



Ion Beam Analysis Activities at the Sandia Ion Beam Laboratory

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**Consultancy Meeting in Support of the New Coordinated Research Project
on Development and Application of Ion Beam Techniques for Materials
Irradiation and Characterization Relevant to Fusion Technology**

IAEA Vienna, Austria 2/24/2020

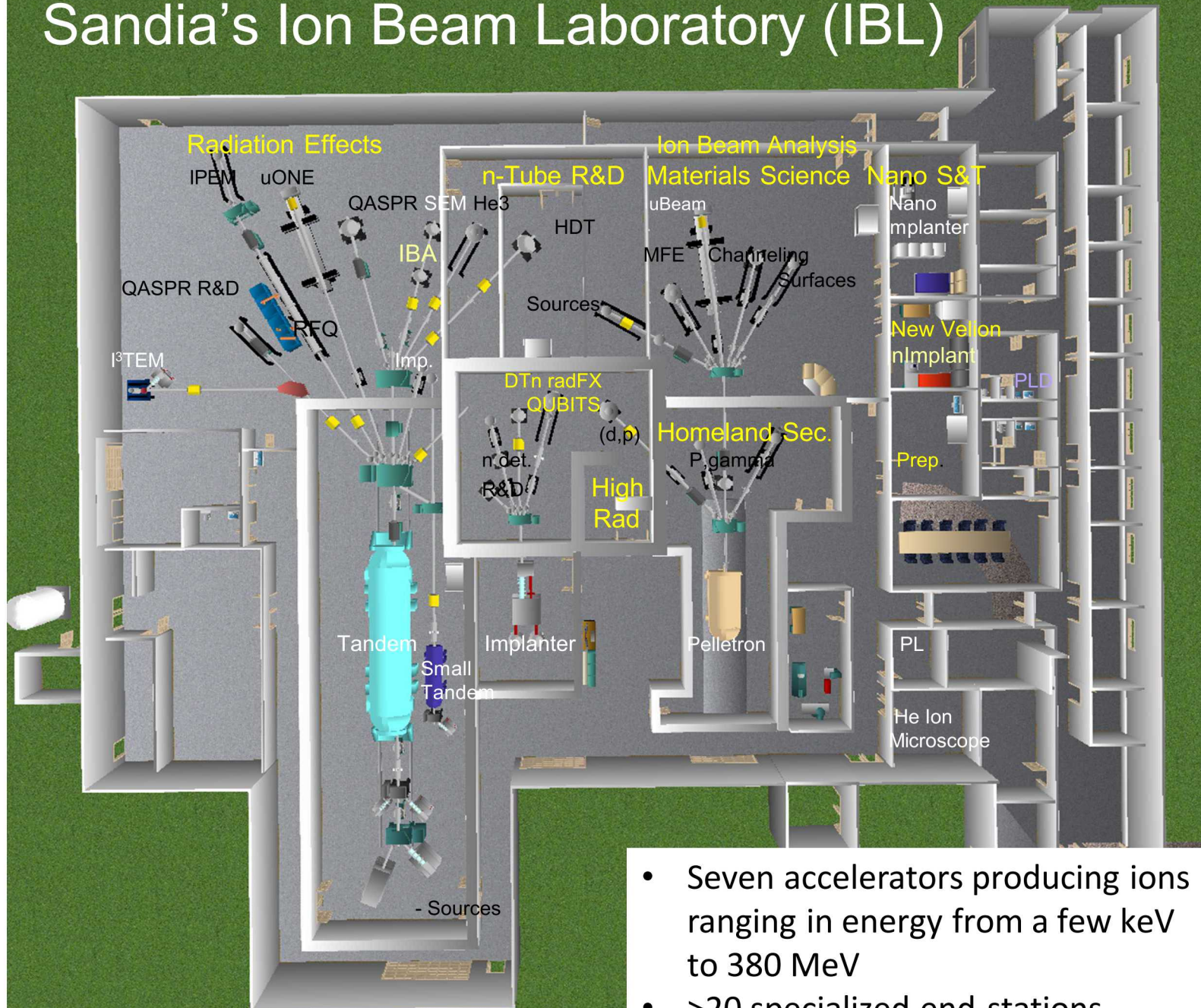


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Outline

- Overview of experimental facilities at the Sandia IBL
 - Accelerators
 - End stations
- Experimental methods used for MFE, illustrated by a few projects
 - DiMES in DIII-D
 - He3 NRA for light-element depth profiling

Sandia's Ion Beam Laboratory (IBL)



- Seven accelerators producing ions ranging in energy from a few keV to 380 MeV
- >20 specialized end-stations

Ion Accelerators

- HVE-Pelletron EN Tandem (6 MV) and negative ion sources for various ion types
- Single-ended NEC Pelletron (3 MV) Model 3UH-2 (MFE)
- Small Tandem (1MV)
- HVEE 300 kV implanter
- Nano implanters
 - A&D 10-100 kV, 150-10 nm beam
 - Raith Velion 5-35 kV, ~ 6 nm beam @ 10 kV (New)
- He ion microscope (New)



End Stations

- IBA for MFE, RBS/NRA on probes or whole tiles from DIII-D (16 x 26 cm translation)
- Irradiation to produce displacement damage, high dpa or short pulsed (>10ns)
- Microbeams ($\sim 1\text{-}5\text{ }\mu\text{m}$ depending on count rate)
- Nanobeams for single ion implantation (QBIT research)
- I^3TEM – TEM with heavy ions from tandem and low-energy light ions (H & He) from Colutron
- Ion Channeling (with UHV, AES, LEED, and gas dosing)
- He ERD for HDT depth profiling
- DLTS to characterize carrier emission rate from defects in semiconductors
- 14 MeV DT neutron production ($\sim 5 \times 10^8\text{ n/cm}^2/\text{s}$) to test semiconductor device response



MFE end station

Work for MFE at the Sandia IBL is long-standing and broad in scope

- DIII-D DiMES Erosion/Redeposition
- EAST/MAPES Erosion/Redeposition for ITER
- W in WEST
- He³ NRA for light-element depth profiling
- C¹³ methane injection in DIII-D
- Li and D retention in NSTX
- Enhanced retention of DT at displacement damage in W
- Kinetics of HDT uptake & release, surface recombination & effect of contaminants
- He ERD for HDT profiling

IBA is used to study PWI in Tokamaks

- To measure changes in near-surface composition of PFCs, providing insight into material erosion/transport/redeposition by the plasma, and data for development and validation of codes used to extrapolate plasma-material response to reactor conditions.
- Main limitation is that IBA is ex-situ and hence not real-time.
Analysis of PFCs are typically either:
 - on tiles from many locations removed after long exposure to various plasma conditions, or
 - on probes exposed to well-defined plasma conditions, but only at one location.

Table of Selected Nuclear Reactions

1 H 1 (19F,2)

1 H	1	(19F, γ)
1 H	2	(3He,p)
1 H	3	(p,n)
2 He	3	(3He,pp')
2 He	4	(11B,n)
3 Li	7	(p, α)
4 Be	9	(p, α)
5 B	11	(p, α)
6 C	12	(3He,p)
7 N	14	(α ,p)
9 F	19	(p, γ)
10 Ne	20	(p, γ)
11 Na	23	(p, γ)
13 Al	27	(p, γ)
15 P	31	(p, γ)

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Table of Selected Nuclear Reactions

Z A Reaction

1 H 1 (19F,γ)

1 H 2 (3He,p)

1 H 3 (p,n)

2 He 3 (3He,pp')

2 He 4 (11B,n)

3 Li 7 (p,α)

4 Be 9 (p,α)

5 B 11 (p,α)

6 C 12 (3He,p)

7 N 14 (α,p)

9 F 19 (p,γ)

10 Ne 20 (p,γ)

11 Na 23 (p,γ)

13 Al 27 (p,γ)

15 P 31 (p,γ)

U.S. DOE User Facility 975

ION BEAM MATERIALS RESEARCH Lab

Sandia National Laboratories


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MASS RESOLUTION OF He RBS

specific generic

RBS has ~2.1x more mass and depth resolution and 10x more sensitivity than IBA.

TOF-ERD has 10x better Resolution and worse Sensitivity than ERD.

X-IBA has 10x worse Resolution and Sensitivity than IBA.

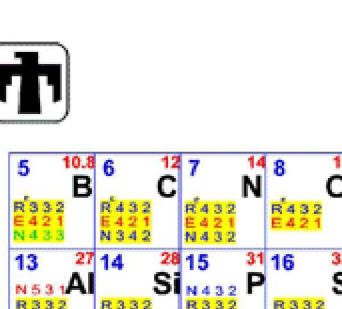
Micro-IBA with 2 μm diameter beam spot has ~10x worse sensitivity than IBA.

DEPTH RESOLUTION COLOR SCALE

1-10 nm RED

10-100 nm BLUE

100-1000 nm GREEN



*DEPTH RESOLUTION COLOR SCALE

1-10 nm RED
10-100 nm BLUE
100-1000 nm GREEN
> 1000 nm BLACK

* Depth resolution in Si substrate.
Resolution is better in higher Z substrate,
worse in lower Z substrates.

The diagram illustrates the components of a Key-Value pair for an element. The 'Key' (left) and 'Value' (right) are shown with their respective atomic numbers and weights. The 'Key' is 74 (Atomic Number) and 184 (Atomic Weight). The 'Value' is W (Symbol) and 184 (Atomic Weight). The 'Key' is also associated with a RANGE (<10³ nm) and SENSITIVITY (#10 AT. FRAC.). The 'Value' is associated with a DEPTH RESOLUTION* (<10 nm). The 'Key' is also associated with an IBA Technique (P = PIXE, R = RBS, E = ERD, N = NRA, H = HBS). The 'Value' is associated with an X-RAY LINE and an X-RAY ENERGY (keV). The 'Key' is also associated with a PREVIOUS ISMRL EXPERIENCE.

Key

ATOMIC NUMBER: 74

ATOMIC WEIGHT: 184

RANGE: <10³ nm

SENSITIVITY: #10 AT. FRAC.

IBA Technique: P = PIXE, R = RBS, E = ERD, N = NRA, H = HBS

Value

SYMBOL: W

DEPTH RESOLUTION*: <10 nm

X-RAY LINE: L 3.096

X-RAY ENERGY (keV): 1.84

PREVIOUS ISMRL EXPERIENCE

☆	58	140	59	141	60	144	61	145	62	150	63	152	64	157	65	159	66	163	67	165	68	167	69	169	70	173	71	175	
	Ce		Pr		Nd		Pm		Sm		Eu		Gd		Tb		Dy		Ho		Er		Tm		Yb		Lu		
	H8.2		H8.2		H8.2		R5.32		R5.32		R5.32		R5.32		R5.32		R5.32		R5.32		R5.32		R5.32		R5.32		R5.32		R5.32
	P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44		P5.44
	L.4.840		L.5.034		L.5.230		L.5.431		L.5.636		L.5.846		L.6.059		L.6.275		L.6.495		L.6.720		L.6.948		L.7.181		L.7.414		L.7.654		
☆☆	90	232	91	231	92	238	93	237	94	244																			
	Th		Pa		U		Np		Pu																				
	H8.3		H8.3		H8.2		R5.32		R5.32		R5.32																		
	P5.44		P5.44		P5.44		P5.44		P5.44		P5.44																		
	L.12.968		L.13.291		L.13.613		L.13.945		L.14.279																				

PIXE = Particle Induced X-ray Emission
RBS = Rutherford BackScattering
ERD = Elastic Recoil Detection
HIBS = Heavy Ion BackScattering

TOF = Time-of-Flight
X-IBA = eX-vacuo Ion Beam Analysis
R[±] = Enhanced Rutherford Backscattering

https://www.sandia.gov/research/facilities/technology_deployment_centers/ion_beam_lab/ion_beam_analysis_periodic_table.html or Google *IBA Periodic Table*

RBS and He³ NRA are the principle IBA techniques used for MFE

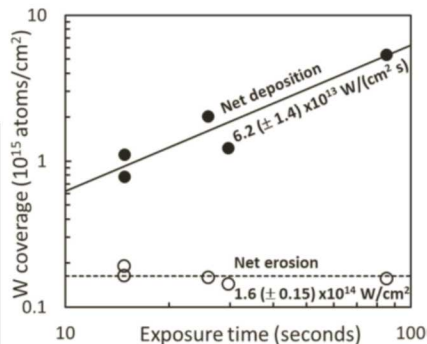
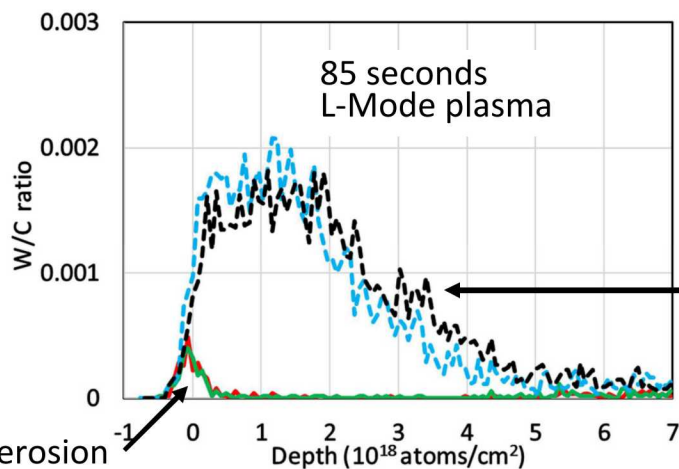
Factors influencing measurement precision for RBS & NRA

- Spectrum analysis is simpler if thin-target approximation is adequate. Otherwise, SIMNRA with energy dependent cross section and stopping may be adequate.
- Best to use RBS for heavy elements on light substrates, NRA for light elements.
- Coverage ratios are more accurate than absolute quantities since some uncertainties cancel.
- For absolute quantities, need reaction cross section, number of incident particles (from integrated beam current) and detector solid angle. Good practice to also use a thin-film reference sample of known coverage, which becomes the only option when the cross section is unknown.
- Cross sections for RBS are more likely to be accurate when they are near Rutherford, which limits the incident ion energy and depth of analysis.
- Thickness of a surface-layer may be determined from reaction or scattering yield, or from ion energy loss if composition is known.

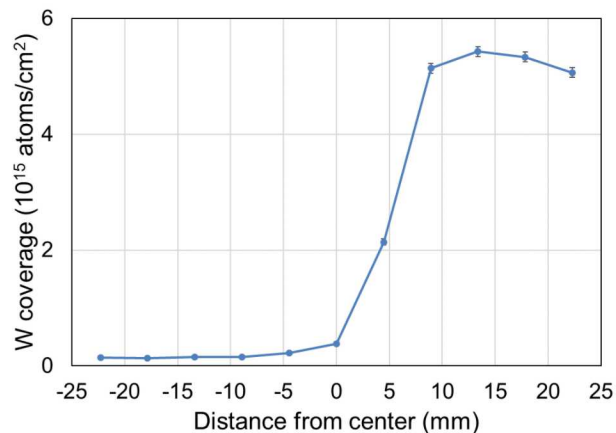
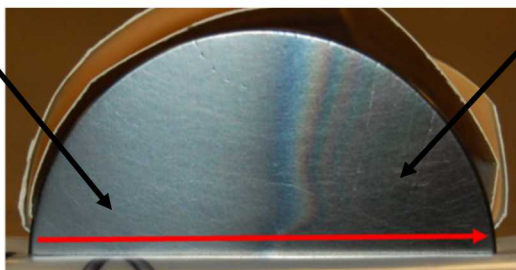
DIID-D DiMES Erosion/Redeposition (RBS)

Toroidal Tungsten Ring Experiment

Phys. Scr. T170 (2017) 014041 PFM16

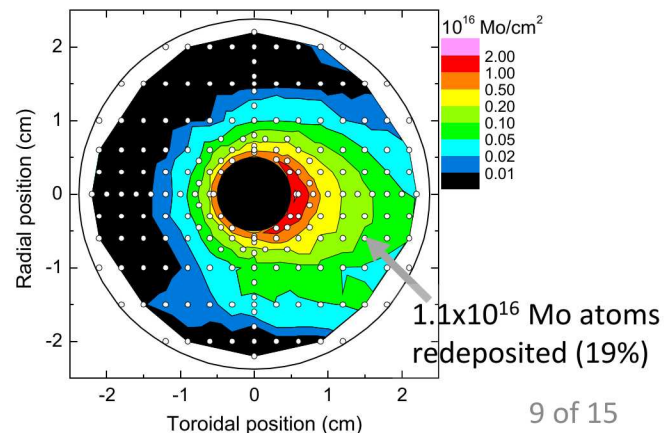
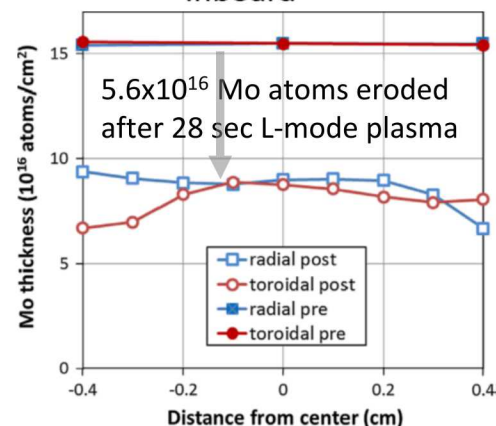
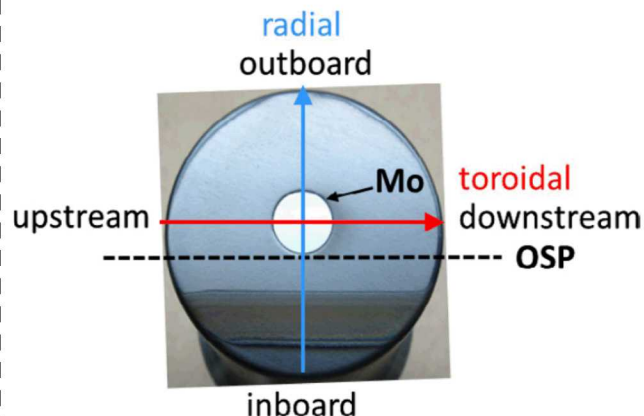


Net deposition on outboard side. Thickness & W coverage increase with exposure.

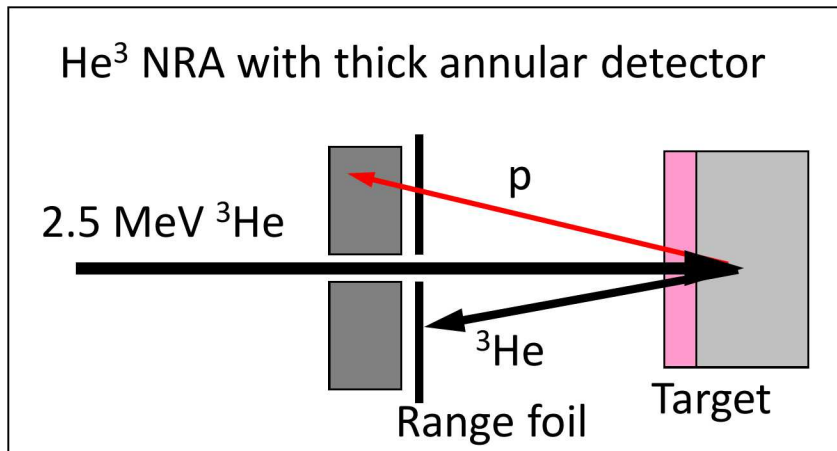


Mo erosion study

JNM 438 (2013) S822 PSI20

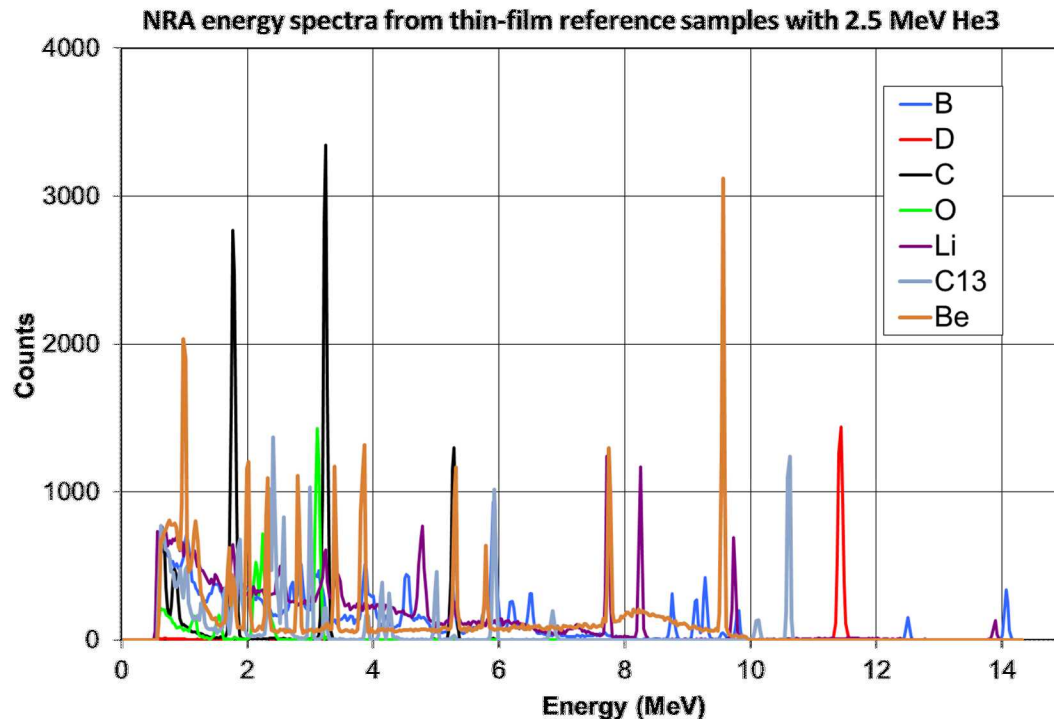


He³ NRA for light element depth profiling



Detector must be:

- Annular to maximize sensitivity and minimize kinematic energy broadening.
- Thick to measure full particle energy (> 1mm for 12 MeV proton from He³D)



He³ NRA for light element depth profiling

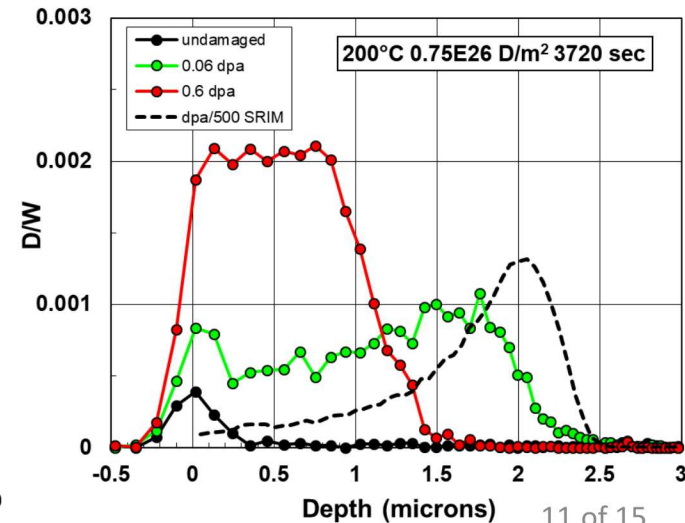
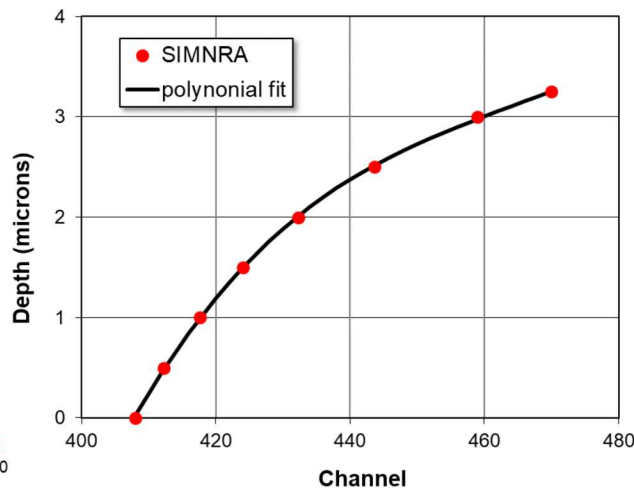
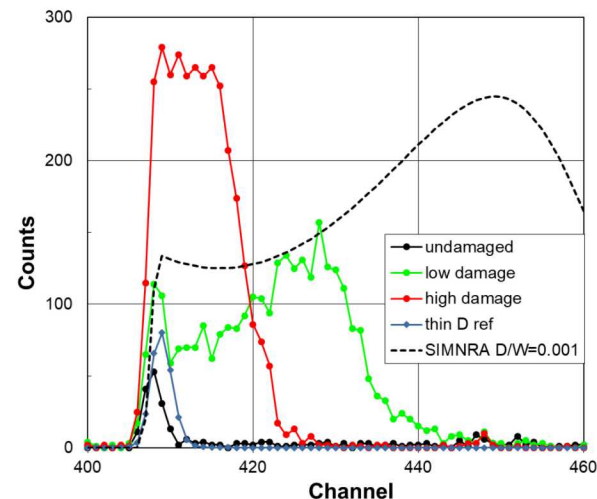
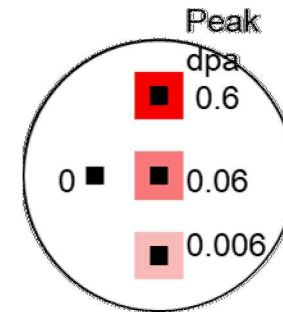
- Particle energy spectrum is measured at a single incident energy.
- Energy scale is transformed to a depth.
- Yield (counts/channel) is transformed to a concentration.
- Transforms obtained from SIMNRA with reaction cross section & stopping power.
- This method was developed at the Sandia IBL is used often for MFE.
- Simpler than alternative method of deconvoluting yield vs incident particle energy.

Study of D retention at displacement damage in W

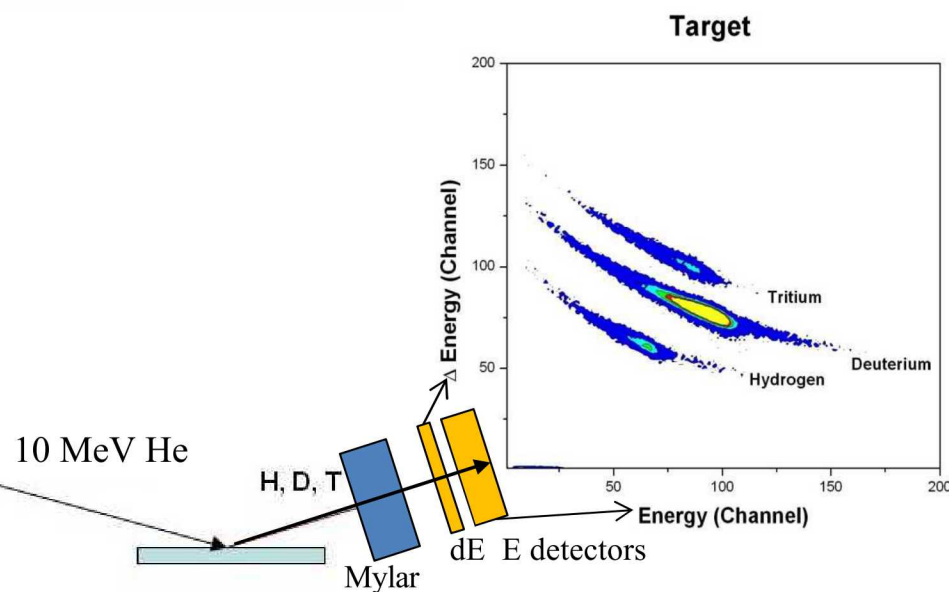
Wampler and Doerner, Nuclear Fusion 49 (2009) 115023

Wampler, Physica Scripta T180 (2020)

- W damaged with 12 MeV Si ions at the IBL
- Exposed to D plasma in PISCES-A at UCSD.
- Energy spectrum of protons from D(He³,p)α measured at the IBL
- Counts vs channel transformed to concentration vs depth.
- Detection limit D/W ~ 10 appm, depth resolution ~ 0.3 μm.
- Many experiments were done including exposure in DIII-D.



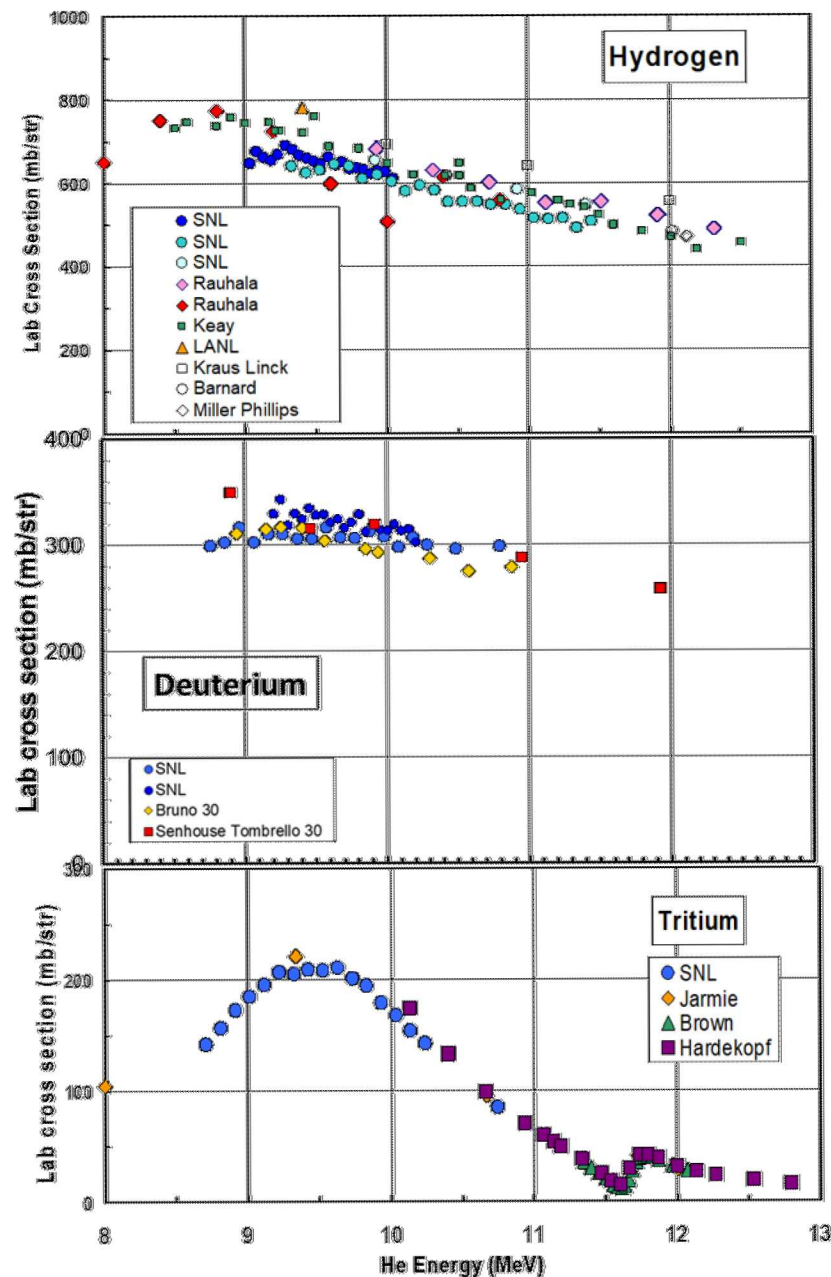
The HDT System can depth profile all three H isotopes in solids to depths of several microns



The HDT system uses 10 MeV He Elastic Recoil Detection (invented in the IBL) to quantify the concentration of H, D and T in solids. Best with smooth surfaces.



ERD cross sections for HDT were measured at the Sandia IBL in 2002

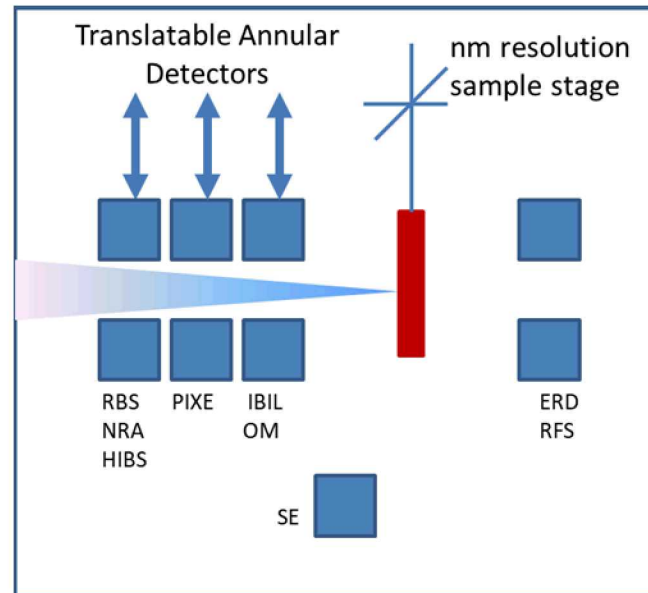


Scattering angle = 30°

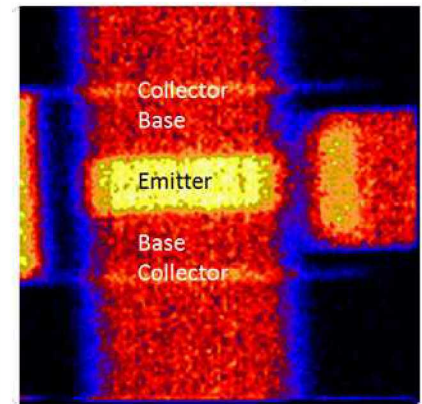
Used metal-hydride thin-film samples fabricated at Sandia in which the quantity of H,D,T was independently measured by thermal desorption with mass spec analysis.

Microbeam

The micro-beam on the Pelletron provides a capability of quantitatively measuring the composition with high sensitivity in 3D at micron lateral resolution and submicron depth resolution.



5 μm Au on HBT emitter 10x50 μm



- Annular detectors provide large solid angles, enabling high sensitivity
- OM-40 “Annular” optical microscope used to position samples and for IBIL
- PIXE detector developed by Rontec and Sandia
- RBS detector also used for NRA and HIBS

Summary

- The Sandia IBL has a broad range of experimental capabilities used for many application including IBA.
- These are used for a broad range of programs which include support for the US MFE program.
- Although a small program, the IBL has done most of the IBA for the US MFE program for decades.
- The IBL has broad experience with IBA methods and some unique equipment and capabilities for MFE:
 - End station for IBA on large objects (e.g. whole Tokamak tiles)
 - Depth profiling of light-elements by He3 NRA
 - Simulation of neutron damage by heavy-ion irradiation
- Other capabilities that have not been used extensively for MFE, but could be, include:
 - System for depth profiling HDT by He ERD (can analyze samples which contain tritium)
 - Microbeams
 - I³TEM
 - 14 MeV DT neutron source