SAND2006-5011C

Overview of Sandia/NM Tritium Capabilities and Research

29th Tritium Focus Group

Oak Ridge National Laboratory

August 15-17, 2006

Clark S. Snow and James F. Browning

Sandia National Laboratories/NM

Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company,
for the United States Department of Energy's National Nuclear Security Administration
under contract DE-AC04-94AL85000.

Sandia's Mission is National Security

- **Core Purpose:** Help our nation secure a peaceful and free world through technology
- **Five key mission areas**:
  - Nuclear weapons
  - Nonproliferation and Assessments
  - Military technologies and applications
  - Homeland security
  - Energy and infrastructure assurance

Our Highest Goal: become the laboratory that the United States turns to first for technology solutions to the most challenging problems that threaten peace and freedom.

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Sandia & Neutron Generators

Sandia has been involved in the area of designing and manufacturing neutron generators for over 50 years. Since that time the Laboratories have developed a wealth of knowledge related to these devices.

Today Sandia continues to manufacture neutron generators in support of National security programs, maintaining a comprehensive vertical infrastructure to address all aspects of neutron generator technology including:

- design
- manufacturing and assembly
- testing and quality assurance
- product support

Reliable

Repeatable

Robust

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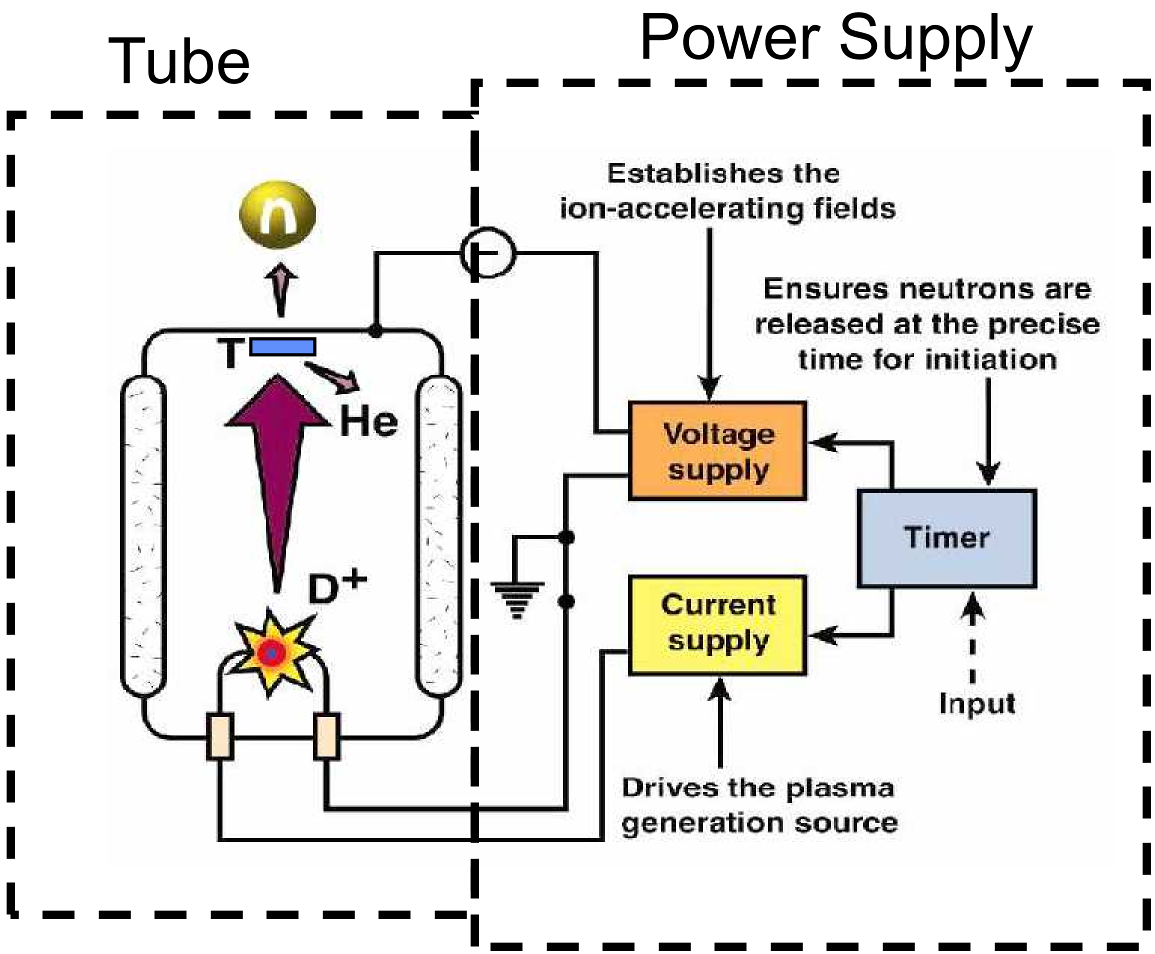
How does a neutron generator work?



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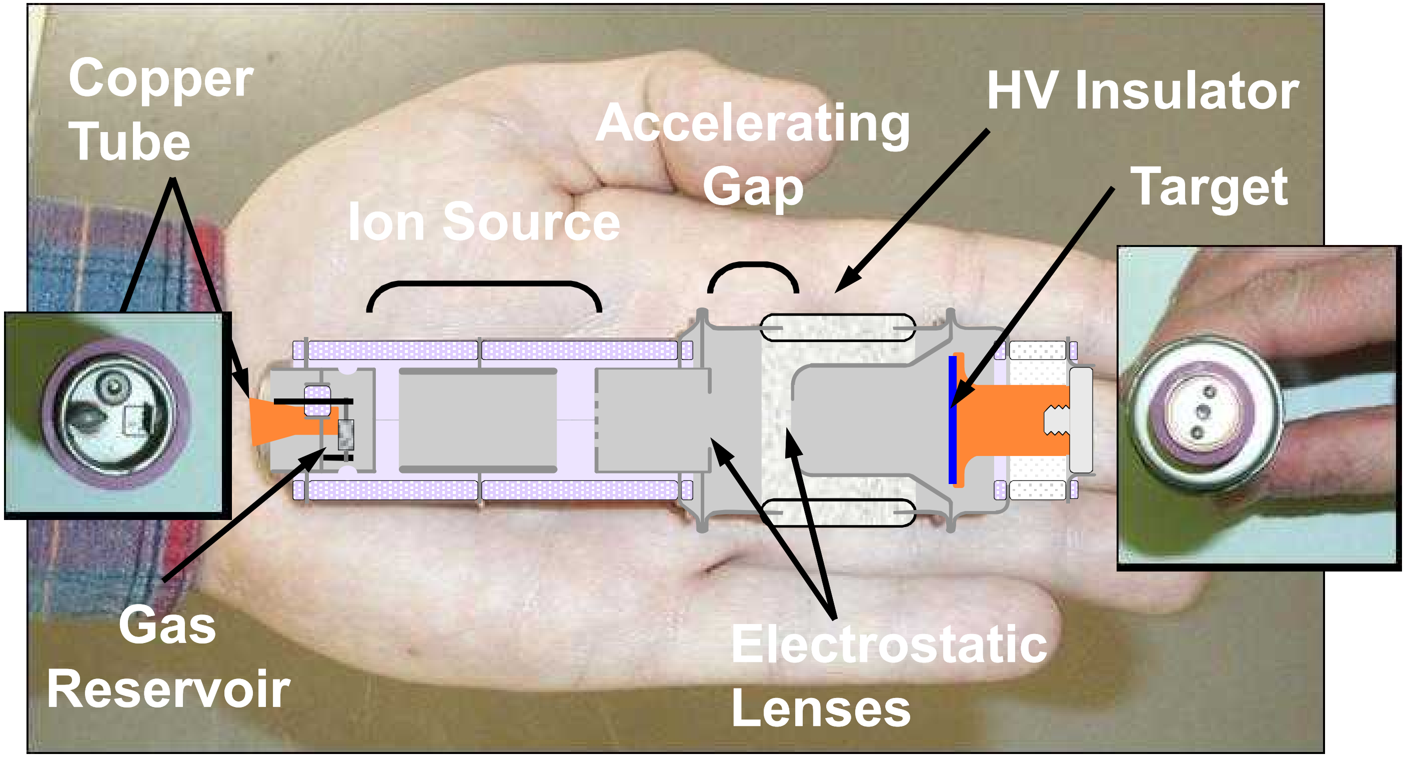
Essential Elements of a Neutron Tube

Neutron Tube

- Ion Source
- Gas Reservoir
- Accelerating Gap
- Electrostatic lenses
- Target
- HV insulator
- Vacuum envelope



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Overview of Sandia/NM Tritium Capabilities

- **All tritium operation is performed inside a "tritium envelope".**

- **Equipment can be connected to both**
  - **Tritium Exhaust (smoke stack release).**
  - **Tritium Capture System**

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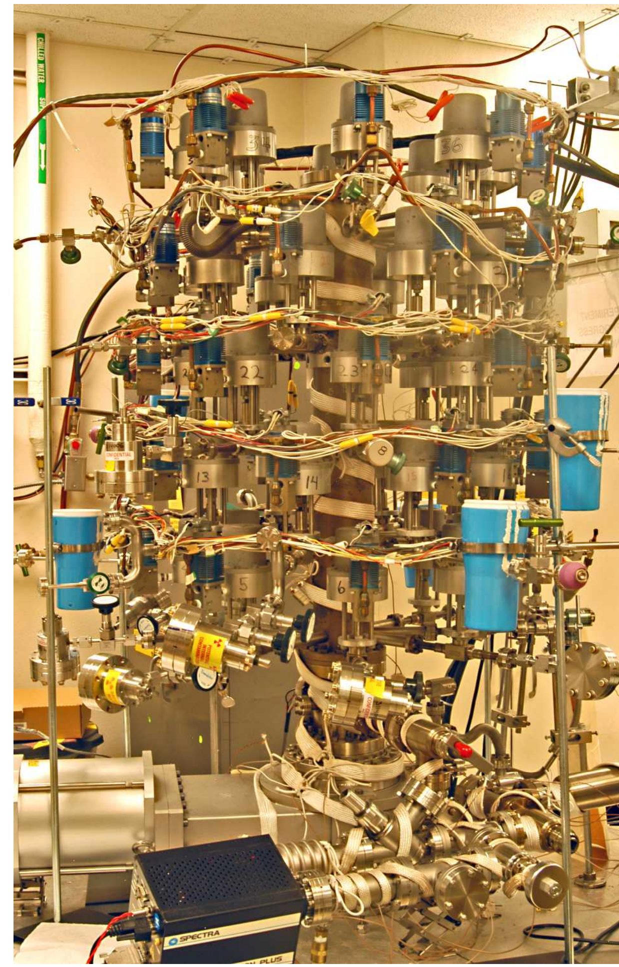
Helium Release "Tree"

- 40 automated sample ports
- RGA measures mass 2-4
- Calibrated by PSL
- UHV system, 1x10^{-9}Torr
- Measures Instantaneous Release Fraction (RFi)

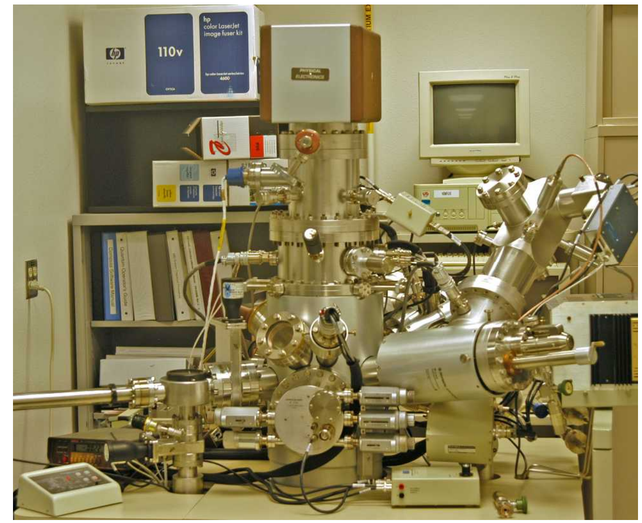
$$RF_i = \frac{ARR}{G(t)}$$

  - Samples build up He for one month
  - Amount of He determined
  - Average Release Rate determined
  - Divided by generation rate



Surface Analysis and Characterization

- **XPS**
  - PHI Quantum 2000 Scanning ESCA Microprobe
    - Energy resolution 0.5 eV
    - Minimum x-ray spot size 8 um
    - Hemispherical Mirror Analyzer (mean radius 279.4 mm)
    - 16 Channel MCD
    - Differentially pumped ion gun
    - Electron neutralizer
    - Ion neutralizer
    - Aluminum anode
    - Maximum sample size 75 mm x 75 mm x 25 mm high
    - (max height 40 mm if sample mounted on platen base)
    - Electron gun filament LaB6
    - Beam voltage 0 - 20 kV
    - Electron beam diameter minimum 5 um, maximum 500 um
    - Scan raster maximum 1500 um x 1500 um
    - Sample introduction chamber isolation from main analysis chamber
    - Sample preparation chamber attached
    - Residual Gas Analyzer
    - Base pressure 5x10 (exp-10) Torr



- **Auger**
  - PHI Model 680 Scanning Auger Nanoprobe
    - Cylindrical Mirror Analyzer
    - Energy resolution < 0.6% as measured by the FWHM of a 1keV elastic peak from clean copper
    - Schottky Field Emission Electron Source
    - Minimum beam diameter 8 nm, at 20 kV, 1 nA
    - Spatial resolution < 10 nm, at 20 kV, 1 nA
    - Maximum Beam Voltage 25 kV
    - Image magnification 13x to 500,000x
    - Differentially pumped ion gun
    - Residual Gas Analyzer
    - Maximum sample size 50 mm x 50 mm x 20 mm high
    - Sample introduction chamber isolation - less than three minutes from air to analysis position for non-out gassing samples
    - Base pressure <2x10 (exp-10) Torr

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X-Ray Diffraction Analysis

- **Bruker AXS D8 DISCOVER with GADDS**
  - **Monochromatic and parallel x-ray beam**
  - **HI-STAR area detector – Peak to back-ground close to theoretical limits**
  - **High temperature stage for in-situ XRD**
  - **Texture analysis**
  - **Reflectivity**
  - **Phase identification**
  - **Grazing incidence**
  - **Structure refinement**

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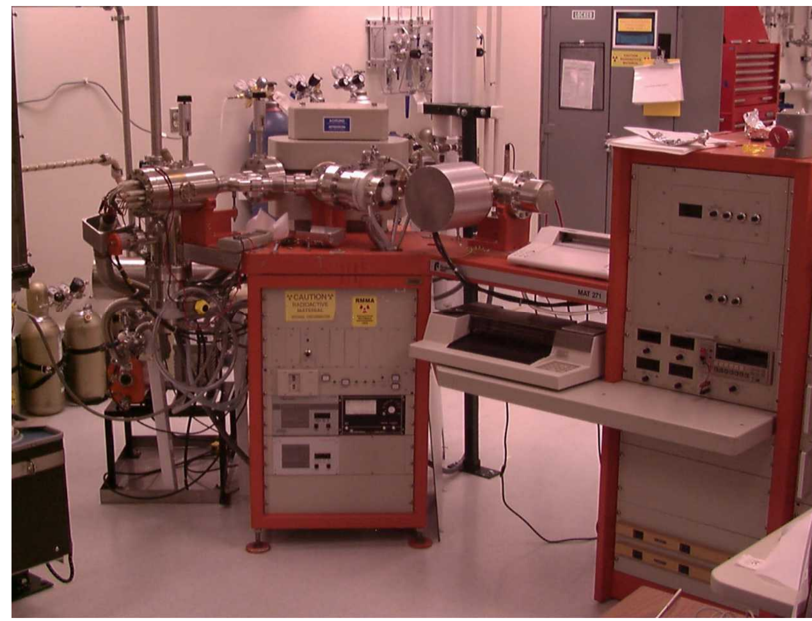
Mass Spectrometry Laboratory

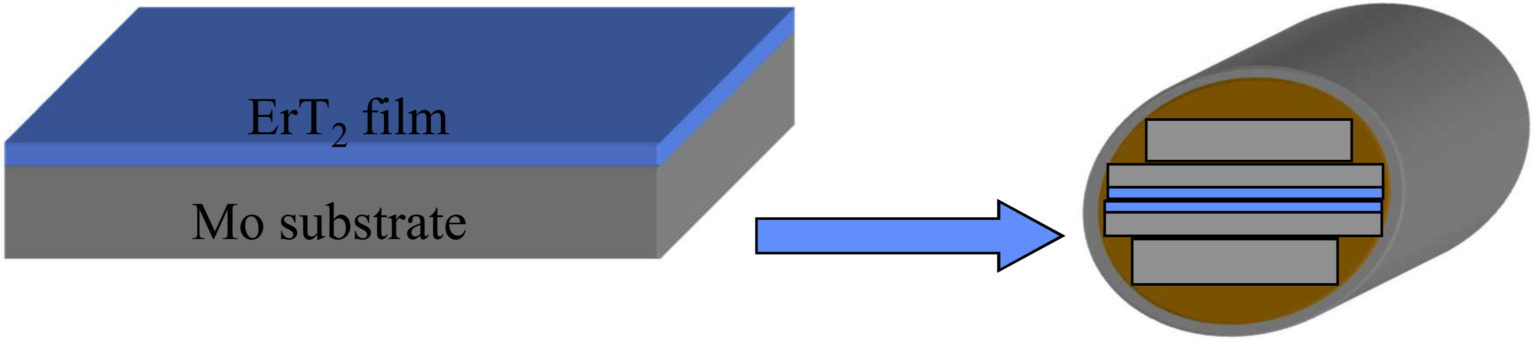
- **Finnigan MAT 271 Mass Spectrometers**
  - **Measurement uncertainty < +/- 2%**
  - **1-150 mass range**
  - **Capable of separating and independently measuring all hydrogen isotope combinations (Resolution > 1500)**
  - **No other technology demonstrates as good a sensitivity/resolution in the low 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**



Transmission Electron Microscopy

- JEOL 2000FX 200keV TEM
  - Noran EDX SiLi detector

  - Stacked geometry used for sample
    - Pair of films glued face to face
    - Sandwich inserted into Mo tube with Mo bracing strips
    - Tube back-filled with epoxy
    - After curing, tube cut into disks
    - Resulting samples dimpled to ~20μm in thickness
    - Ion milled at +/- 4° with 3-5keV Ar ion beam until perforation



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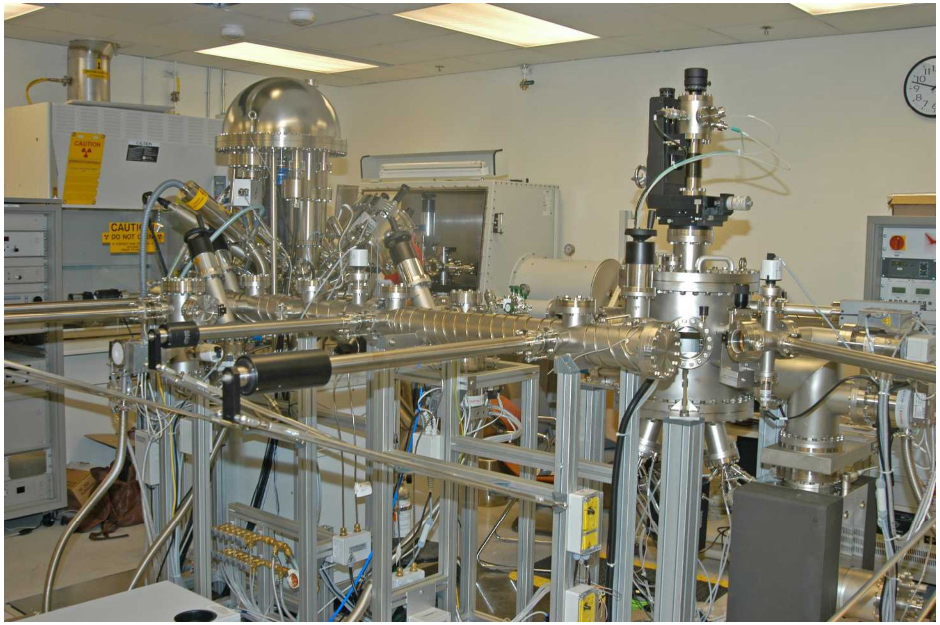
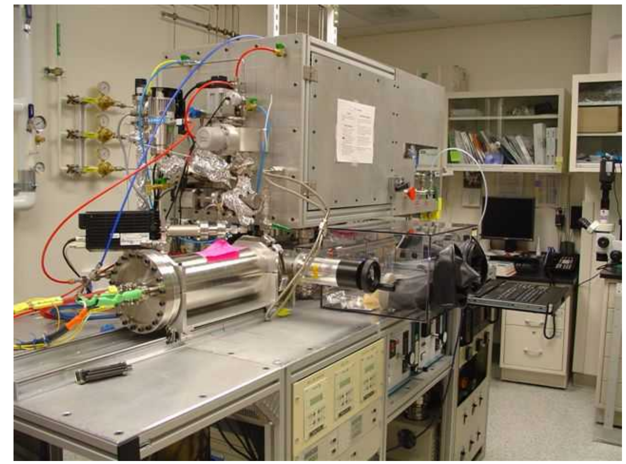
Thin Film Sciences and Surface Analysis

- Dual chamber system
  - Deposition chamber
    - 2 e-beam evaporation cells
    - 1 high temperature effusion cell
    - RHEED deposition monitor
    - RGA
    - Flux monitor
    - Substrate temp from 70-1000K
    - Can deposit 1-3 materials in single run
  - Surface Analysis chamber
    - Mono-chromatic XPS
    - Dual anode XPS
    - Auger
    - UPS
    - Secondary Electron Microscopy
    - 70K-1000K
    - Reaction Chamber



Metal Hydride Thermodynamics and Kinetics

- Seivert's type apparatus
  - 0.1 – 1000Torr pressure range
    - Sensitivity 0.1 Torr
  - 20 to 900C temperature range
    - Sensitivity of 0.25C



- TOS Thermal Desorption System
  - angular reflectron time-of-flight mass spectrometer (R.M. Jordan Company, Grassvalley, CA)
  - m/Dm @ m/q 4 ~ 212 (1000 theroetical limit)
  - ~750C upper temperature limit

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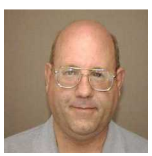
Who to contact for more information

- Group Manager – Rich Antepenko (505) 284-2086

- XRD/TEM – James Browning (505) 284-2700

- Surface Analysis – George Moore (505) 284-2828



- Mass Spectrometry – Henry Peebles (505) 844-2033

- Thermodynamics – Rob Ferrizz (505) 844-1449

- Helium Release Measurements – Clark Snow (505) 845-9548

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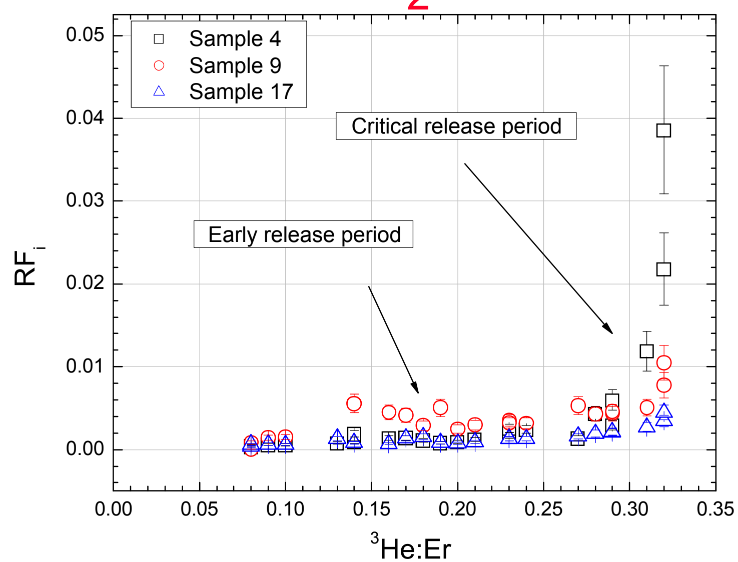
Fundamentals of Helium Release from Metal Tritides

- **Very small fraction of helium evolves from film.**
- **Release is characterized by two phases**
  - **Early release**
  - **Critical release**
- **Measurements made on helium release tree discussed earlier.**

ErT_2



$$RF_i = \frac{-\frac{d}{dt}N(t)}{G(t)}$$

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This is how other RE metal tritides behave

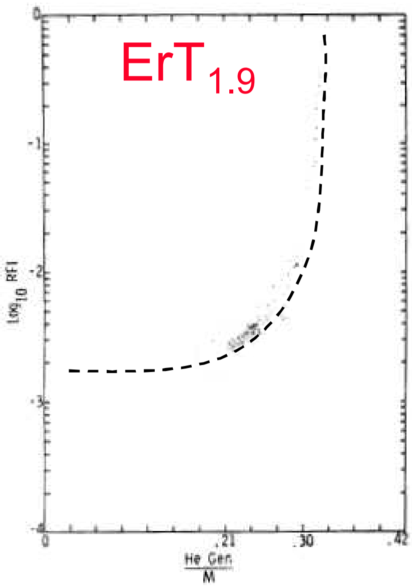

Fig. 3. RFI versus He/M for erbium tritide film A.R. 1.9 with no air exposure from film deposition to tritiding and minimal air exposure thereafter.

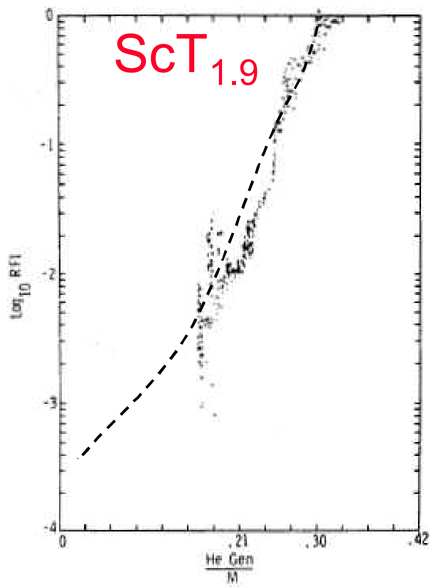

Fig. 11. RFI versus He/M for scandium tritide film. A.R. = 1.9.

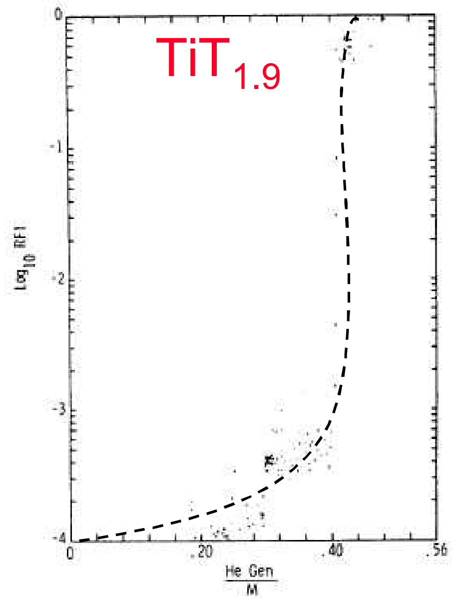

Fig. 14. RFI versus He/M for titanium tritide film. A.R. = 1.9.

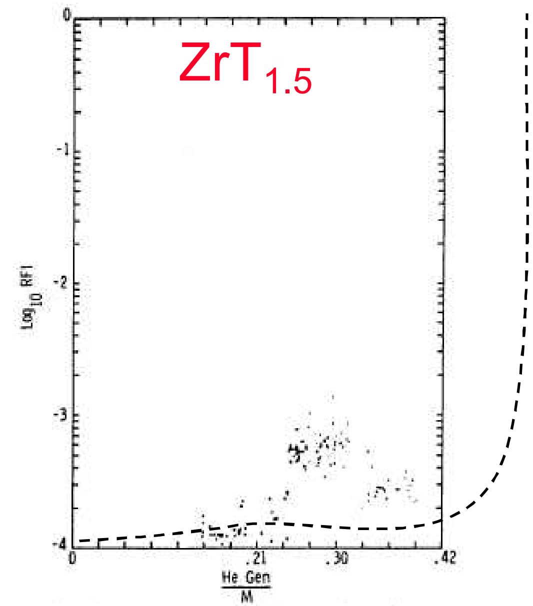

Fig. 15. RFI versus He/M for zirconium tritide film. A.R. = 1.5

Reported by Len Beavis, SAND79-0645.

Material	RFI	Crit.
$ErT_{1.9}$	0.2%	~0.3
$ScT_{1.9}$	N/A	N/A
$TiT_{1.9}$	0.03%	0.42
$ZrT_{1.5}$	0.03%	0.48

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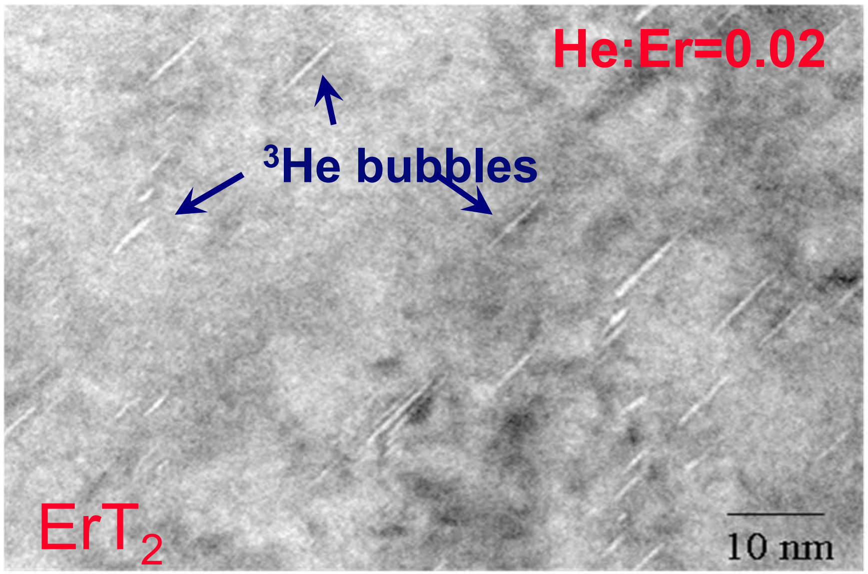
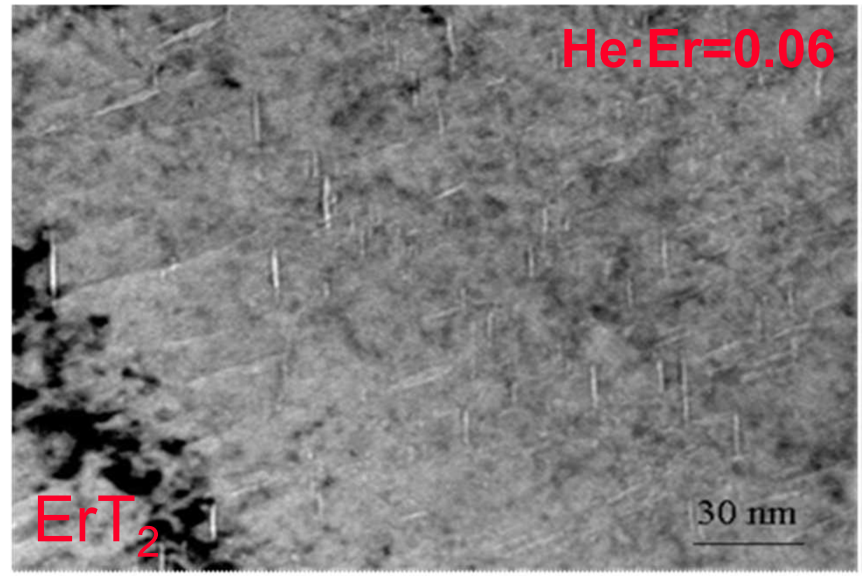
The remaining ^{3}Helium forms bubbles.

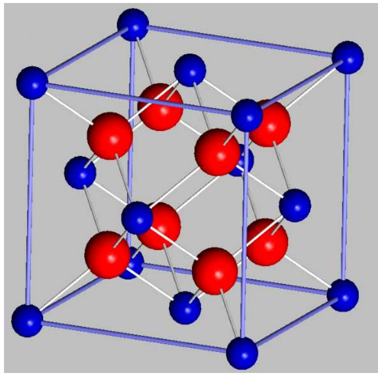




- TEM cross-section and plan view, bright-field, ~{110} zone:

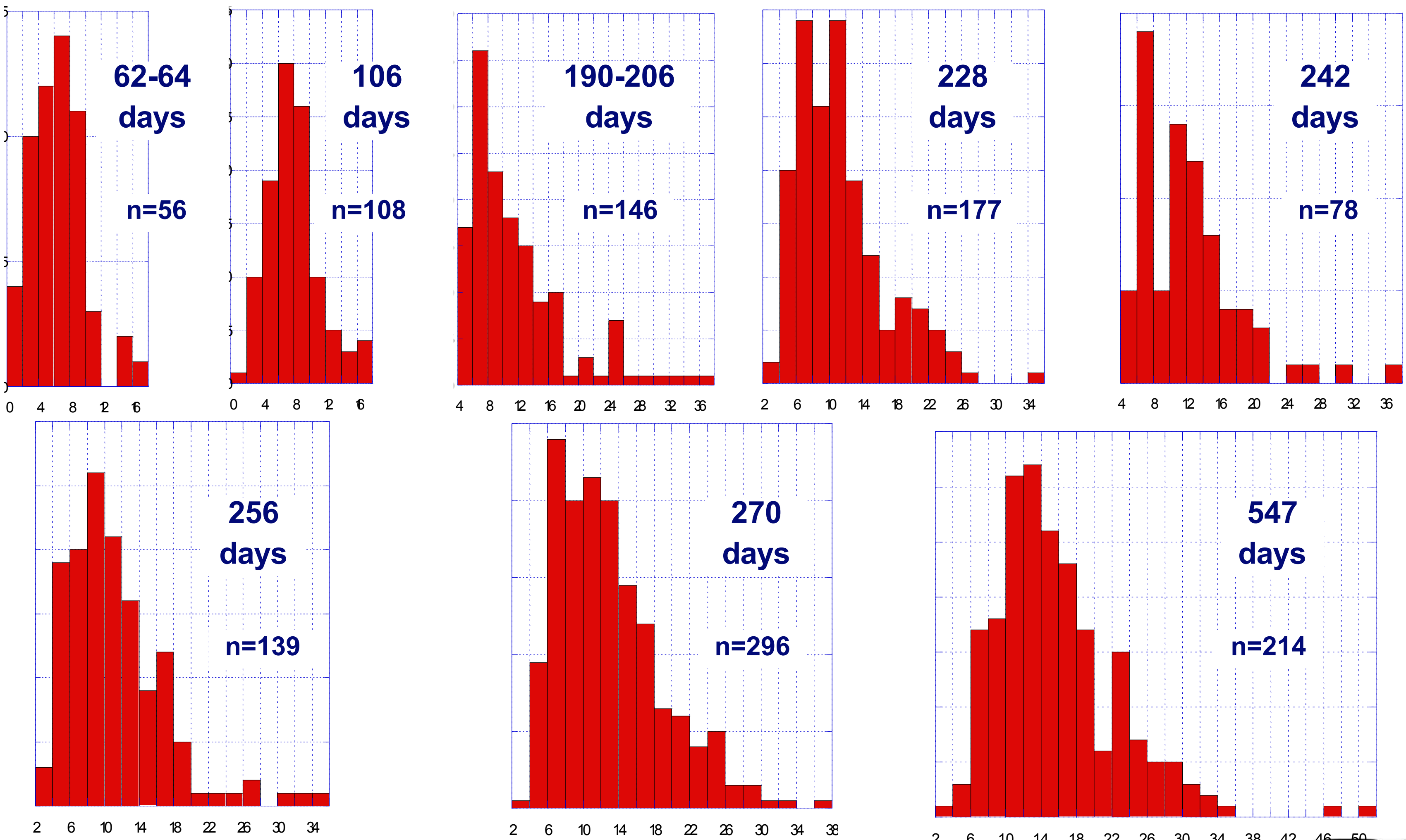
- Bubble populations are visible on two different sets of {111} planes.

β-phase fluorite structure



H:M = 2.0

Helium platelet diameter grows with age.



Data by Gillian Bond NMTech and James Browning SNL/NM

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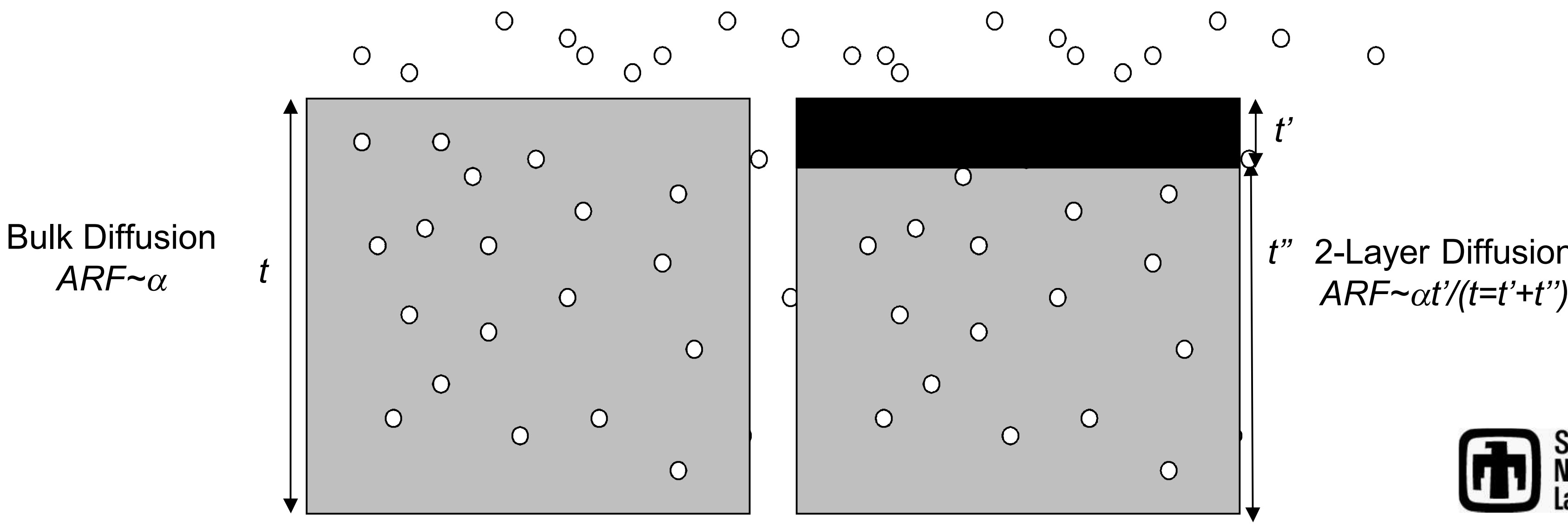
Where does the helium that is released come from?

- To answer this question, ErT_2 films of various thickness were grown.
- If the helium evolved from the films uniformally, i.e. from all depths of the film equally, then the helium release fraction should scale with thickness.
- If the helium only evolved from the surface then the release fraction should be constant.



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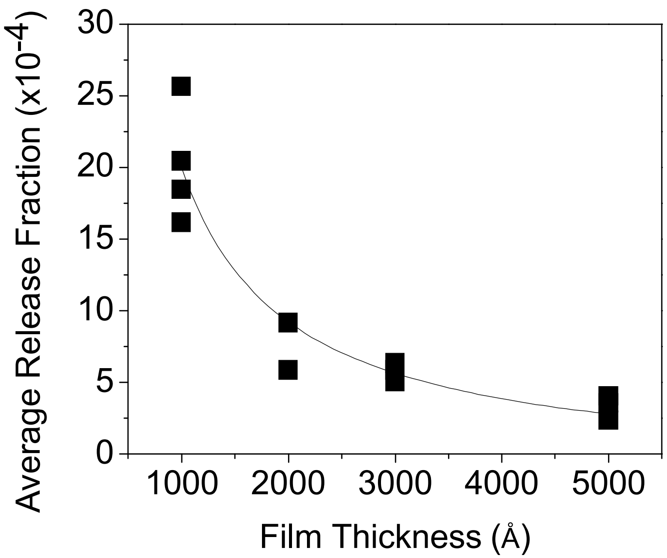
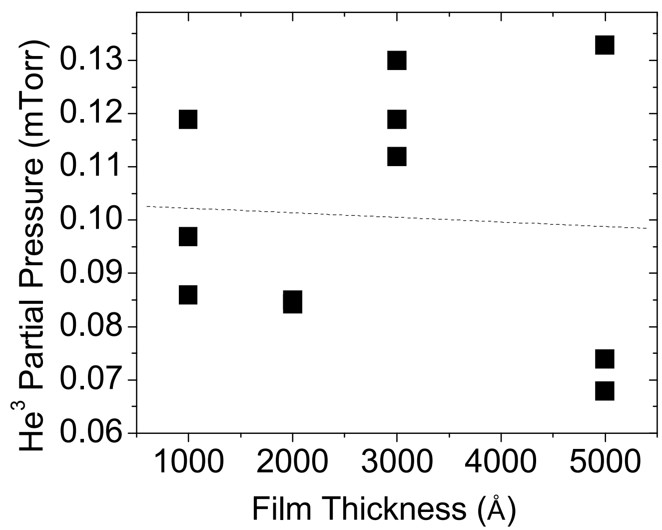
The amount of helium released from film is constant



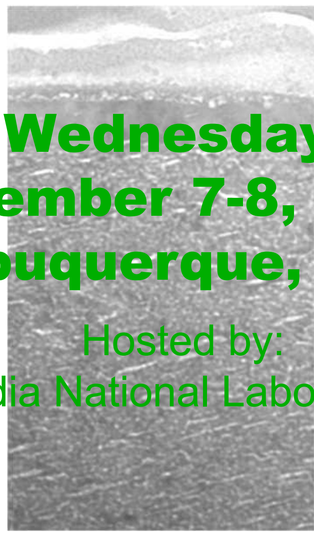


- Total helium released is constant.
- ARF varies inversely with thickness.

If you want to control "early helium release" you need to understand the surface.

We invite you to participate in the "Hydrogen and Helium Isotopes in Materials" Conference

Hydrogen and Helium Isotopes in Materials



Wednesday
November 7-8, 2006
Albuquerque, NM

Hosted by:
Sandia National Laboratories

Proposed topics include:

- Optical, mechanical, and electronic properties of materials.
- Surface/interfacial phenomena.
- Thin film properties.
- Bulk properties.
- Helium retention/helium release from materials.
- Aging effects on materials.

Both experimental and theoretical studies are encouraged.

Deadlines

To be a presenter	------------	September 30, 2006
Turn in Abstract	-------------	October 7, 2006
Hotel Reservation (special rate)--		October 2, 2006
Presentation Deadline	-------------	October 21, 2006
Attendance Registration	-------------	October 26, 2006
Meeting Date	-------------	November 7-8, 2006

Contacts:
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