Quantitative high-resolution depth profiling of light and heavy elements with low-energy high-resolution ERDA

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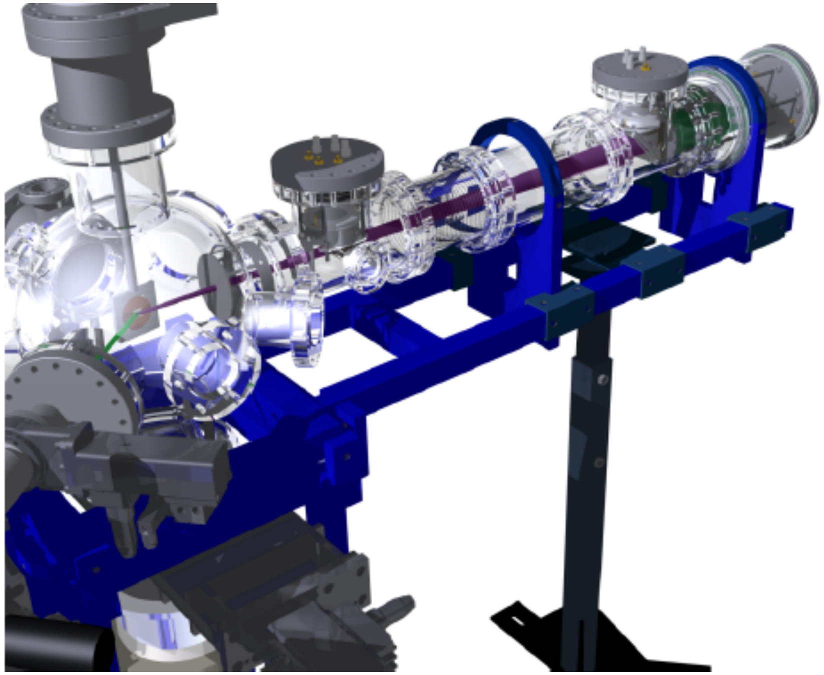

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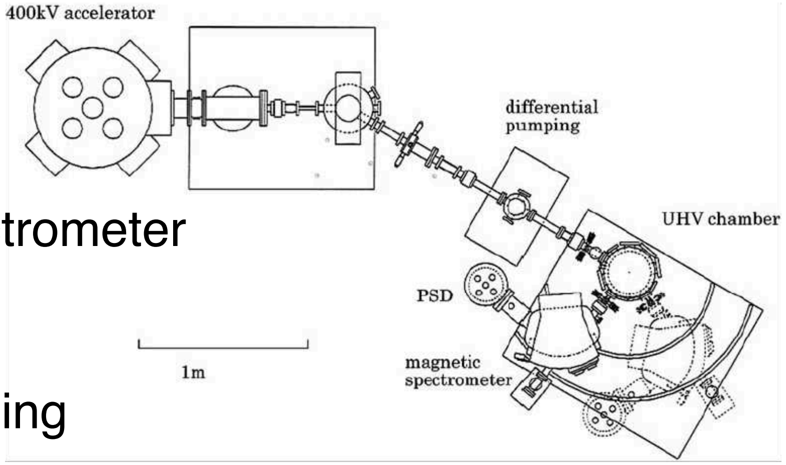
Outline

- Introduction to high resolution ion beam analysis
- Development of low-energy heavy ion elastic recoil detection analysis tools at IMEC and Jyväskylä
- Analysis examples from hundreds of nanometers thick films to single monolayer films

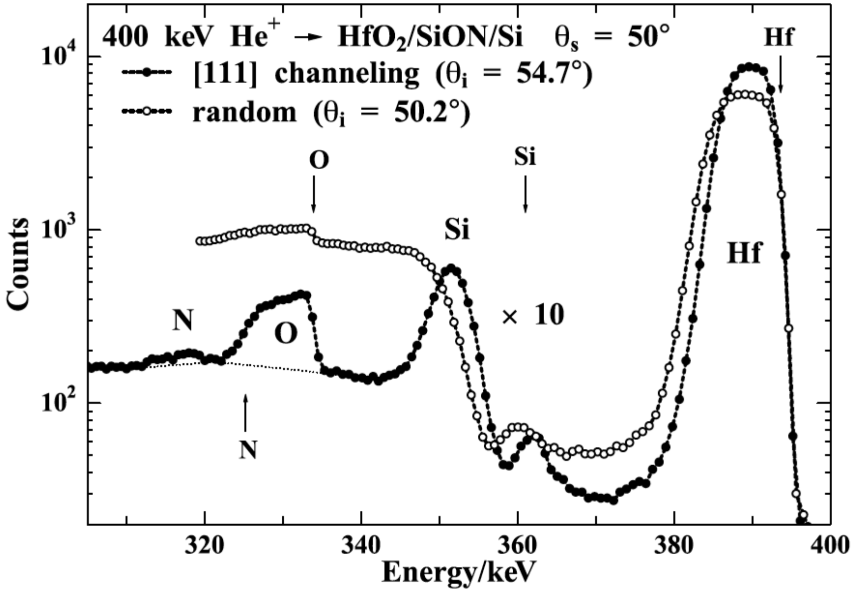
Ion beam techniques for elemental depth profiling

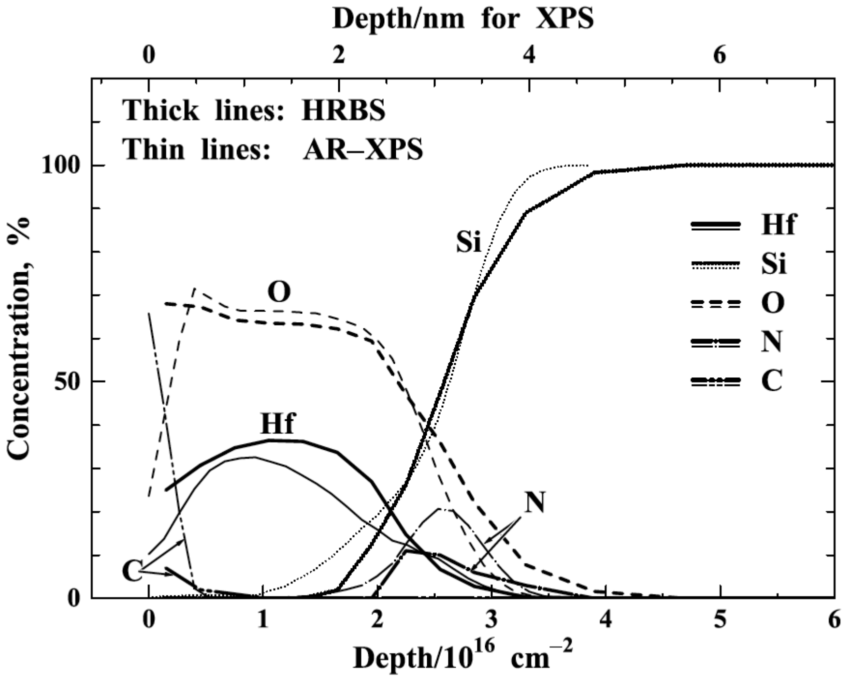
- Incident ion energy range 0.1-30 keV
  - SIMS, LEIS (Low Energy Ion Scattering)
    - Very sensitive for all elements, depth information based on sputtering
- Incident ion energy range 50-200 keV
  - MEIS (Medium Energy Ion Scattering)
    - Backscattered H or He ions used, depth information based on ion energy loss
    - High depth resolution, limited sensitivity for lighter elements
- Incident ion energy range >200 keV
  - RBS (Rutherford Backscattering Spectrometry)
  - He-ERDA (Elastic Recoil Detection Analysis)
  - NRA (Nuclear Reaction Analysis)
  - **HI-ERDA (Heavy Ion ERDA)**

High resolution RBS



- Typically 300–500 keV He incident ions
- Best energy resolution with magnetic spectrometer
  - Limited energy window
  - Charge state sensitive
- Background reduced by means of channeling
- Limited sensitivity for lighter elements

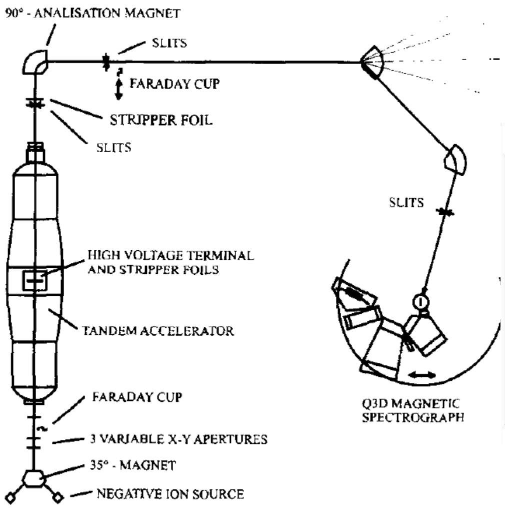


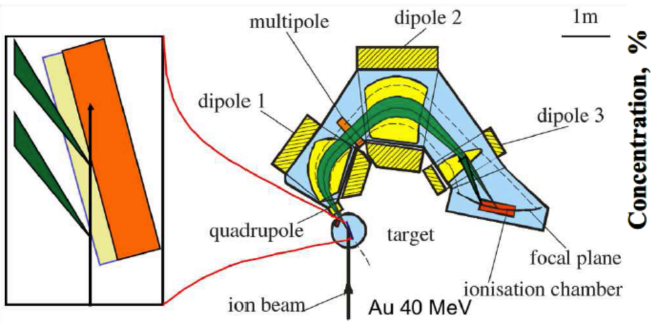


Spectrum and depth profile from: K. Kimura, K. Nakajima, T. Conard, W. Vandervorst, A. Bergmaier, G. Dollinger, ANALYTICAL SCIENCES 26 (2010) 223.

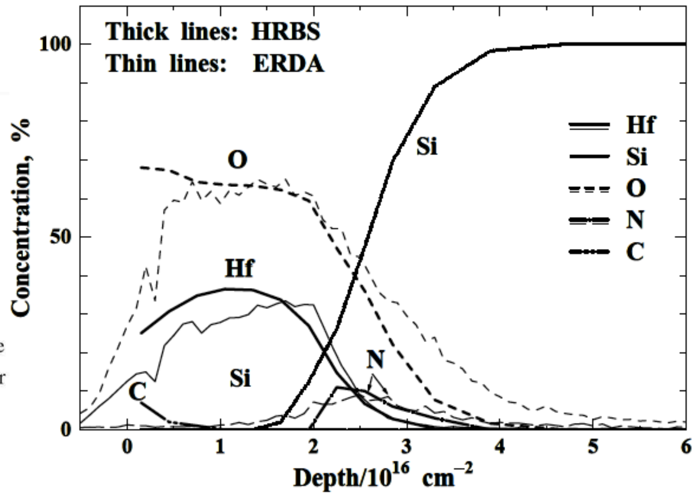
High energy heavy ion ERDA

- Typically 30–200 MeV Cl, Br, I or Au ions
- Gas ionization, time-of-flight–energy or magnetic spectrometers for elemental or isotopic identification
- Magnetic spectrometer offers superior energy and therefore depth resolution, kinematic correction needs to be applied
- Magnetic spectrometers are large and expensive instruments and multiple charge states for heavier elements need to be considered





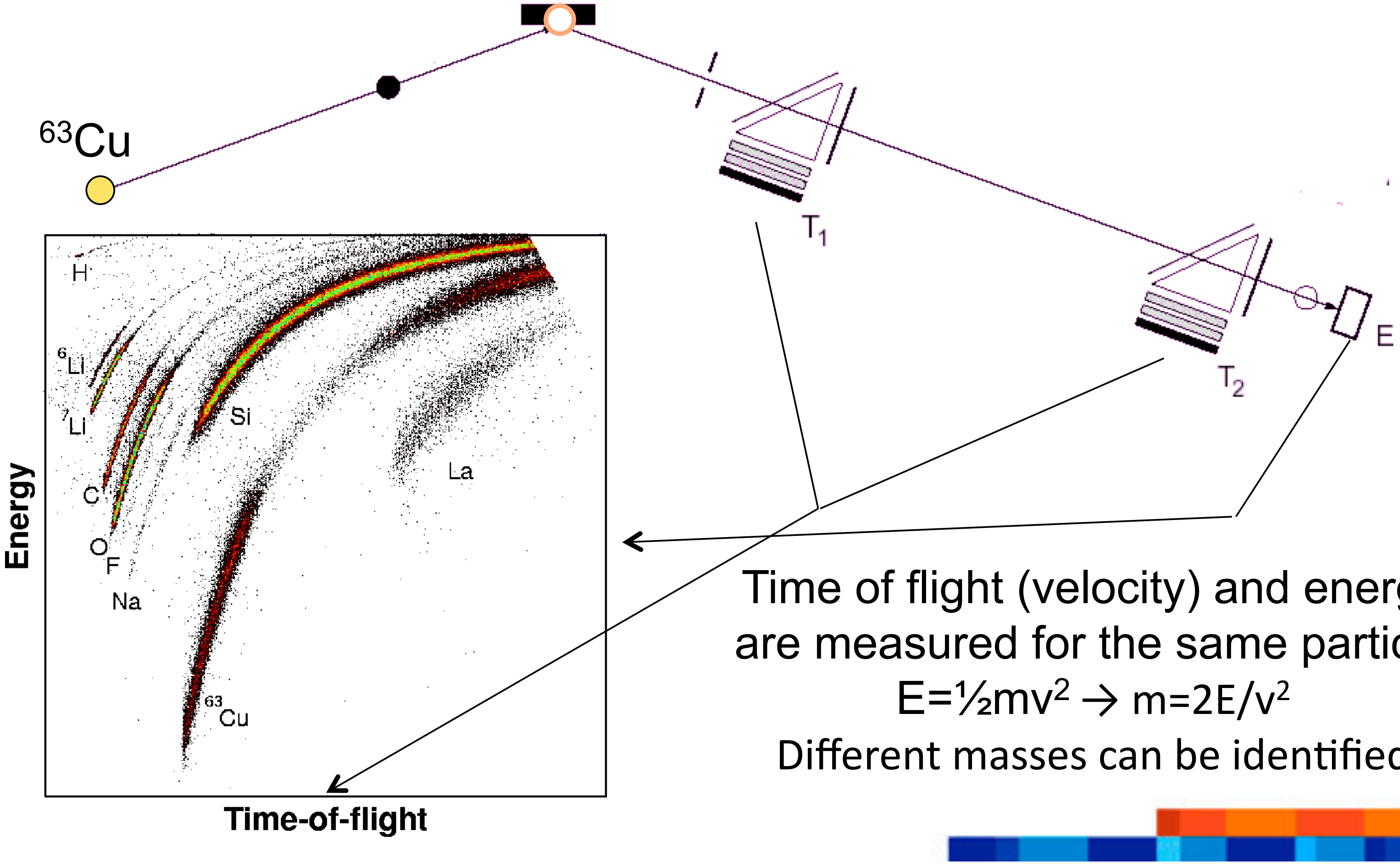
Munich spectrograph



Depth profile from: K. Kimura, K. Nakajima, T. Conard, W. Vandervorst, A. Bergmaier, G. Dollinger, ANALYTICAL SCIENCES 26 (2010) 223.

Low energy heavy ion ERDA

- Typically 1–20 MeV Cl, Br, I or Au ions from 1–3 MV tandem accelerator
- Time-of-flight–energy spectrometers for isotopic identification and energy spectrum measurement

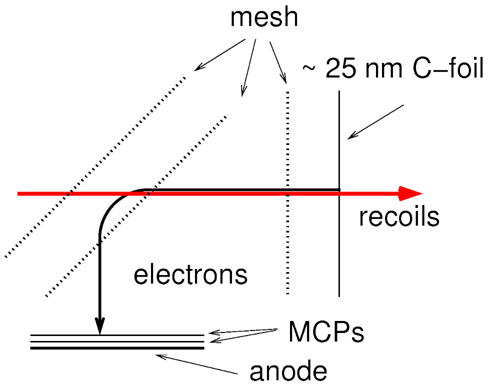


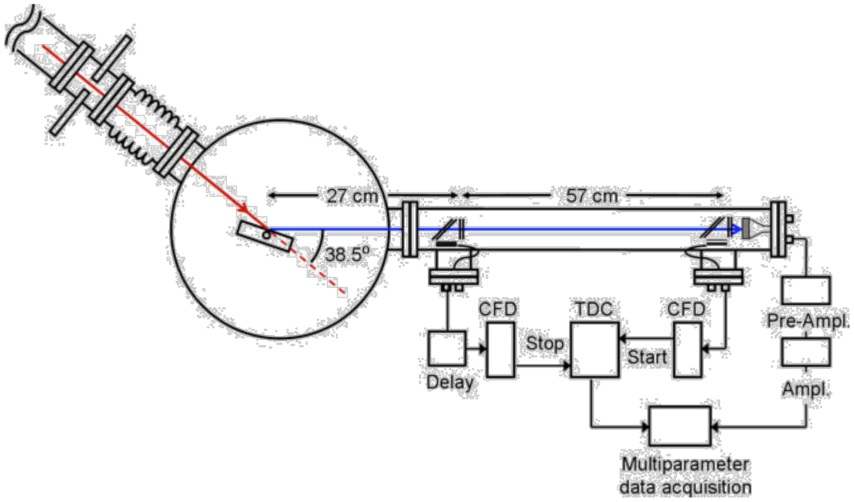
Time of flight (velocity) and energy are measured for the same particle

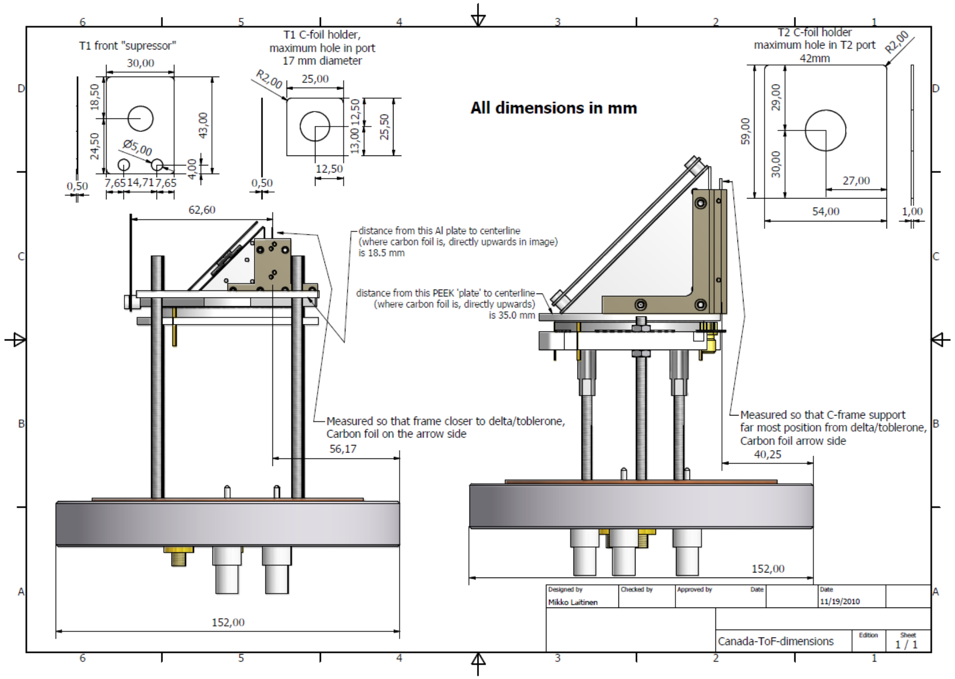
$E=\frac{1}{2}mv^2 \rightarrow m=2E/v^2$

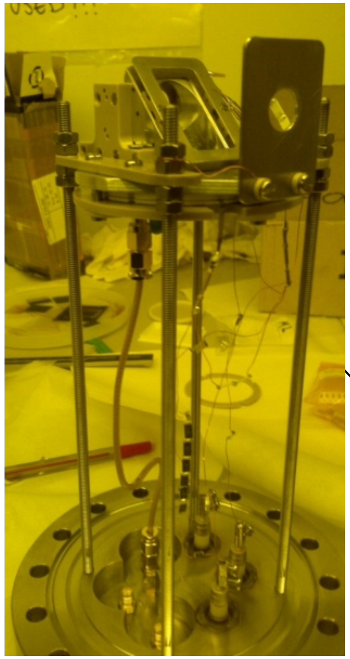
Different masses can be identified

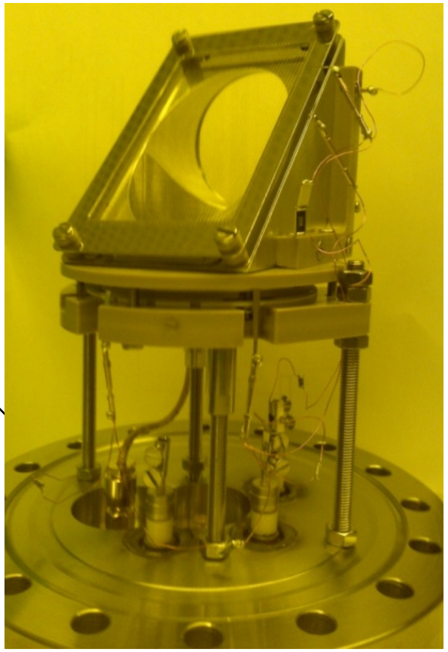
Time-of-flight-ERDA telescope





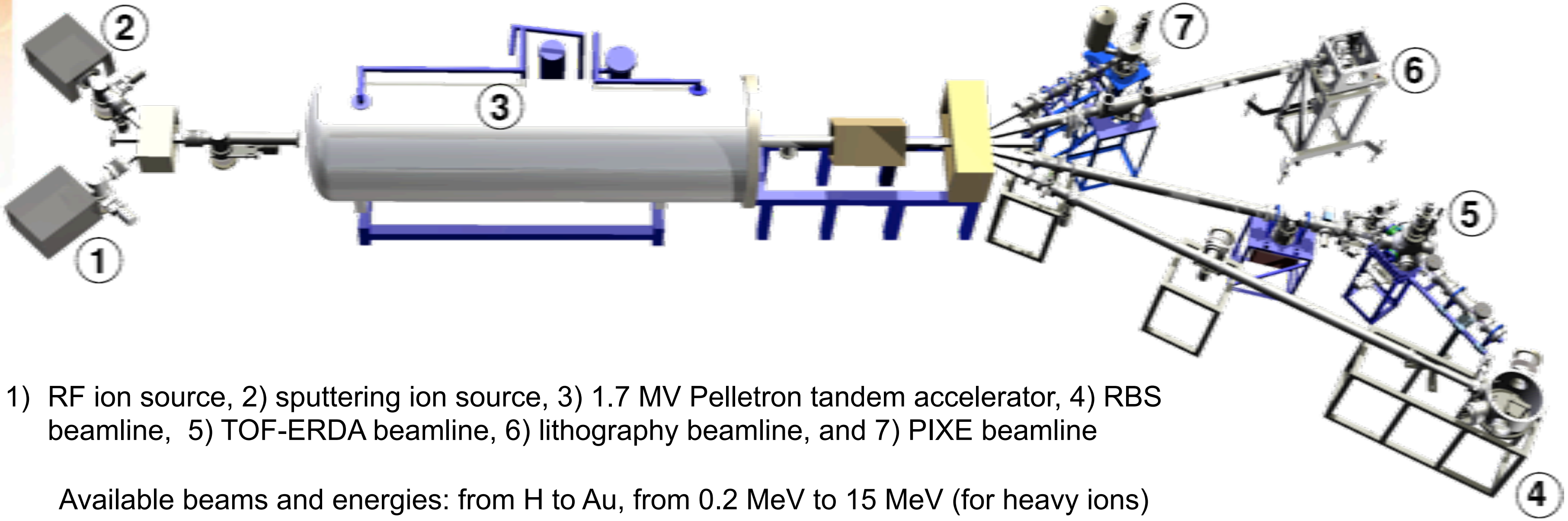






Why or why not low energies?

- Pros of low energies
  - Better energy resolution with TOF with smaller energies
  - Higher scattering cross-section, more events form the layer of interest
  - Only a small accelerator is needed
- Cons of low energies
  - Probing depth is more limited
  - Both ingoing ions and out-coming recoils suffer from the multiple scattering
  - Separation of heavier masses is poorer mainly due to Si energy detector

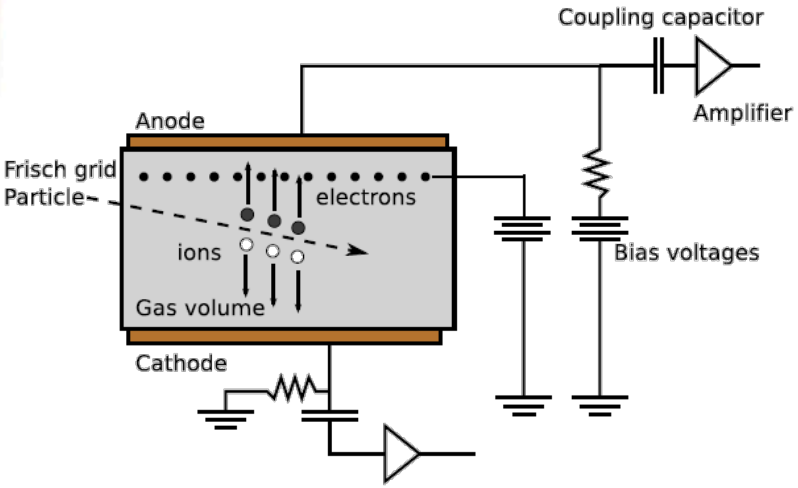


1) RF ion source, 2) sputtering ion source, 3) 1.7 MV Pelletron tandem accelerator, 4) RBS beamline, 5) TOF-ERDA beamline, 6) lithography beamline, and 7) PIXE beamline

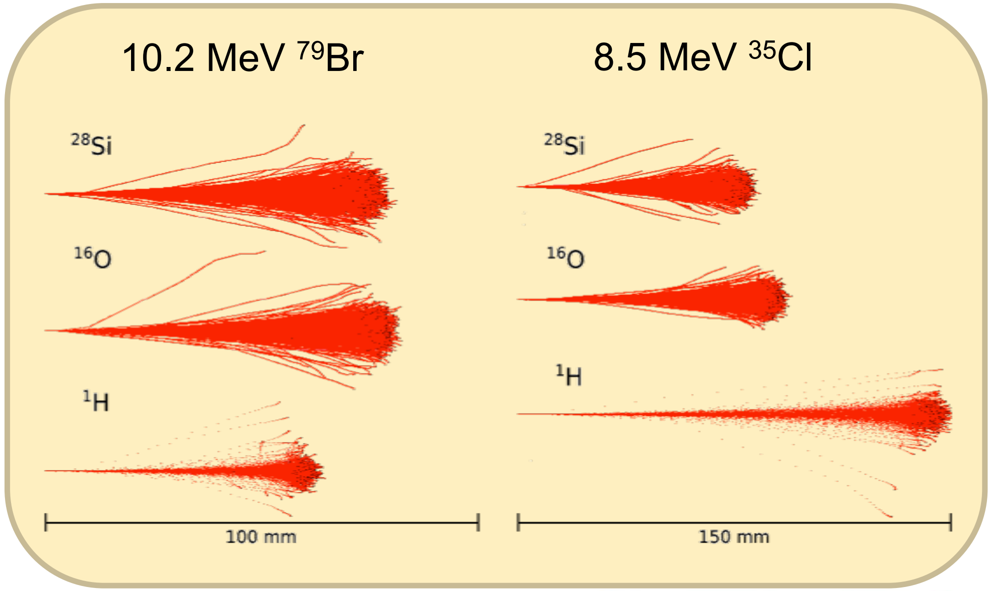
Available beams and energies: from H to Au, from 0.2 MeV to 15 MeV (for heavy ions)

Gas ionization detector to replace Si-energy detector

- Why replace a well working system?
  - Greatly improved energy resolution for low energy heavy ions → heavier masses can be resolved
  - Gas detector is 1D position sensitive by nature → possibility for kinematic correction and therefore larger solid angles possible
  - Gas detector does not suffer from ion bombardment

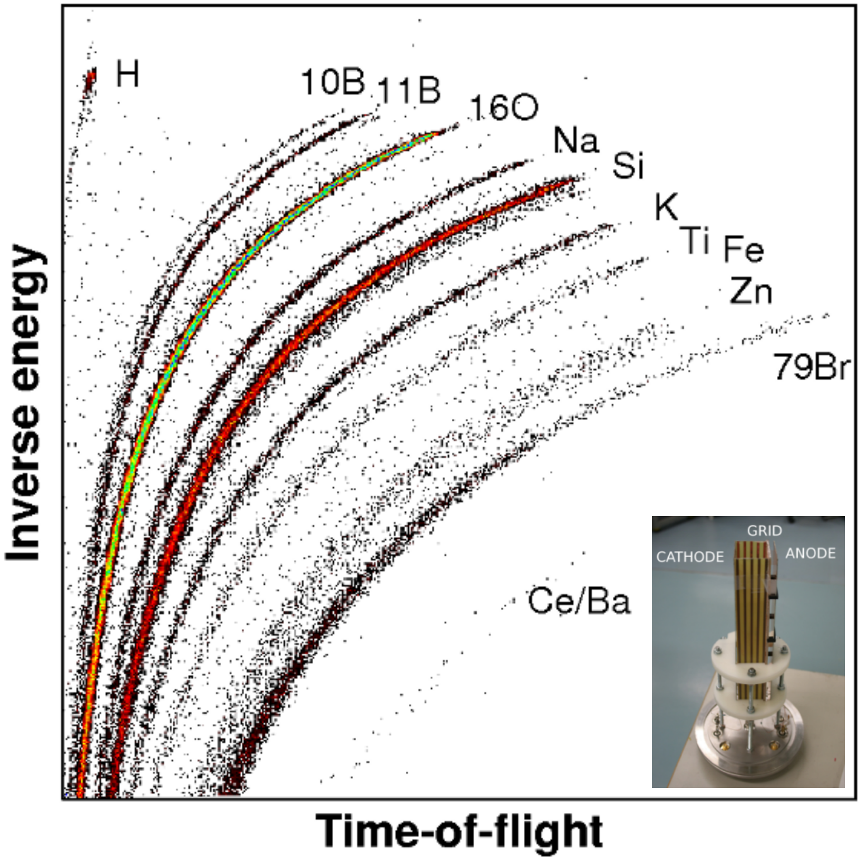


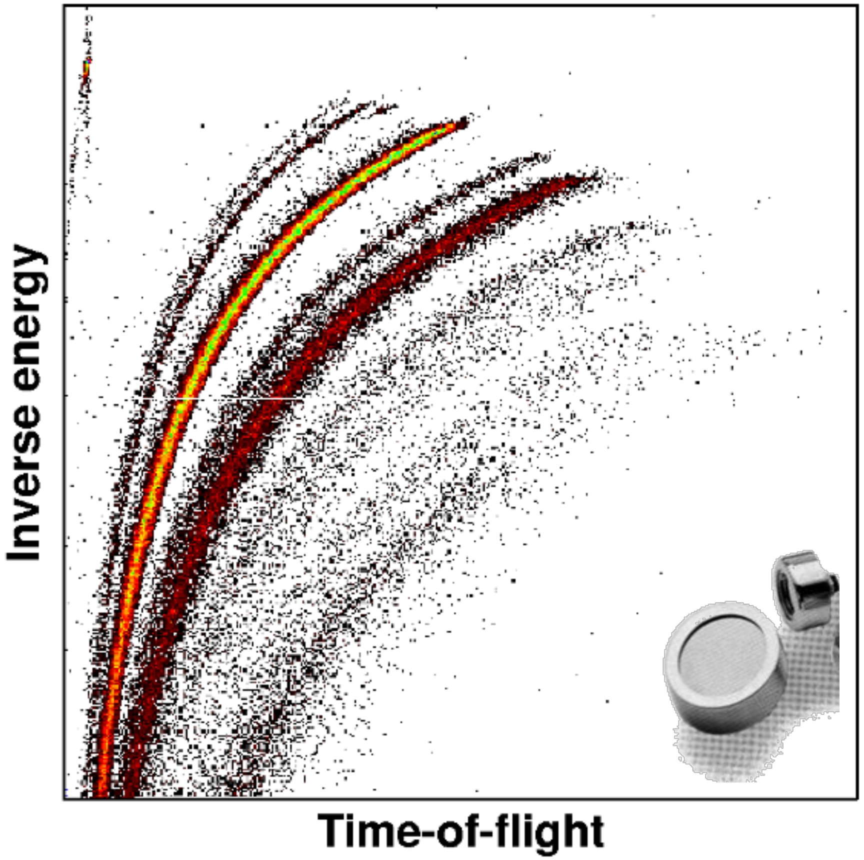
Recoil ranges in isobutane



Gas detector performance

- Same borosilicate sample is measured with two different energy detectors: gas ionization detector and new Si-detector





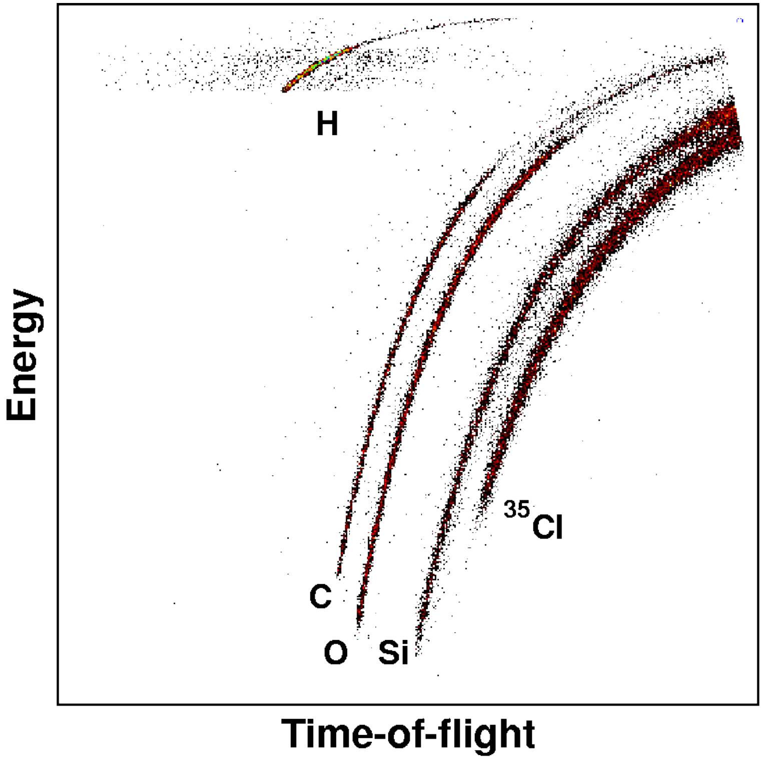
Quantification of the measurements

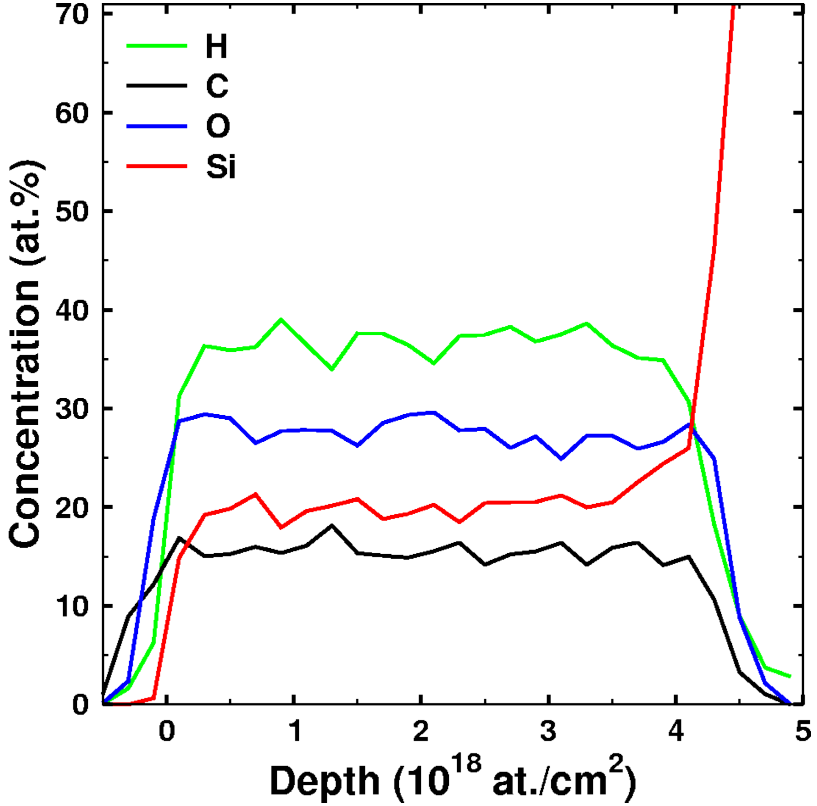
- Scattering process (kinematics and cross-sections) are well known, and energy loss fairly well
- Three means of quantification
  - In case of homogenous films direct comparison of elemental yields normalized with the cross-sections
  - Conversion of energy spectra directly to depth profiles using the information above
  - Analytical or Monte Carlo simulation of energy spectra

ANALYSIS EXAMPLES

Low-*k* materials

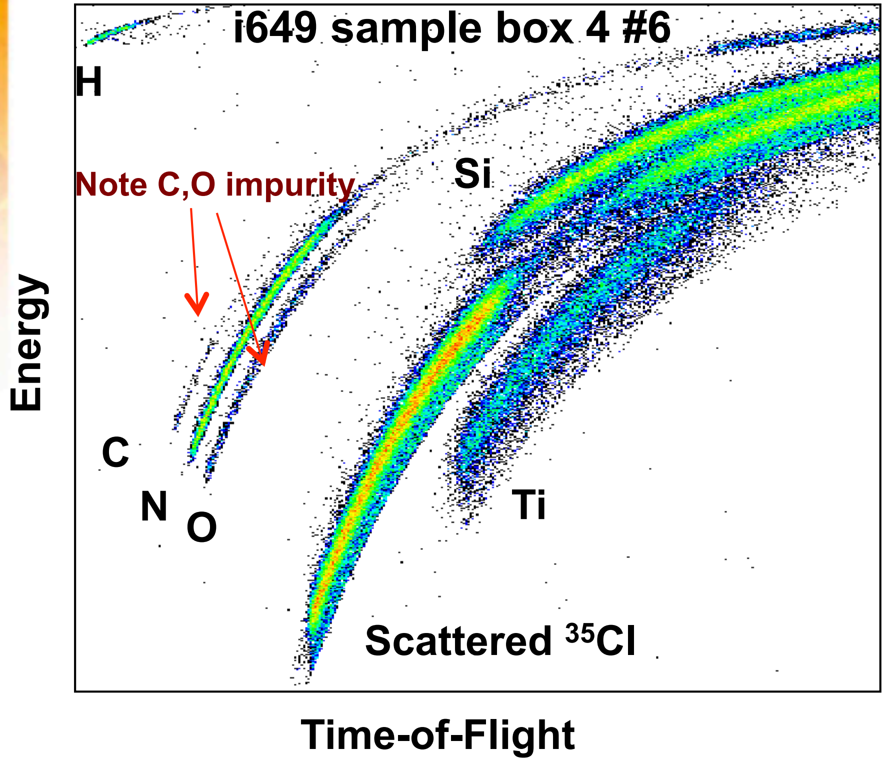
- Analysis example: 600 nm low-*k* (Black Diamond)
- Beam: 16 MeV $^{35}Cl^{7+}$, geometry:19+19=38°
- Note: H and C elemental losses during the measurement





Round Robin experiment : TiN films on Si

- Similar samples sent to several IBA laboratories for detailed analysis through EU-funded SPIRIT program
- Perfect samples for TOF-ERDA: relatively light elements, nice thickness, uniform, some impurities.
- Measured with 10.215 MeV $^{35}Cl^{6+}$



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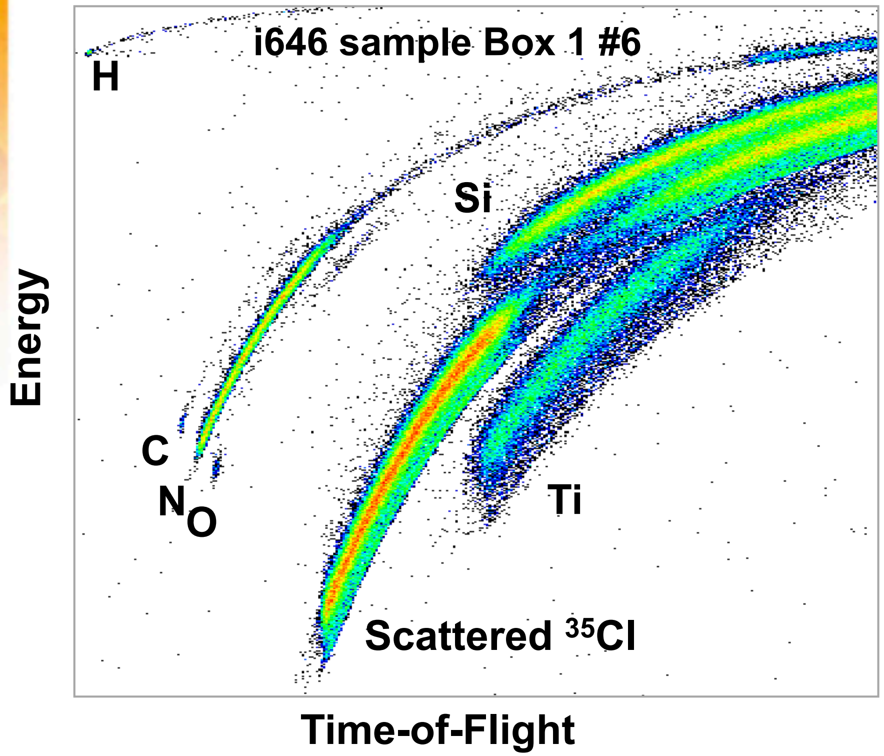
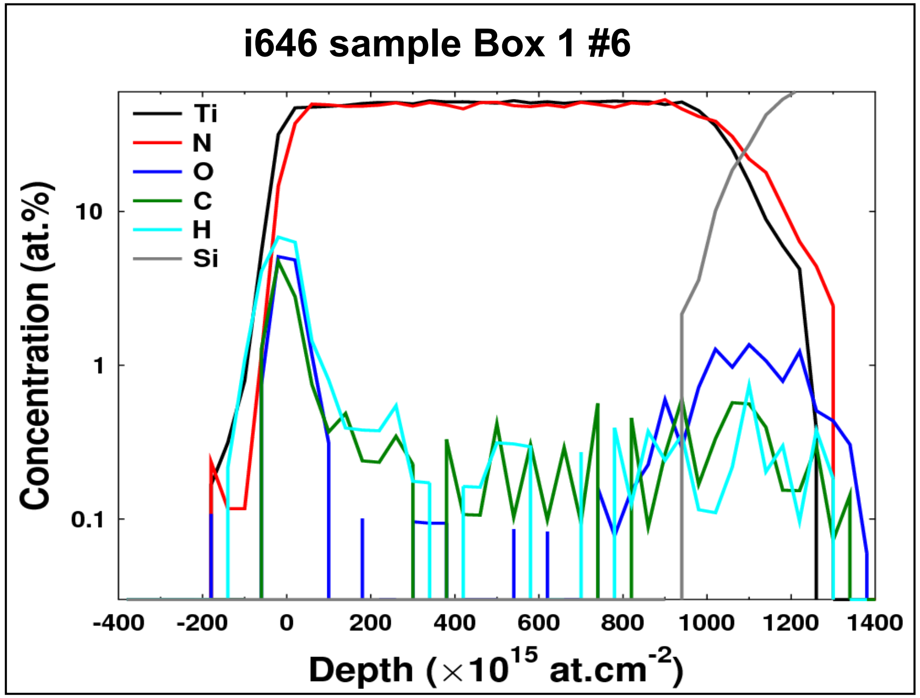
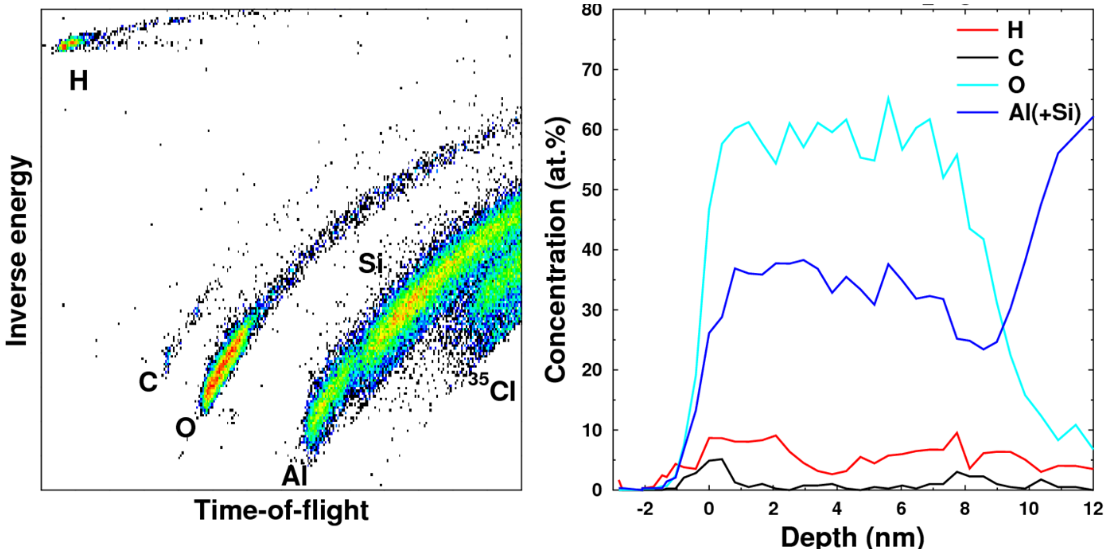


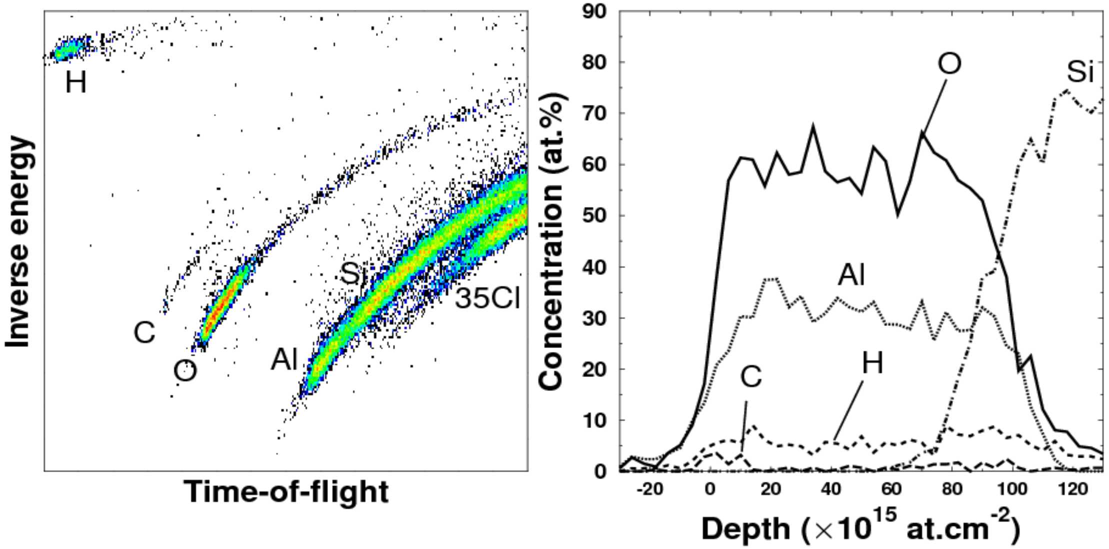


8.6 nm thick ALD-Al_2O_3 on Si

- TOF-ERD analysis with low energy incident ions

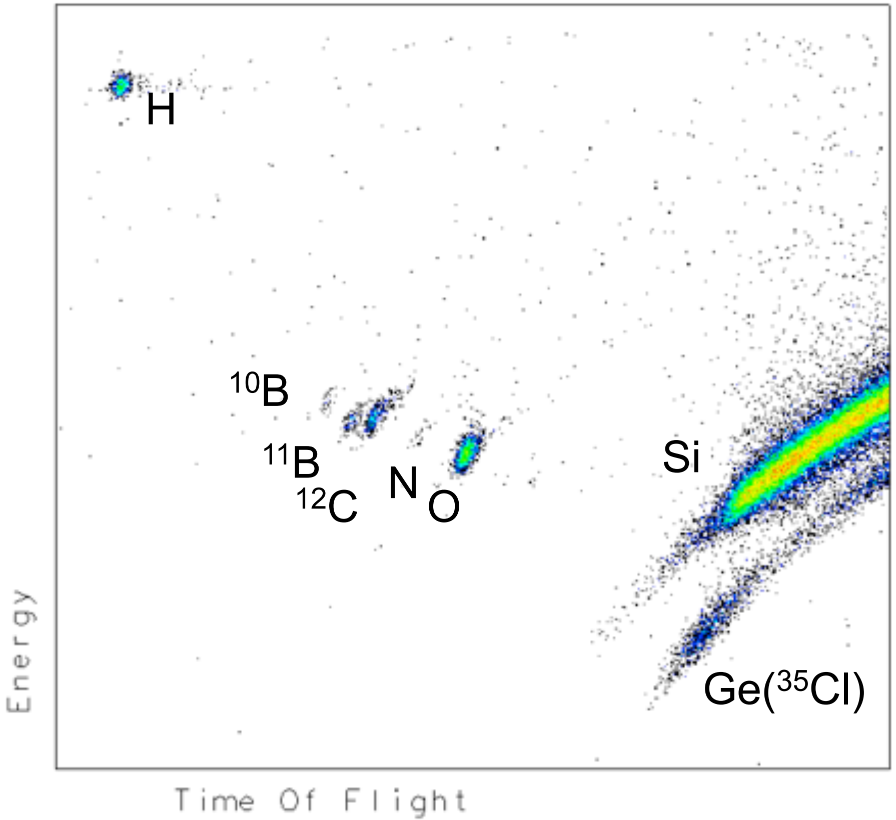


5 MeV ^{35}Cl
with Si detector

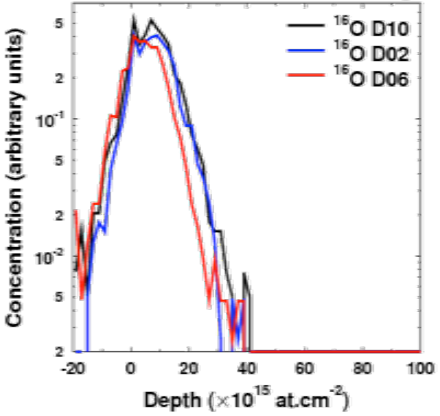


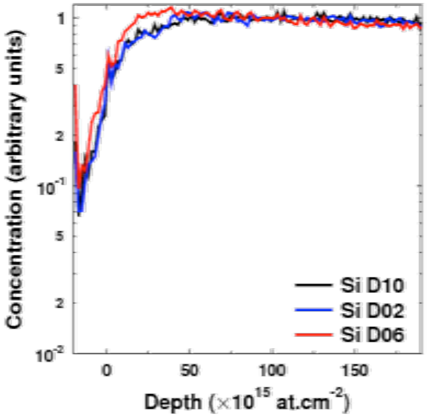
3 MeV ^{35}Cl
with GIC detector

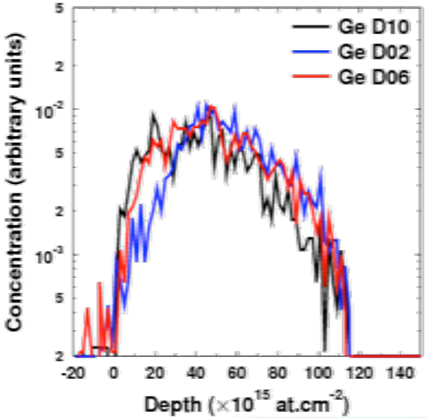
B, C and Ge implanted SiO_2/Si



- 3 MeV ^{35}Cl beam and 31°+10°=41° geometry
- ^{11}B areal density 8.8e14 at./cm^2
- C areal density (without the surface peak) 4.3e14 at./cm^2
- Ge areal density calculated from scattered ^{35}Cl 5.2e14 at./cm^2

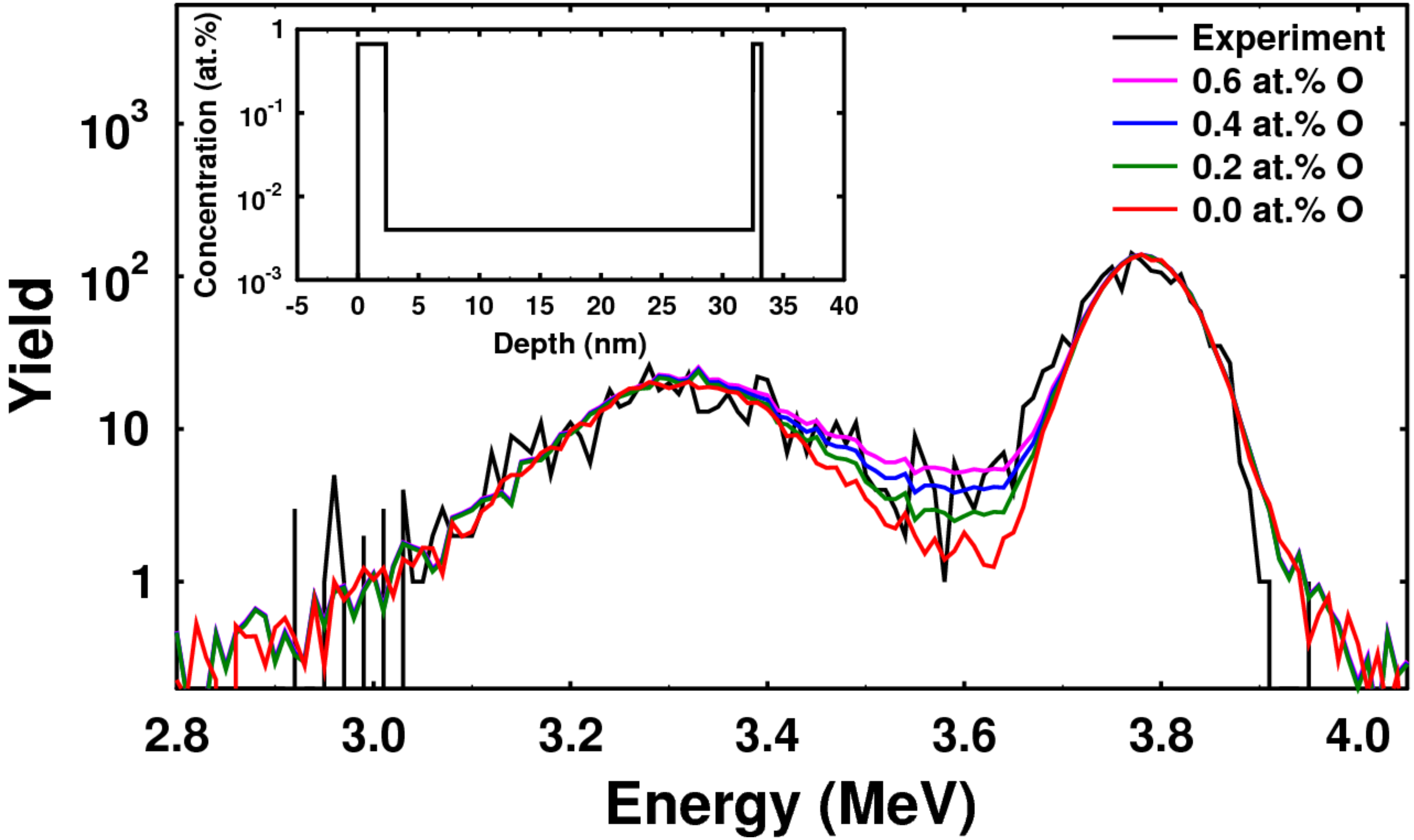






O in 32 nm thick UHV evaporated Ti-film

- Measured with 8 MeV ^{79}Br incident beam
- Energy spectra simulated with Monte Carlo program MCERD



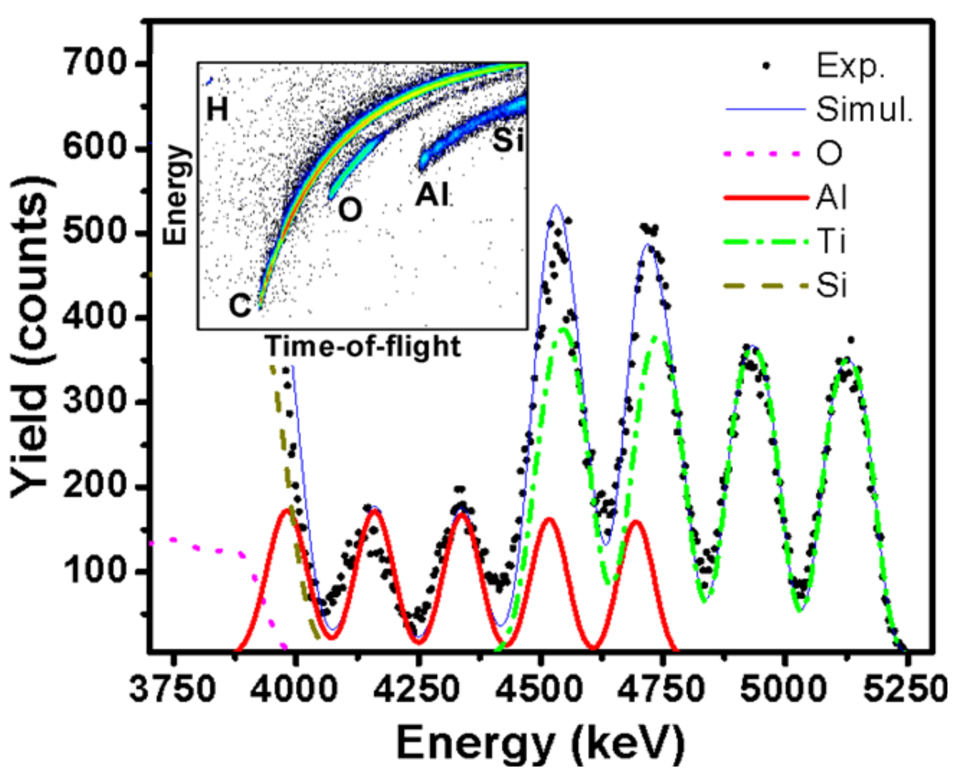
Surface peak 1.7e16 at./cm^2 and interface peak 5.5e15 at./cm2.
Oxygen content in the film 0.4 at.%

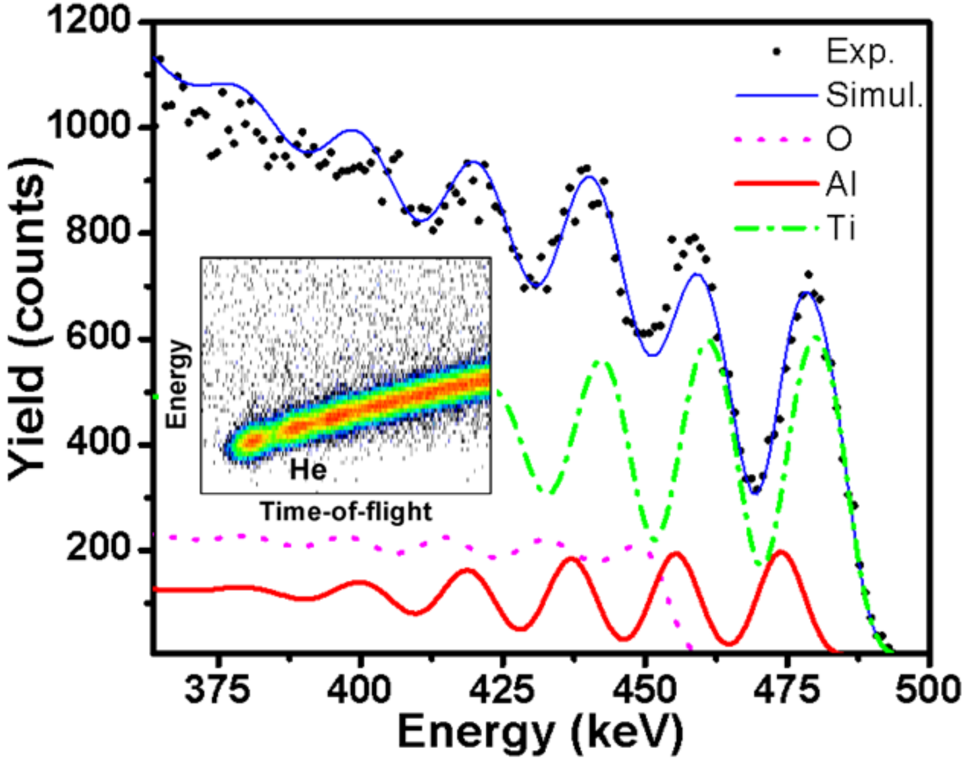
Al_2O_3/TiO_2 nanolaminate samples

- 6.0 MeV $^{12}C^{3+}$ - incident angle 35°
- 0.5 MeV $^{4}He^{1+}$ - Rutherford Scattering to forward angles up to 39° angle
- Detector angle 41°
- Simulated using SIMNRA code

5 nm layers, ^{12}C beam

2 nm layers, ^{4}He beam





M. Laitinen, T. Sajavaara, M. Rossi, J. Julin, R.L. Puurunen, T. Suni, T. Ishida, H. Fujita, K. Arstila, B. Brijs, H.J. Whitlow, http://dx.doi.org/10.1016/j.nimb.2011.04.074

Summary

- Quantitative depth profiling of all elements, including hydrogen, can be performed with low energy heavy ions for very different sample types and film thicknesses
- Better mass resolution can be reached by means of energy detector based on gas ionization
- Energy broadening due to multiple scattering can be reproduced by Monte Carlo simulations, which results in more reliable sample compositions

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