Ion Beam Facility: unveiling the power of Ion Beam Analysis and Ion Irradiation for industry advancements

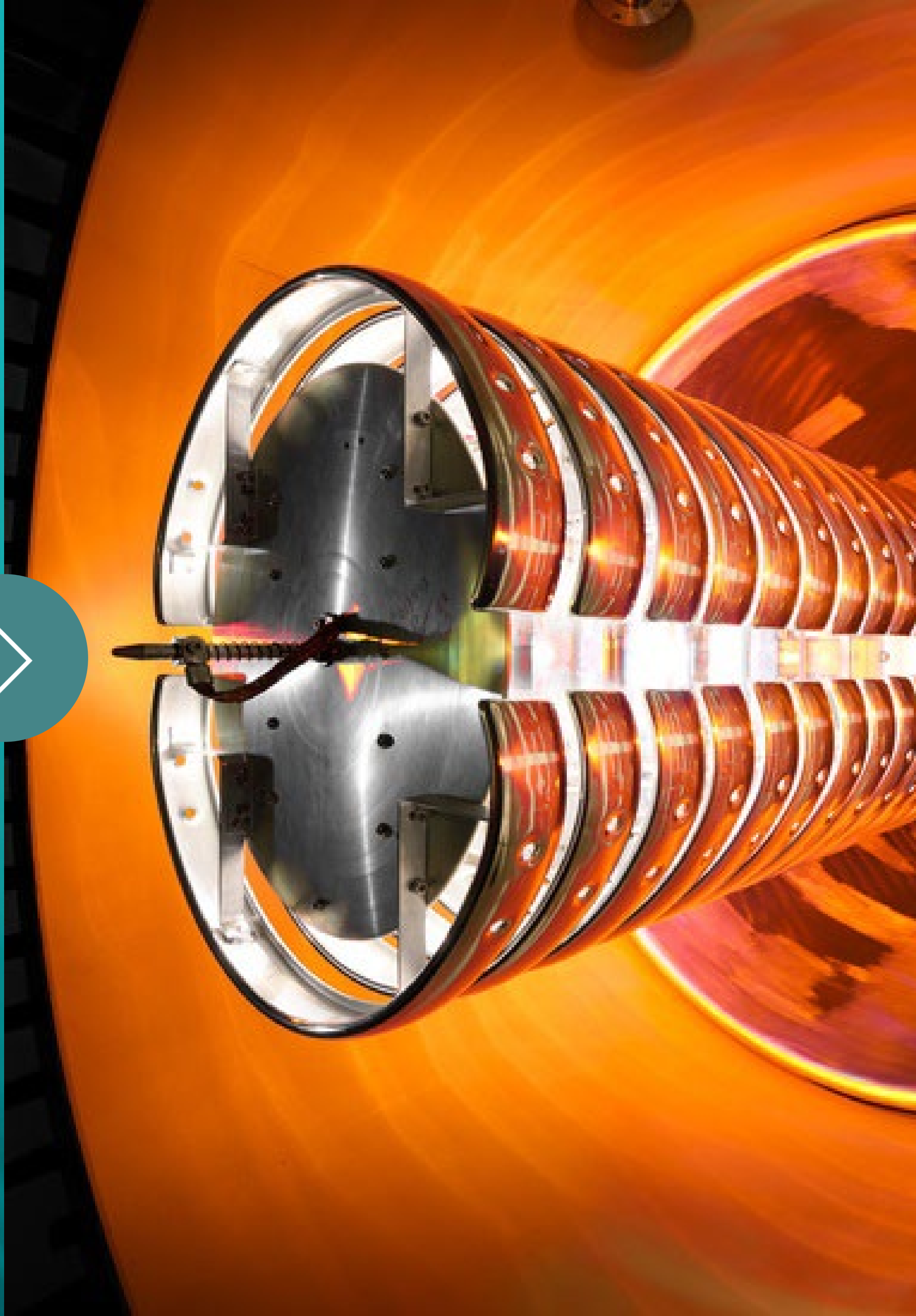
Dr. Beata Tyburska-Pueschel

Dutch Institute For Fundamental Energy Research

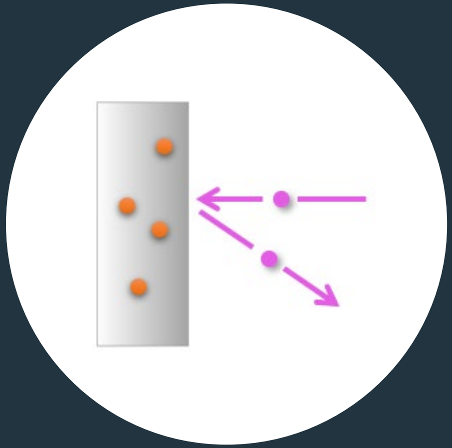
Precision Fair

DIFFER

NWO

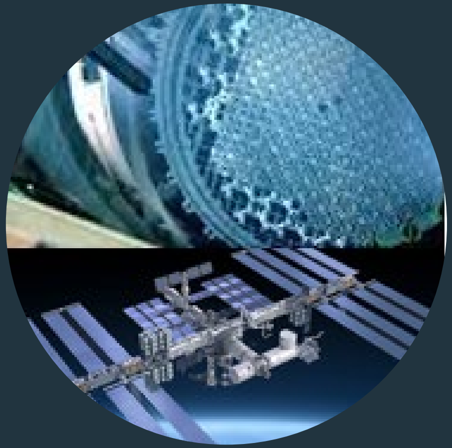


IBF lab



Physics

Material Analysis



Irradiation

Collaboration

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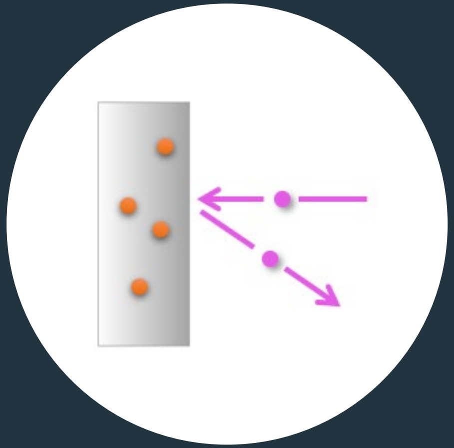

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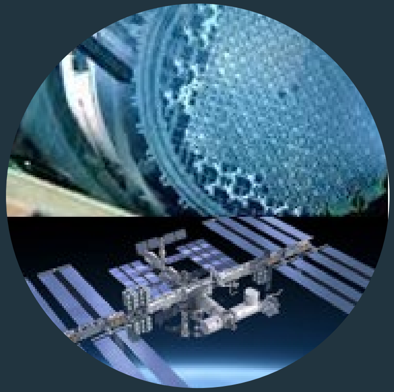


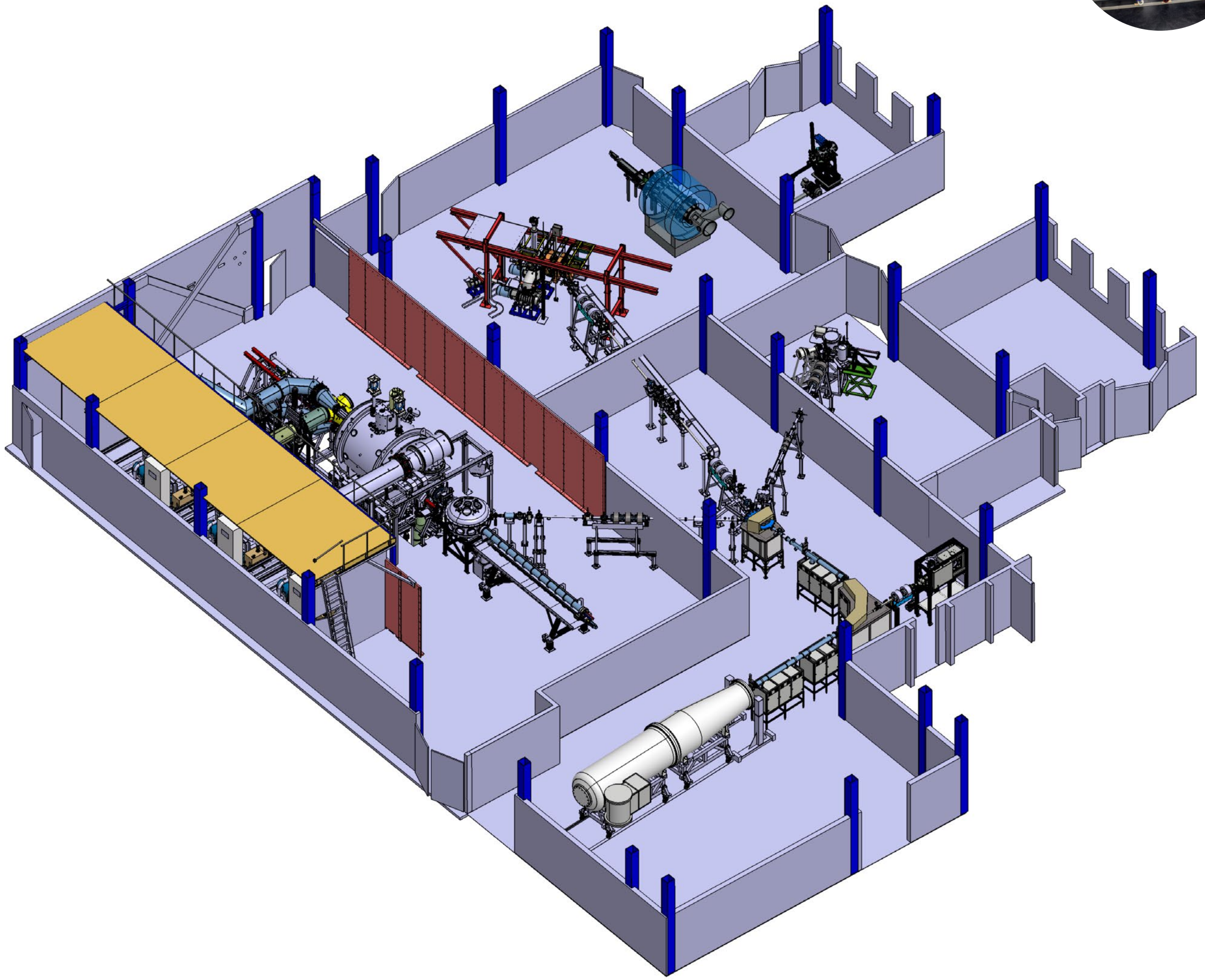
Irradiation

Collaboration

DIFFER Ion Beam Facility

- 3.5 MV Singletron - max. energy 4 MeV for $^{4}He^{2+}$
- Gas ions – H, 3He, 4He, plan to add D
- High-current up to 50 μC/cm ^{2}
- Four beamlines

- User-facility
- 30% time for industry
- Measurements & analysis



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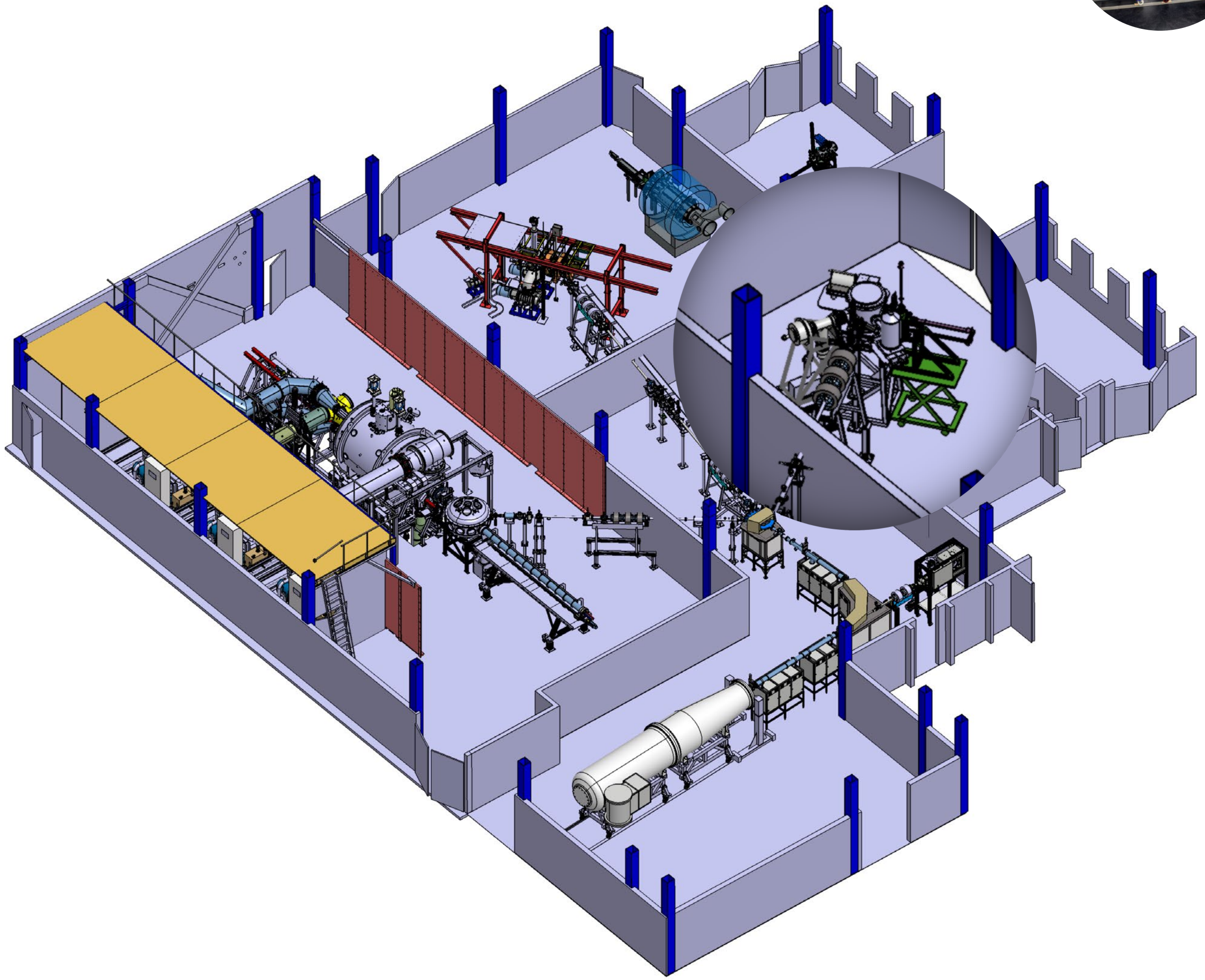

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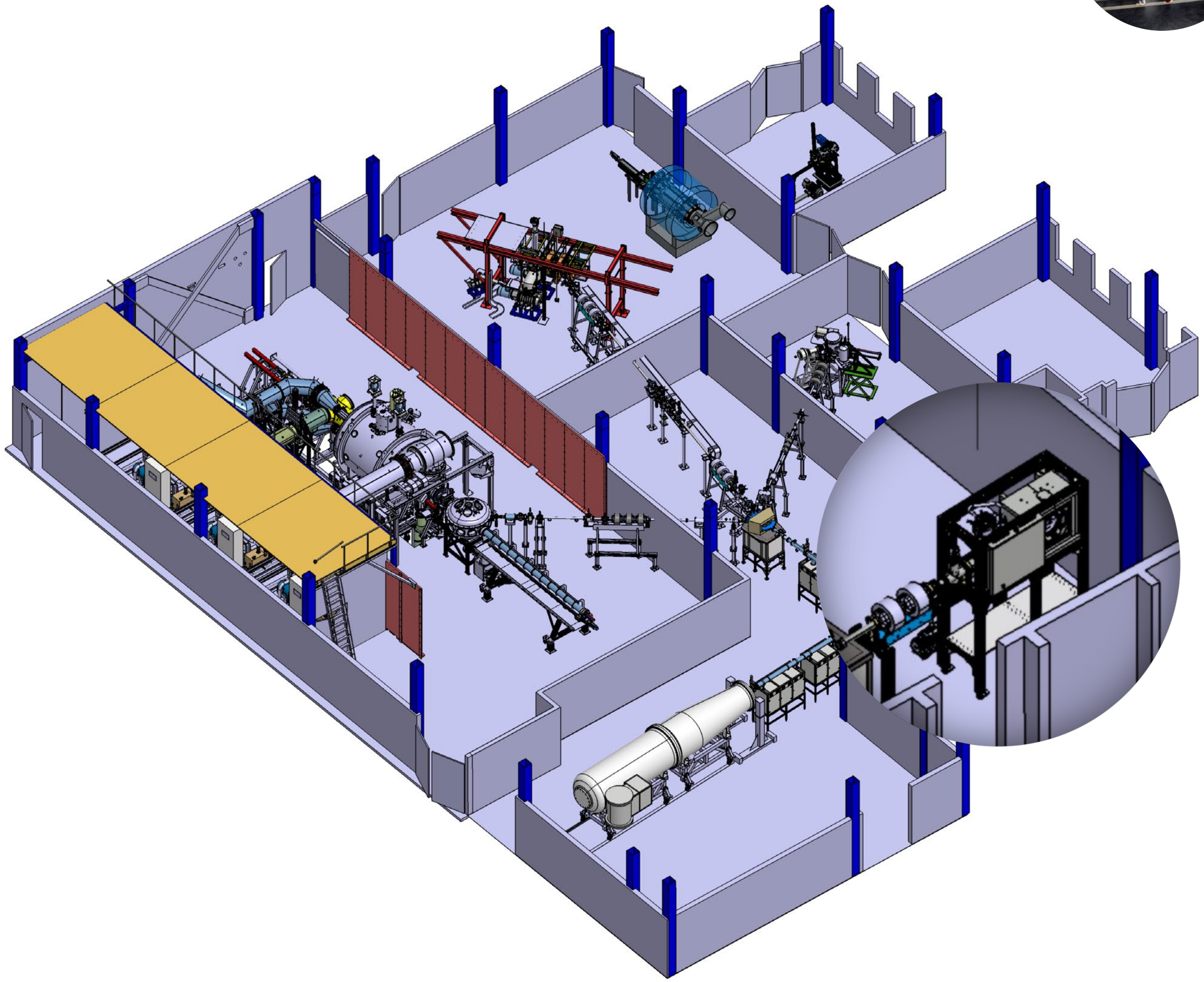

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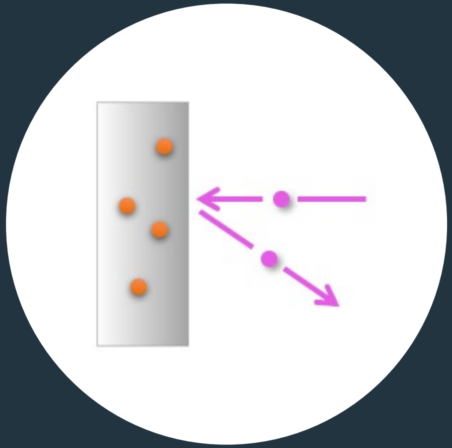

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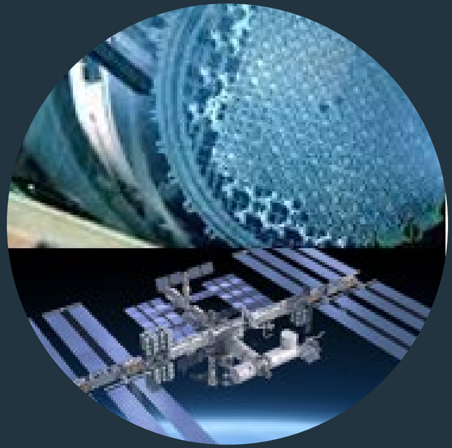

Irradiation

Collaboration

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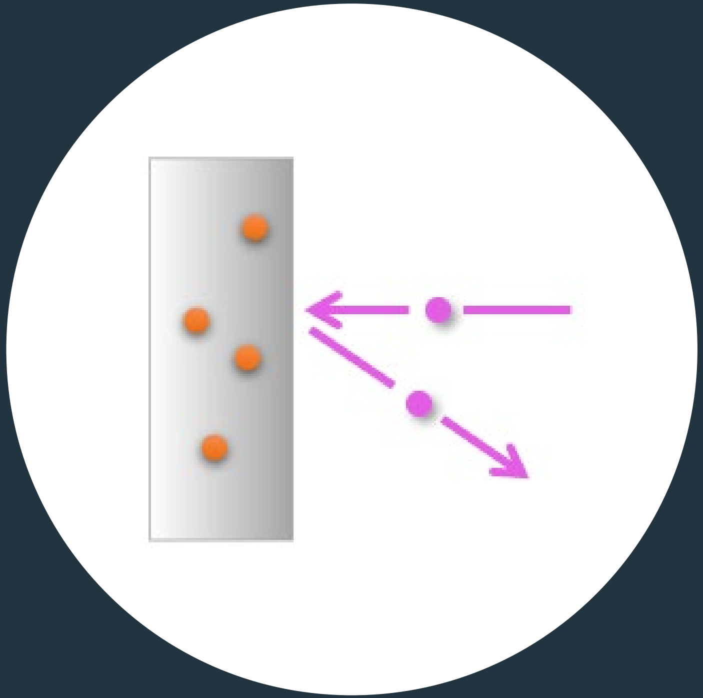

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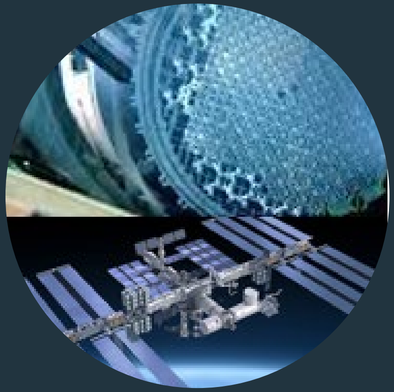
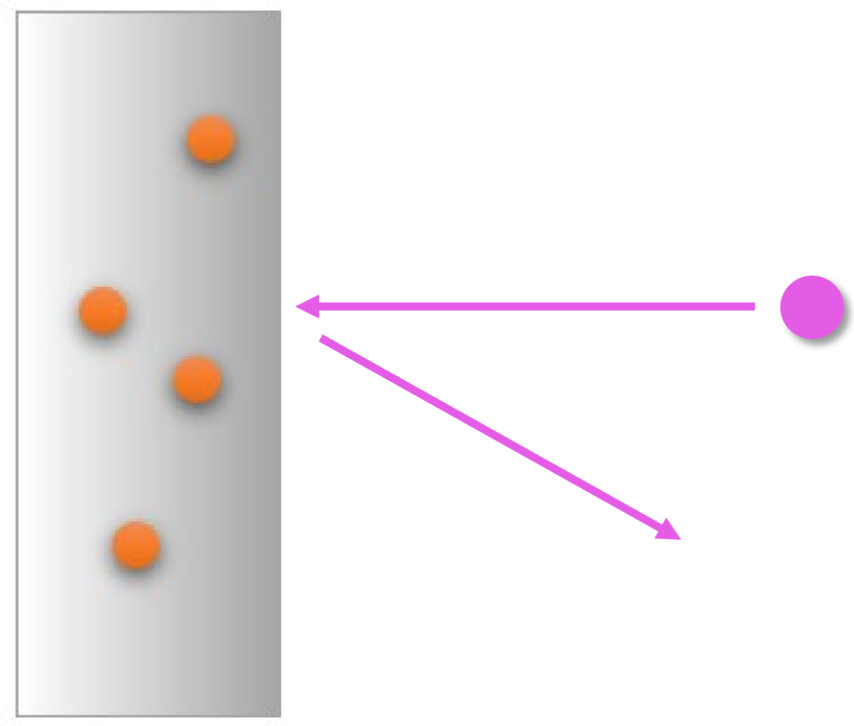
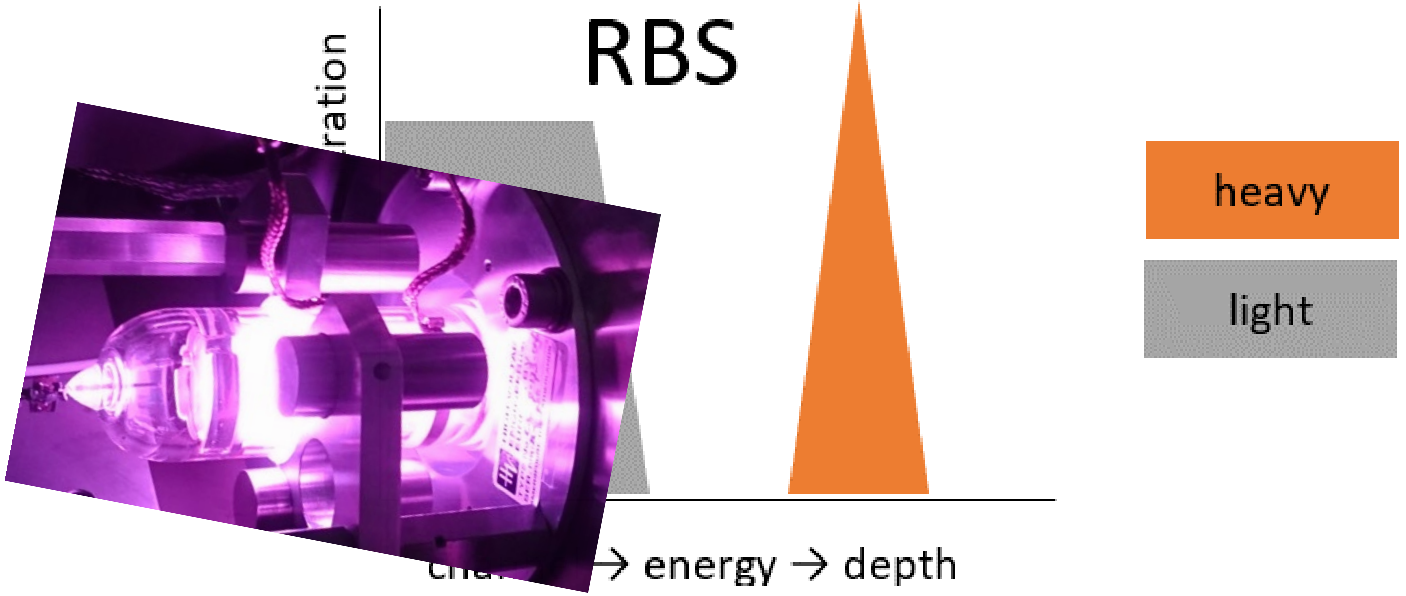


Irradiation

Collaboration

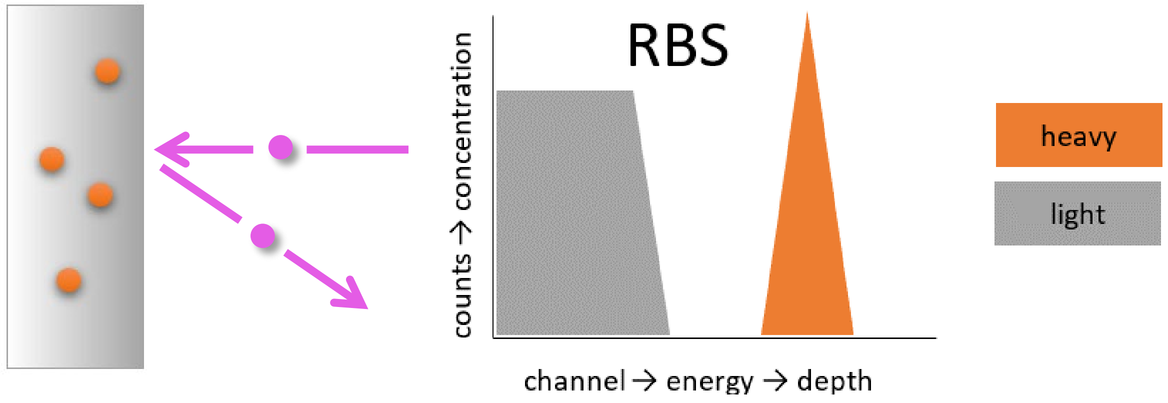
IBA: Rutherford Backscattering





Peak height	→ concentration
Peak width	→ layer thickness
Peak area	→ total amount
Spectrum shape	→ porosity, roughness

IBA techniques



IBA techniques

RBS: heavy element in light matrix

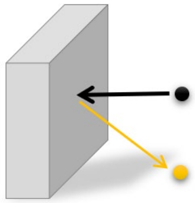
NRA: light element in heavy matrix, trace impurities

ERD: hydrogen quantification

PIXE: multi-elemental, high signal-to-background ratio, detection limits close to 1 ppm

PIGE: every single isotope of low-Z elements

Ion Beam Analysis



IBA

- Depth profiling & elemental composition
- Quantitative without standards
- Non-destructive
- No sample preparation
- Fast and cheap

Parameters

Information depth: up to 15 µm

Sensitivity: 1 – 1000 ppm

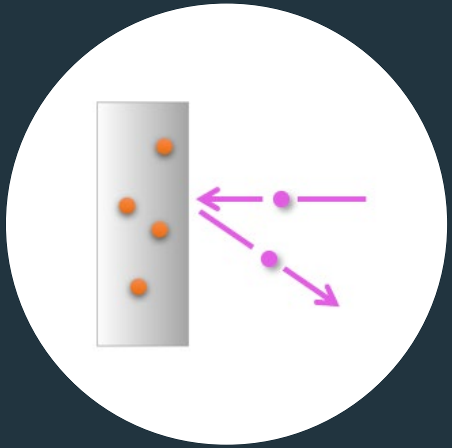
Accuracy: down to 1%

Depth resolution: down to 2 nm

Measurement time: 5 to 60 min per point

Applications: material science, geology, art, surface quality, etc.

IBF lab



Physics

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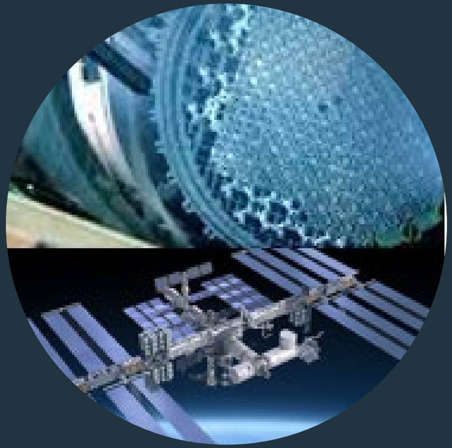
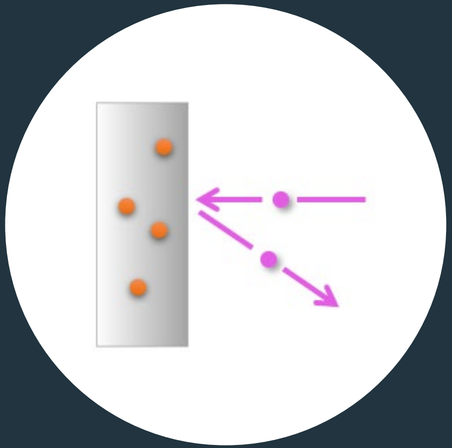


Irradiation

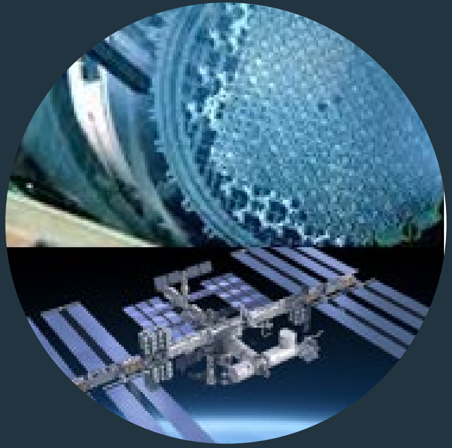
Collaboration

IBF lab



Physics

Material Analysis



Irradiation

Collaboration

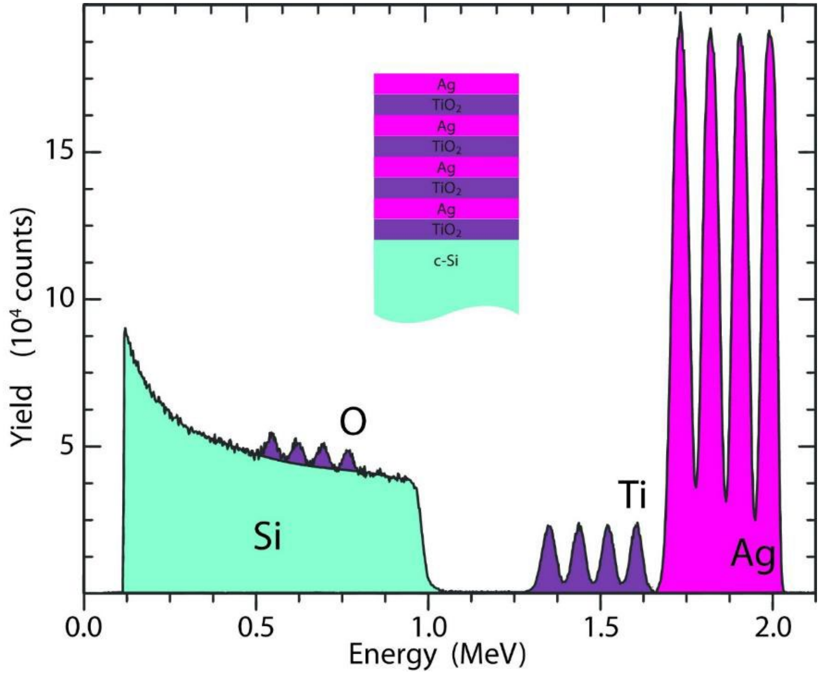
Case 1: multi layer mirrors

Objective: test the accuracy of the deposition technique

~40 nm thin layers are resolved with accuracy of 0.1 nm (RBS)

layer#	10^{15} at/cm^2	comp.	nm*
1	268	Ag	46.2
2	369	TiO_2	39.2
3	281	Ag	48.4
4	371	TiO_2	39.4
5	281	Ag	48.4
6	379	TiO_2	40.2
7	285	Ag	49.1
8	399	TiO_2	42.3

*) Assuming bulk densities



Courtesy of Wim Arnold Bik, www. detect99.nl

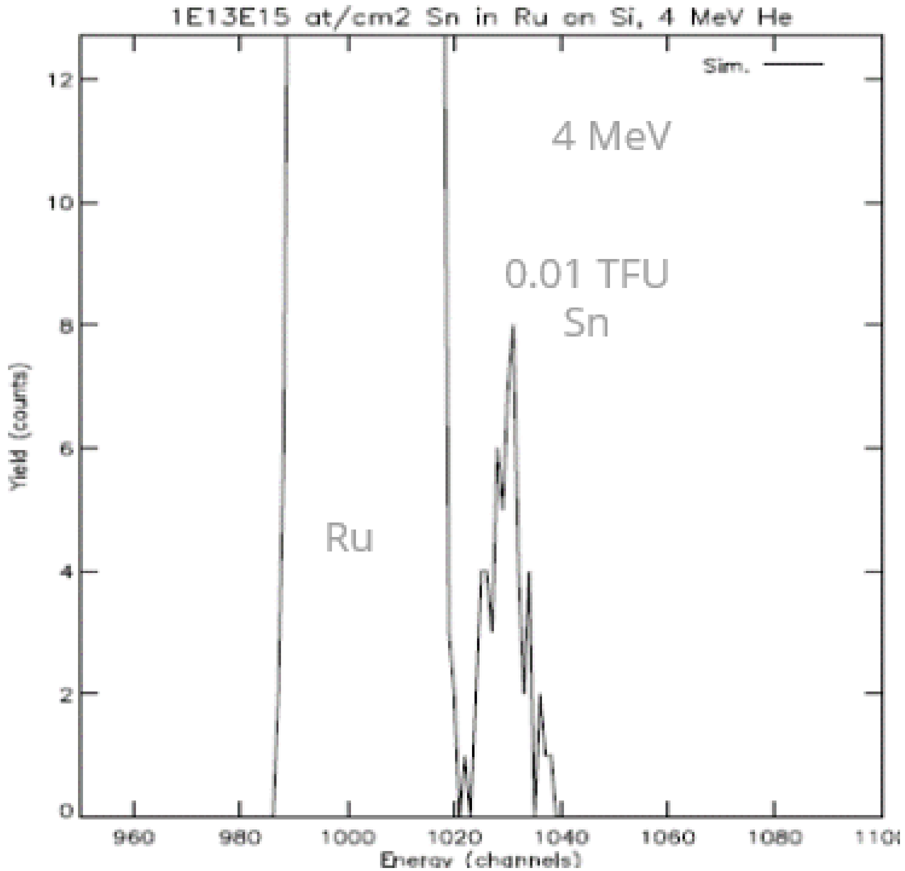
Case 2: tin contamination of EUV mirrors

Objective: test the efficiency of the cleaning technique

Courtesy of Wim Arnold Bik, www. detect99.nl

Material: Ru-capped Si/Mo MLM contaminated by Sn plasma

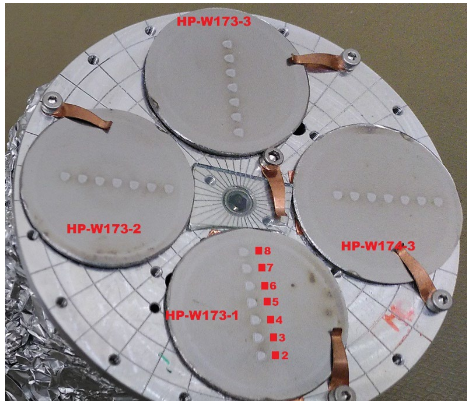
Result: 2 nm of Sn

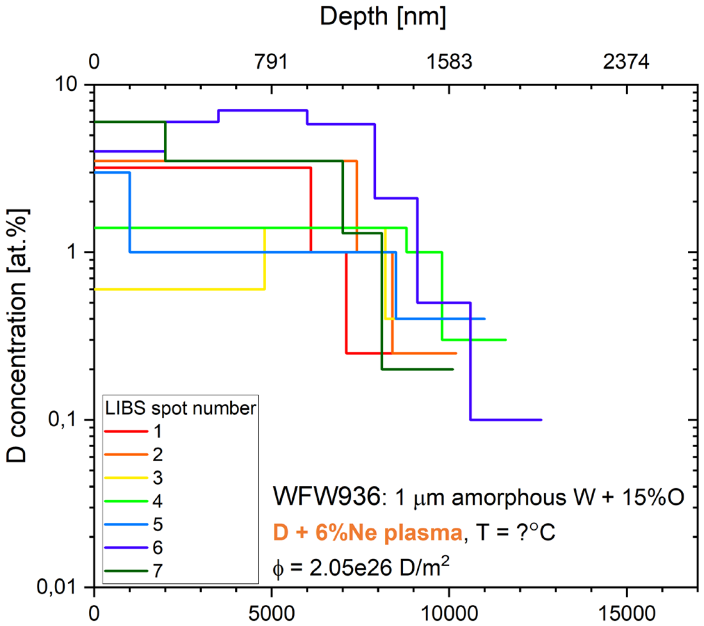


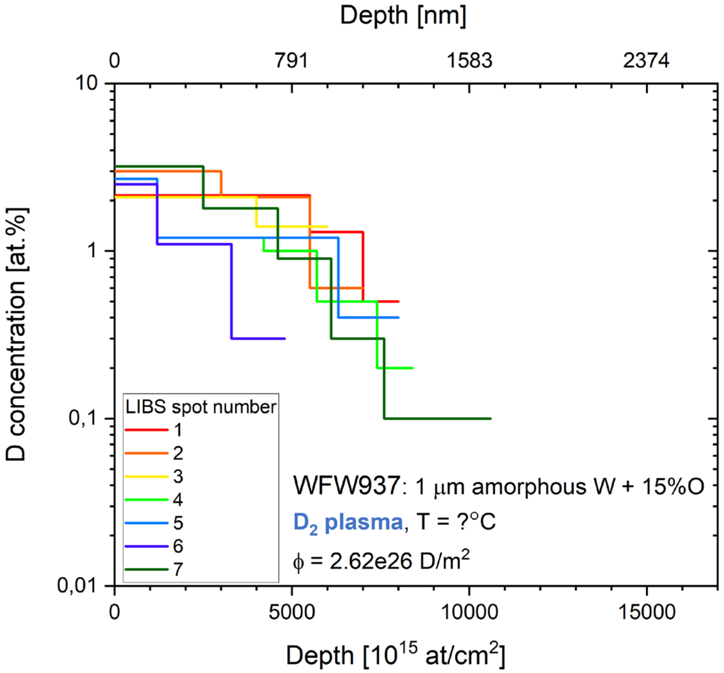
Case 3: fusion tungsten

Objective: determine deuterium depth profiles, Compare results to LIBS

Material: 3 cm ø W discs exposed to deuterium plasma seeded with impurities





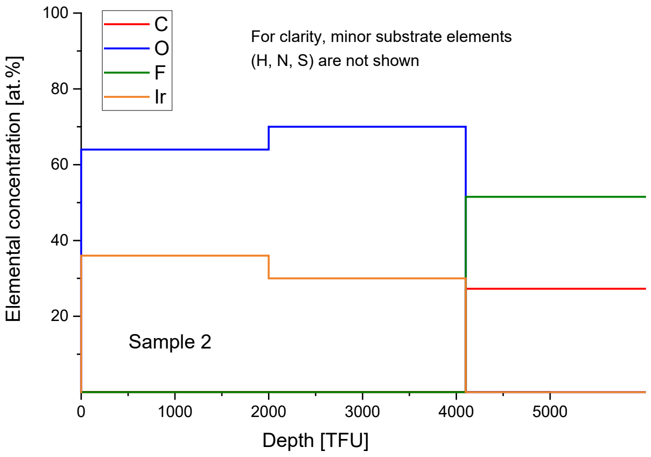


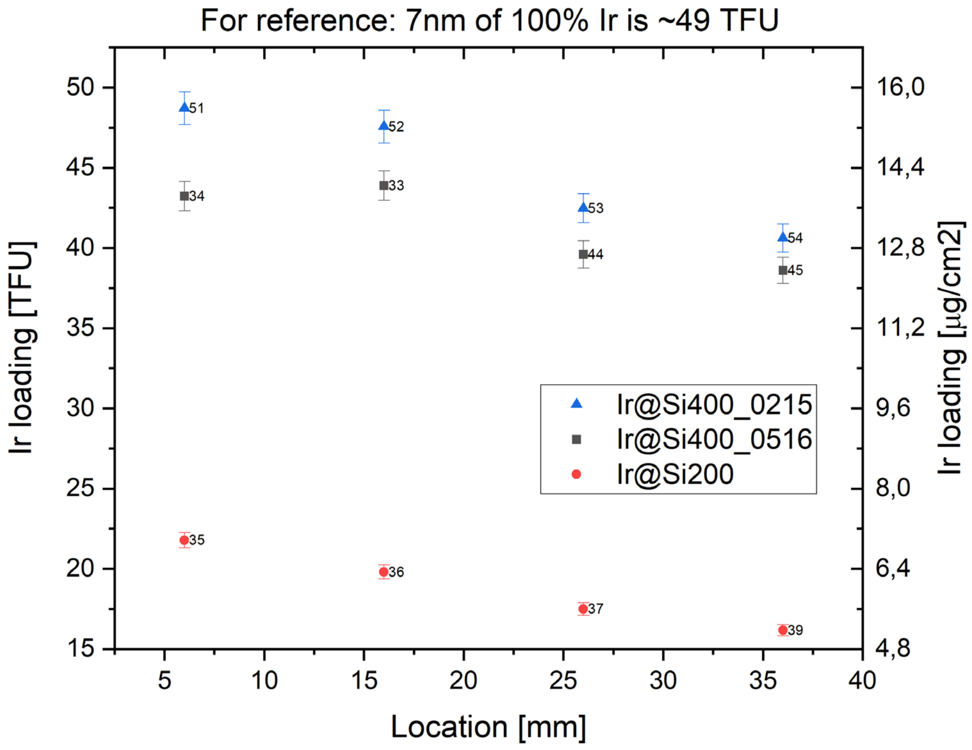
Case 4: catalyst -coated membrane

Objective: determine Ir loading, depth profiles & deposition uniformity

Aim: engineer CCMs with homogeneous catalyst layers at low Ir loadings

Material: IrOx on polymeric membrane



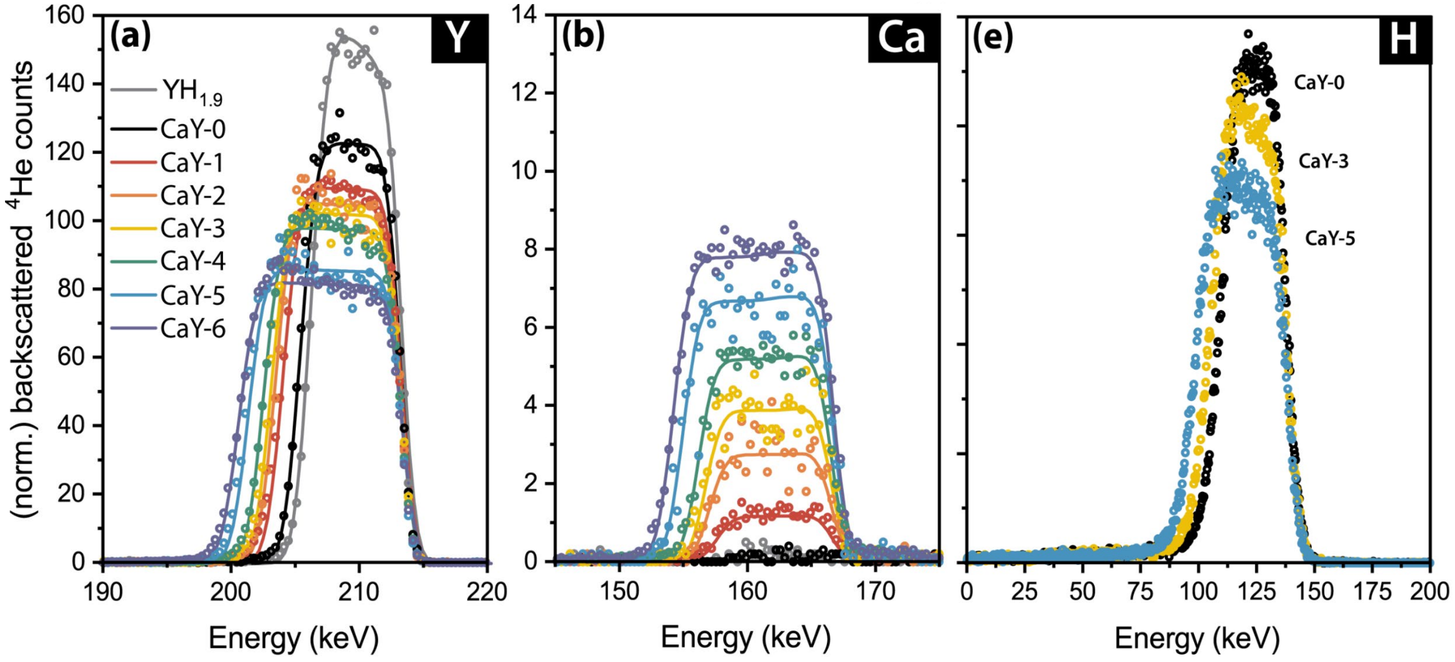


Case 5: Photochromism

Objective: correlate the amount and type of anion vacancies to photochromic properties

Material: Y oxyhydride doped with Ca to create O^{2-} and H^{-} anion vacancies

When Ca↑, O remains constant and amount of H↓ → H vacancies ↑



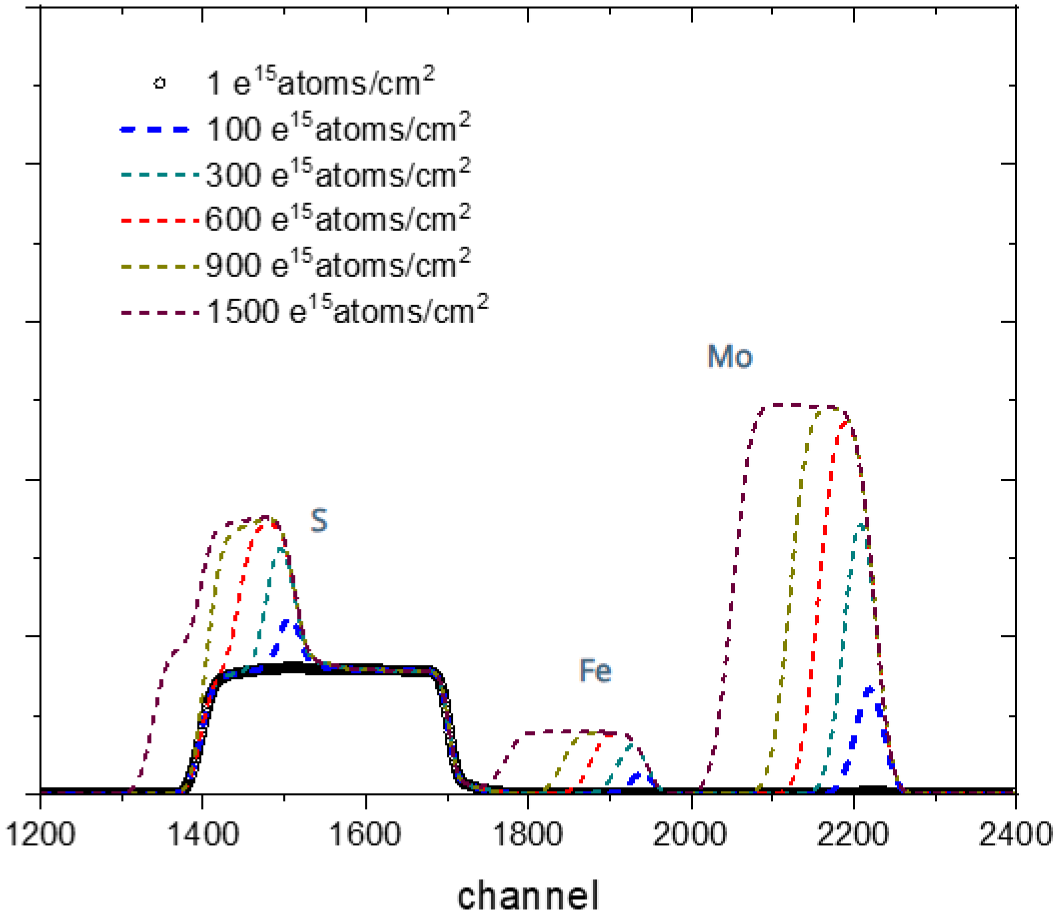
Case 6: electrolysis (June ‘24)

Objective: measure dynamic changes to the electrode and correlate it with the electrolysis parameters

Material: FeMoSx

Electrode build up from 10 nm to 150 nm

Continuous measurements for 10 hours



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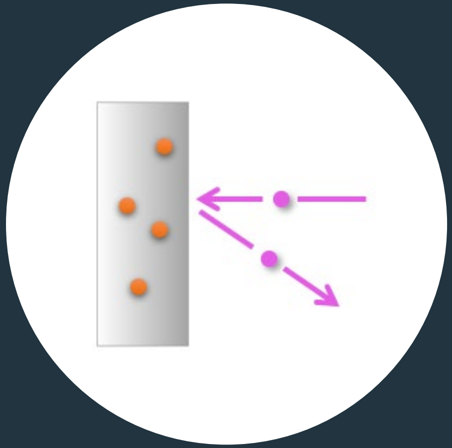
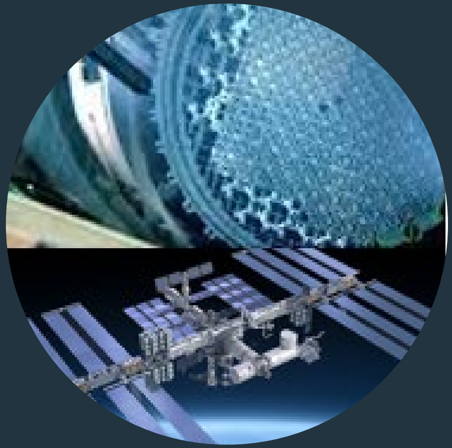
IBF lab



Physics

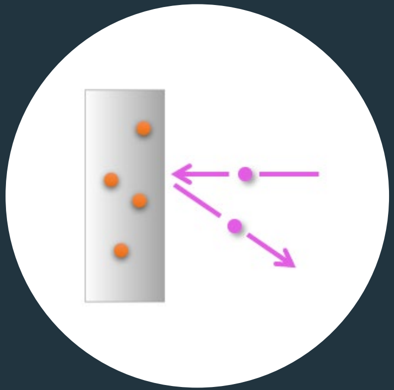
Material Analysis



Irradiation

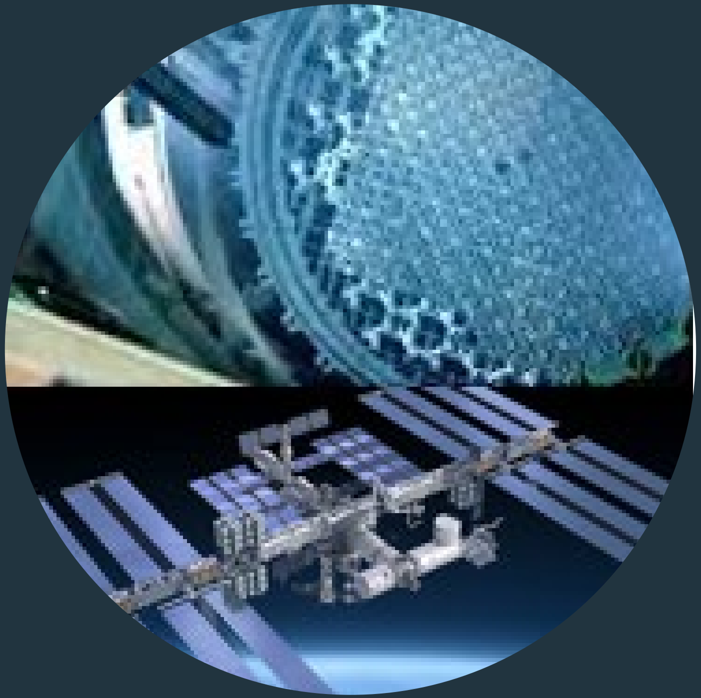
Collaboration

IBF lab



Physics

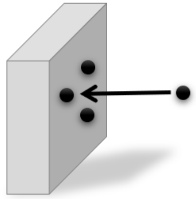
Material Analysis



Irradiation

Collaboration

Irradiation



Ion irradiation

- Defect creation & material modification

Applications

- Proxy for fusion/fission neutrons
- Cosmic rays (FPGAs, diamond detectors)
- Plant mutation
- Semiconductors → not at DIFFER

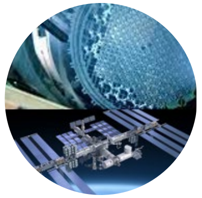
Parameters

Particles: protons or 4He

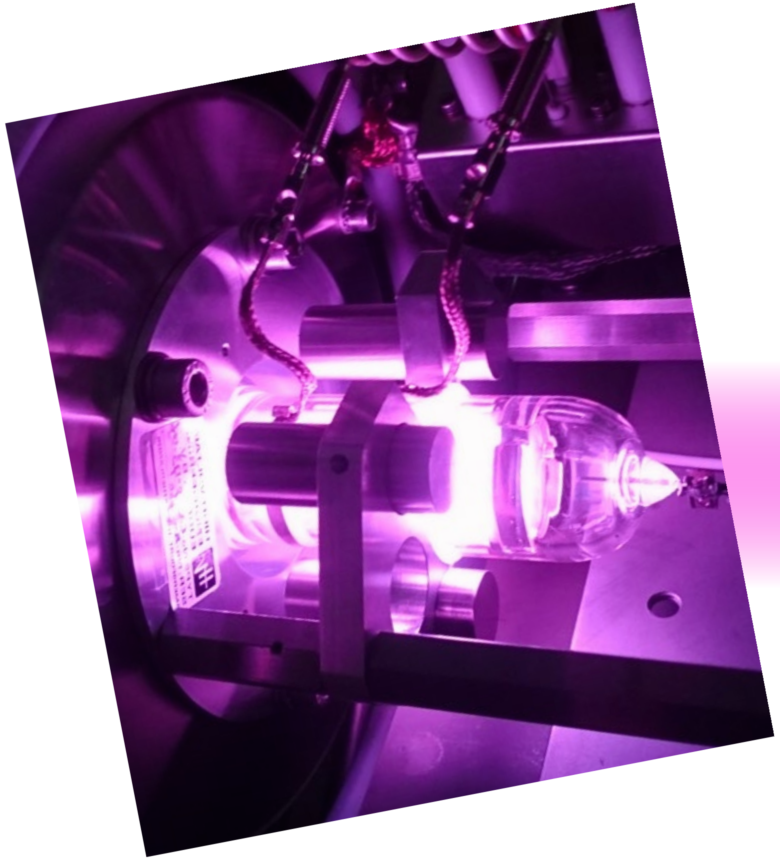
Energy: up to 3 MeV p, 4 MeV 4He

Temperature: up to 1000°C

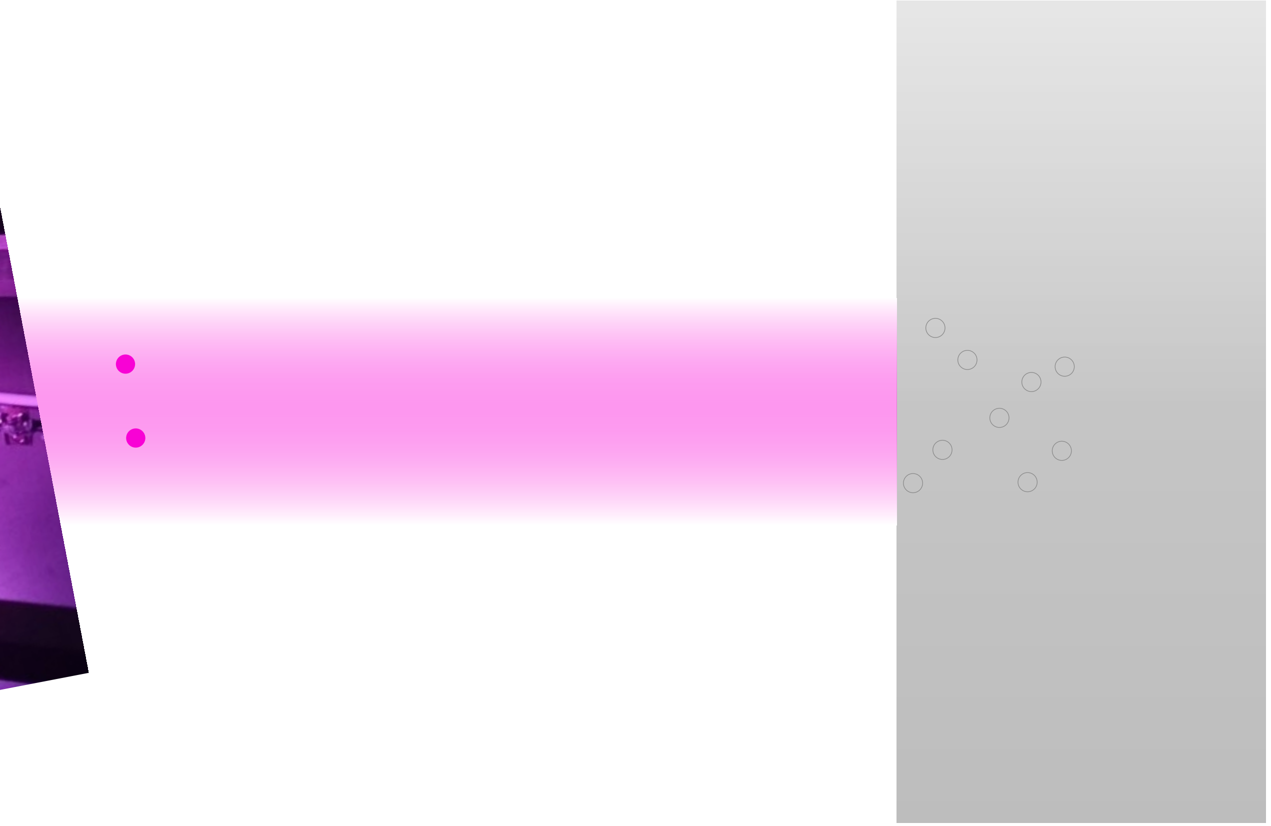
Current: 100 pA/cm^2 up to 50 µA/cm^2



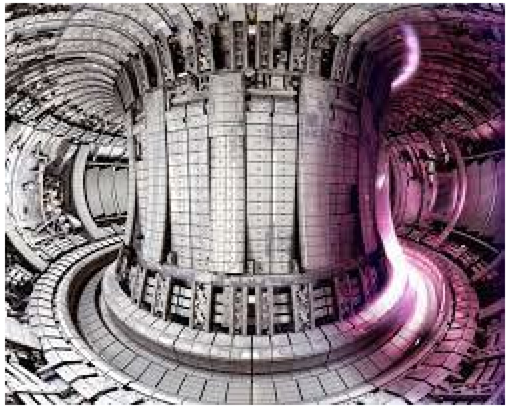
Case 1: only irradiation



fast particles



material



Case 2: cosmic rays in FPGA

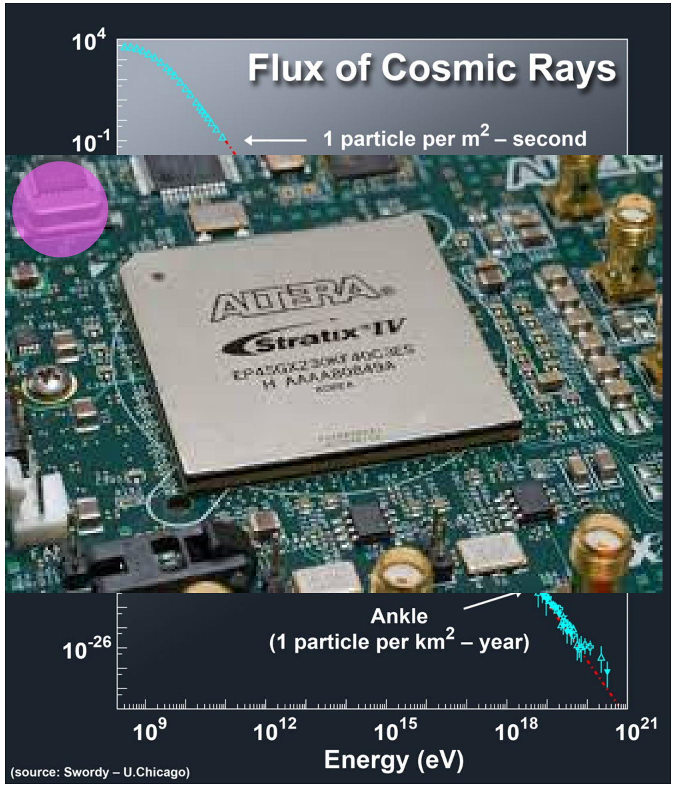
Objective: Use proton -irradiation as a failure generator to test SEU mitigation techniques

Cosmic rays

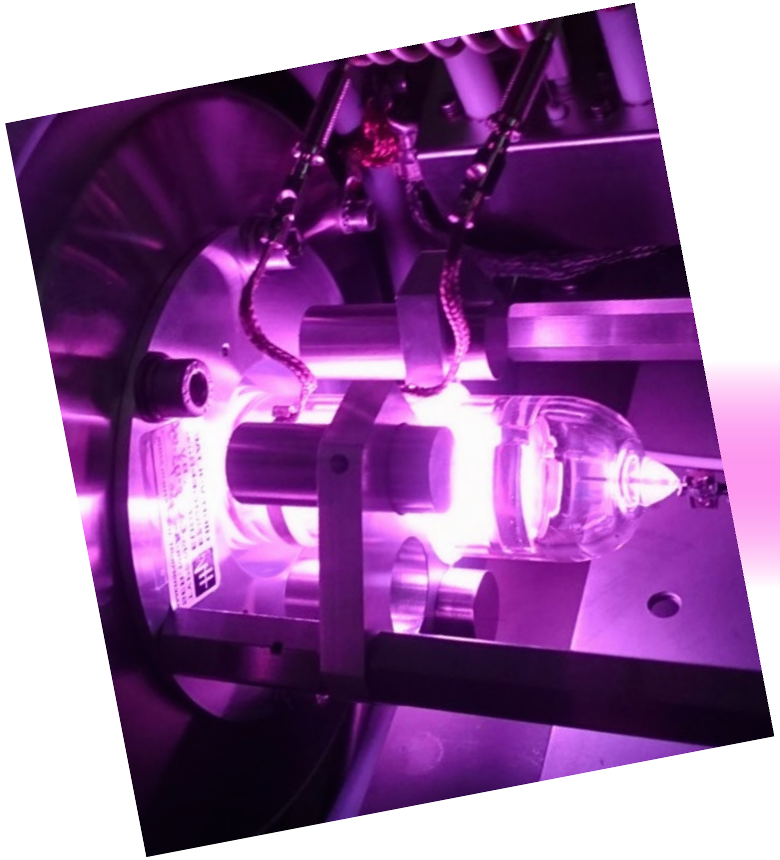
- Protons, atomic nuclei
- Energy: 1 GeV+😱😱
- Flux: 6e4 p/(m^2s) at 1 GeV p
- SEU/month = 1
- Random spot

Singletron

- Protons
- Energy: 3 MeV
- Flux: 6e11 p/(m^2s) (100 pA)
- SEU/s = 4
- Targeted spot



Case 3: simultaneous irradiation -corrosion (June '24)



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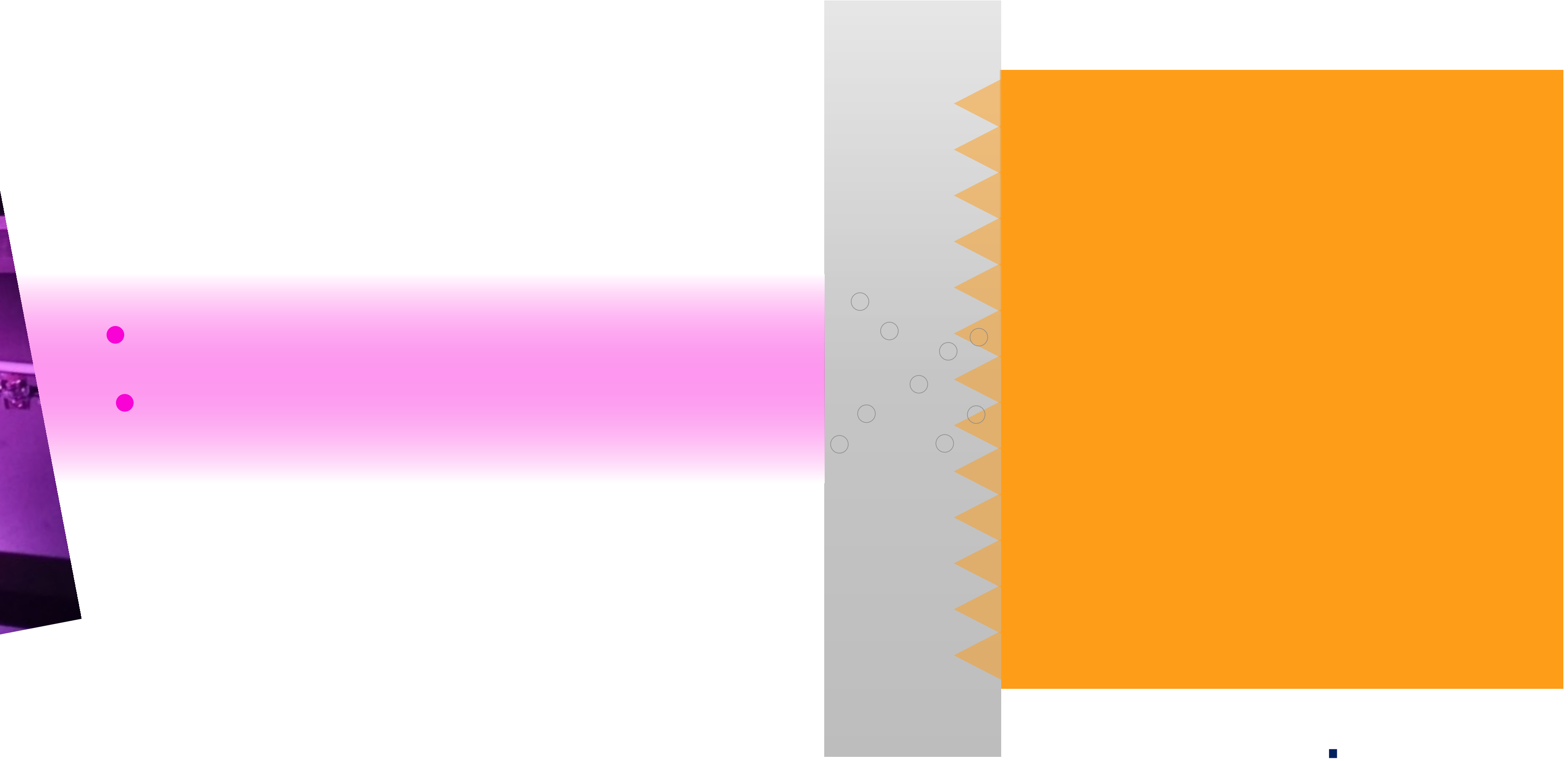
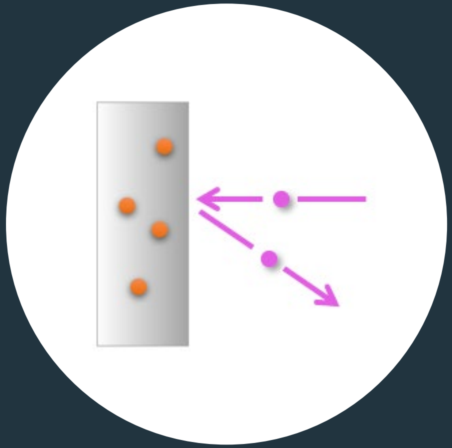


30-60 µm sample

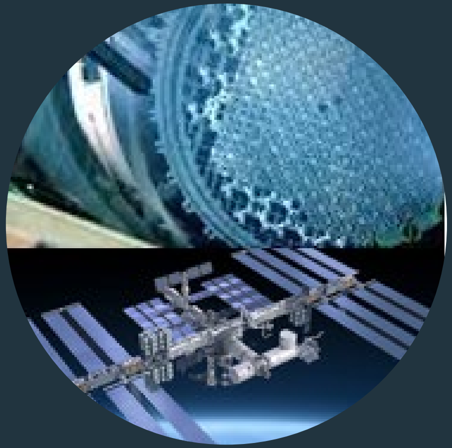
corrosive material

IBF lab



Physics

Material Analysis



Irradiation

Collaboration

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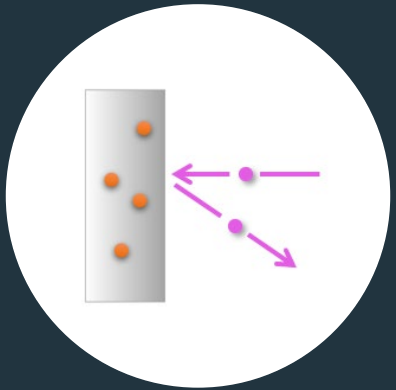

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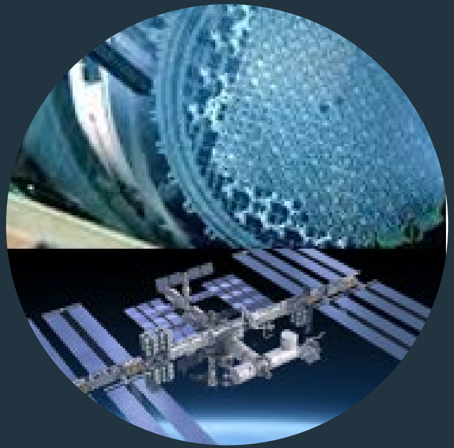

Irradiation

Collaboration

Summary

Share your research problem with me!

- Material composition
- Depth profiling
- Trace impurities
- Interest in isotopes
- Defects – simulate fission/fusion/cosmic rays etc. damage
- He bubbles

Simulation before measurements

Contact

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Request beam time

collaboration ↔ share students ↔ commissioned research

Backup

Case 3: plant mutation

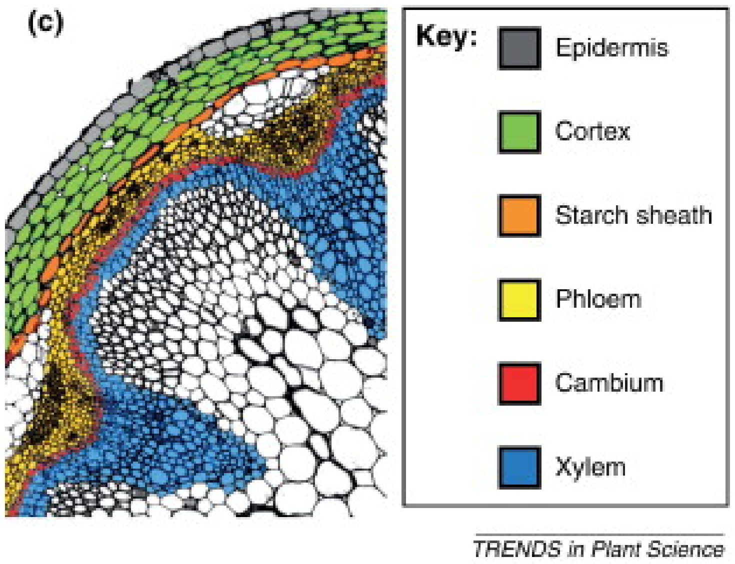
Objective: Use ion -irradiation to change the color and texture of ornamental plants

Cyclotron in Japan

- 220 MeV carbon
- R_p = 1,2 mm
- LET = 107 keV/µm
- 1 Gy = 6e6 at/cm^2

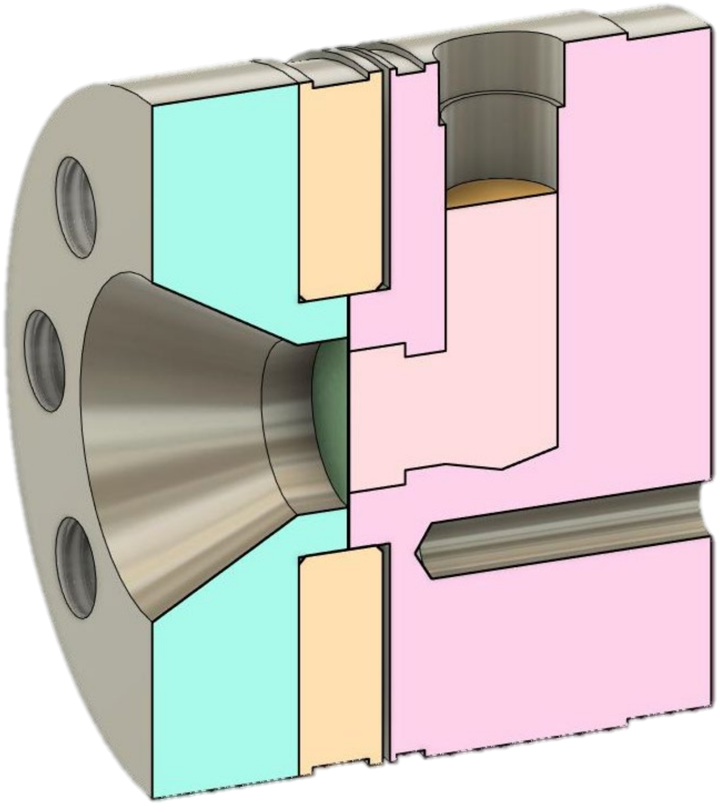
Singletron

- 3 MeV hydrogen
- R_p = 150 µm
- LET = 160 keV/µm
- 1 Gy = 4e6 at/cm^2 → 1s/cm^2



https://doi.org/10.1016/j.tplants.2011.11.004

DICE parameters

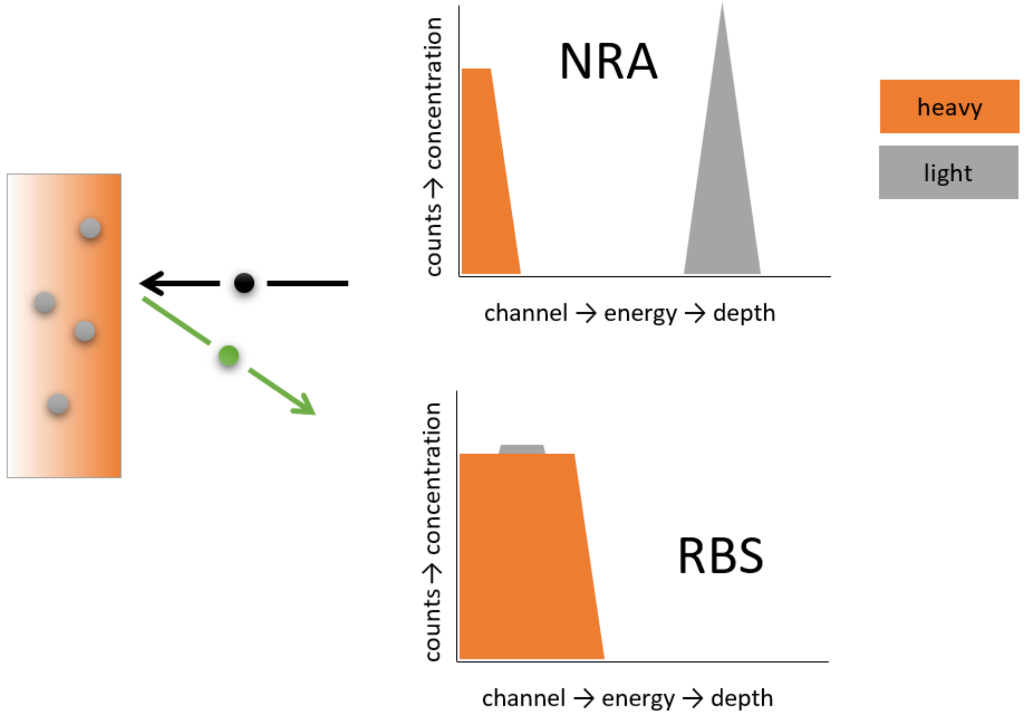


- **Simultaneous** irradiation -(salt) corrosion experiments
- Salt circulation, i.e. dynamic salt
- Thorium salt
- Operation time: up to 100 hours
- 1 year MSR = 5 days DICE
- Temperatures: up to 1000ºC
- High beam currents: up to 50 $\mu A/cm^2$
- Easy sample/salt loading
- Control temperature
- Reliable and absolute current measurements
- Shielding to protect against gammas and neutrons
- No cell corrosion products in the salt
- Adaptable design for other experiments
- Vacuum 10^{-6} mbar

IBA – Nuclear Reaction Analysis

NRA – light elements in heavy matrix

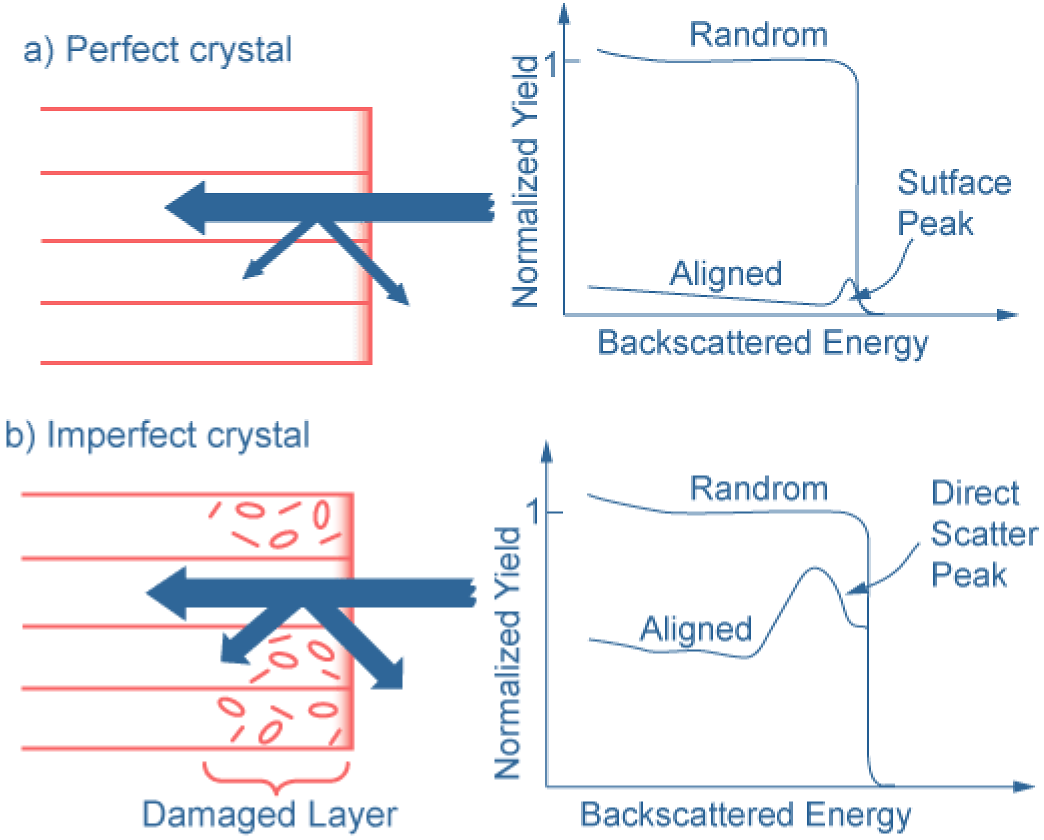
- Low Z elements
- Tracing impurities
- Isotope sensitive
- No high -Z background
- Requires expensive detectors
- Needs measured cross -sections



IBA – RBS/channeling

RBS/C – crystal perfection

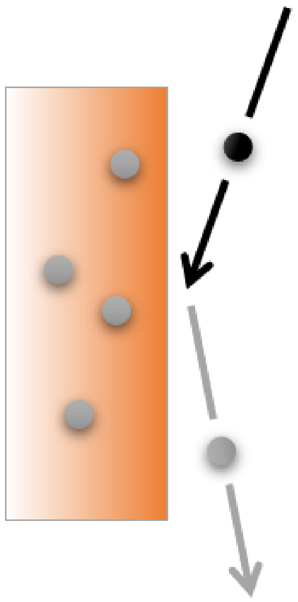
- Degree of damage
- Amorphization

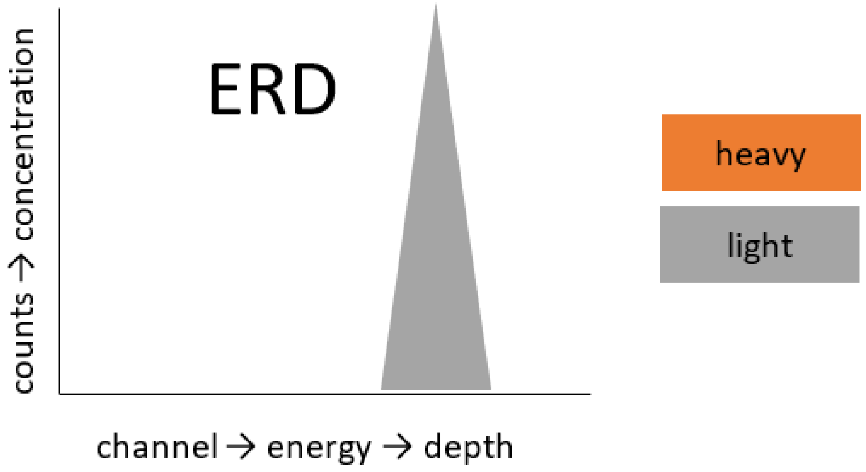


IBA – Elastic Recoil Detection

ERD – light element profiling

- Simpler than NRA
- Simultaneous multi -element depth profiling
- Shallow depth





PIXE and PIGE

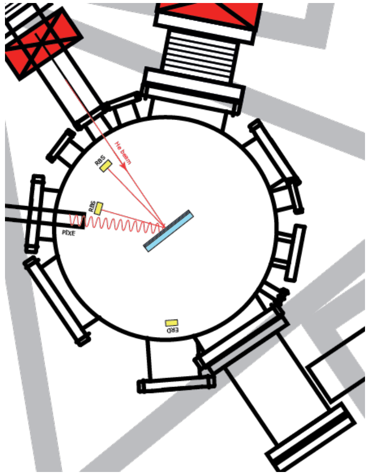
PIXE – particle -induced X-ray emission

Multi -elemental, high signal to background ratio, nondestructive, very sensitive   - detection limits close to 1 ppm.

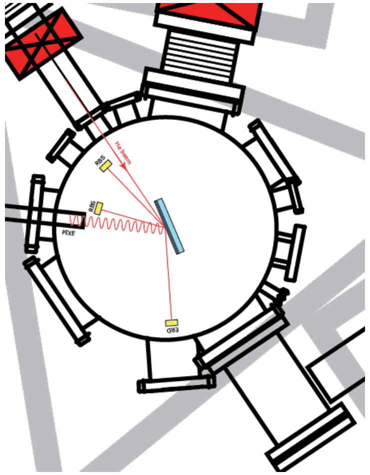
PIGE – particle -induced gamma emission

For low -Z elements, gamma -ray energies, are a characteristic “fingerprint” of every single isotope

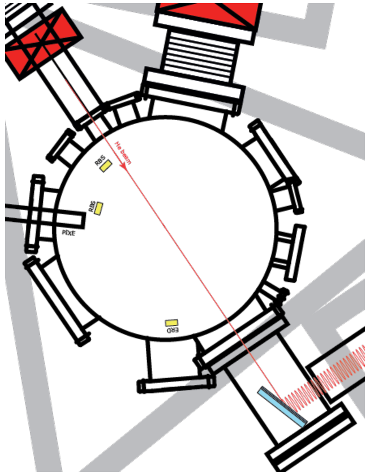
RBS+NRA+PIXE



ERD+RBS+PIXE



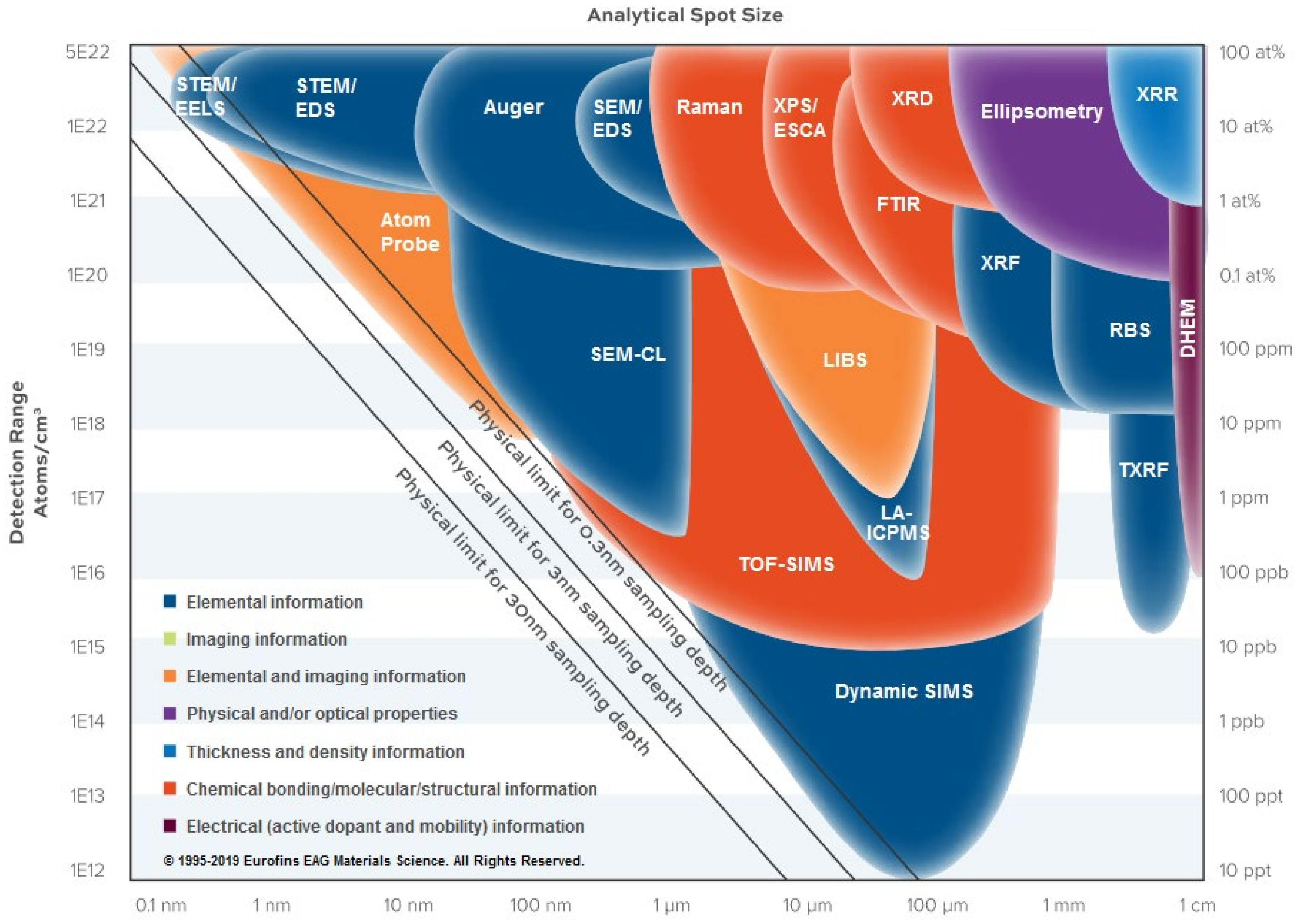
PIGE



Sensitivity comparison

RBS vs. SIMS

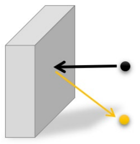
- Quantitative
- Non-destructive
- Larger beam spot
- Faster
- More accurate depth profiling



Technique comparison

	SIMS	XTEM	SAM	GD-OES	XPS	LA-ICP-MS	IBA
Primary beam	keV ions	~100 keV electrons	~100 keV electrons	Plasma	X-rays[a]	Pulsed laser	~3 MeV light ions ~30 MeV heavy ions
Detected signal	Sputtered ions	Primary electrons in phase contrast	Auger[a] electrons	Visible photons[h]	Photo-electrons	Evaporated ions	X-rays[a]; nuclear reaction products: scattered primaries, target recoils and γ-rays
Destructive[b]	Yes	Yes	Yes	Yes	Yes	Yes	No
Depth resolution[c]	2 nm	0.1 nm	2 nm	20 nm	2 nm	50 nm	2 nm
Information depth[d]	500 nm	100 nm	500 nm	50 μm	500 nm	~5 μm	15 μm
Lateral resolution[e]	50 nm	0.1 nm	2 nm	1 mm	3 μm	10 μm	500 nm
Elemental imaging	Yes	EELS, EDX[f]	Yes	No	No	No	Yes
Molecular information	Yes	No?[k]	Yes	No?[k]	Yes	No?[k]	No[l]
Ambient analysis	No	No	No	No	No	Yes	Yes
Sample preparation	No	Yes	UHV	No	UHV	No	No
Quantitative	?[g]	No	Yes	Yes	Yes	Yes	Yes
Standards needed	Yes	—	Yes	Yes	Yes	Yes	No
Elemental sensitivity[h]	10^{-8}	10^{-1}	10^{-3}	10^{-6}	10^{-3}	10^{-9}	10^{-6}
Accuracy	—[i]	—	10%	10%	5%	5%	1%

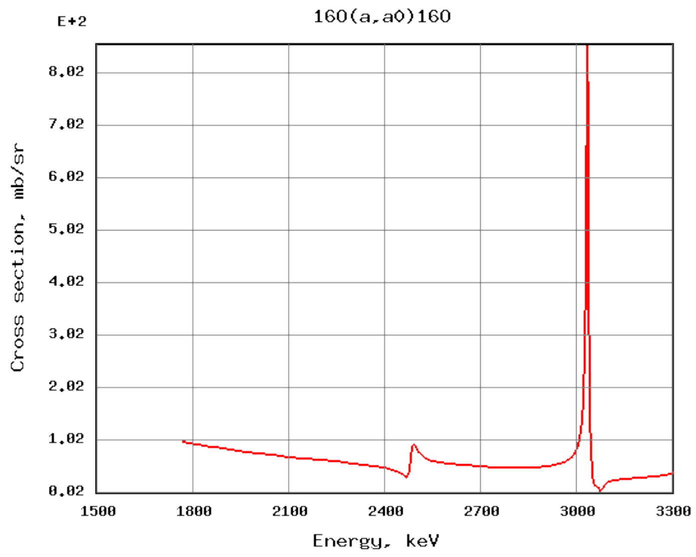
Chris Jeynes& Julien L. Colaux, Analyst, 2016, 141, 5944–5985

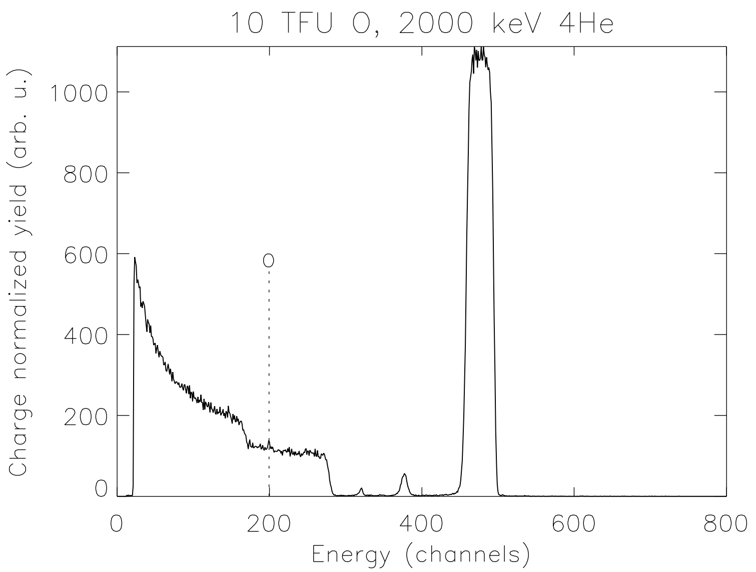


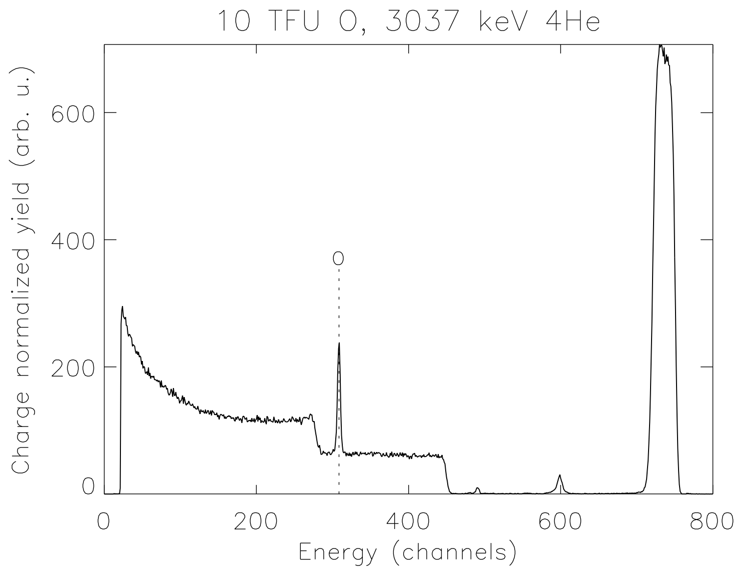
Case 5: resonances

Resonances are used for

- thin -film depth profiling with a very high depth -resolution
- finding traces of elements (impurities) – ultra -high sensitivity







IBF parameters at DIFFER

Technique	Elements	Sensitivity	Depth	Depth resolution
RBS	$Z \geqslant 5$	10 ppm (large Z) - few at.%	2 - 120 µm	> 5 nm
NRA	Z: 1 - 15	Isotope dependent 10^{-3} at.%	< 10 µm	> 50 nm
ERD	H, D	H: 0.1 at.%, D: 100 ppm	< 2,5 µm	~20 nm
PIXE	$Z \geq 13$	1 – 10 ppm for F, Li, B, N	few tens of µm	poor
PIGE	Z: 1 - 15	10 ppm (large Z) – 1 at.%	few tens of µm	Isotope dependent
Irradiation	$Z \geq 3$	--	< 150 µm	--

Final parameters depend on the combination of energy, ions, target, setup, and experimentalist ability to optimize them.