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IAEA-CN301-109

Ion accelerators for modification and analysis of materials

Present status and an outlook towards the future

A perspective from the Uppsala Tandem Laboratory

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International Conference on

Accelerators for Research and Sustainable Development:

From Good Practices Towards Socioeconomic Impact

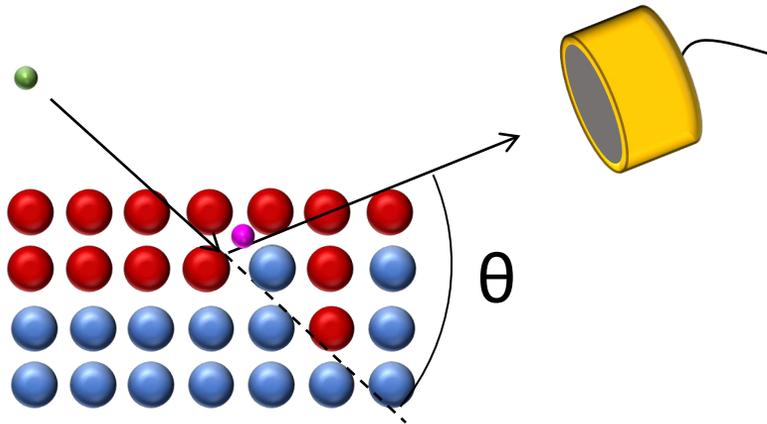


23–27 May 2022

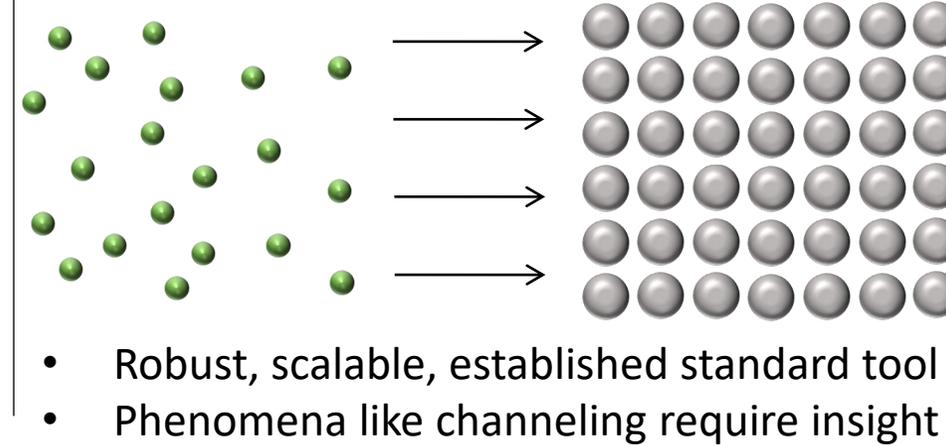
IAEA Headquarters, Vienna, Austria

INTRODUCTION: WHAT CAN WE DO WITH eV – GeV IONS?

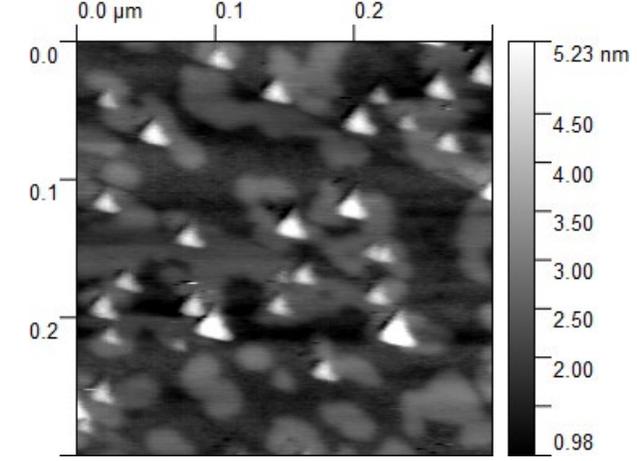
Materials characterization



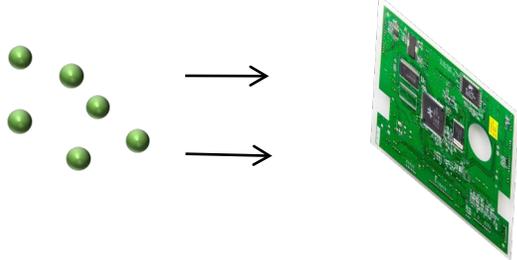
Ion implantation



Materials modification

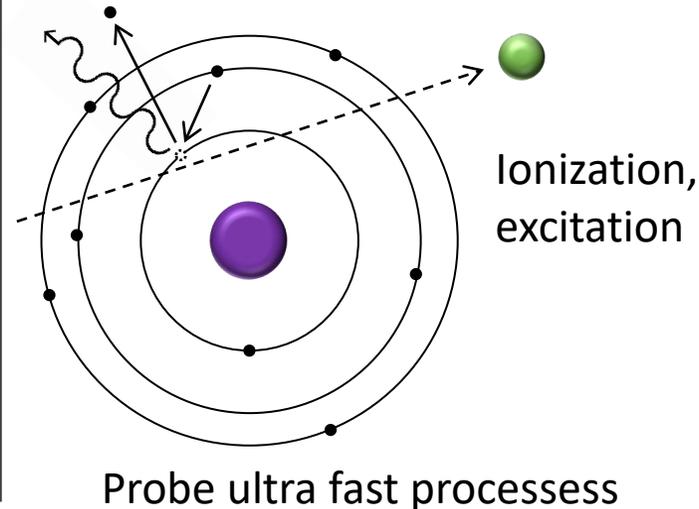


Irradiation

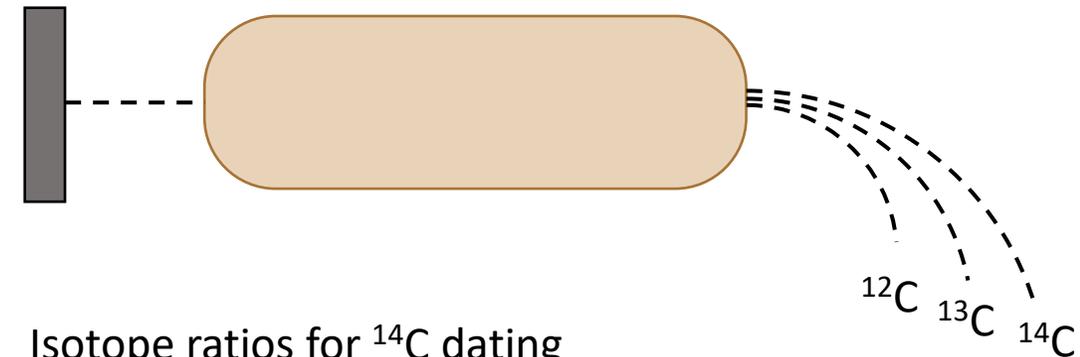


- Damage simulation
- Device testing
- Medical treatments (hadron therapy)

Fundamental physics

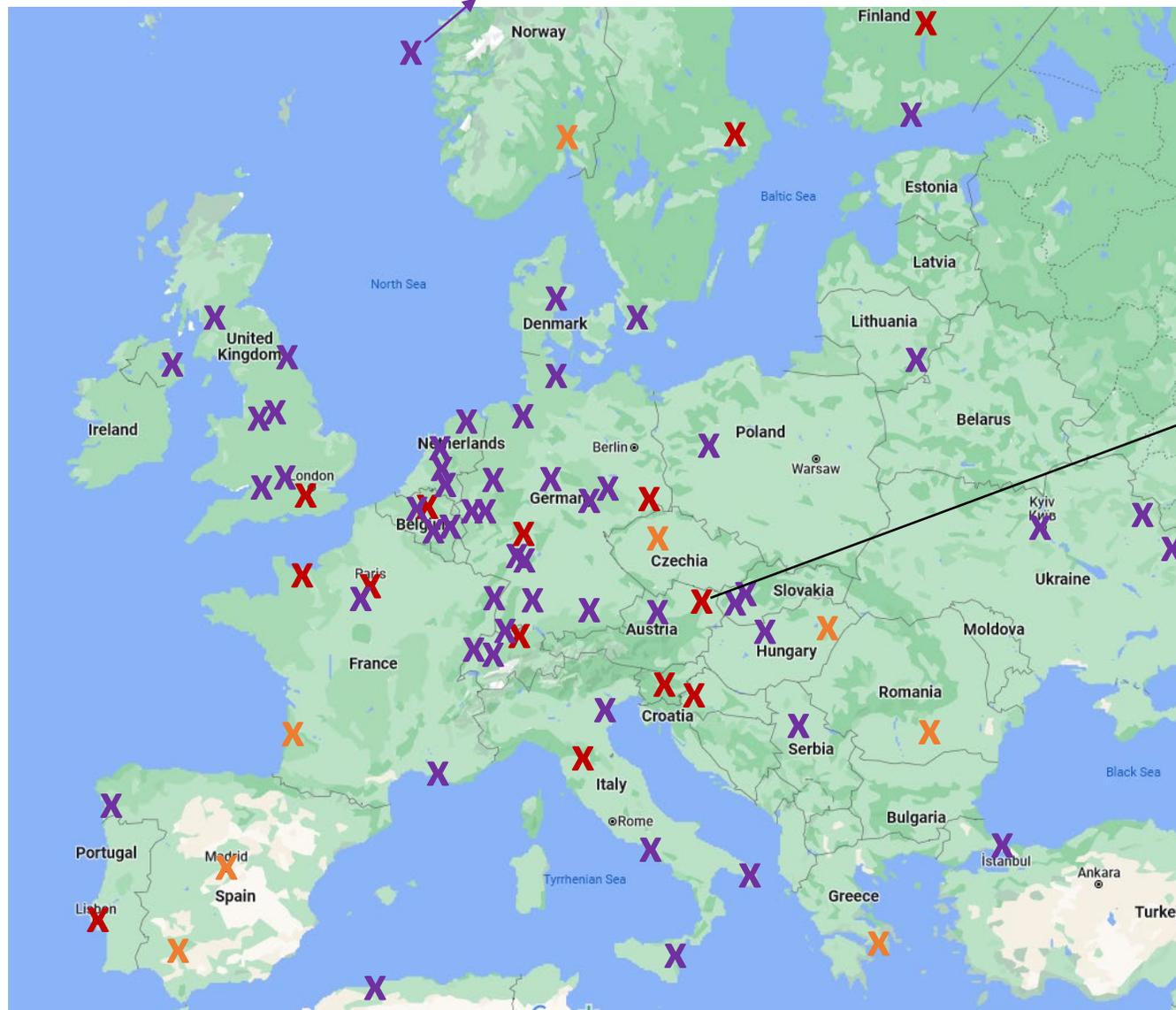


Accelerator mass spectrometry



- Isotope ratios for ^{14}C dating
- Geological surface exposure dating

EUROPEAN ION BEAM INFRASTRUCTURES



[1] IonBeamCenters.eu & RADIATE transnational access, 2022-05-09.

[2] M.A. Ramos, J. Gómez-Camacho, *Focus point on small and medium particle accelerator facilities in Europe*, Eur. Phys. J. Plus **136** (2021), 1219.

[3] IAEA Accelerator Knowledge Portal, Electrostatic Accelerators, <https://nucleus.iaea.org/sites/accelerators/>



Isotope Physics
Research Group

Vienna Environmental
Research Accelerator
(VERA), AMS

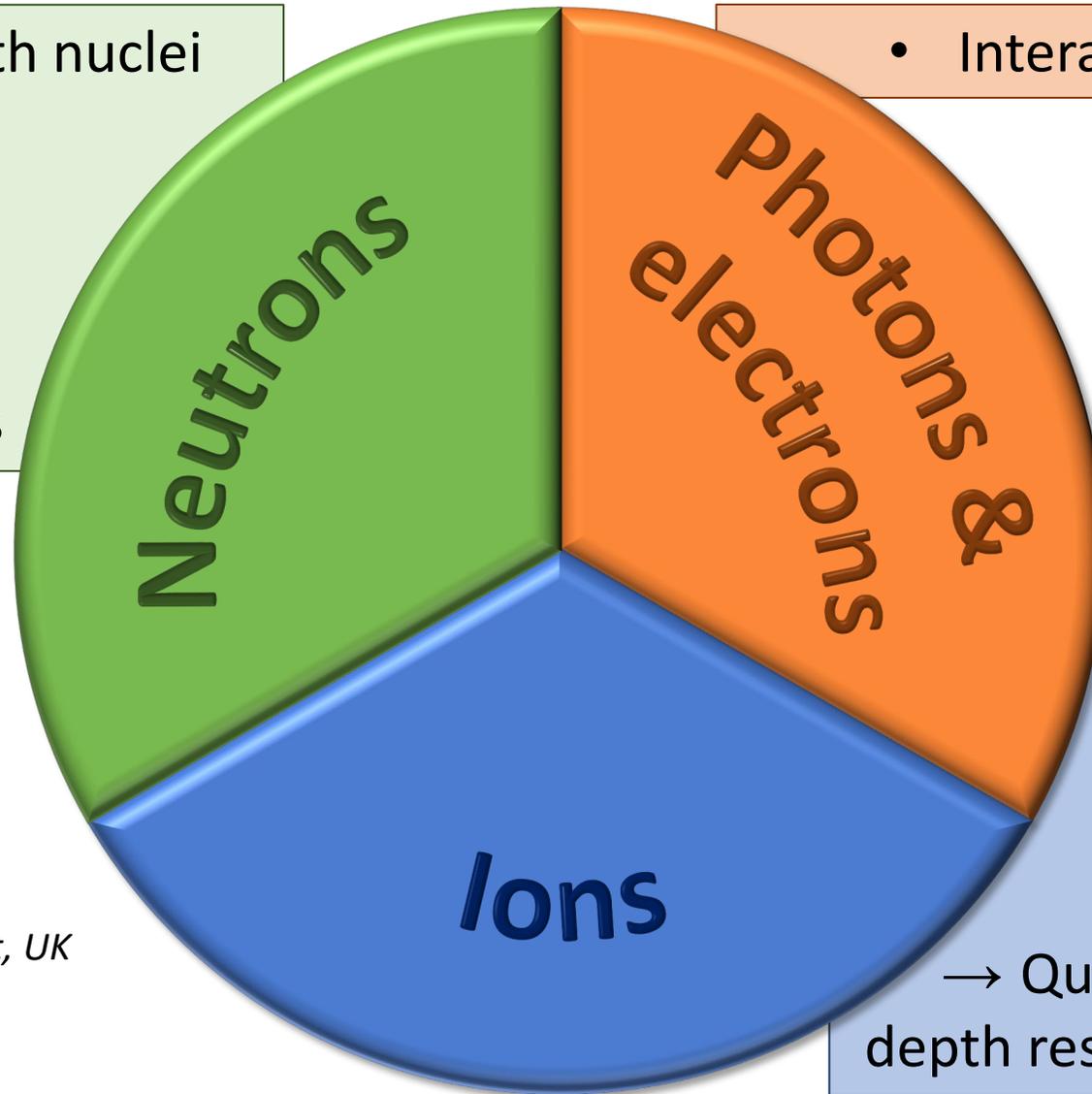


MATERIALS CHARACTERIZATION: WHY IONS AS PROBES?

- Interacts primarily with nuclei
- No charge, but finite magnetic moment
→ Useful for probing magnetic materials



ISIS Neutron and Muon Source, Didcot, UK
Image: <http://neutronsources.org>



- Interact primarily with electrons



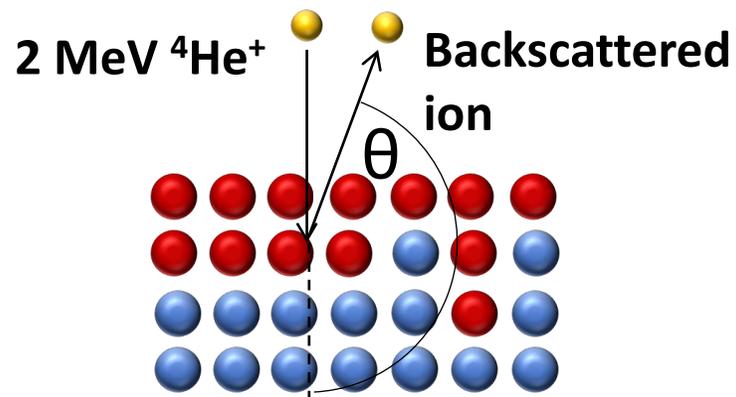
MAX IV, Lund, Sweden
Image: <http://maxiv.lu.se>

Combined probe
Significant interaction with both electrons and nuclei

→ Quantitative, non-destructive, depth resolved composition analysis

OVERVIEW OF ION BEAM ANALYSIS METHODS

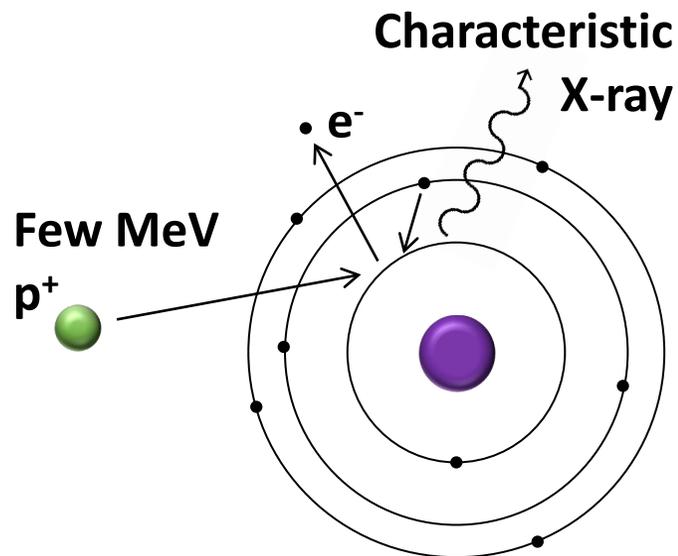
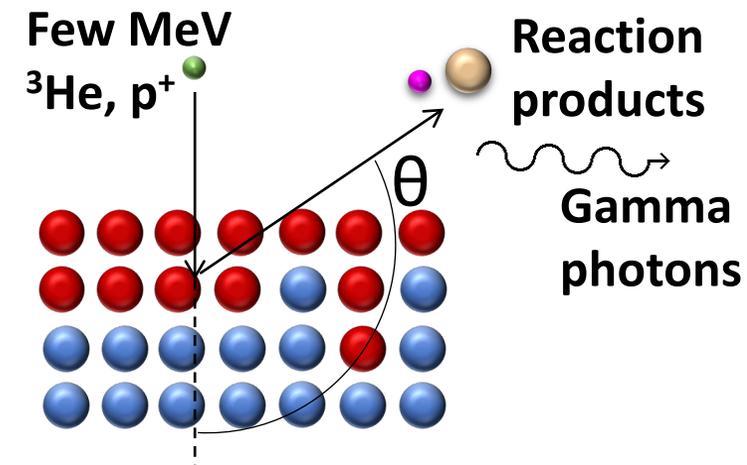
Rutherford Backscattering Spectrometry (RBS)



- Measure: Particle energy spectra
- Reaction **cross sections** give composition
- Tabulated **stopping powers** give depth scale.

Recall presentation by: **Marek Rubel**

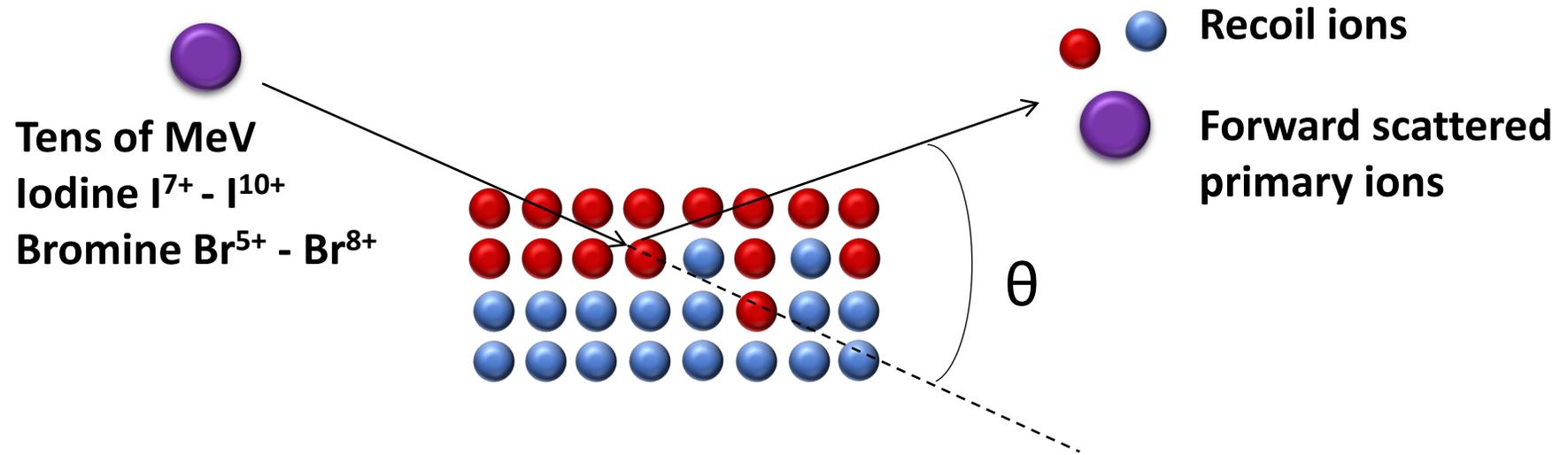
Nuclear Reaction Analysis (NRA)



Particle-Induced X-ray Emission (PIXE)

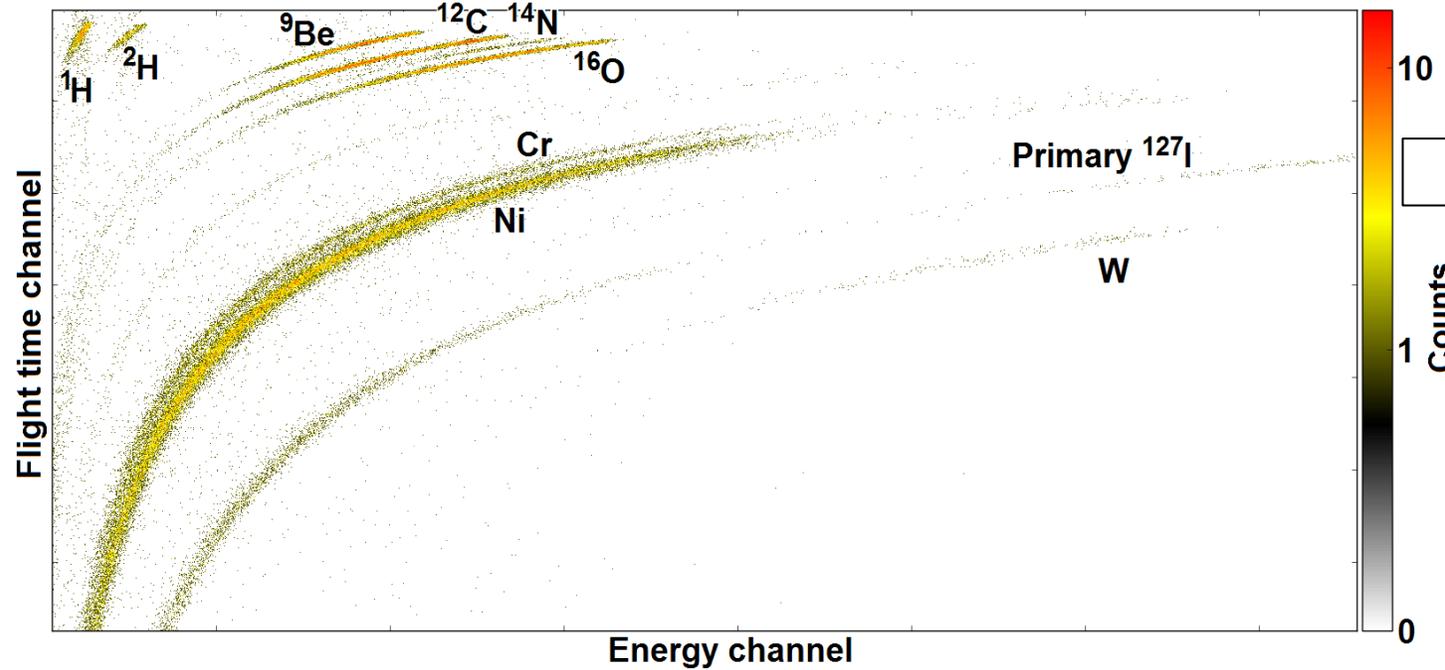
METHOD FOCUS: ToF-ERDA

Time-of-Flight Elastic Recoil Detection Analysis

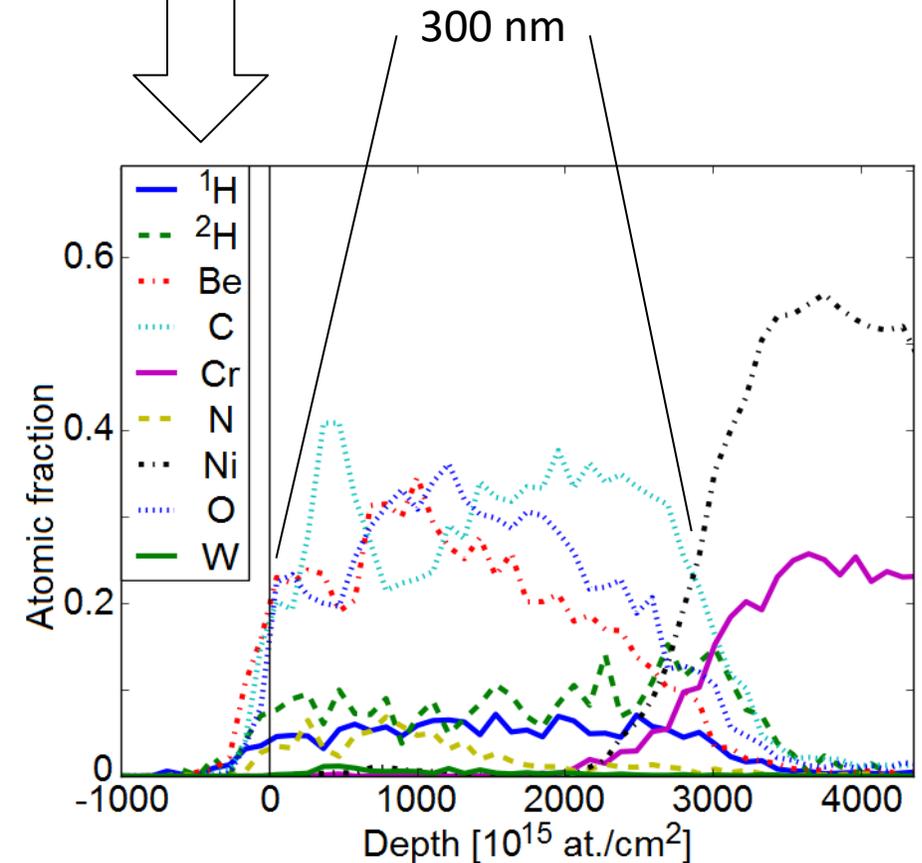


- Detect: Ion velocity and energy \rightarrow mass-based identification
- Full composition depth profiling to $\approx 1 \mu\text{m}$; 10-20 nm resolution
- Quantification across periodic table (including H)
- Provides $< 1 \text{ at.}\%$ sensitivity
- Ideal for light elements in heavy matrix

METHOD FOCUS: ToF-ERDA



P. Ström et. al., J. Nucl. Mater.
516, 202-213 (2019)



Example: Mixed layer deposited inside plasma confinement device for **nuclear fusion research**.

Depth variation of elemental concentrations in 300 nm thick layer: ^1H - ^{186}W simultaneously.

See upcoming presentation by: **Anna Widdowson**

UPPSALA UNIVERSITY TANDEM LABORATORY

A national infrastructure for ion beam-based research in:

- Accelerator Mass Spectrometry - AMS
- Ion Beam Modification of Materials - IBMM
- Ion Beam Analysis - IBA

4 accelerators, 9 ion sources, 11 beamlines



Operation time 2020

2800 h

Tandem accelerator NEC 5 MV Pelletron (2001)

IBA, IBMM, AMS

2000 h

Linear 350 kV Danfysik accelerator (2003 & 2015)

IBA, IBMM

MICADAS - 170 kV, ETH (2014)

AMS

3500 h

Dedicated cleanroom for sample preparation

AMS, IBA, IBMM

Low-Energy Ion Scattering set-up (2018)

IBA, IBMM

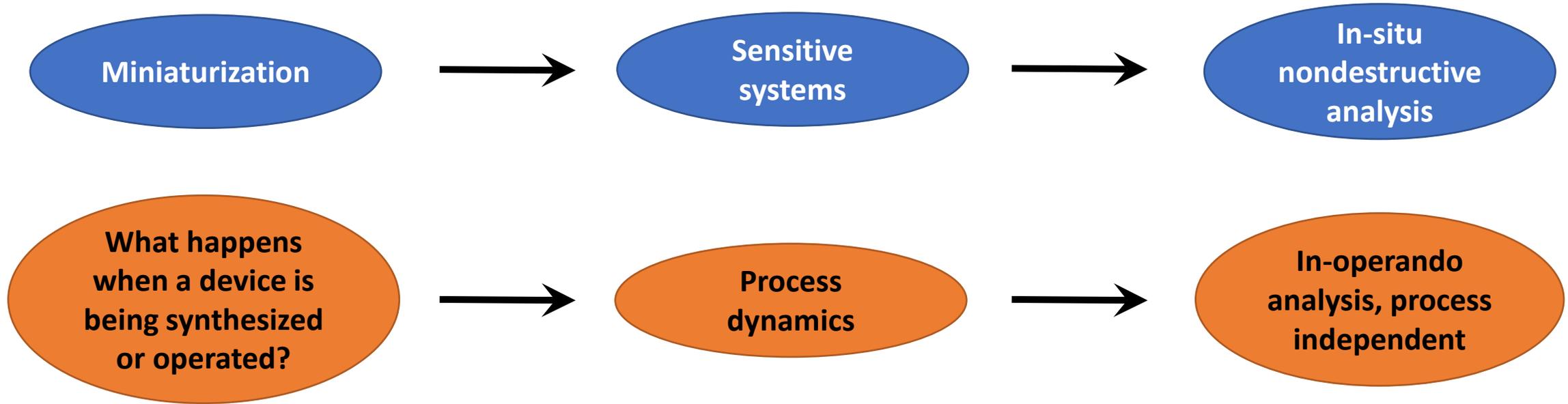
Low-Energy Ion Implanter (2022)

Under commissioning

UPPSALA UNIVERSITY TANDEM LABORATORY



EMERGING NEEDS: IN-SITU AND IN-OPERANDO CHARACTERIZATION



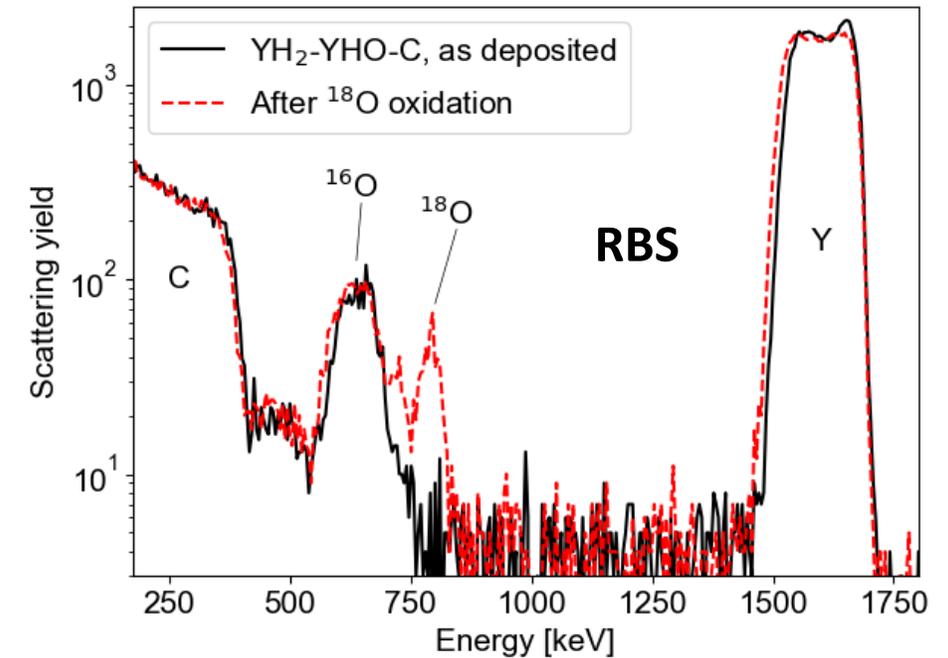
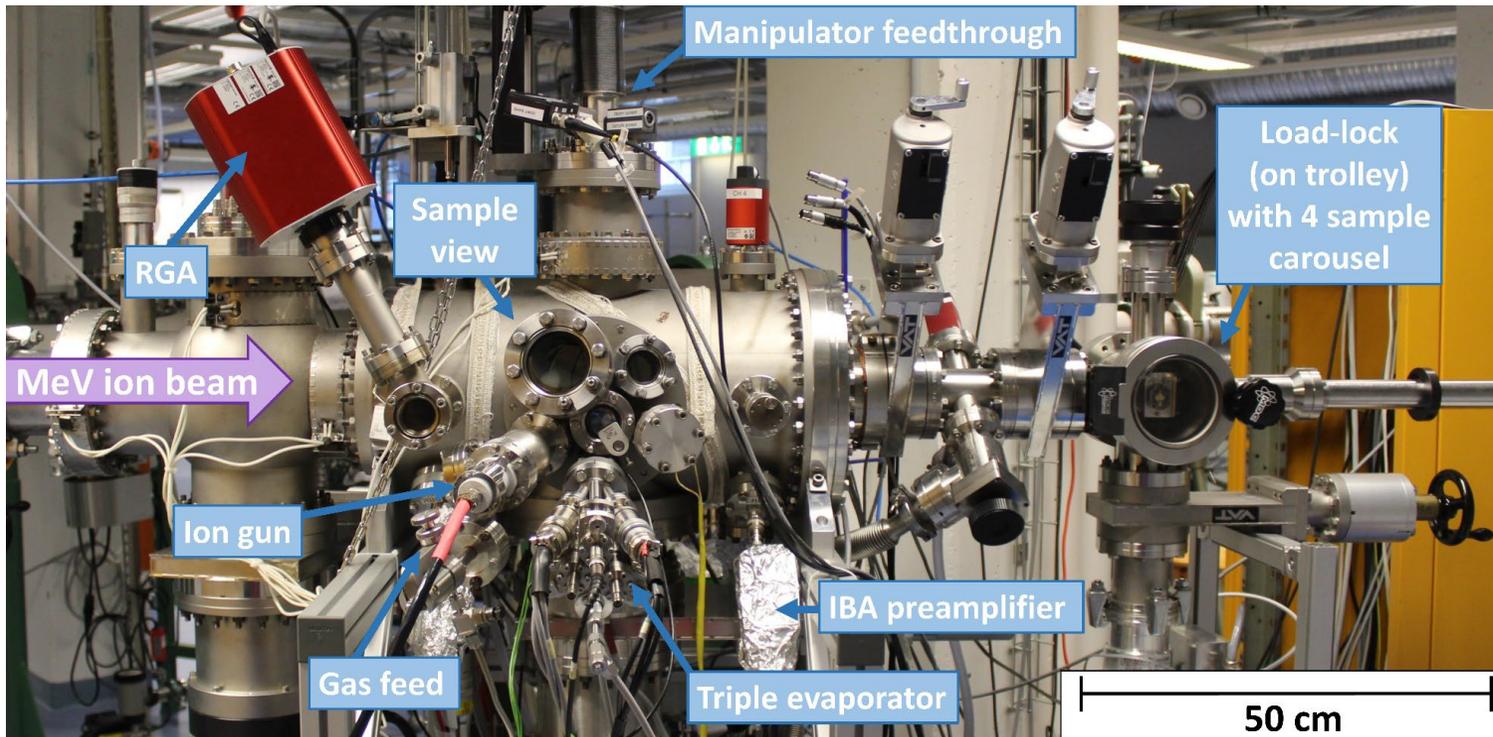
WHEN IS ION BEAM ANALYSIS THE BEST CHOICE?

- Thin film characterization with nondestructive composition depth profiling.
- Robust in-situ, and in-operando analyses that are independent of the process under study.
- Films or bulk materials for full quantitative information about light and heavy constituents.

RESEARCH HIGHLIGHTS

SIGMA set-up

- UHV chamber with undockable load-lock
- In-situ growth of thin films with triple e-beam evaporator
- Controlled gas exposure and keV ion implantation & sputtering
- Annealing to 1000°C and desorption spectroscopy
- MeV ion implantation and ion beam analysis



In-situ ¹⁸O-oxidation example

- YH₂-YHO bilayer grown in-situ
- Oxidized with ¹⁸O tracer
- Enables study of oxygen mobility

D. Moldarev et al., *Oxygen mobility in yttrium hydride films studied by isotopic labelling*, Eur. Phys. J. Web Conf. **261** (2022) 01001.

RESEARCH HIGHLIGHTS

In-operando Li depth profiling in thin film battery

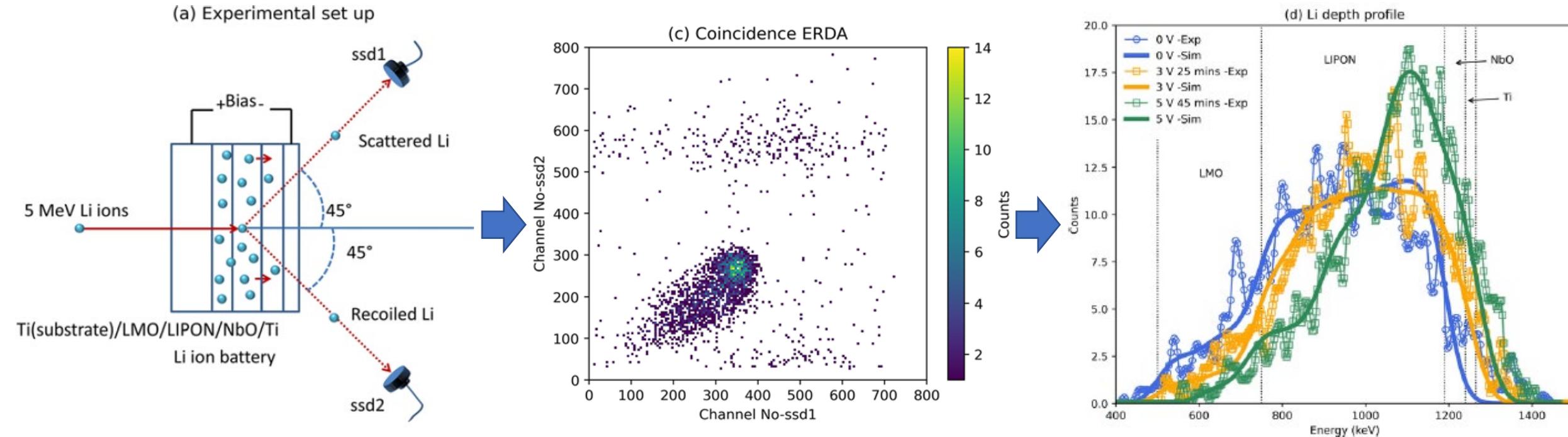
- Coincidence measurements → selection of faint signal
- Outgoing energy → depth information
- < 100 nm resolution in 5 μm stack
- Migration of Li tracked during operation of battery

V. Mathayan et al.

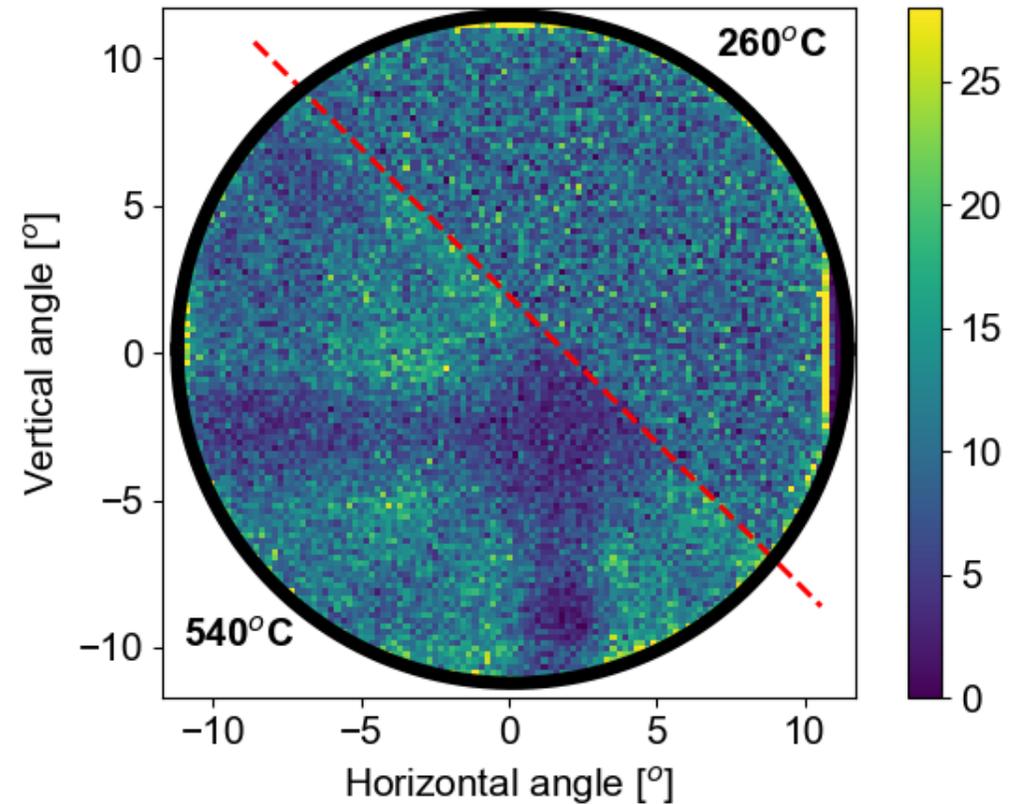
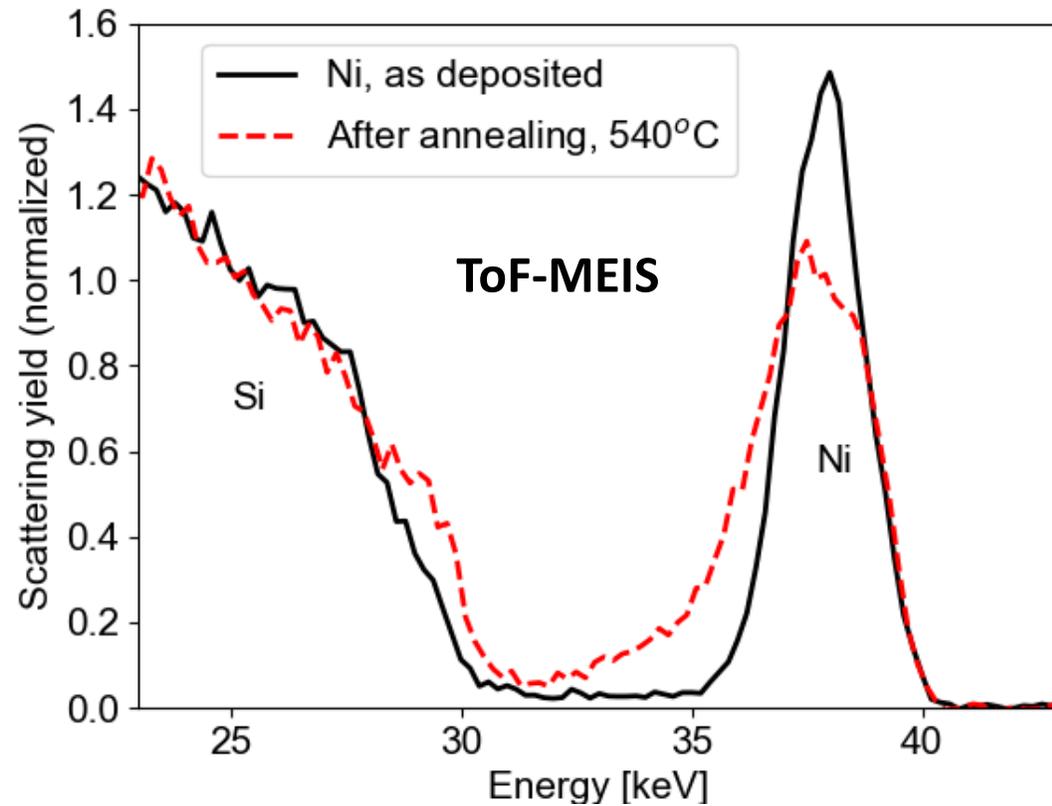
In-operando observation of Li depth distribution and Li transport in thin film Li ion batteries, Appl. Phys. Lett. **117** (2020), 023902.

Sensitive in-operando observation of Li and O transport in thin-film Li-ion batteries, Mater. Today Energy **21** (2021), 100844.

Assessing the potential of ion beam analytical techniques for depth profiling Li in thin film Li ion batteries, J. Appl. Phys. **130** (2021), 125306



RESEARCH HIGHLIGHTS

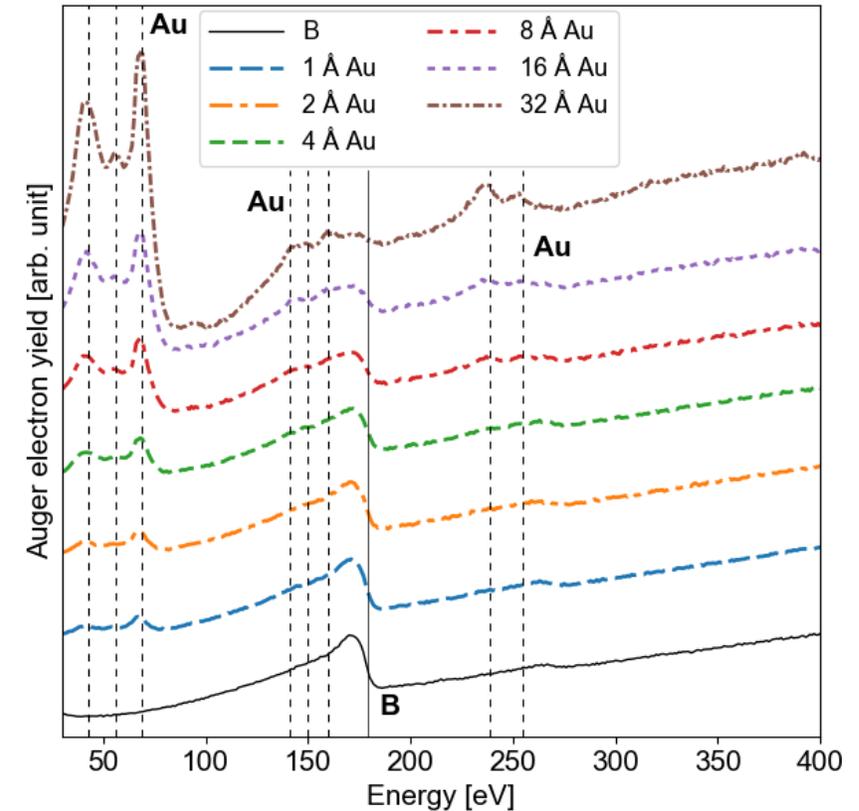
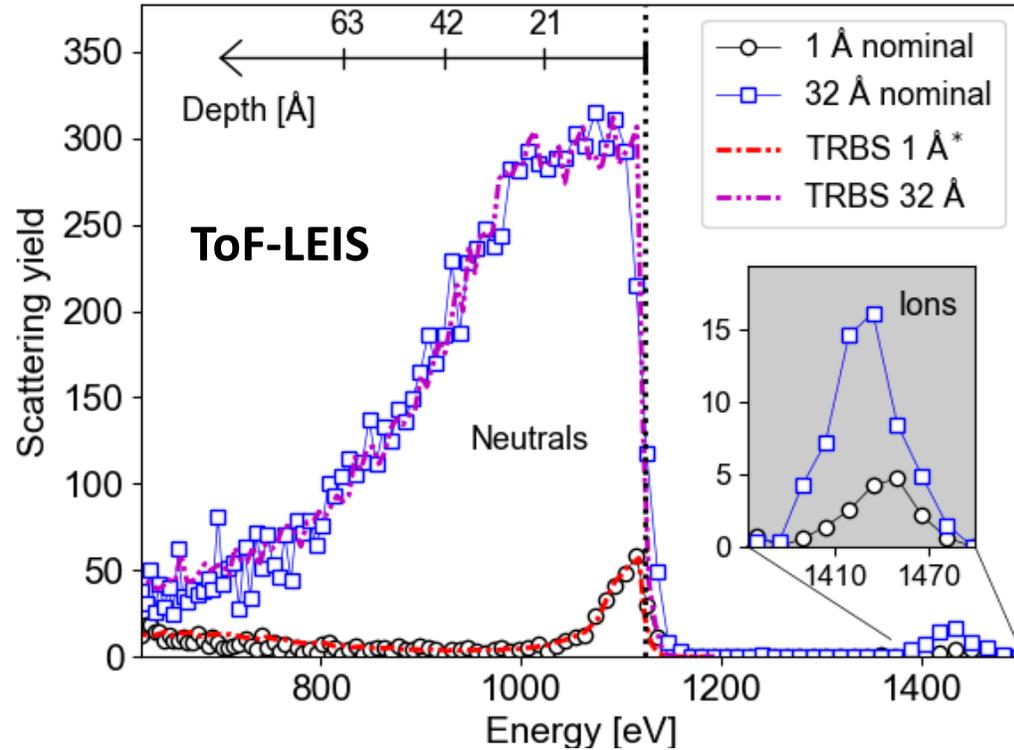


In-situ annealing and study of nickel silicide formation

- Ex-situ magnetron sputter deposition of polycrystalline Ni film, 3 nm
- In-situ annealing → stepwise silicide formation Ni→NiS→NiS₂
- Energy spectra revealed degree and depth dependence of Ni/Si mixing
- Position sensitive detector (right image) indicated epitaxial silicide final film

T. T. Tran et al., *In-situ nanoscale characterization of composition and structure during formation of ultrathin nickel silicide*, Appl. Surf. Sci. **536** (2021) 147781.

RESEARCH HIGHLIGHTS



Investigation of Au growth mechanism on B, all steps in-situ

- Sputter cleaning of Si substrate
- Auger electron spectroscopy → impurity monitoring
- Boron coating and deposition of Au film
- Ion scattering for composition in first few nm

D. Primetzhofer et al., *Quantitative analysis of ultra thin layer growth by time-of-flight low energy ion scattering*, Appl. Phys. Lett. **92** (2008) 011929.



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Thank you!

Contributions and figures: D. Primetzhofer,
T. T. Tran, D. Moldarev, V. Mathayan

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