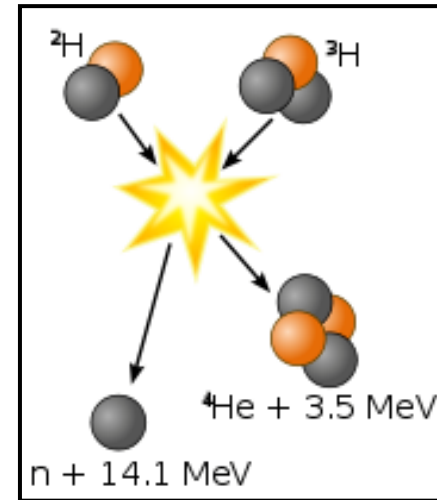
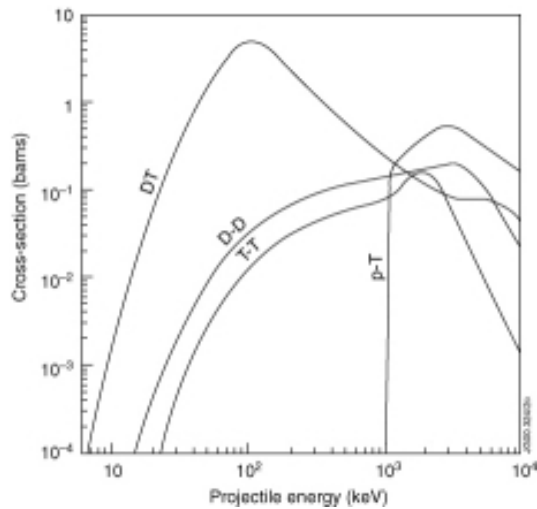


*Exceptional service in the national interest*



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

Analytical and Materials Sciences, Org. 07576

Raj Tandon @ Component Sc., Engg., and Production Center

Sandia National Lab, NM



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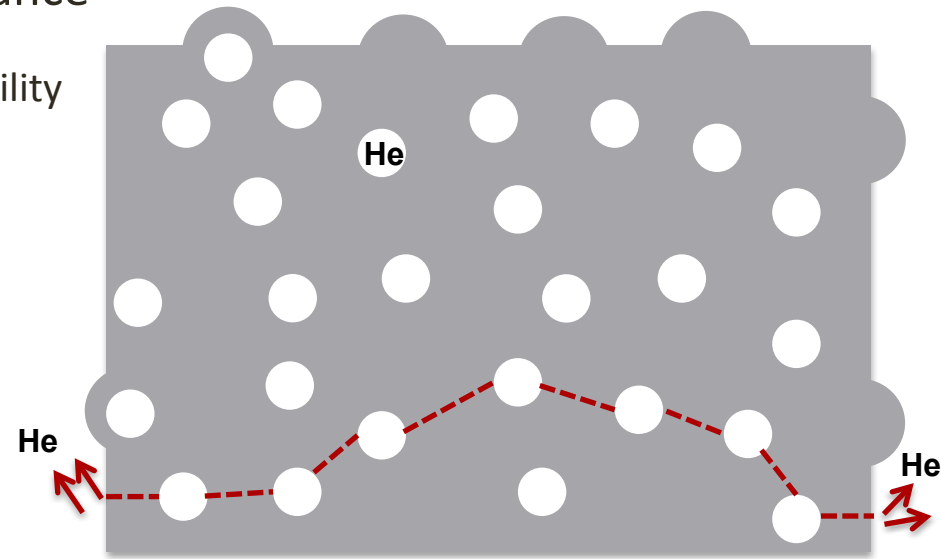
# Outline

- Tritium materials interactions, and tube operations
- Tritium Safety
- Tritium Loading
- Tritium Forensics Tools and examples
- Ion-Beam and CINT facilities

# Tritium-Material Interactions

- Tritium dissolves in material lattice causing metal embrittlement
- Tritium decays to  $^3\text{He}$ 
  - Insoluble in most materials
  - Forms high pressure nanometer sized bubbles, and swelling
- Detrimental Impact on Material Performance
  - Can cause fracture and impact HDT storage-ability

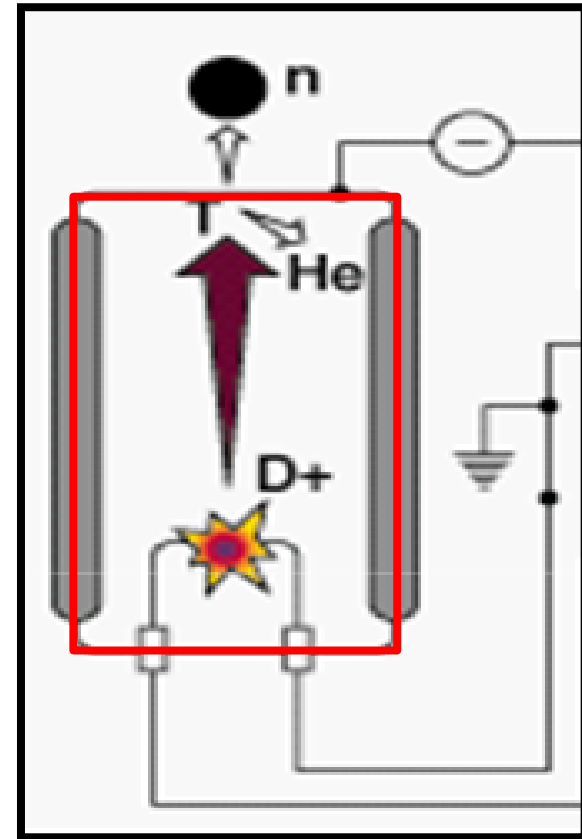
Pressurized bubbles can cause materials to fracture and **release** helium



Schematic of Helium Bubbles Causing Cracks

# Neutron Generators

- Neutron generators are compact linear accelerators, and release neutrons by fusion of hydrogen isotopes.
- Deuterium, tritium, or a mixture is ionized and accelerated under high field
- Energetic ions strike a metal hydride target which also contains deuterium, tritium or a mixture, and fusion reactions release neutrons



**Vacuum Enclosure**

# Sandia NG Tritium Capabilities

- All tritium operations are performed inside a “tritium envelope” with dedicated single-pass & monitored ventilation in a radiological facility
- Equipment can be connected to both
  - Tritium Capture System
  - Tritium Exhaust (smoke stack release)
- Inside tritium envelope
  - Tritium Loaders (U-bed to Films)
  - Mass Spectrometers (analysis of produced parts, then to TCS)
  - Other Characterization Equipment



# Tritium Capture System-Tritium Safety



## 80 ft<sup>3</sup> accumulation tank

- Sub-atmospheric pressure

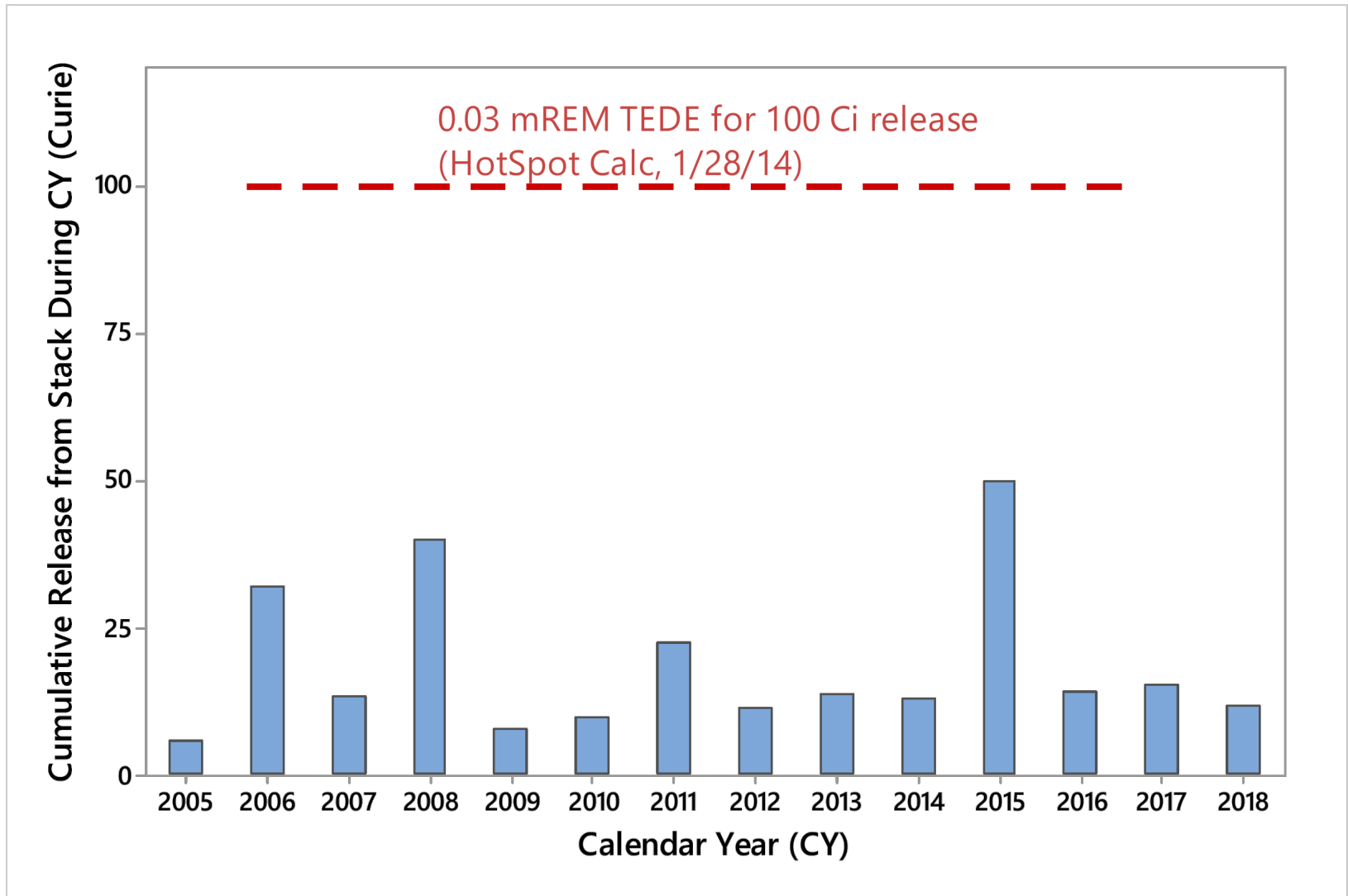
## Valve control panel, sieve beds, catalytic burners

- Effluent tritium verified, then sent to stack

## Calorimeter for sieve bed analysis

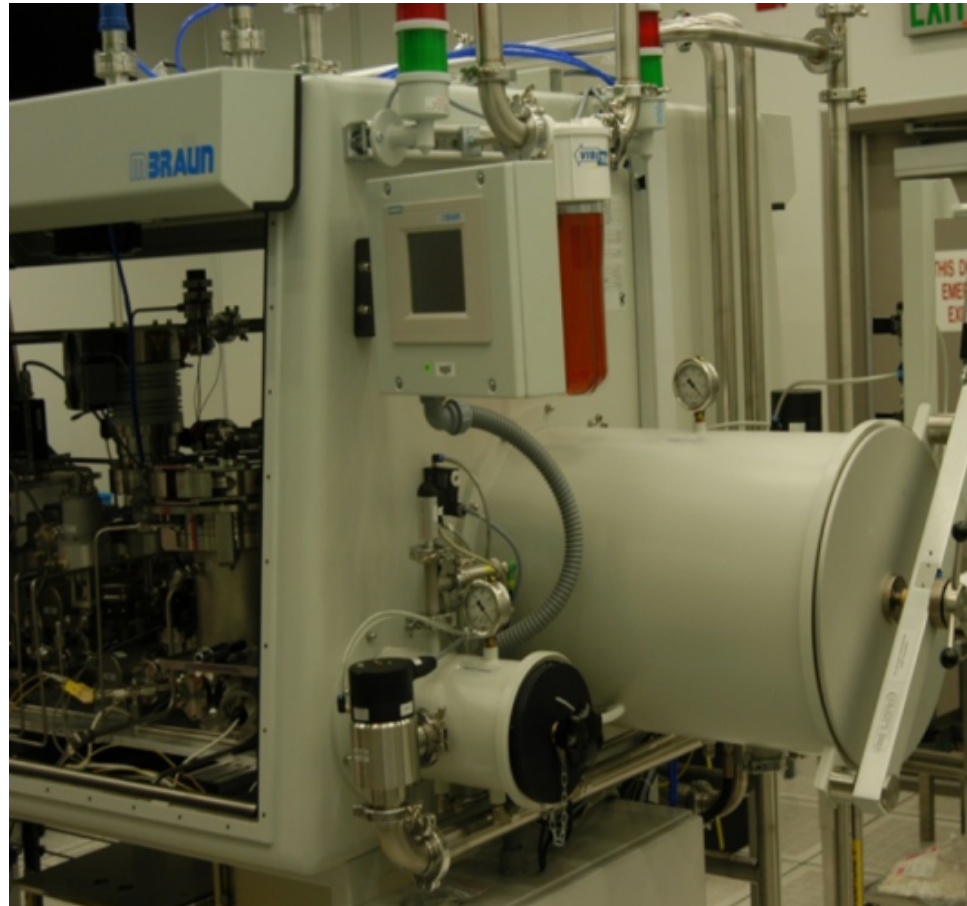
Ensures that effluent gasses have minimal tritium content-tritium safety

# Tritium Releases to Environment



# Tritium Gas Loading

**Tritium Loader – gas manifold built in a glove box for loading metal films with H-D  
-T gases**

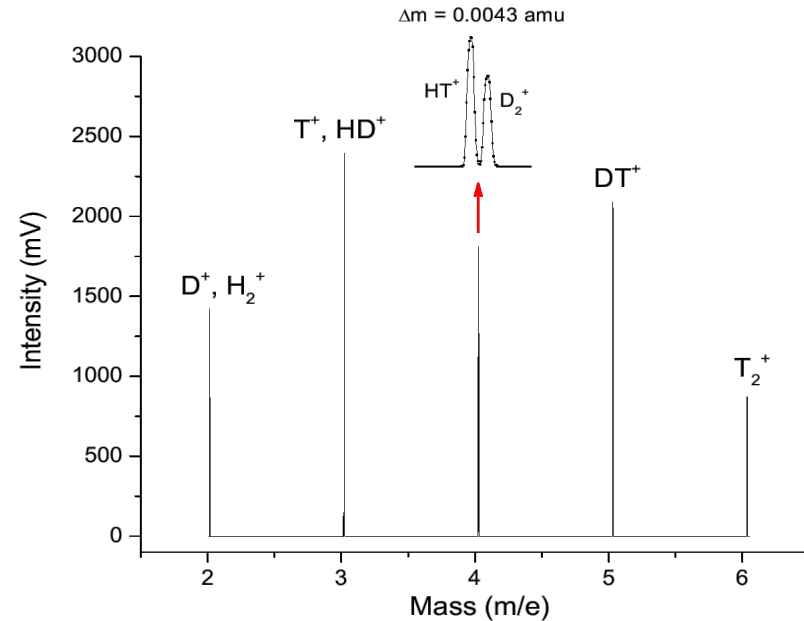
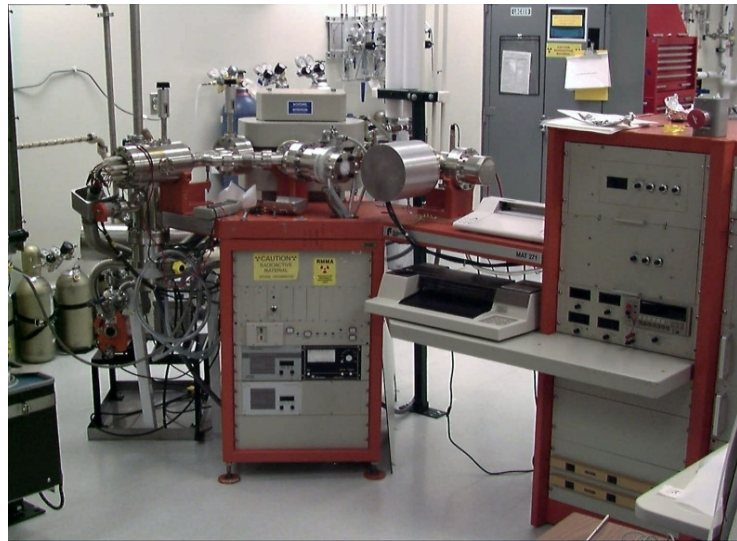


# Gas Analysis Capabilities

Used for thin film analysis



**Mass Spectrometer:** Optimized for analysis of the hydrogen isotopes



## Finnigan MAT 271 Mass Spectrometers

- Measurement uncertainty  $< \pm 2\%$  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

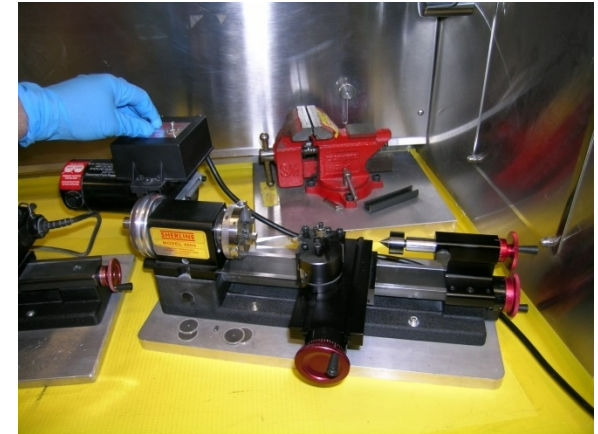
# Tube Disassembly and Inspection



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



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

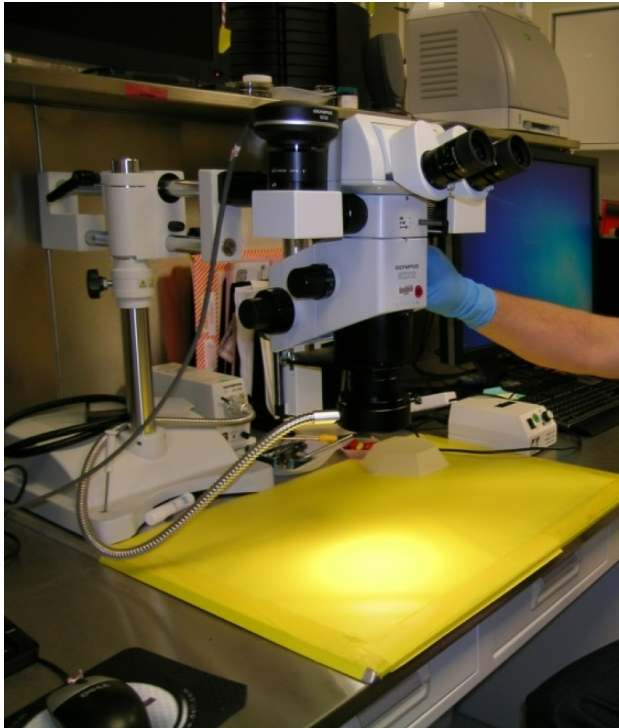


End mill lathe is used cut through the source weld.

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 R&D

# Materials Science Tools



X-Ray Diffractometer (XRD)

- Phase identification
- Structure refinement
- Texture analysis

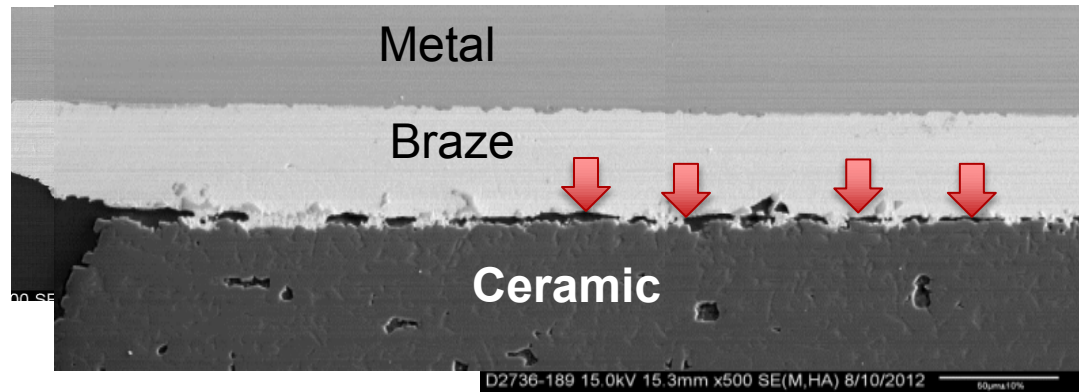


Dual Beam FIB

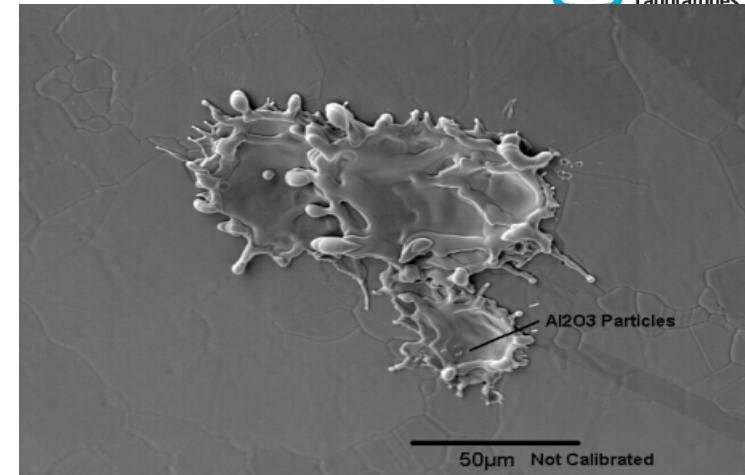
- Rad STEM sample prep

# Troubleshooting

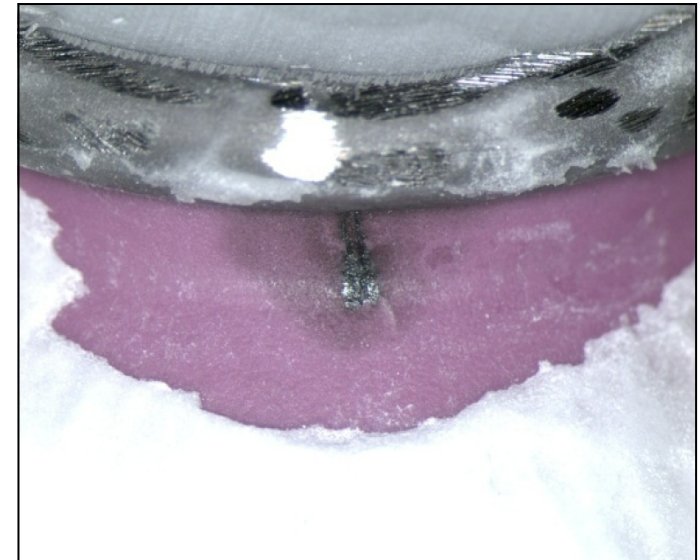
Leak path identified using dye penetrant



Potential leak path



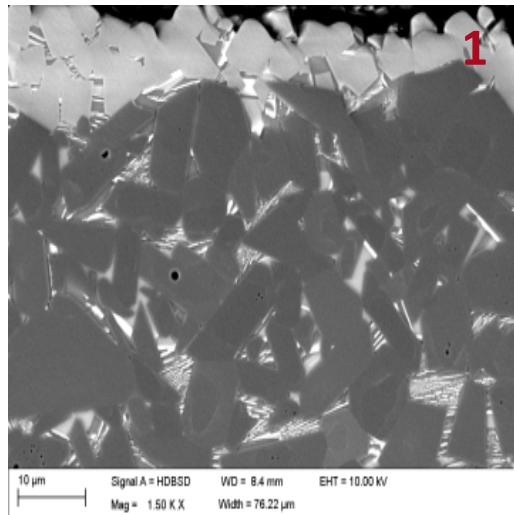
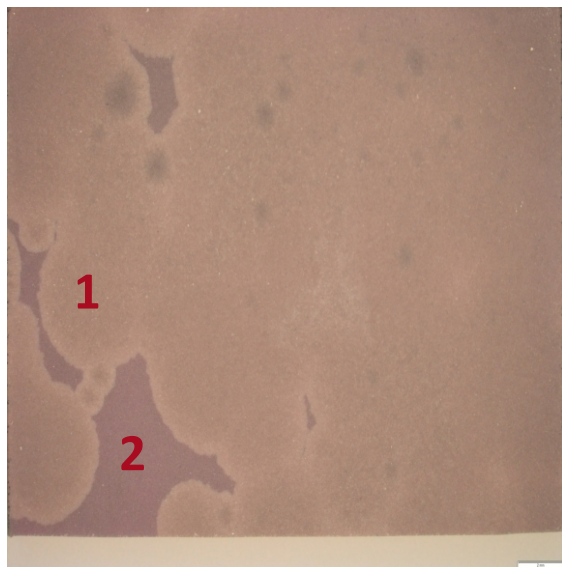
I-High Voltage Breakdown



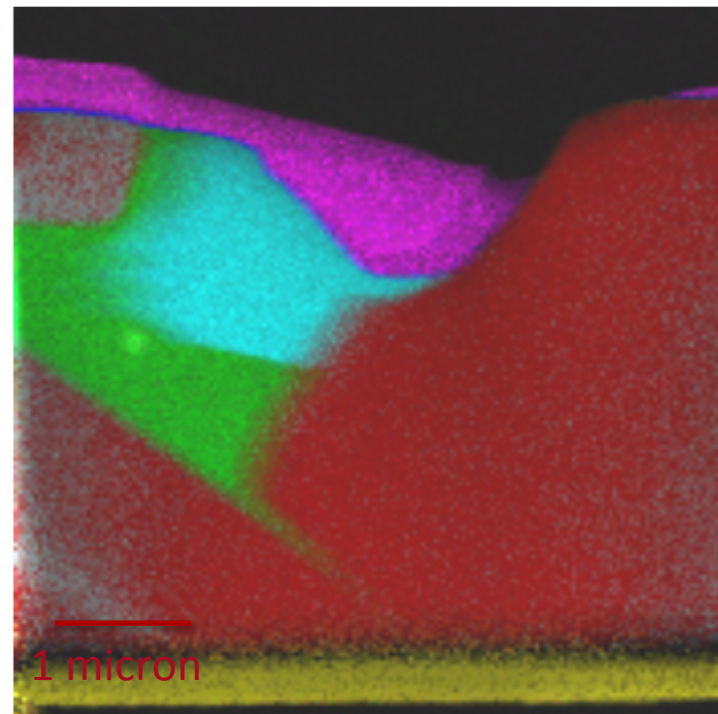
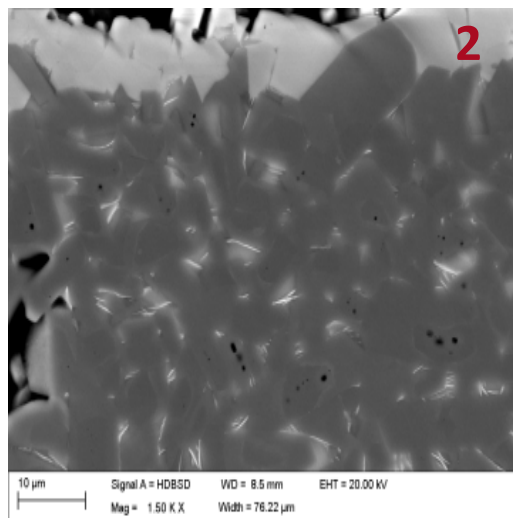
E-High Voltage Breakdown

# Phase Identification

## Optical View



## SEM View



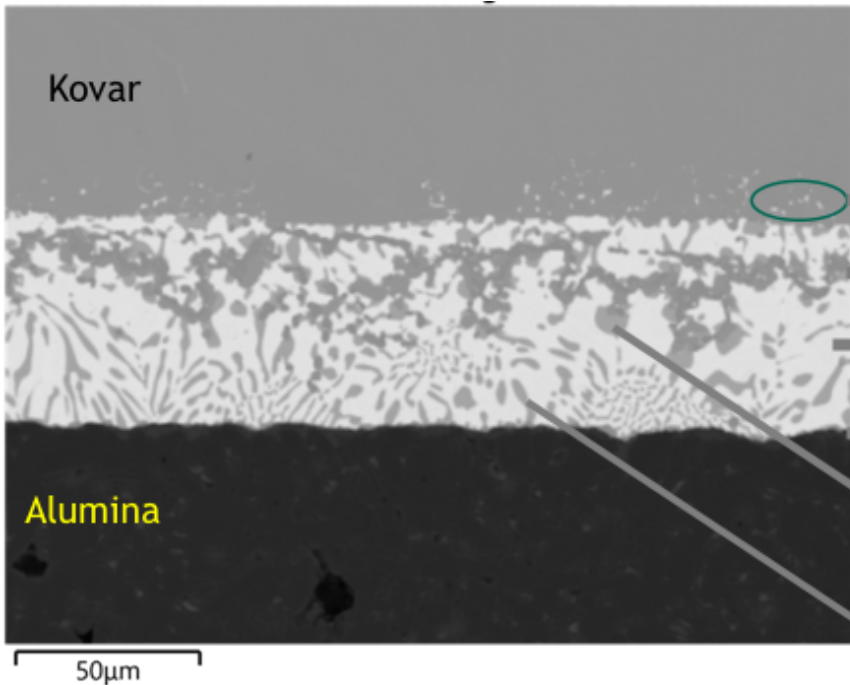
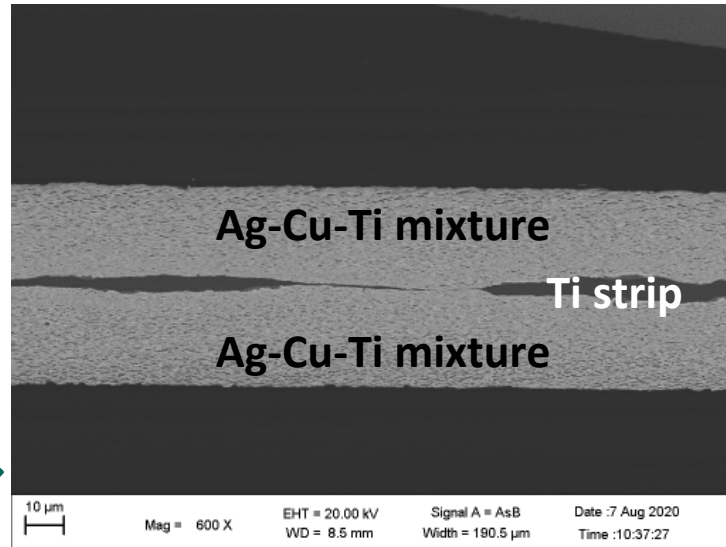
## TEM View

Multi-scale techniques used to identify relevant physical phenomenon

Concentration of Ti-rich precipitates is different

# Microstructure Development

Starting Braze Material

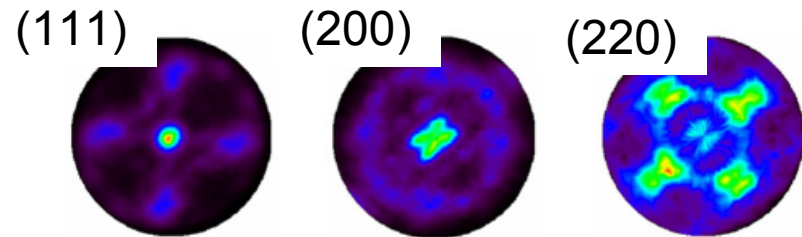
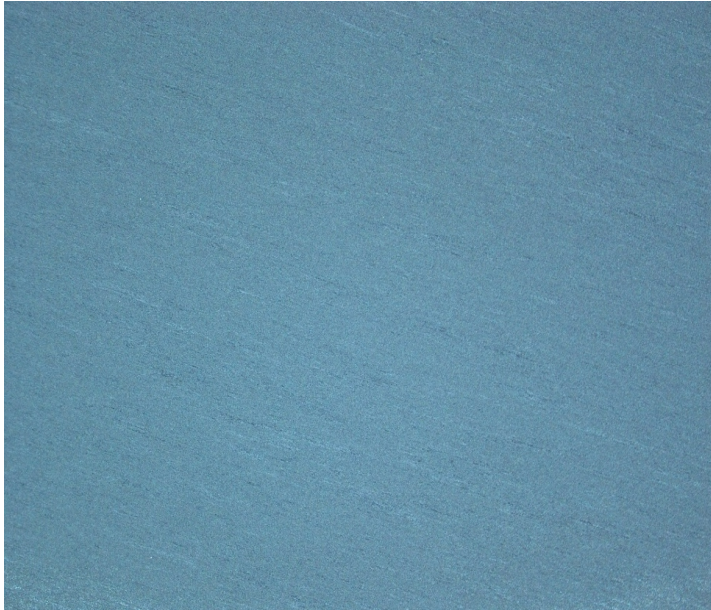


- Silver penetration into Kovar
- Lacy phase  $(\text{FeNiCo})_{2-3}\text{Ti}$
- Silver in braze joint
- Alumina- braze interfacial layer  $(\text{FeNiCo})_2\text{Ti}$
- Cu-Ti-Ag spheres
- Cu-Ag-Ti whiskers

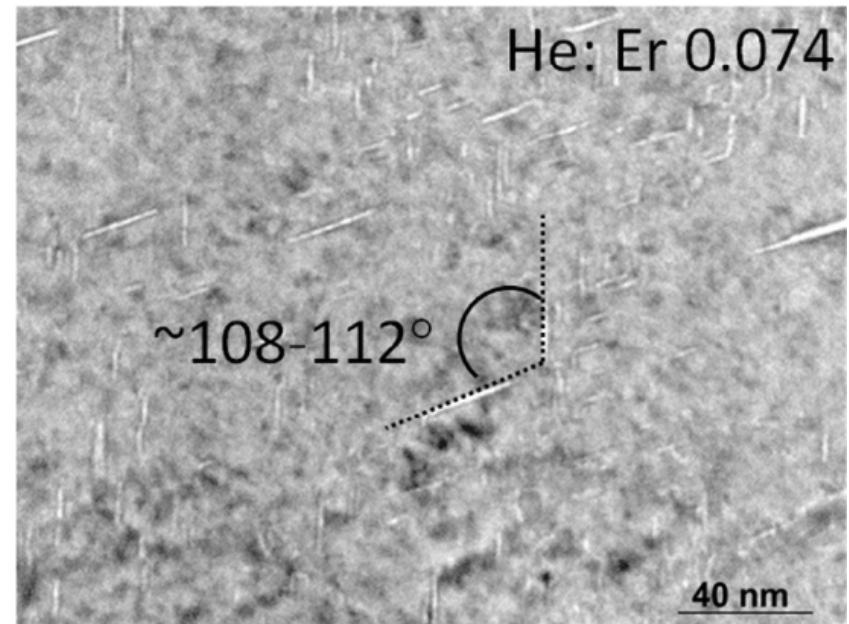
Multiple techniques used to identify relevant physical phenomenon

# Aging of Metal tritides

## ErT<sub>2</sub> loaded film



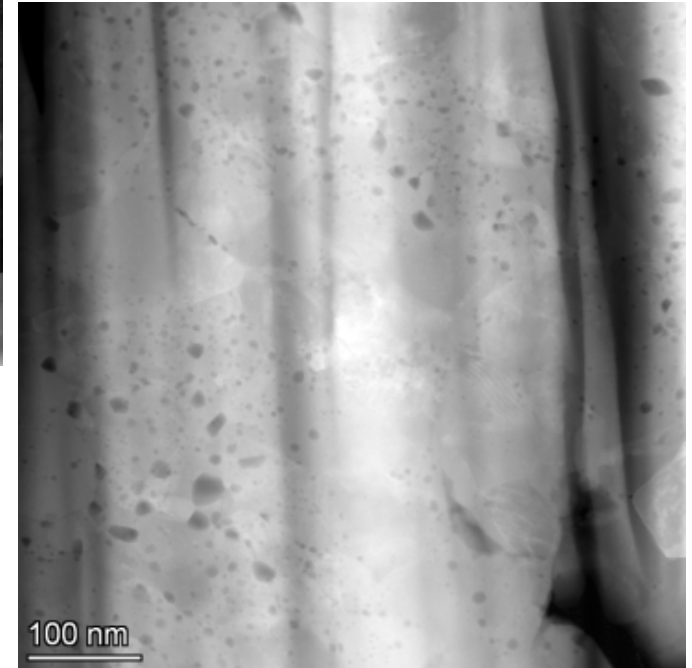
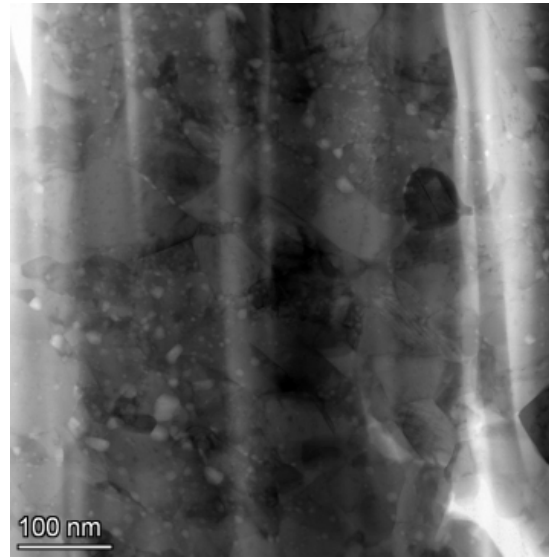
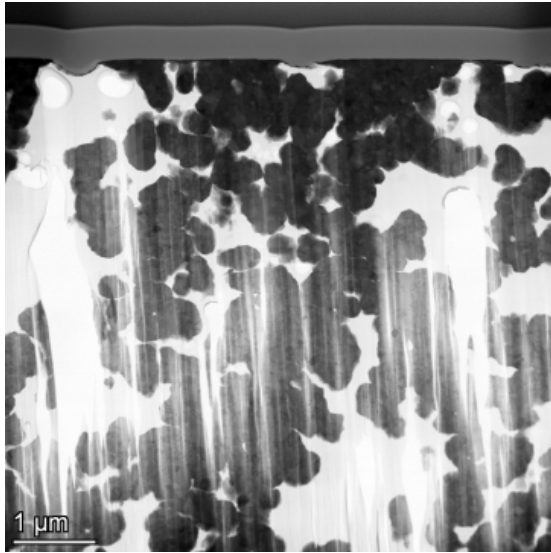
Strong (111) texture with secondary (200) texture



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

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

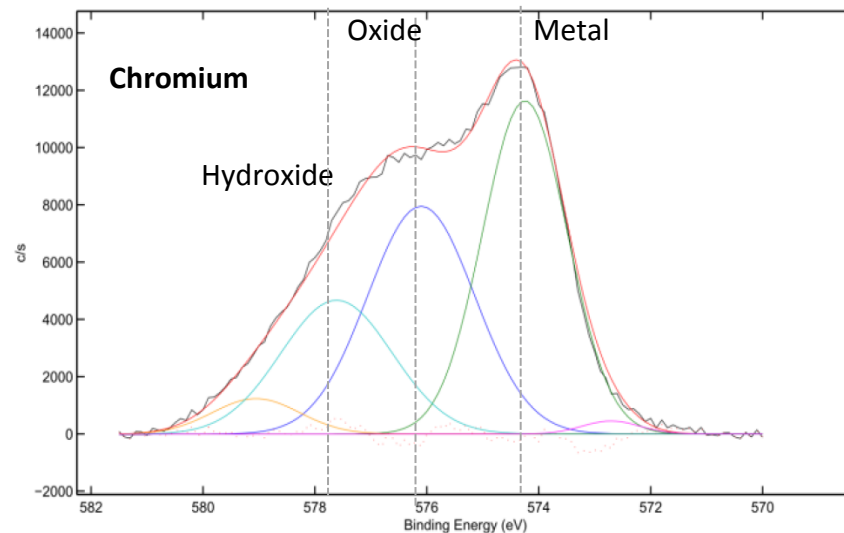
# Aging of metal tritide powder



High resolution TEM imaging  
necessary to understand bubble  
evolution in materials

# Surface Analysis Capabilities

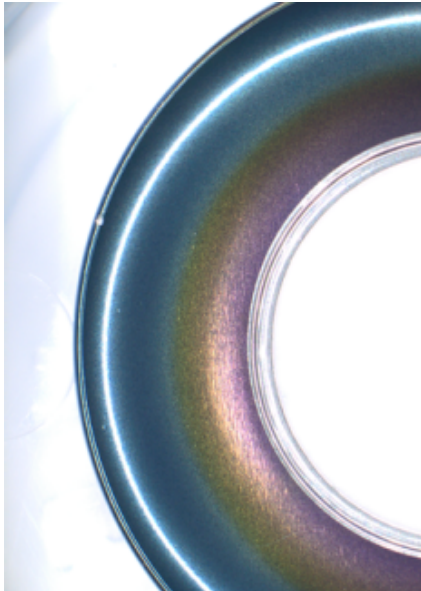
## X-ray Photoelectron Spectrometer



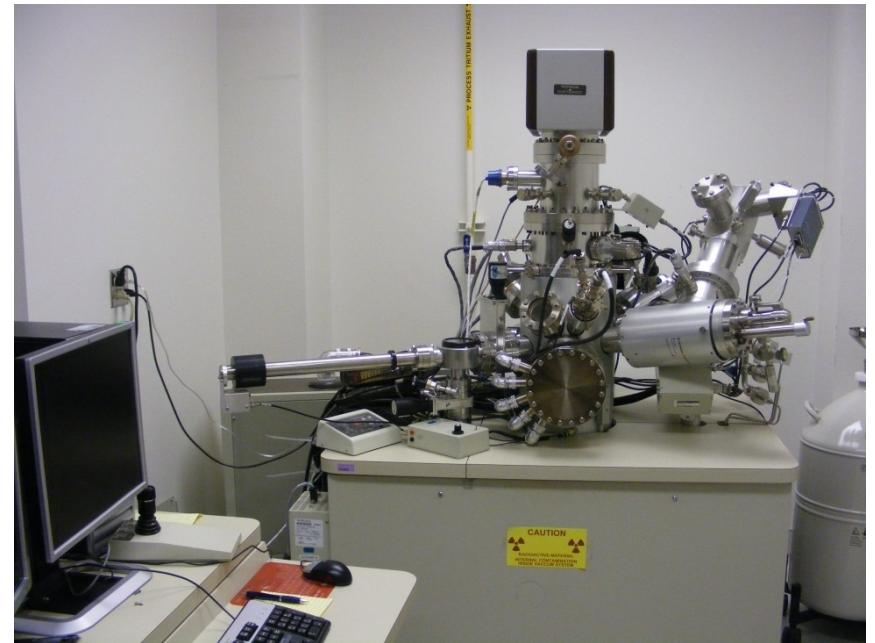
Passivation studies on surfaces of tritium gas bottles

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

# Variable color film after hydrogen loading

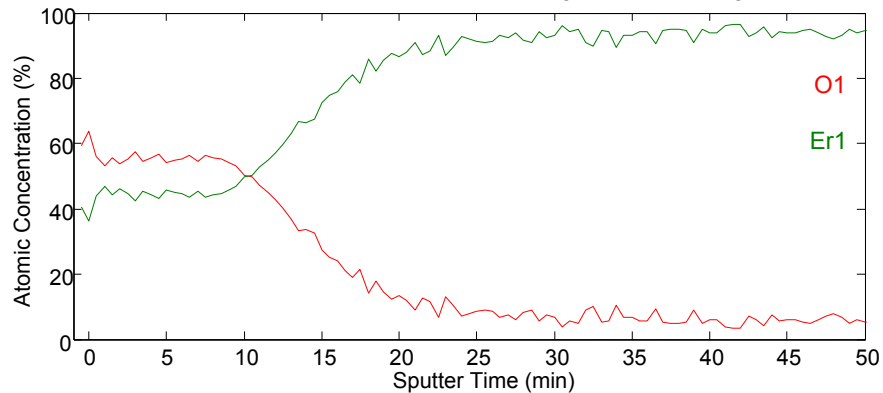


## Auger Electron Spectrometer

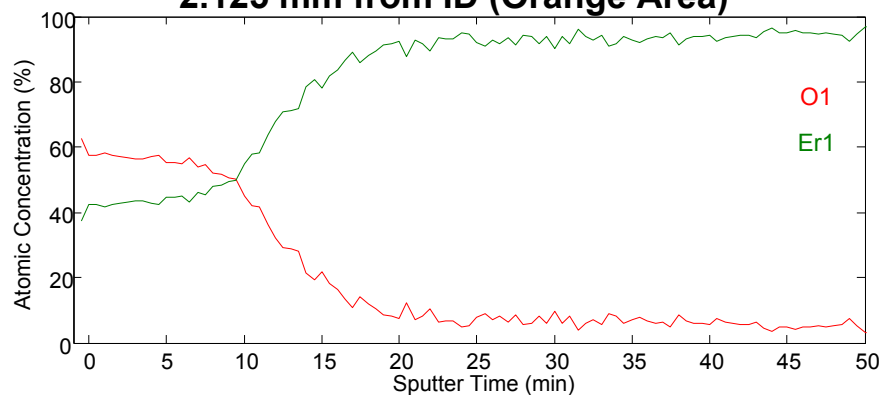


# Auger Profile Results

### 1 mm from ID (Pink Area)



### 2.125 mm from ID (Orange Area)

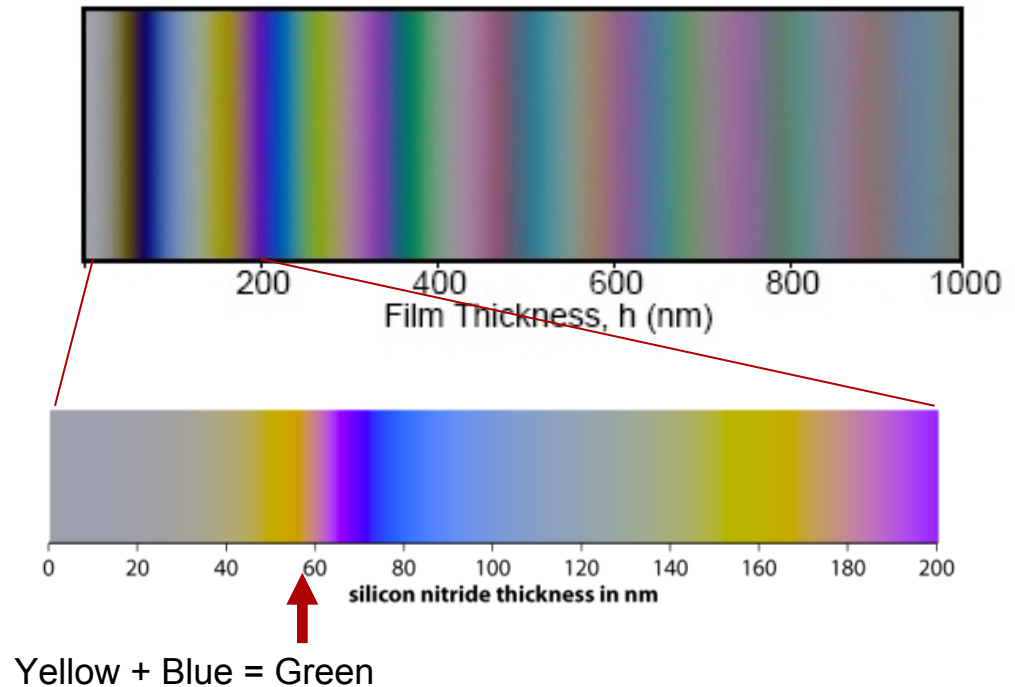
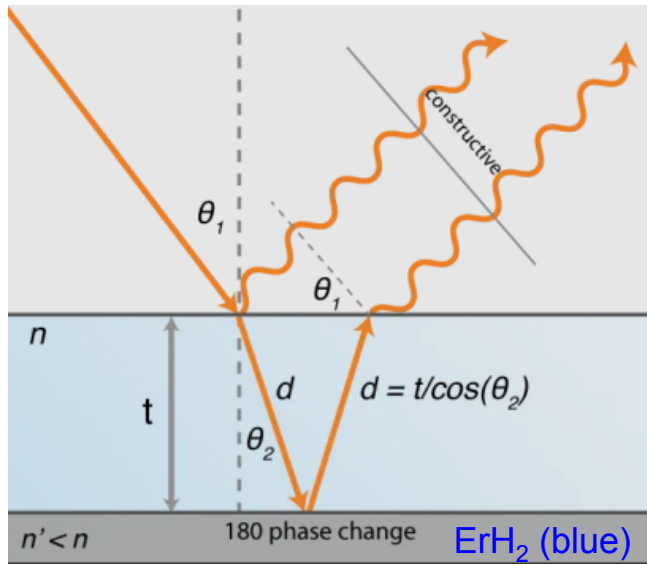


Location from edge of metal ID rim	Thickness from profile	Optical Color
1 mm	680.9 Å	Pink
2.125 mm	549.4 Å	Orange

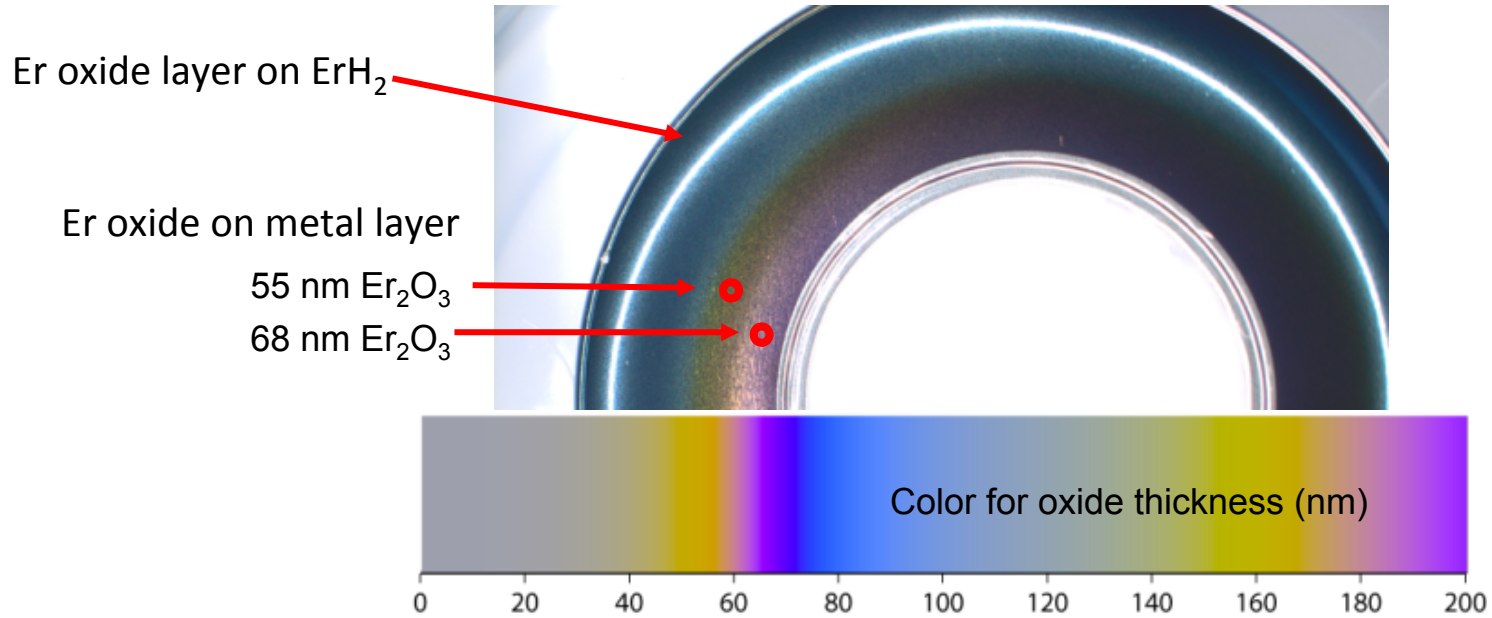
Sputter rate of 45.8 Å / min was used to calculate film thickness.  
Only Er and O signals shown

# Film Color Charts

- Oxide film thickness at multiples of  $\frac{1}{4} \lambda$  are constructive
- Observed color is combination of hydride color (blue) + oxide color (thickness dependent)



# Color clearly related to oxide thickness



OD - Thin oxide on  $\text{ErH}_2$

Detailed description: A schematic diagram showing a thin white layer on top of a blue rectangular block.

ID - Thick oxide on Er metal not hydride  
(oxide prevented loading)

Detailed description: A schematic diagram showing a thick dark grey layer on top of a light grey rectangular block.

# Quantitative and Trace Metal Analysis

## Inductively Coupled Plasma – QQQ Mass Spectrometry (ICP – MS) with Laser Ablation

- Simultaneous multi-element analysis of most metals at ultra-trace ppb levels (**parts per billion,  $\mu\text{g/L}$** ) in aqueous solutions.
- Higher sensitivity than single quad
- Mass sorter for removal of ambiguity in analysis
- Laser Ablator with 3 mm spot allows rapid, high precision analysis without needing to dissolve material
- Photo courtesy: Univ. of Adelaide Website

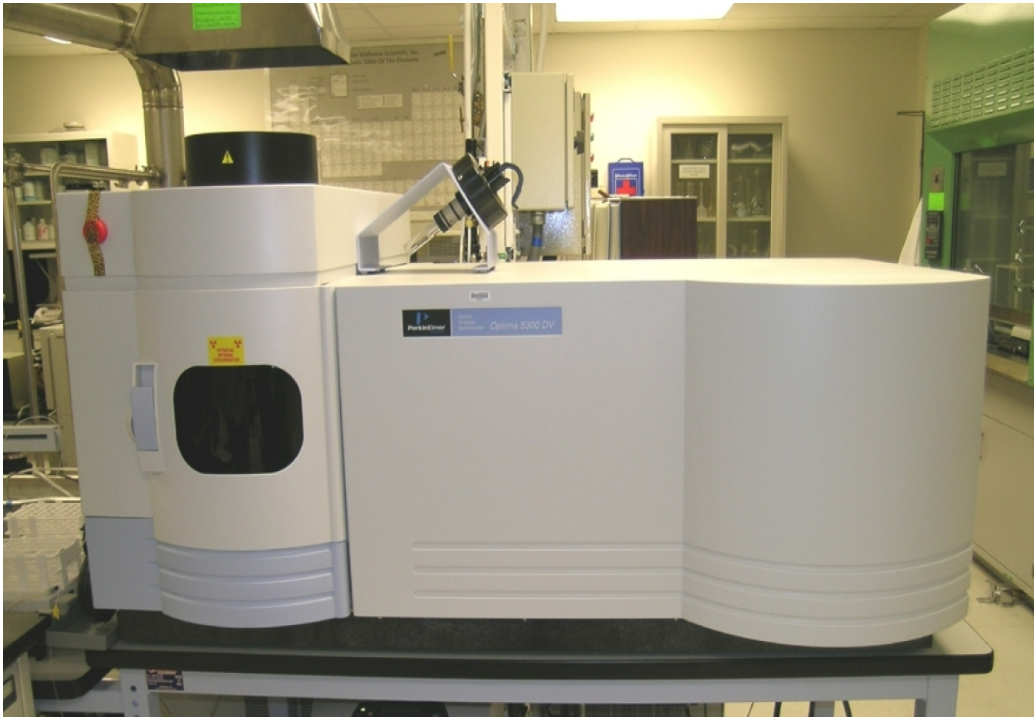


NG thin film metal analyses for gas load  
ratio determination  
Rapid and space resolved ppb level  
analysis

# Quantitative and Trace Metal Analysis

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

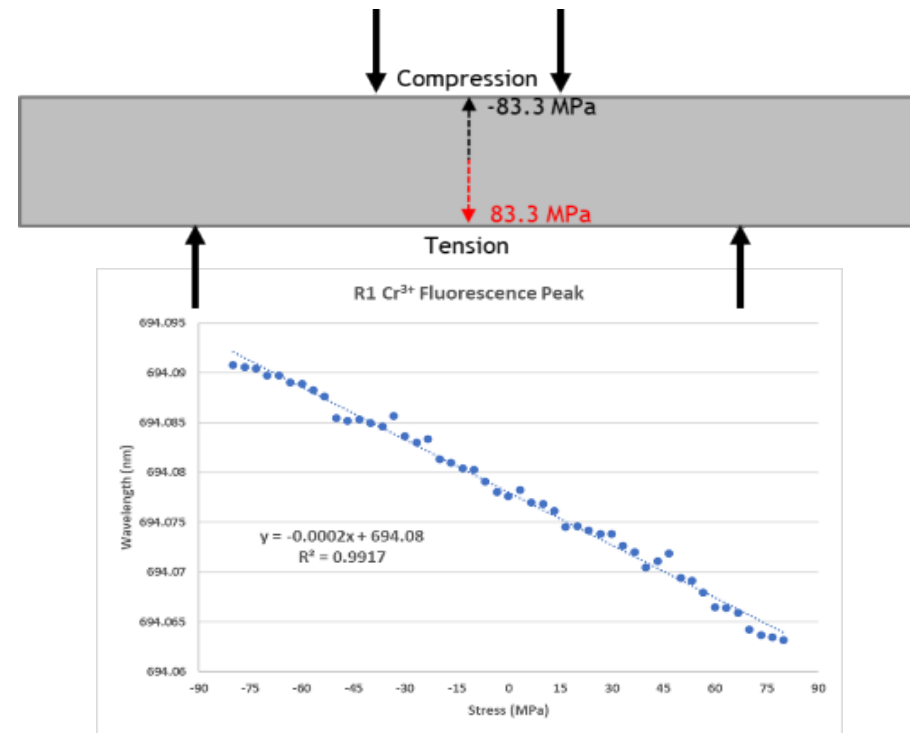
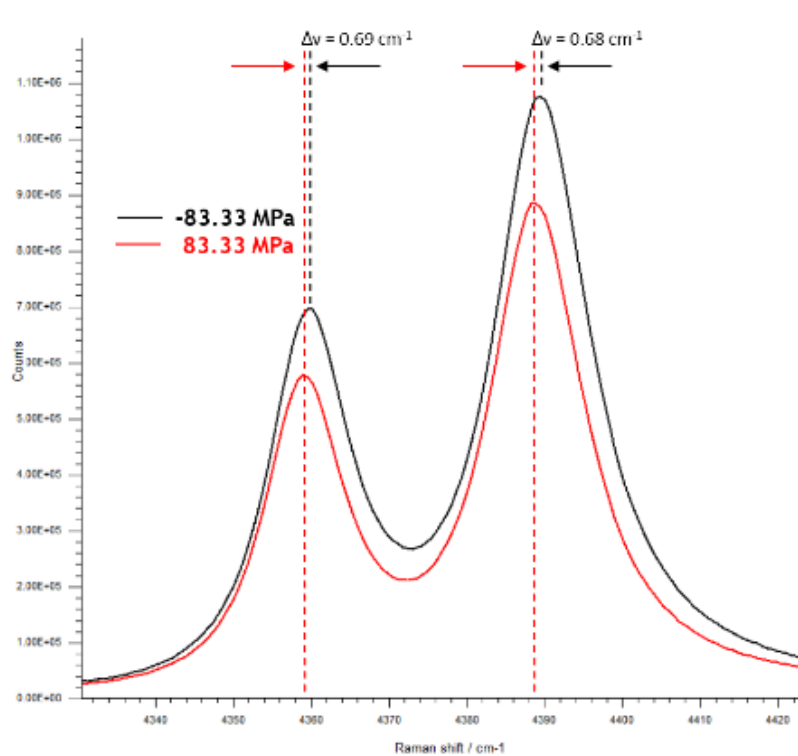
- Utilized to identify and quantify elemental constituents at the ppm level (parts per million, mg/L).



NG thin film metal analyses for gas load ratio

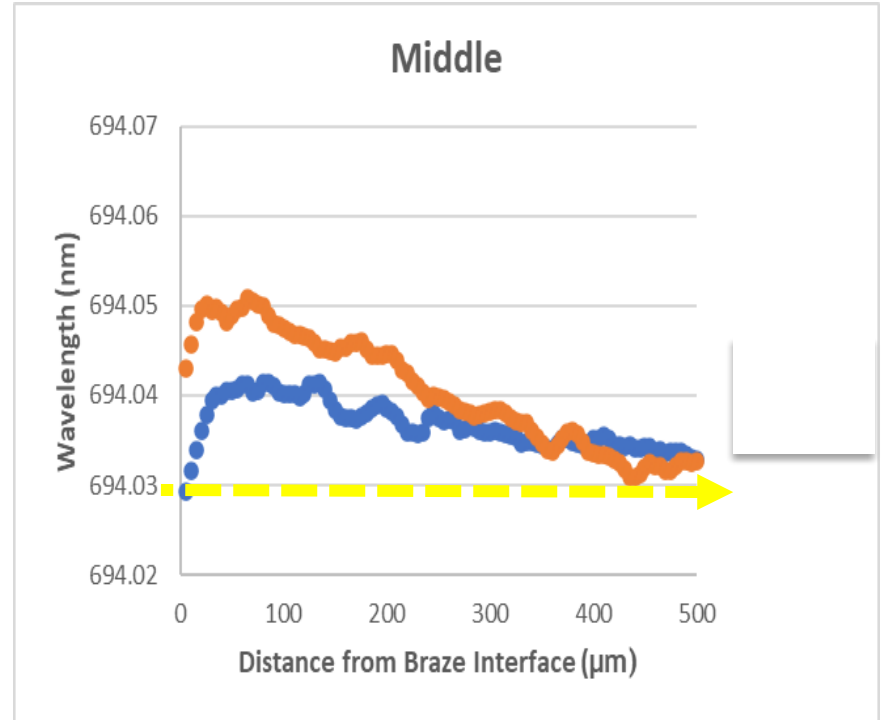
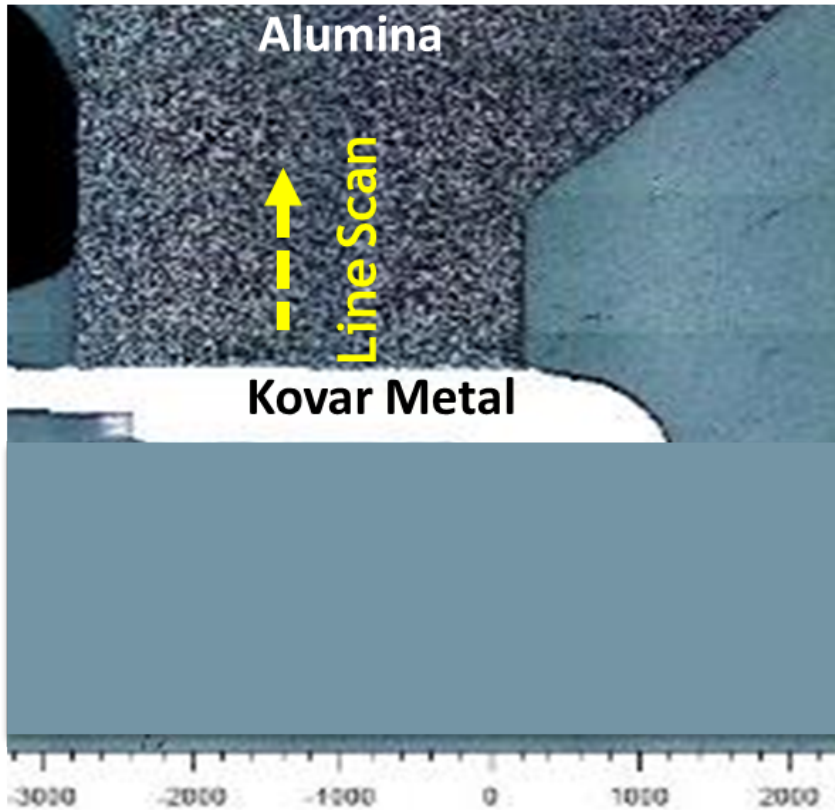
# Stress Measurements at Interface Using Spectroscopy

Cr<sup>3+</sup> impurity in alumina has photoluminescence (PL) spectrum that shifts with stress



Stress calibration

# Stress Measurements at Interface Using Spectroscopy



**Use tensor manipulation to calculate stress as a function of distance from braze interface**

# Thermal desorption

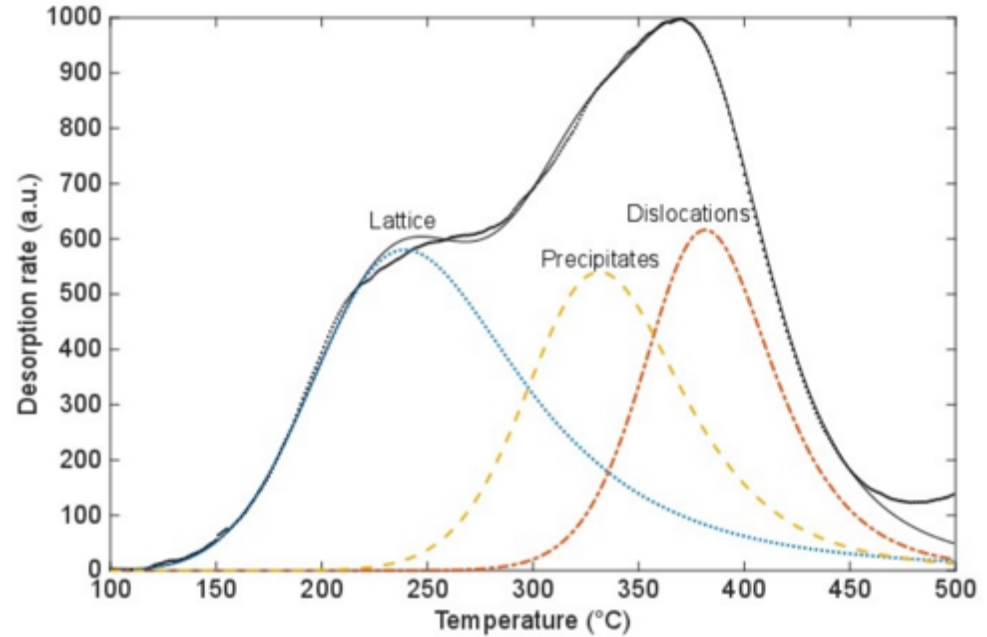
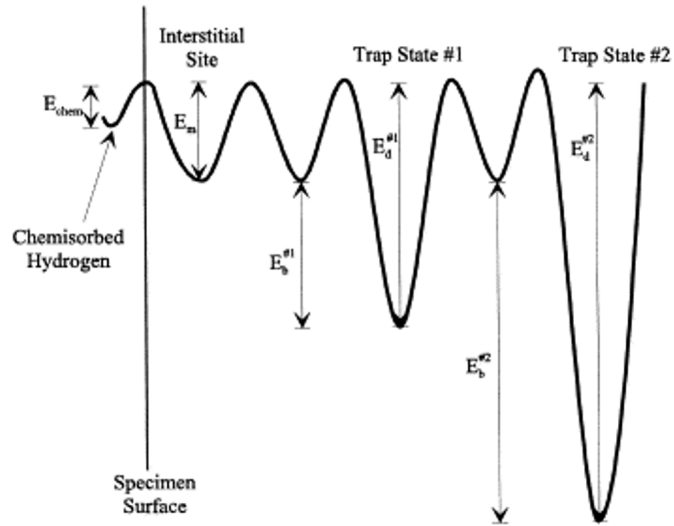


## Thermal Desorption System

- Angular reflection time-of-flight mass spectrometer
- Mass resolution  $\sim 212$
- $\sim 750\text{C}$  upper temperature limit

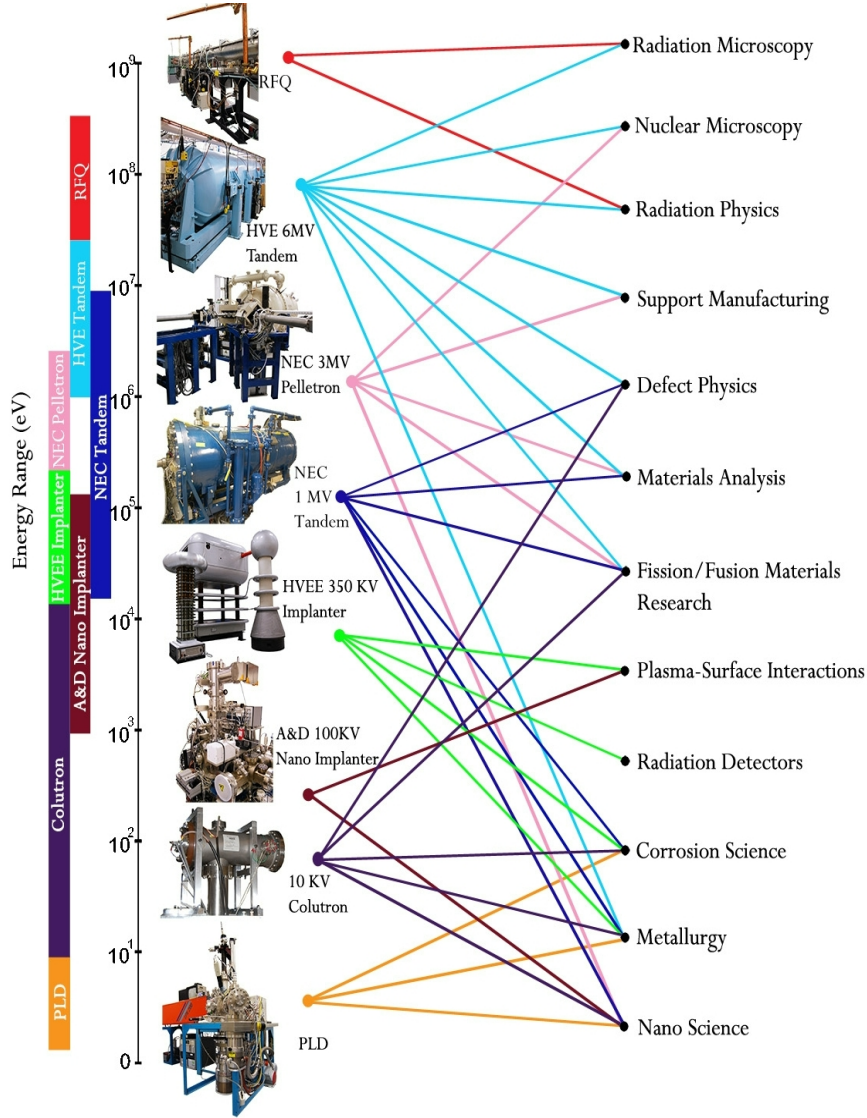
Tools used to develop new materials, and study evolution of tritium from these materials

# Film Deposition, Thermal desorption



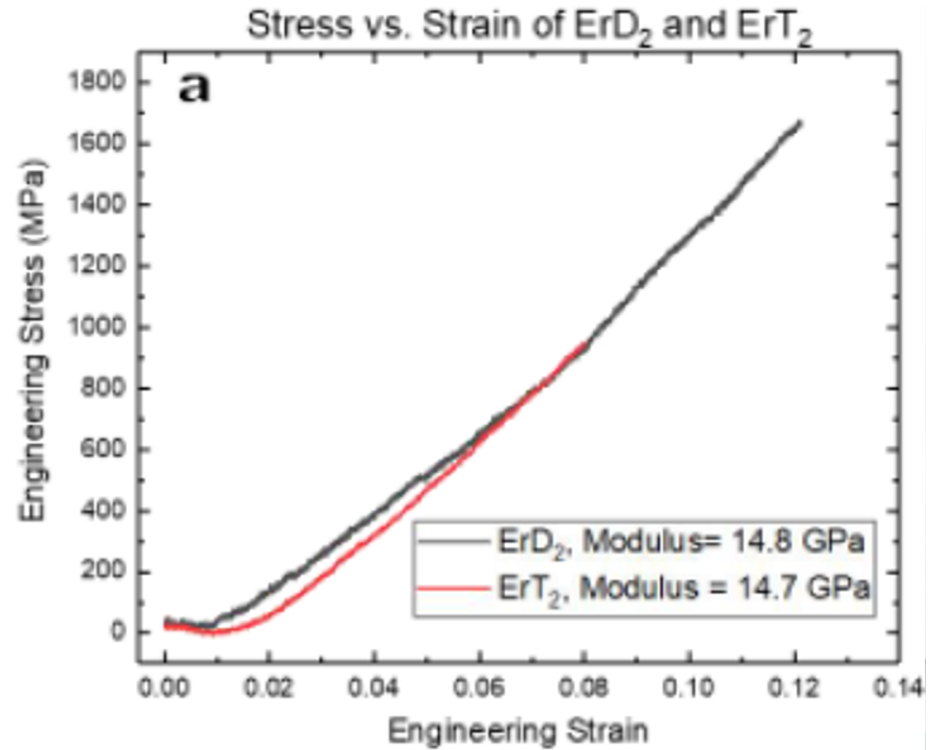
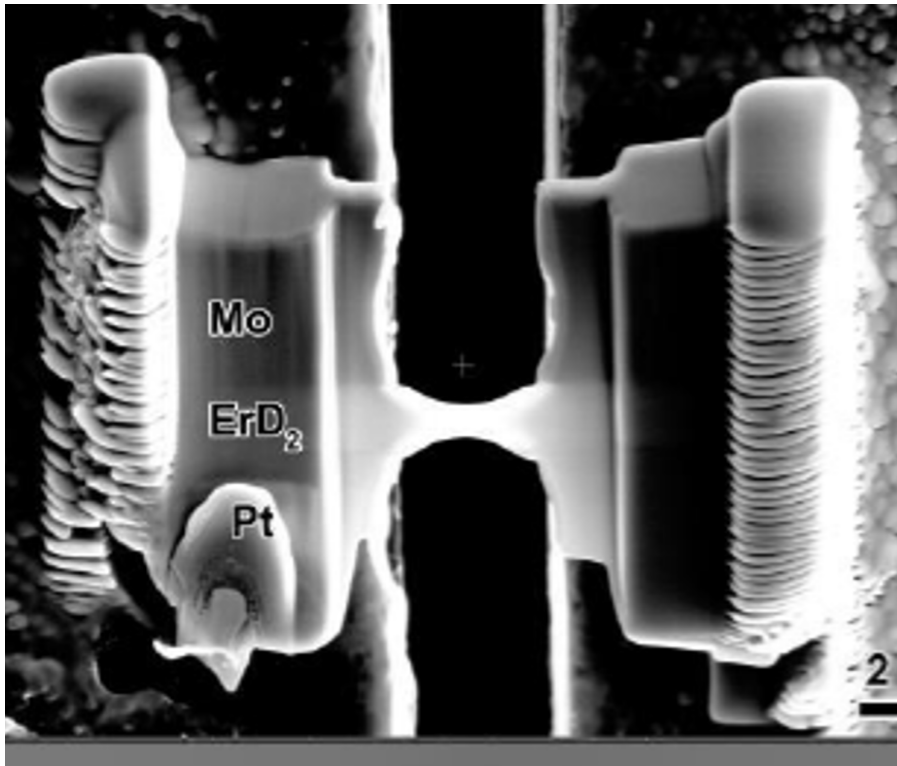
Tools used to develop new materials, and study evolution of tritium from these

# Sandia's Ion Beam Laboratory, CINT



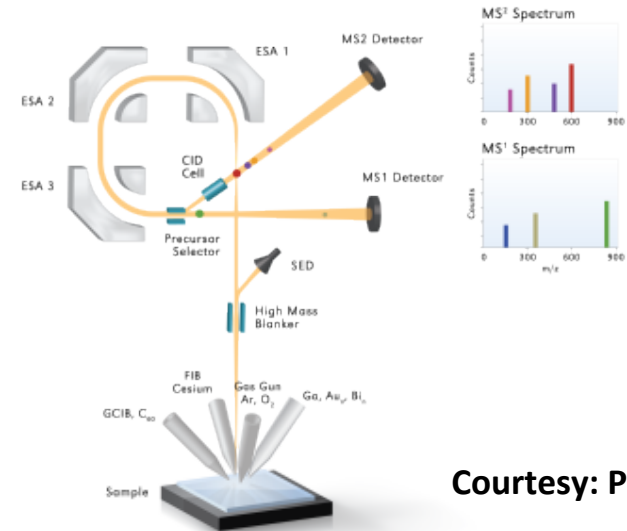
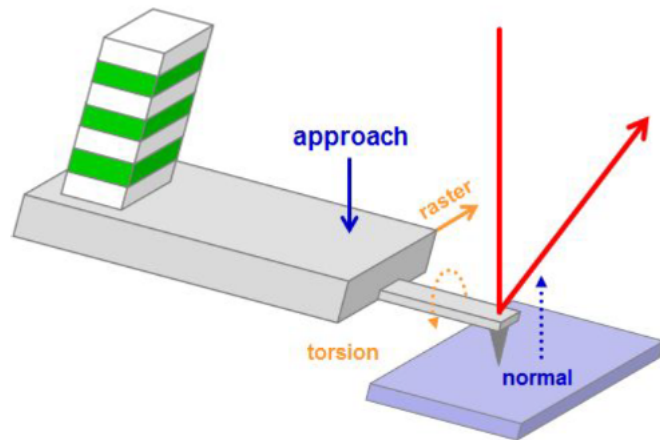
Tools at IBL used to study helium evolution, and H-isotope profiling in materials

# Hysitron Pi-95 Fixture tensile Testing

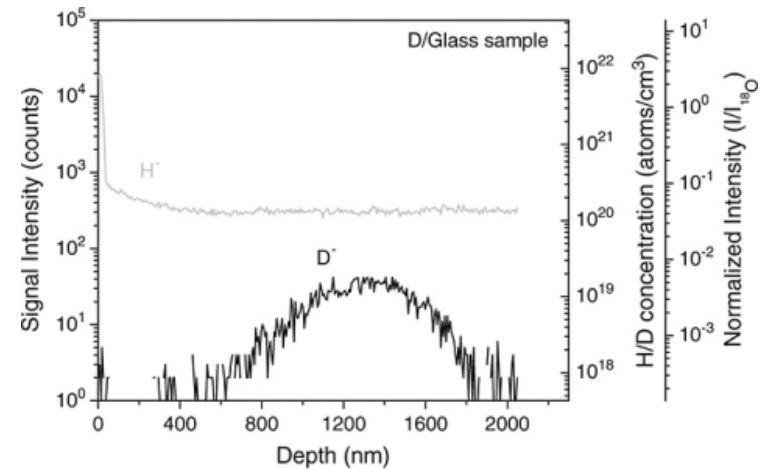
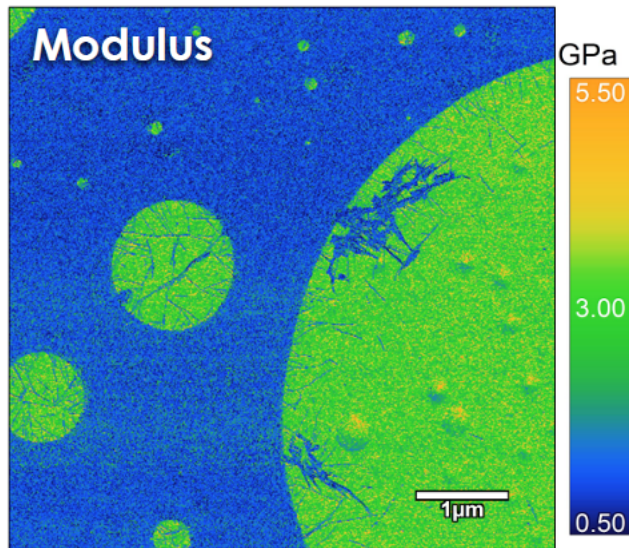


Micro-tensile test on FIB samples reveal that the tritide is ~ half as strong as the deuteride

# New Tools: AFM and TOF-SIMS



Courtesy: PHI Inc



H-D-T depth profiling is possible using TOF-SIMS

Possible to track property degradation of films due to He accumulation over time