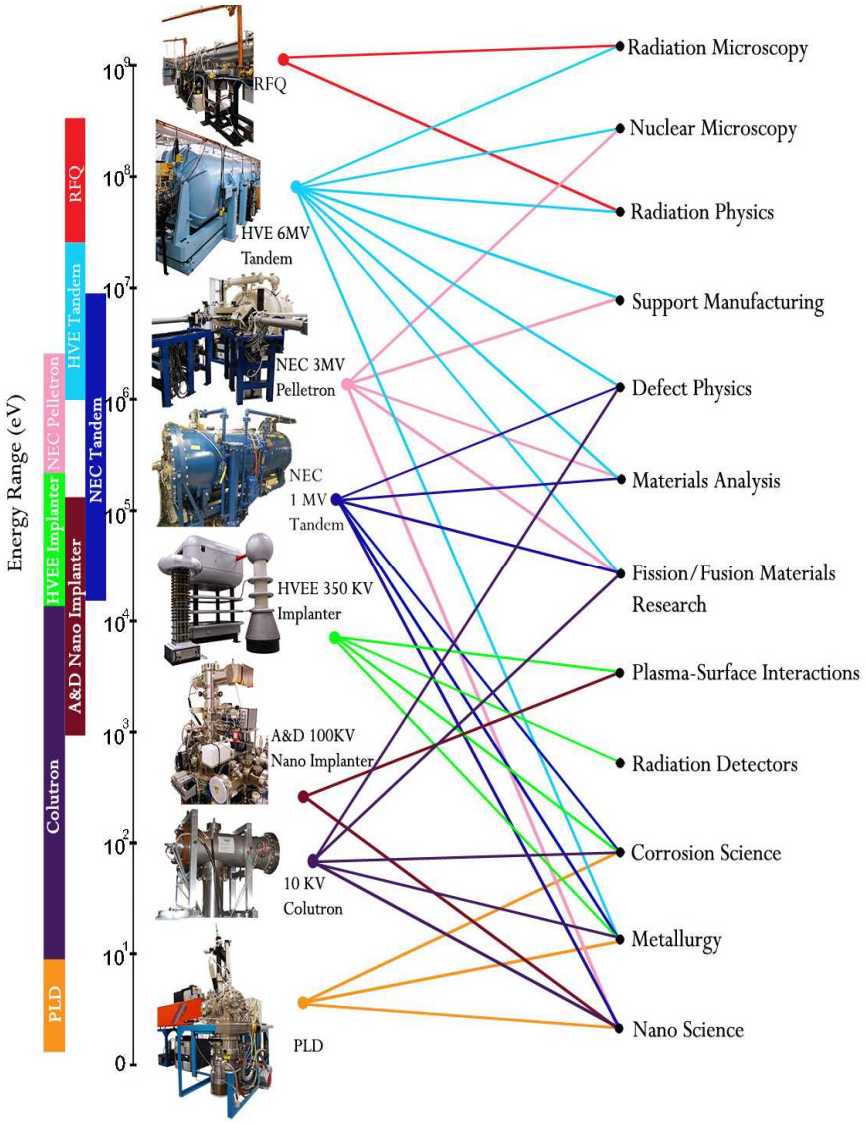
# Film Deposition, Thermal desorption




Tools used to develop new materials, and study evolution of tritium from these materials

# Sandia's Ion Beam Laboratory





Tools at IBL used to study helium evolution, and H-isotope profiling in materials

# END