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Development of research around 1.7 MV Pelletron in Jyväskylä during five years of operation

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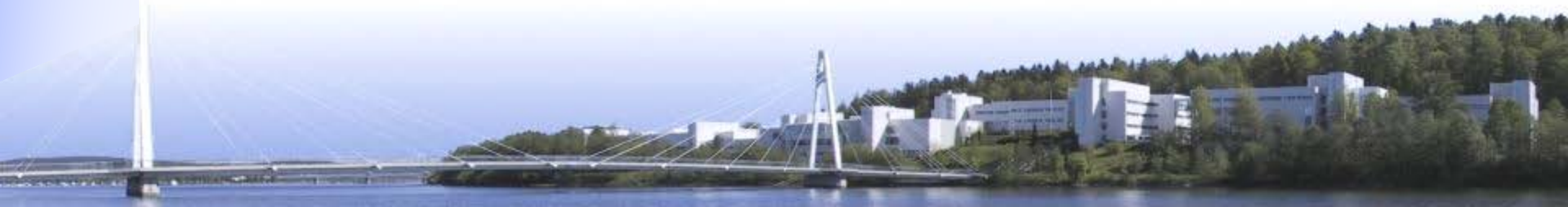

Outlook

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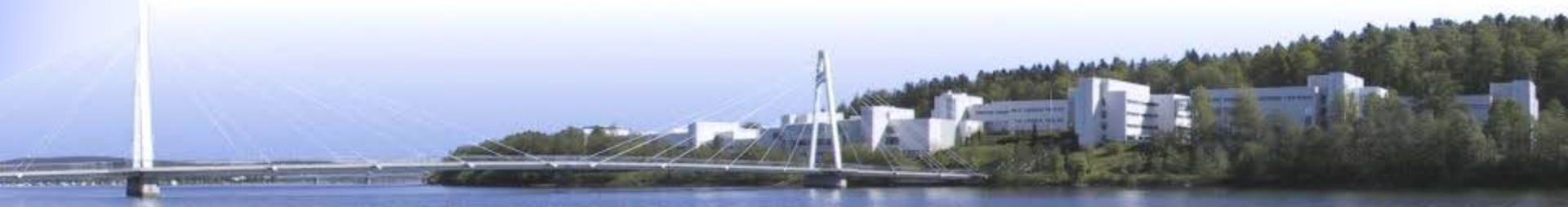
- Background: Who we are
- Moving the 1.7MV Pelletron to Jyväskylä
- Water leak at the SF6 heat exchanger
- From corona needles to resistors
- Turbopumped stripper system installation
- Beamlines and recent developments
- ‘Contaminants’ after analyzing magnet



University of Jyväskylä, Department of Physics (JYFL)

UNIVERSITY OF JYVÄSKYLÄ

- **University**: Staff 2600, 15000 Students, 217 M€ turnover
- **Department:** Personnel 190, including 85 PhD students

Physics department main research areas:

- Nuclear and accelerator based physics
  - One team out of 8 : Accelerator based materials physics
- Materials physics
- High- energy physics

NordForsk 2011: Comparing Research at Nordic Universities using Bibliometric Indicators

Among 30 Nordic Universities,

JyU is among the top four universities

in Physics + Mathematics

("second place" after Aarhus U.)

Accelerator based materials physics

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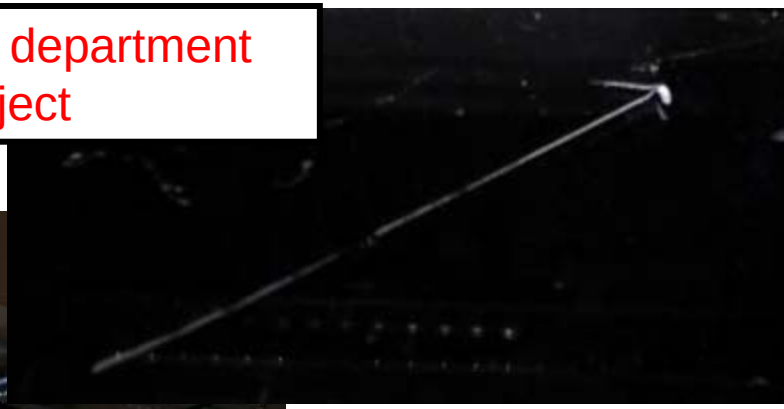
- 1 Senior, **no other staff, including engineers**
- 4-5 PhD students (for example: ion beam lithography, detector development, direct signal digitization by fast digitizers)
- 4-6 Master (and bachelor) thesis students

24/7 working diffusion cloud chamber for physics department permanent exhibiton: Masters thesis project



Commercial system price tag: 25-50k€ depending on size

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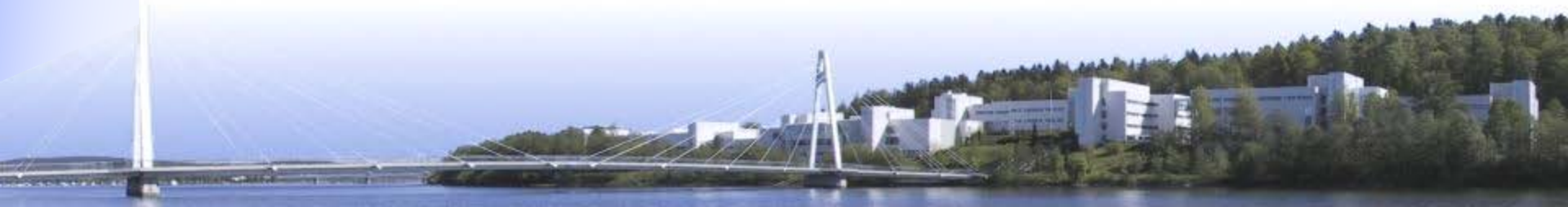
MOVING THE 1.7MV PELLETRON TO JYVÄSKYLÄ



Acquisition of the Pelletron accelerator

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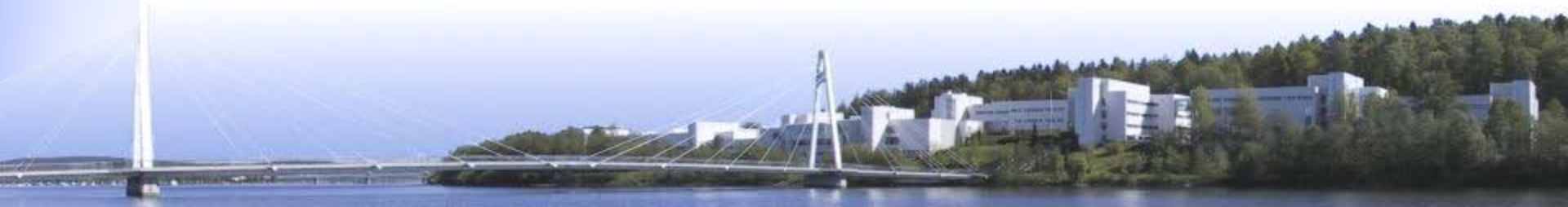
- A coffee table rumor was heard in late 2006…
  … and quickly confirmed by indirect route
- The technical research center of Finland (VTT) had little usage of their accelerator and needed the room space for cleanroom extension.. …And our group in Jyväskylä needed an accelerator.
- 1.7MV 5SDH-2 Pelletron(serial number 002, made in 1985) with one (Alphatross) ion source and one beam line was donated to JYU by VTT



Moving the accelerator from VTT

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Monday 18th of September 2006

Moving the Accelerator from VTT

UNIVERSITY OF JYVÄSKYLÄ

- SF6 purity 88%!
- No moisture 19ppm$_V$
- Purity analysis by Solvay Germany

1ATM of air+ 5bar SF6 overpressure!

Herkunft	Gebinde		Gewicht	Anteil SF6	SF6	Luft	CF4	CO2	SO2F2	SO2	**S2F10**	SF5-O-SF5
	Art	Nr.	kg		Vol-%	Vol-%	Vol-%	Vol-%	ppmv	ppmv	ppmv	ppmv
Wikeström, Helsinki	Flasche	72356	28	18	65.2939	34.706	0.0206					
Wikeström, Helsinki	Flasche	74390	21	16	75.2364	24.231	0.0699	0.0226	0.28837		0.01014	
Wikeström, Helsinki	Flasche	12780	30	28	93.8826	6.1131	0.001				0.00009	0.00516
Wikeström, Helsinki	Flasche	9977952	33	30	89.6052	10.395	0.0022			0.00027		0.00558

S2F10 was considered a potential chemical warfare agent in World War II because it does not produce lacrimation or skin irritation, thus providing little warning of exposure. LD50 levels about 15-25 ppm

Monday 18th of September 2006

Moving the Accelerator from VTT

Tuesday 19th of September 2006

Arriving at Jyväskylä after 330km

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25th of September 2006

Building of the Pelletron lab

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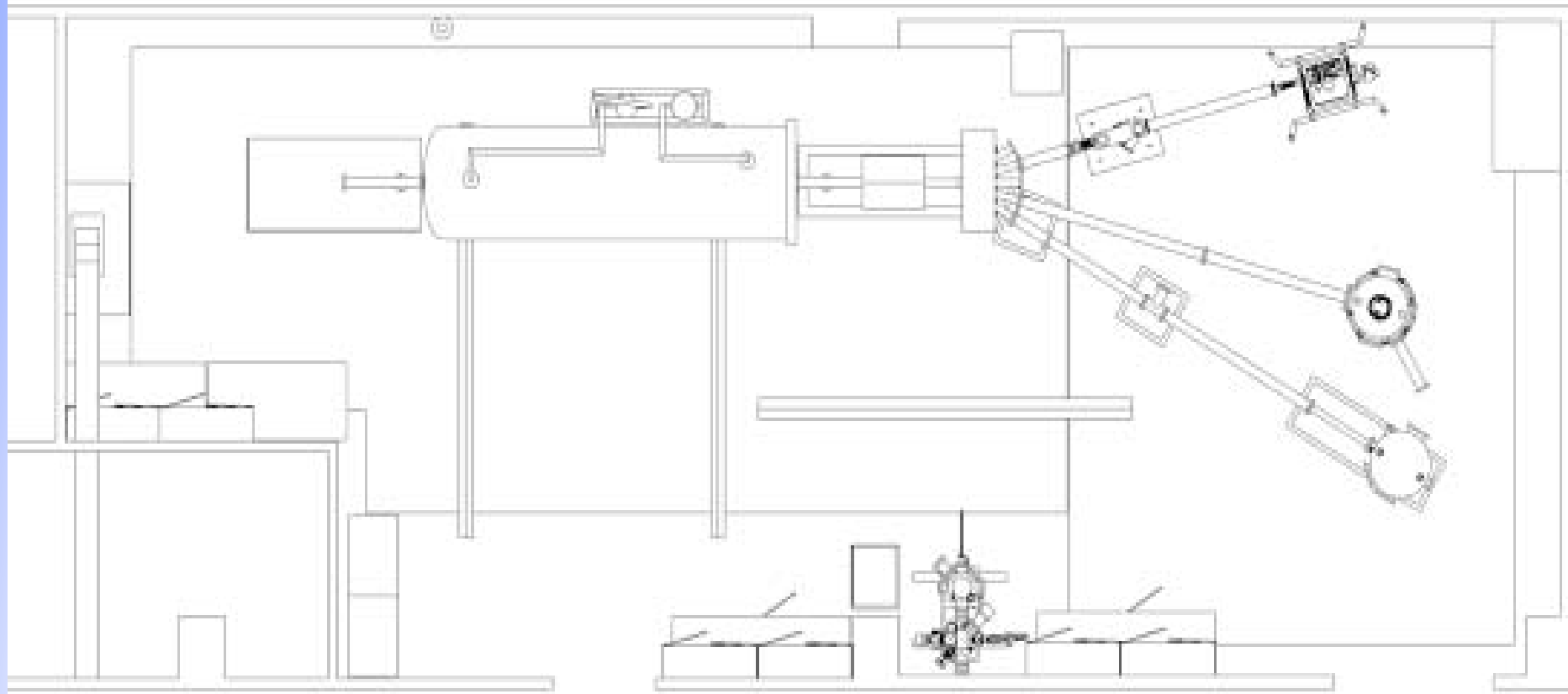

Target hall

30th of November 2006

Installing of the Pelletron

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14th of December 2006

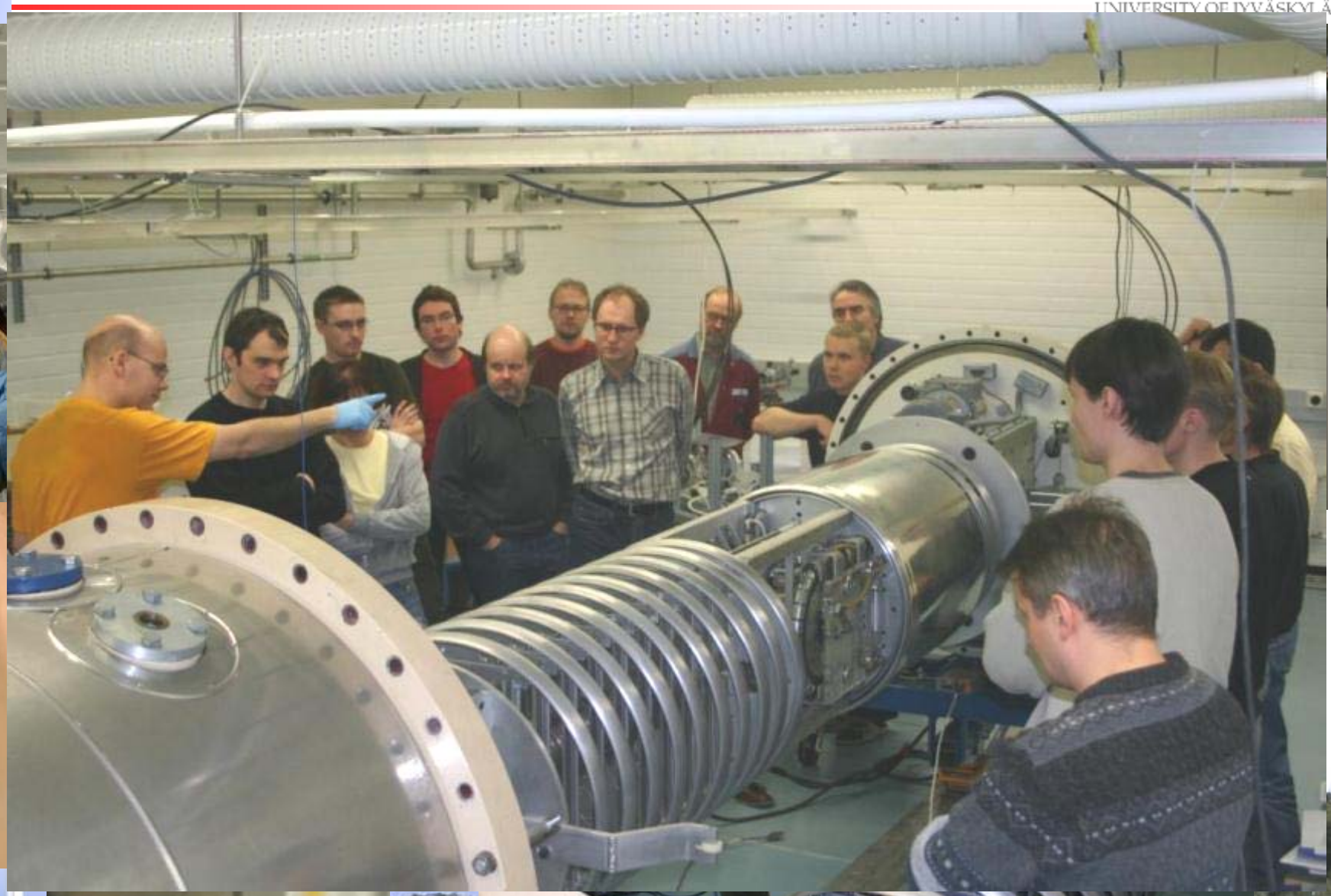
Installing of the Pelletron

UNIVERSITY OF JYVÄSKYLÄ

17th of December 2006

2nd of January 2007

Cleaning of the Pelletron



18th of January 2007

Powering up of the Pelletron acc.

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6th of February 2006

Ion source cleaning + comissioning

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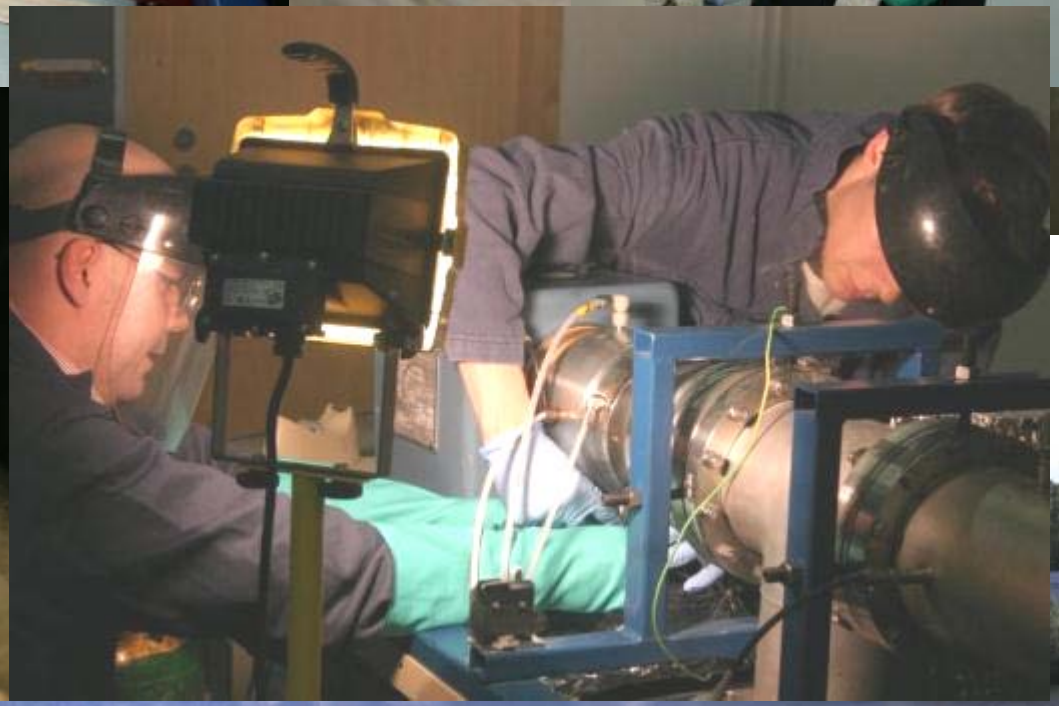

First RBS measurements

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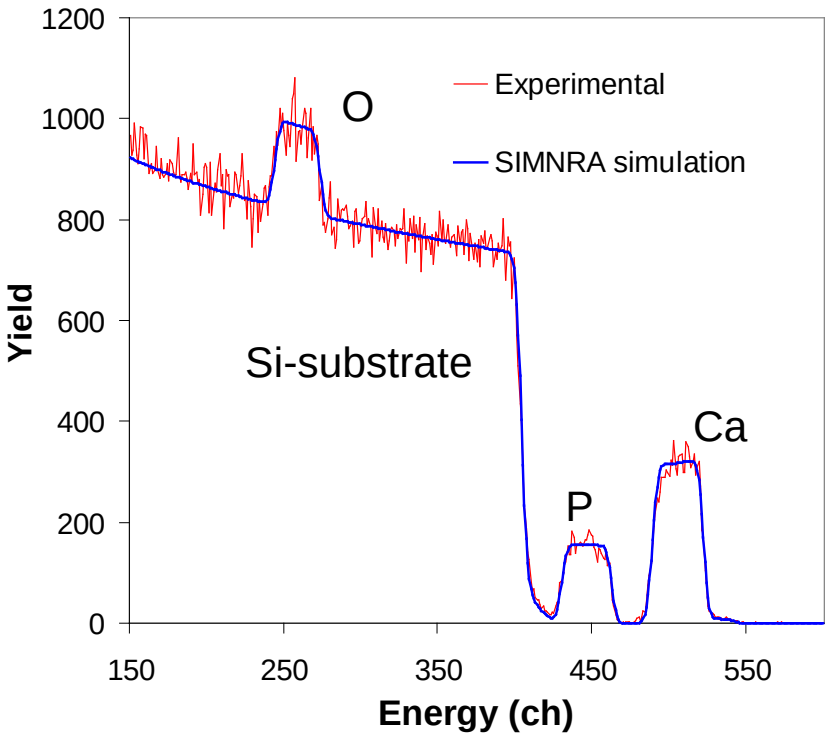
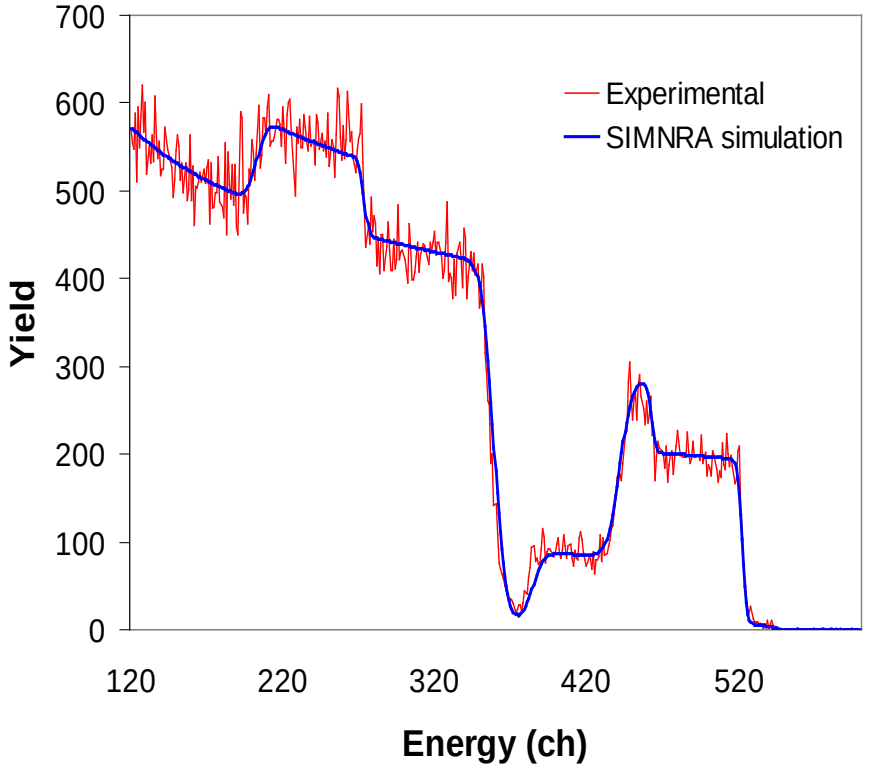
Thin (170 nm) hydroxyapatite



Thick (550 nm) hydroxyapatite



Ca/P ratio 1.18

Ca/P ratio 1.33

Beam on target: 21.2.2007

Total commissioning time from 'amateurs': ~2months

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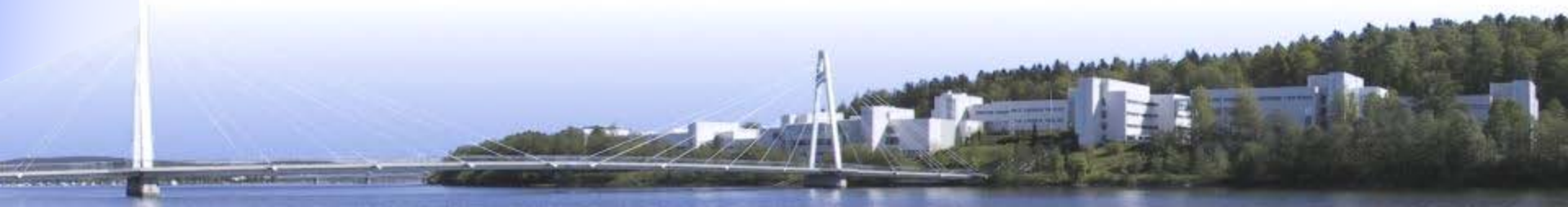
WATER LEAK AT THE SF6 HEAT EXCHANGER



Water leakage from the heat EX

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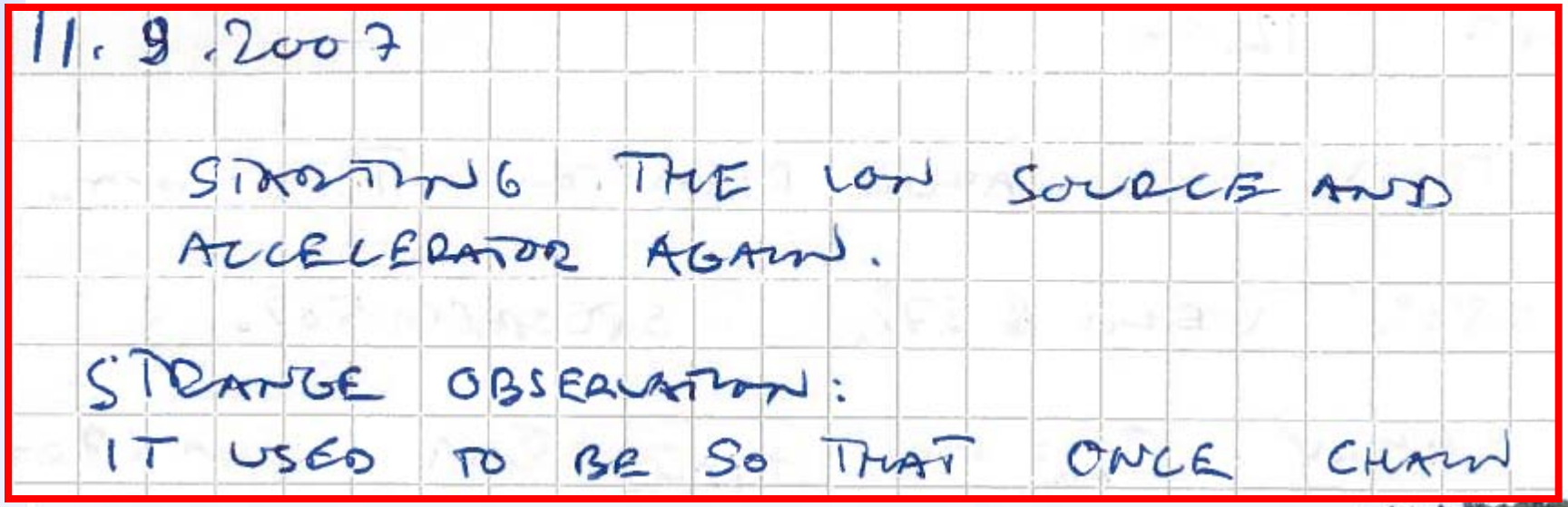
- First symptom: Voltage(GVM) didn't go DOWN when chains were turned off
- Real signal: Voltage rised very poorly, even after long "conditioning" (about 1.5 weeks from first symptoms)


11.9.2007

STARTING THE ION SOURCE AND ACCELERATOR AGAIN.

STRANGE OBSERVATION:
IT USED TO BE SO THAT ONCE CHAIN

Beginning of September 2007

Water leakage from the heat EX

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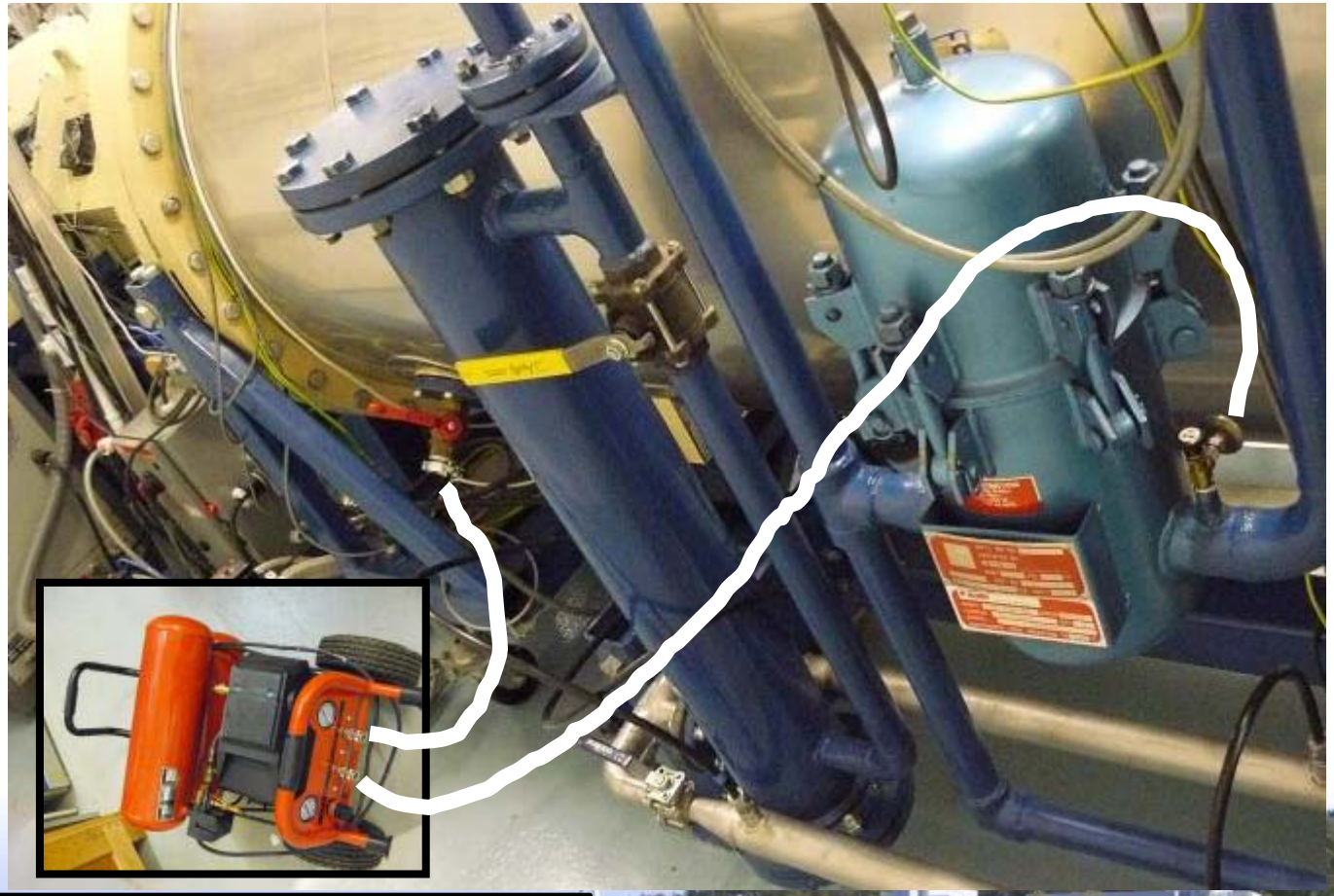
- Measuring the purity of SF6: 99.6% (27.9.2007)
- Water content in SF6: **7500 ppm_V** !!! (19 @ VTT)



5th of October 2007

Water leakage from the heat EX

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5th of October 2007, almost a month from first symptoms

Water leakage from the heat EX

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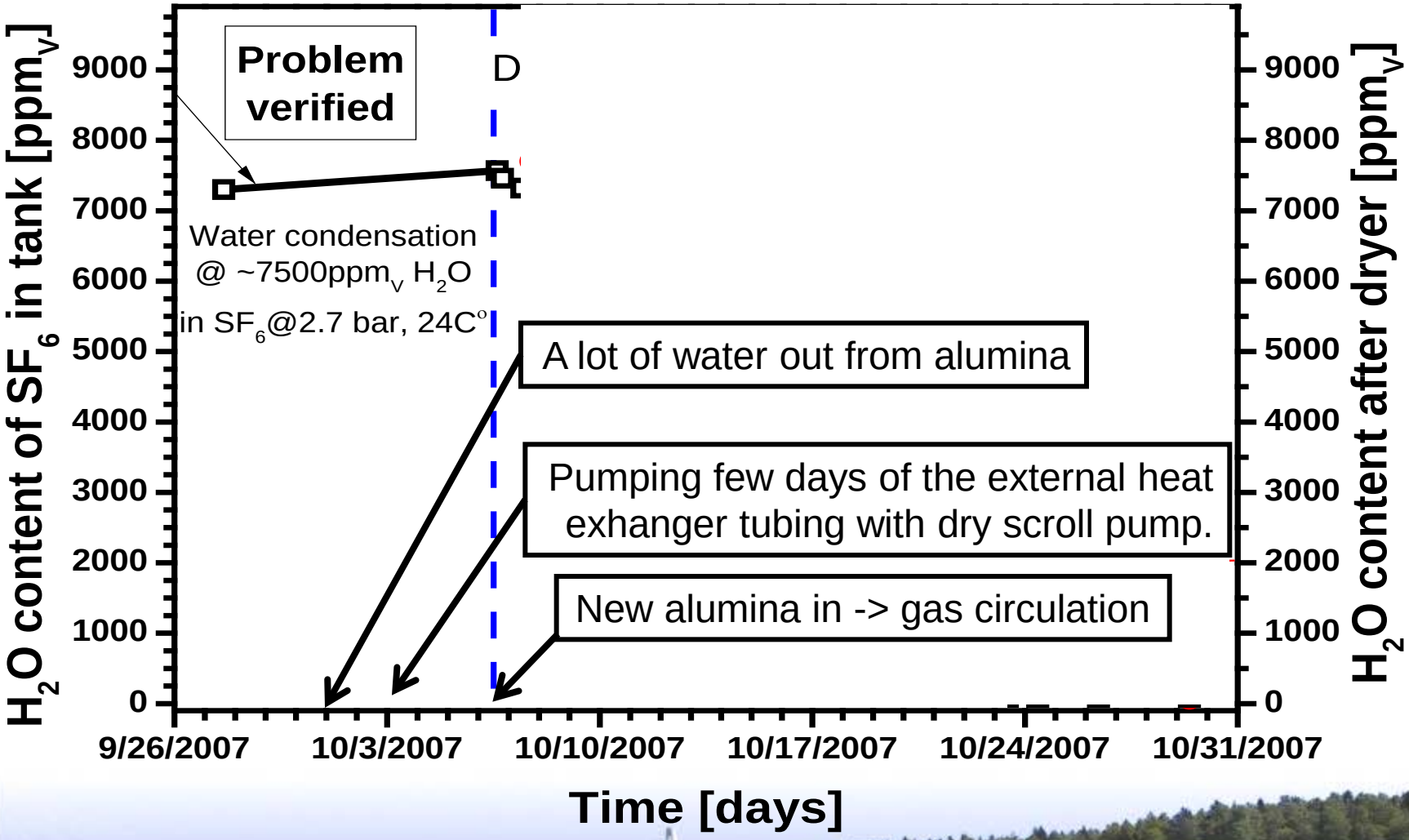

Water leakage from the heat EX

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- Conclusions and aftermath of the water leakage after tank opening + cleaning:
  - There had been few cm of liquid water inside the tank bottom (“high water” -mark)
  - New “pure” ion exchanged water at new lab might enhanced corrosion of the rusted Cu pipes of the original heat exhanger
  - Only aftermath was most likely the GVM bearings that failed less than month later: forced tank opening and gas recovery

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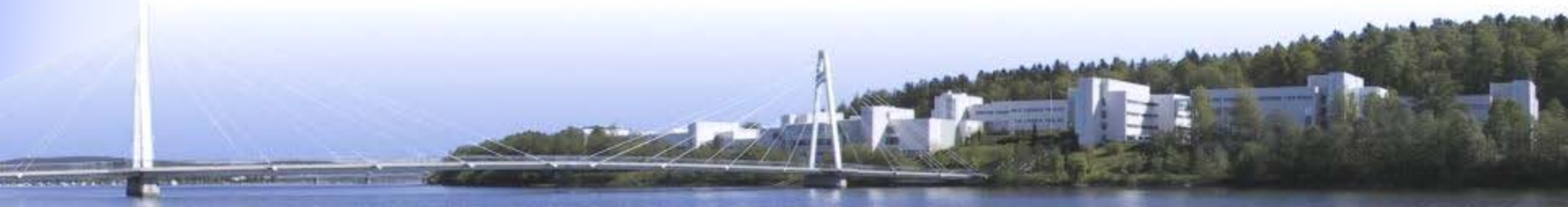
FROM CORONA NEEDLES TO RESISTORS AND TURBOPUMPED STRIPPER SYSTEM INSTALLATION



From corona needles to resistors

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- 550 Mohm resistors ordered from NEC to replace some 60 corona gaps
  - Resistor based charge division change was relatively easy, except tight space
  - Lower voltages far more stable, accelerator has been run with 75kV at terminal

From corona needle based voltage division to resistors

2008

Turbopumped stripper change

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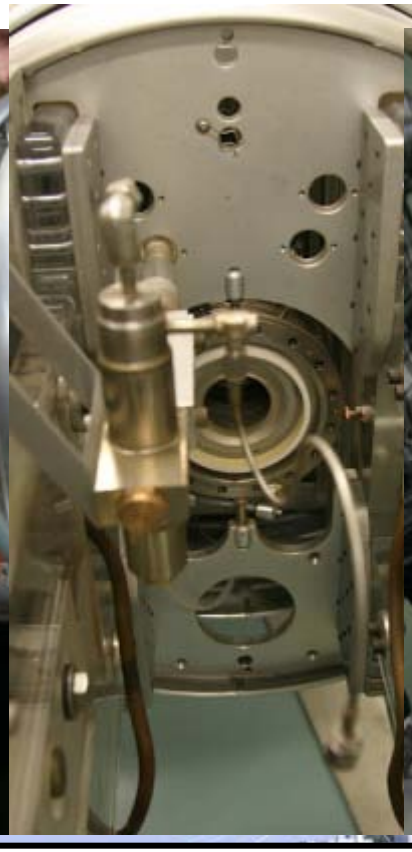
- Original: N_2 gas stripper, extra gas pumped through HE- beam tube.
- Recirculation by turbo: About order of magnitude lower pressures at HE-side for same charge state distributions, even with larger holes at LE and HE terminal.
  - Less beam (charge state-)contamination due to residual stripper gas.



Turbo pumped stripper system 2012: higher transmission

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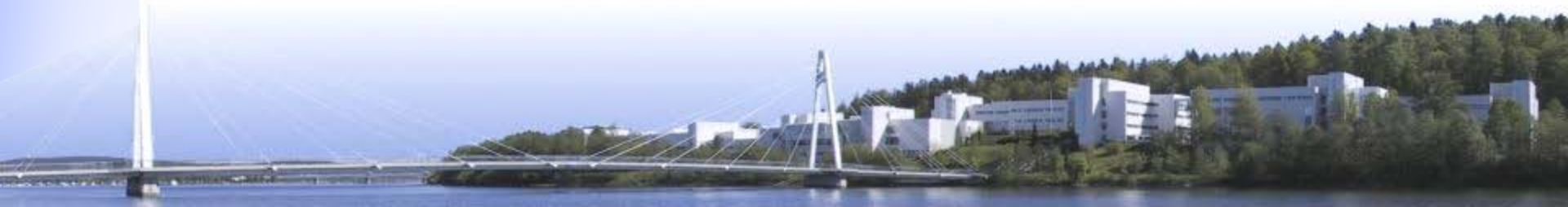
BEAM LINES AND RECENT DEVELOPMENTS



The lab and the beam lines 2009

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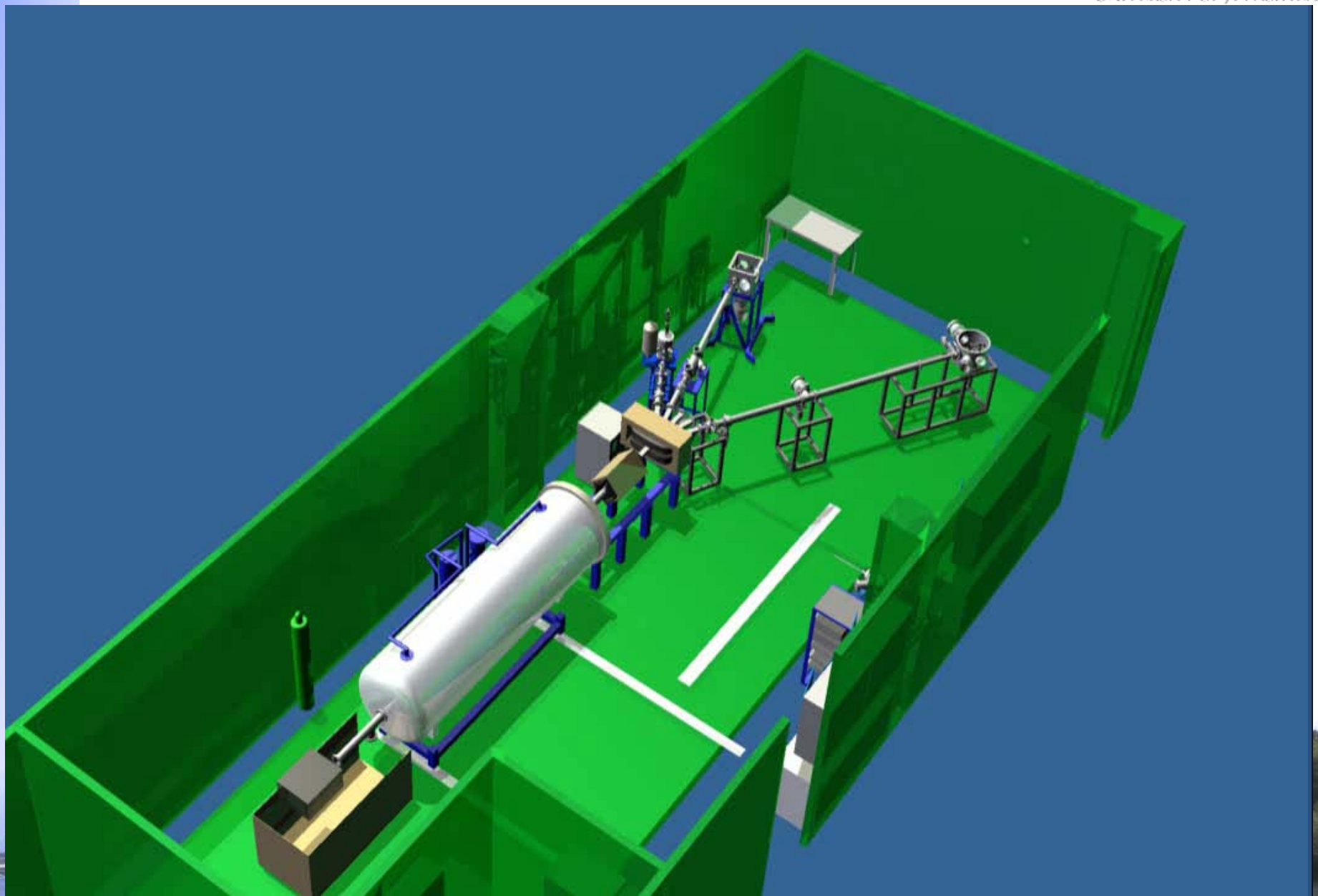

Low energy heavy ion ERDA

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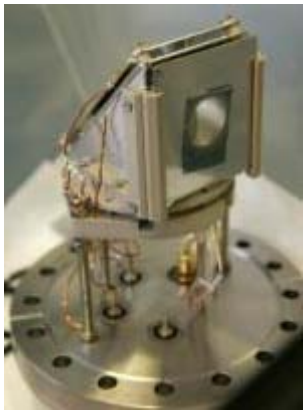
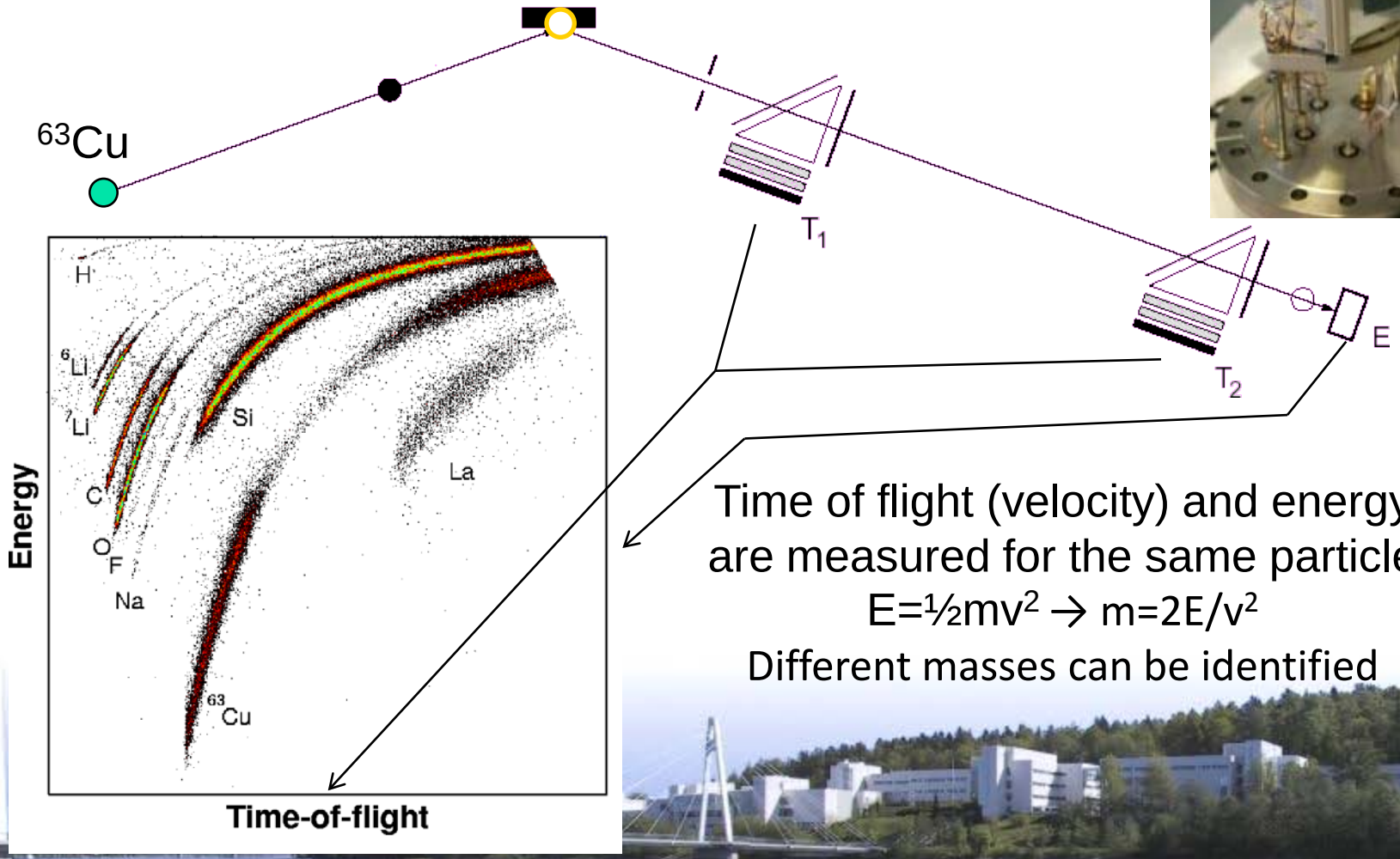
- Typically 1–20 MeV Cl, Br, I or Au ions from 1–3 MV tandem accelerator
- Time-of-flight–energy spectrometers for isotopic identification and energy spectrum measurement





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

Example: Thin film with high mass element

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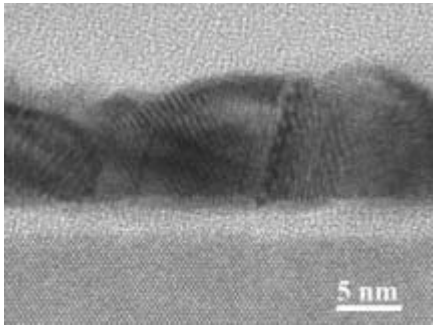
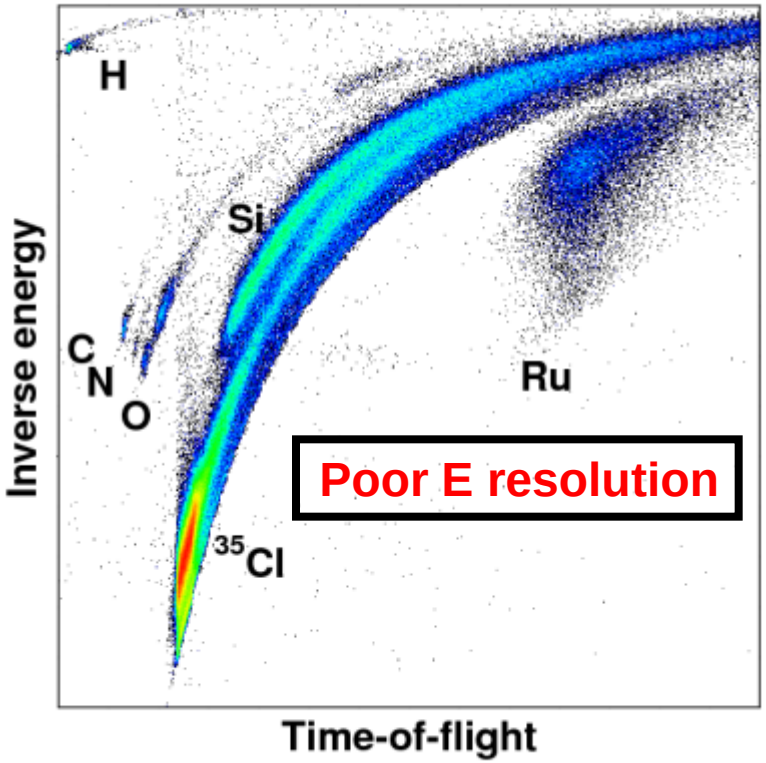
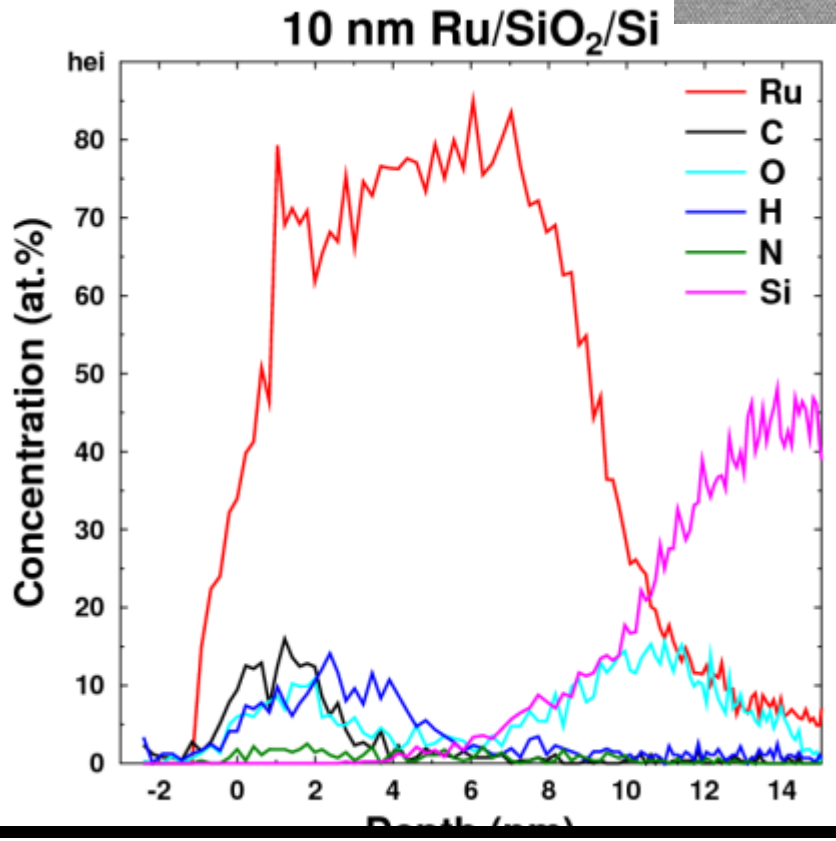
- Atomic layer deposited Ru film on HF cleaned Si
- Scattered beam, ^{35}Cl, used for Ru deph profile
- Monte Carlo simulations needed for getting reliable values for light impurities at the middle of the film







Low energy heavy ion ERDA – See posters!

Gas ionization detector to replace Si-energy detector

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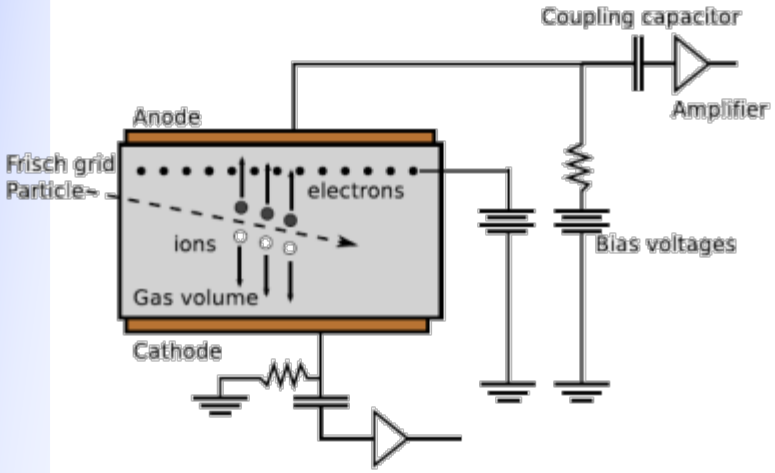
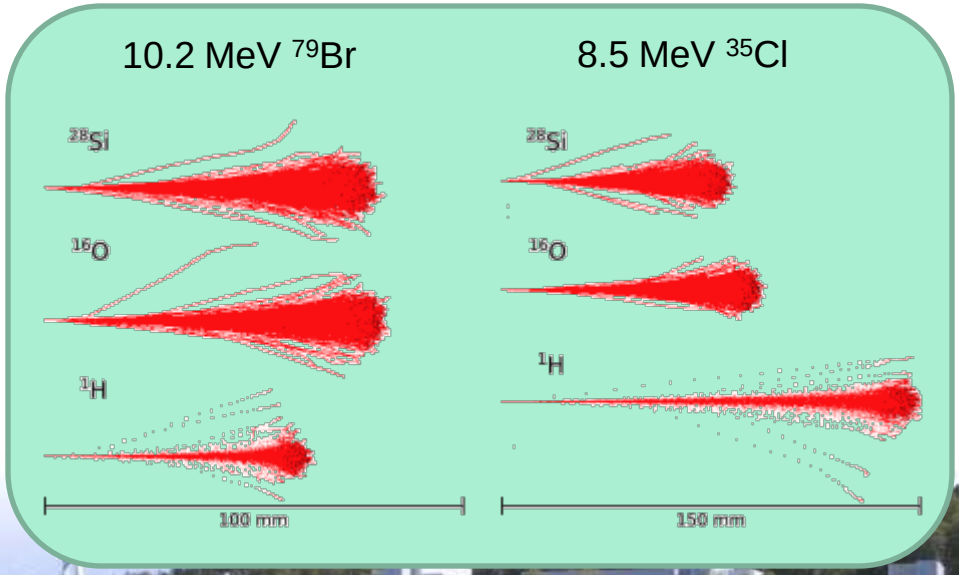
- Why try to fix a well working system?
  - Greatly improved energy resolution for low energy heavy ions → heavier masses can be resolved
  - Gas detector is 1D position sensitive by nature → possibility for kinematic correction and therefore larger solid angles possible
  - Gas detector does not suffer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



Recoil ranges in isobutane



Gas ionization detector develoment – See posters!

Gas detector performance

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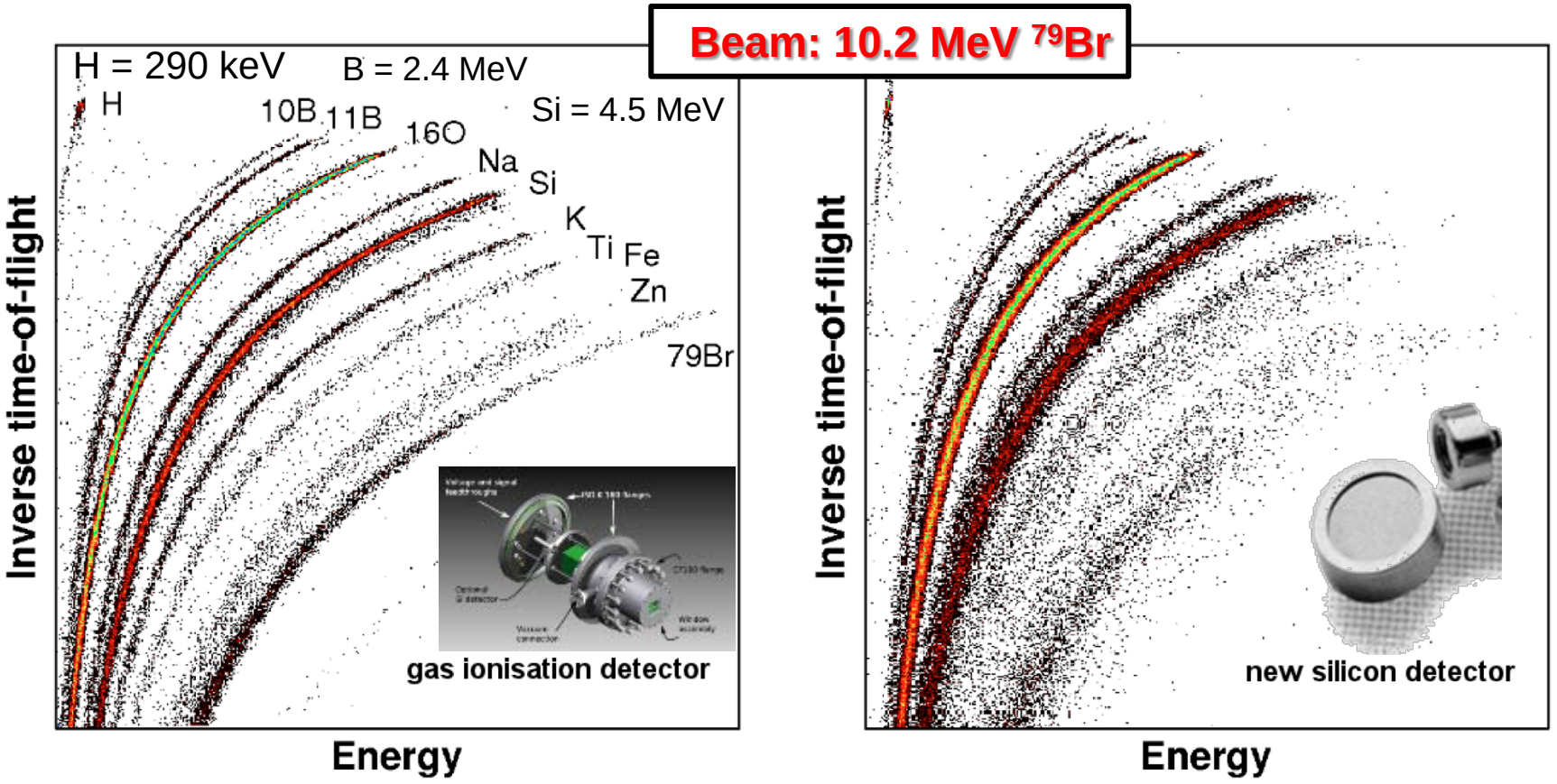
- Same borosilicate sample is measured with ToF-E, with two different energy detectors: a gas ionization detector with thin SiN window and new Si-detector



Gas ionization detector with thin SiN window

‘Contaminants’ after analyzing magnet

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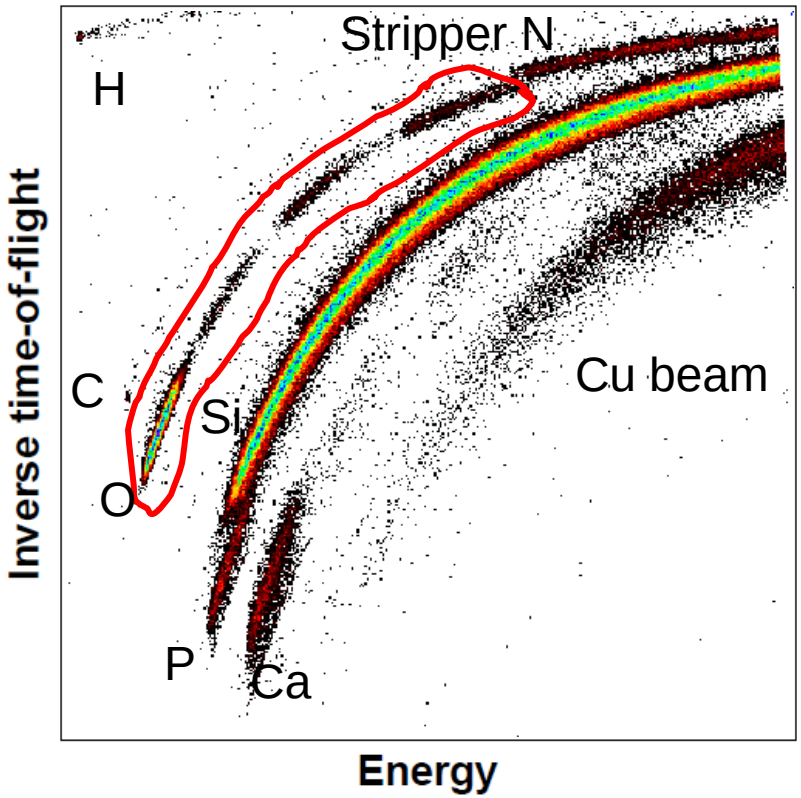
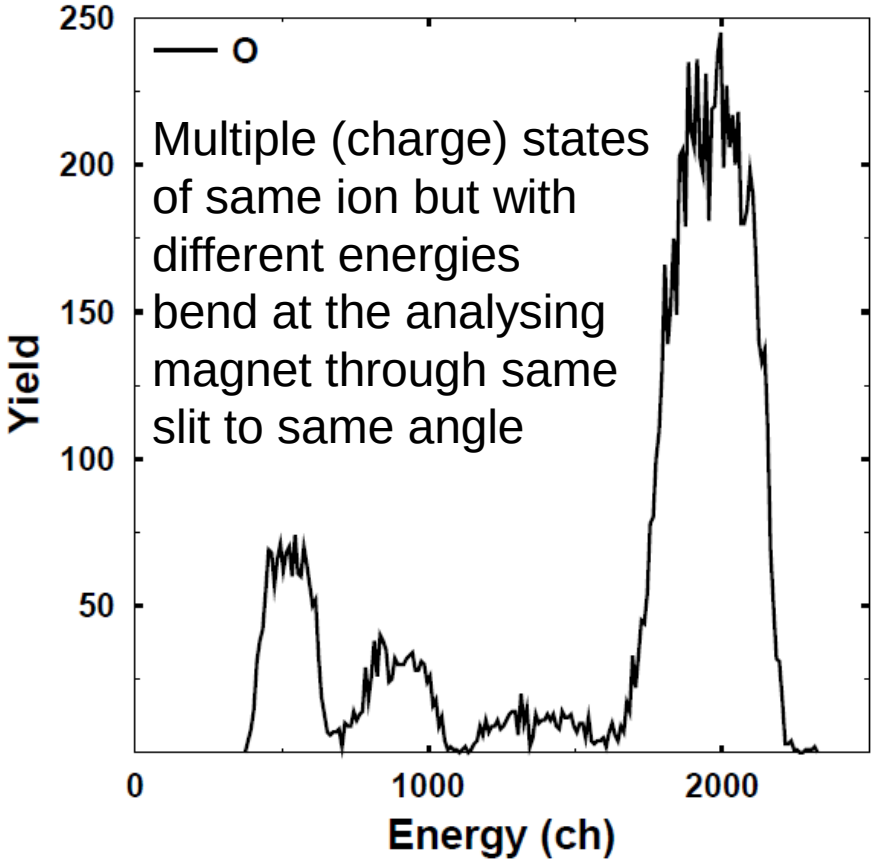
- 13.6 MeV $^{63}Cu^{7+}$ CaPO (hydroxyapatite)





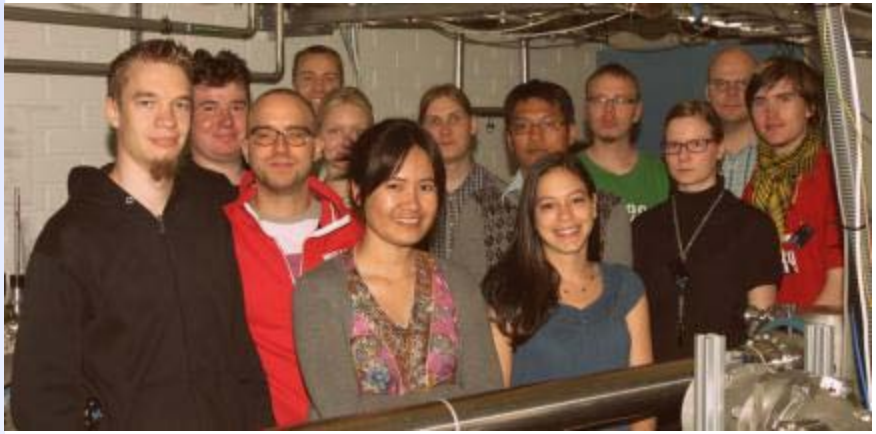
Is this mostly due to stripper gas effect in HE column?

Acknowledgements

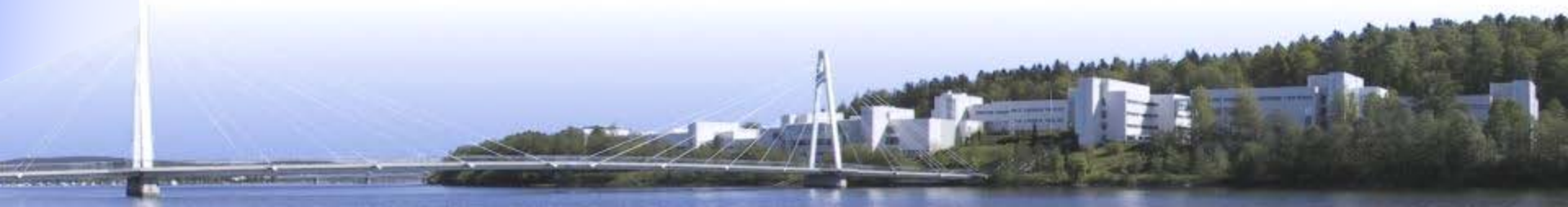
TEKES-EU Regional Funds

Academy of Finland

TEKES



Accelerator based materials physics group at JYFL



Pelletron Laboratory

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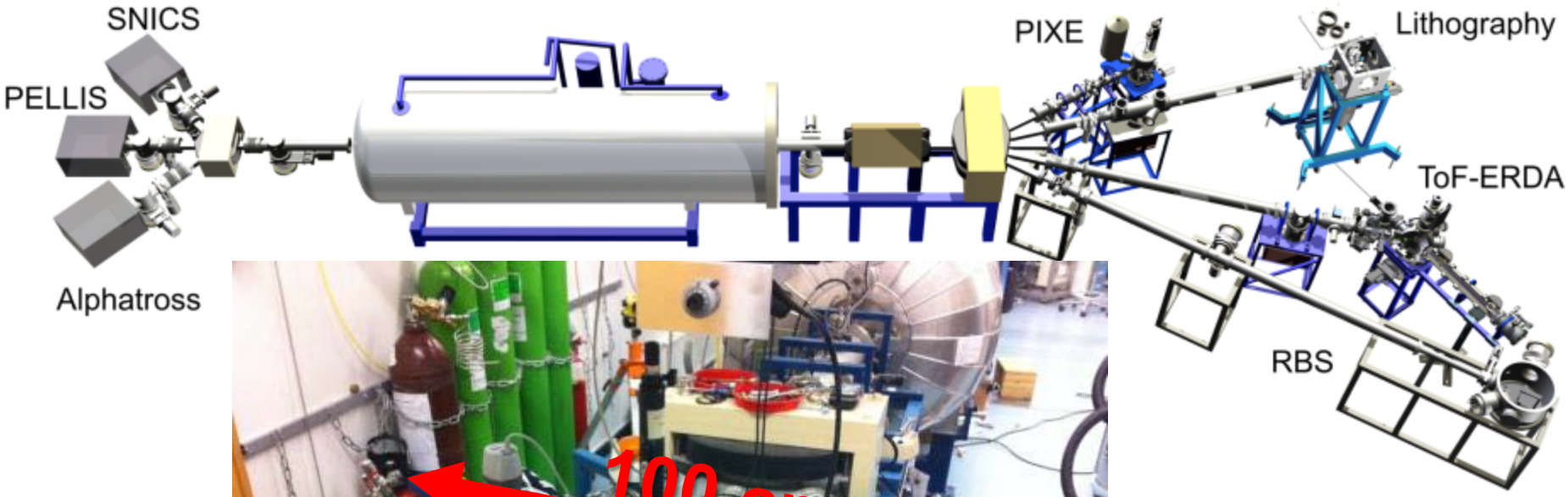
- 25 years old 1.7 MV Pelletron accelerator, in Jyväskylä since 2006
  - Available beams and energies: from H to Au, from 0.2 MeV to 20+ MeV



Three ion sources within ~2 m^2 !

Ion sources

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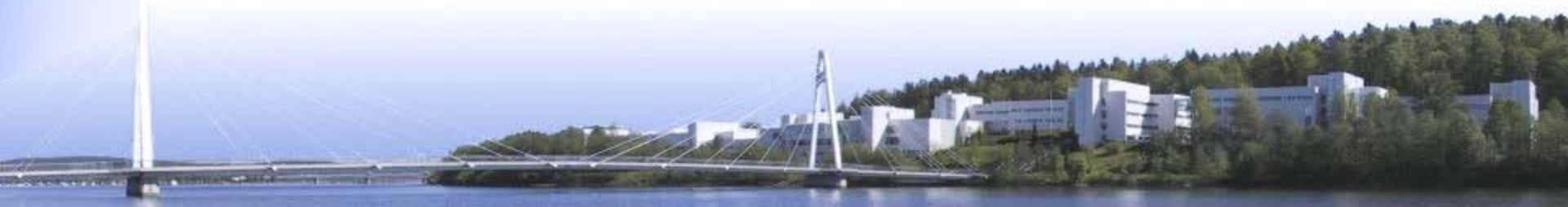
- Mean life time of the ion source more important than 2 more beam
- Maintained by group, development together with ion source team

Ion source name	Ion source type	primary ion(s)	Typical intensities
Alphatross	100 MHz RF, Rb charge exhange	He^-	250 nA
SNICS '1'	Cs sputtering, single cathode	C^-, Cl^-, Cu^-, Br^-	2 000 nA
PELLIS	Filament driven multicusp	H^-	15 000 nA

Ion source name	Typical operational parameters			
	"cathode" HV, current	filament P, oven T	Einzel focusing HV	'Extraction' lens HV
Alphatross	+5 kV, 0.75 mA	250 °C / 55 °C	none	-8 kV
SNICS '1'	-4 kV, < 0.2 mA	19 A, 6 V	-8 kV	-8 kV
PELLIS	+100 V, 1 A	70 A, 3 V	-5.5 and -3.4 kV	-10 kV

Where does the ions end up



Example: Thin film with high mass elements

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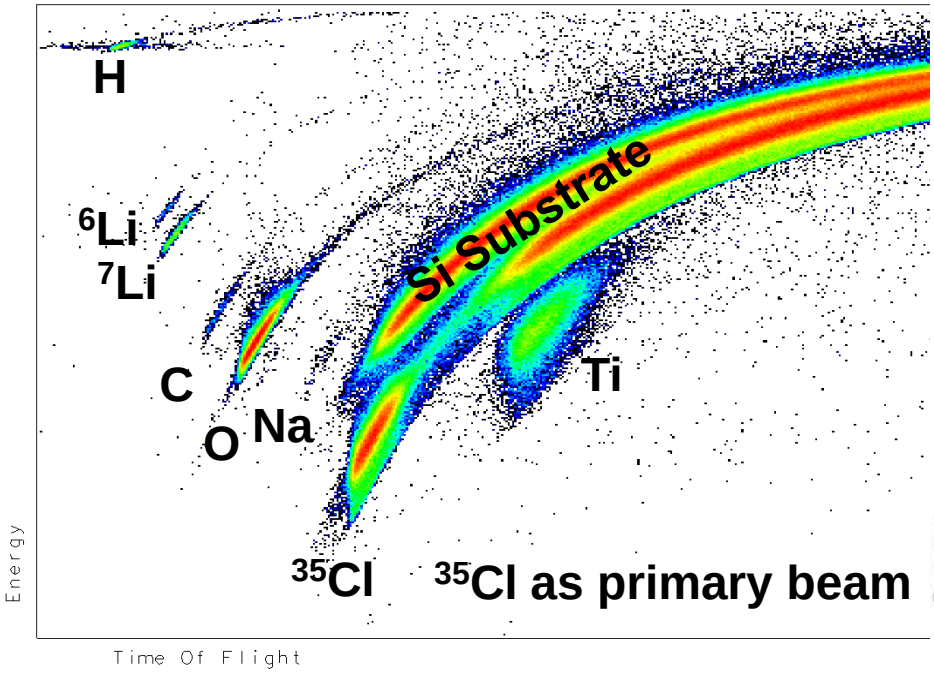
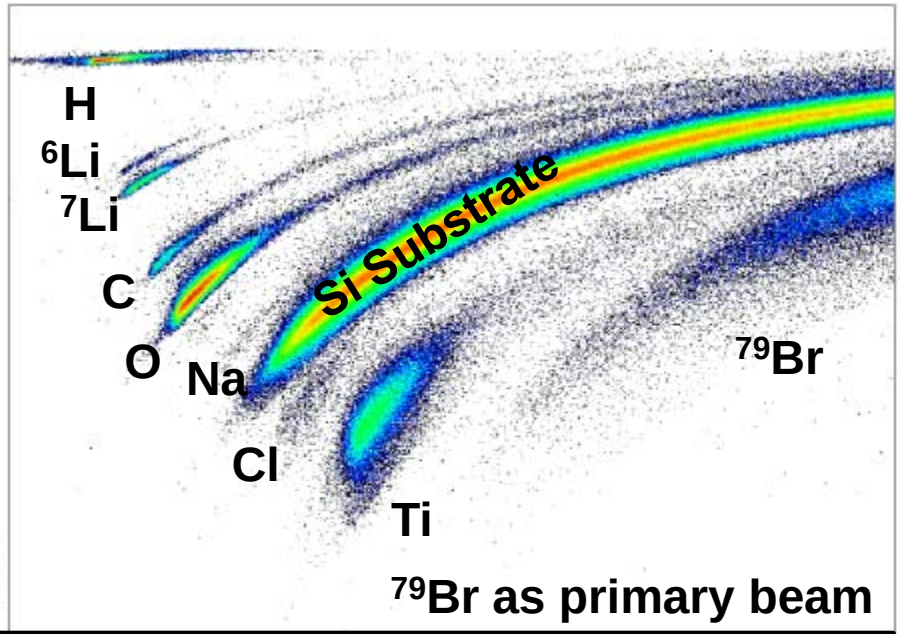
- In ALD impurites from carrier gas or precursors may interfere in the process

EXAMPLE: LiTiO thin film, ~50 nm : Cl^{35} VS Br^{79}

- **^{35}Cl close to perfect beam but <u>cannot probe the Cl impurities</u> in the film**
- **Heavier ^{79}Br beam needed for this 'same as beam mass impurity' search**
- **Br beam suffers from multiple scattering and cannot probe whole film**





Poor E-resolution for heavy elements

Low energy heavy ion ERDA

Lithography with light ions

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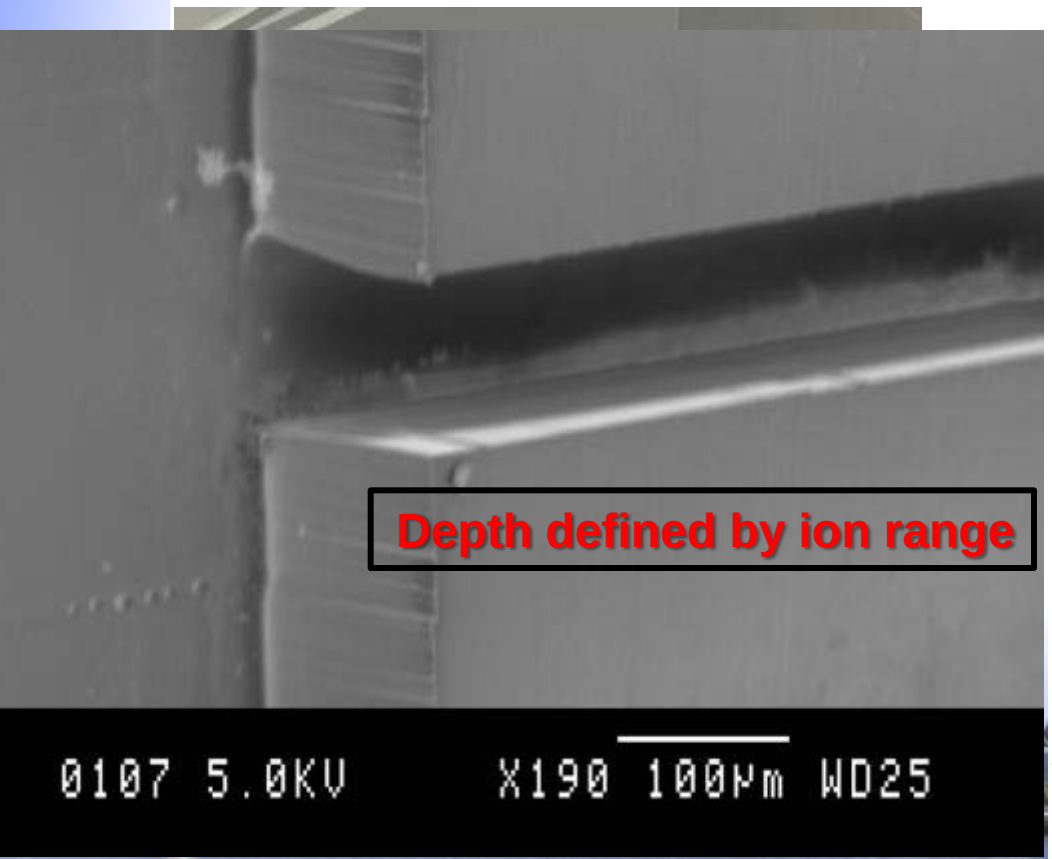
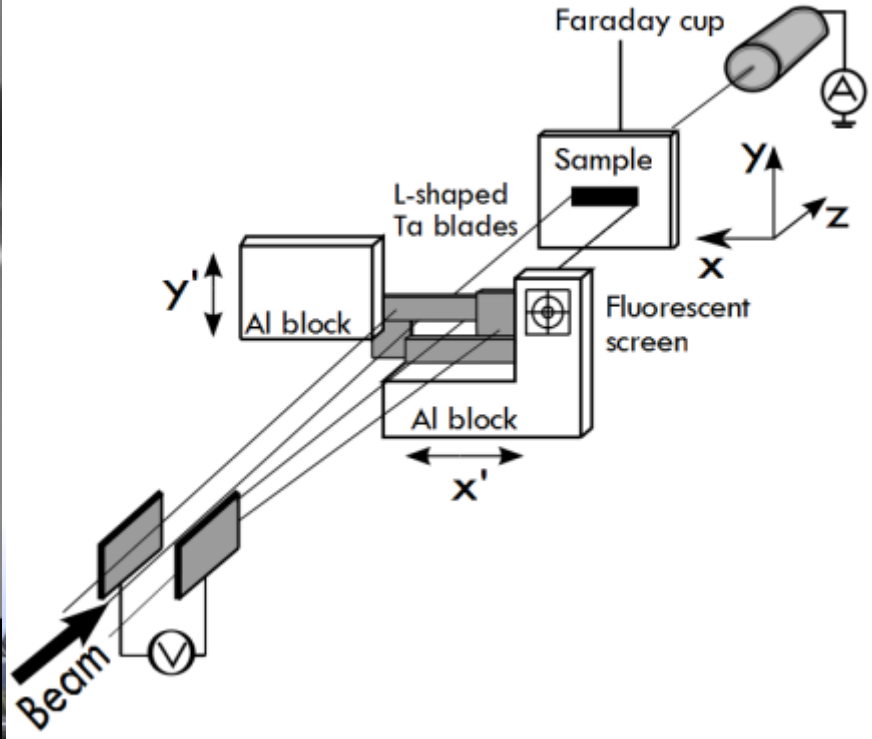
- Ion beam lithography enables direct writing of deep ’3D’ structures, for example microfluidistic channels to the resists (PMMA) or even quartz.
- Stability in energy and fluence most critical, as for very small beam sizes the online monitoring is challencing.
- Uniform beam(up to ~30%), parallel exposure trough slits → fast prototyping.





Lithography with light ions

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- Ion beam lithography enables direct writing of deep ’3D’ structures, for example microfluidistic channels to the resists (PMMA) or even quartz.
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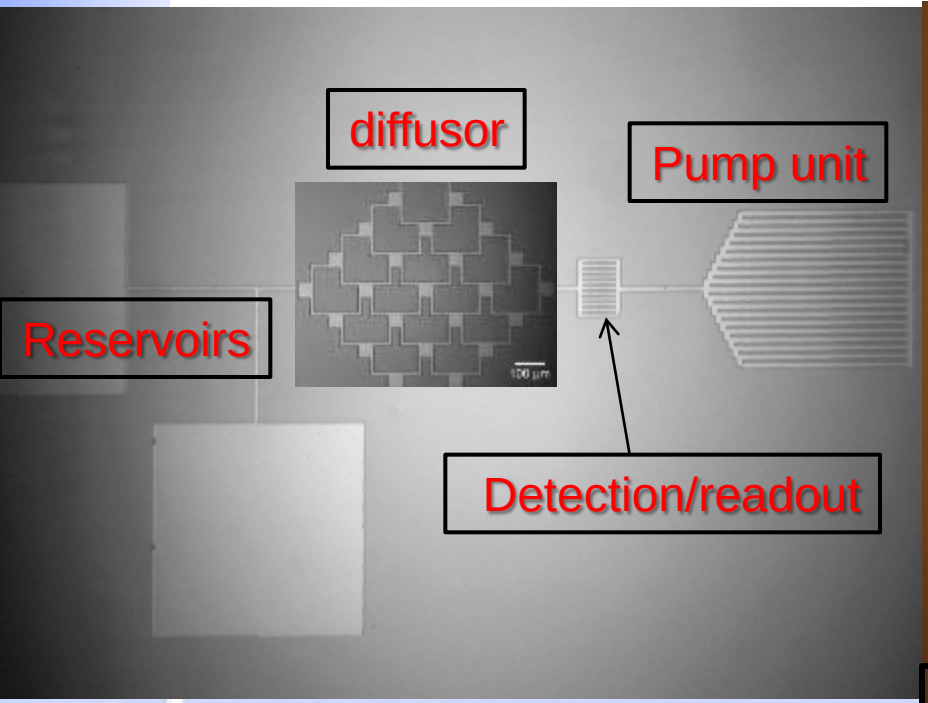
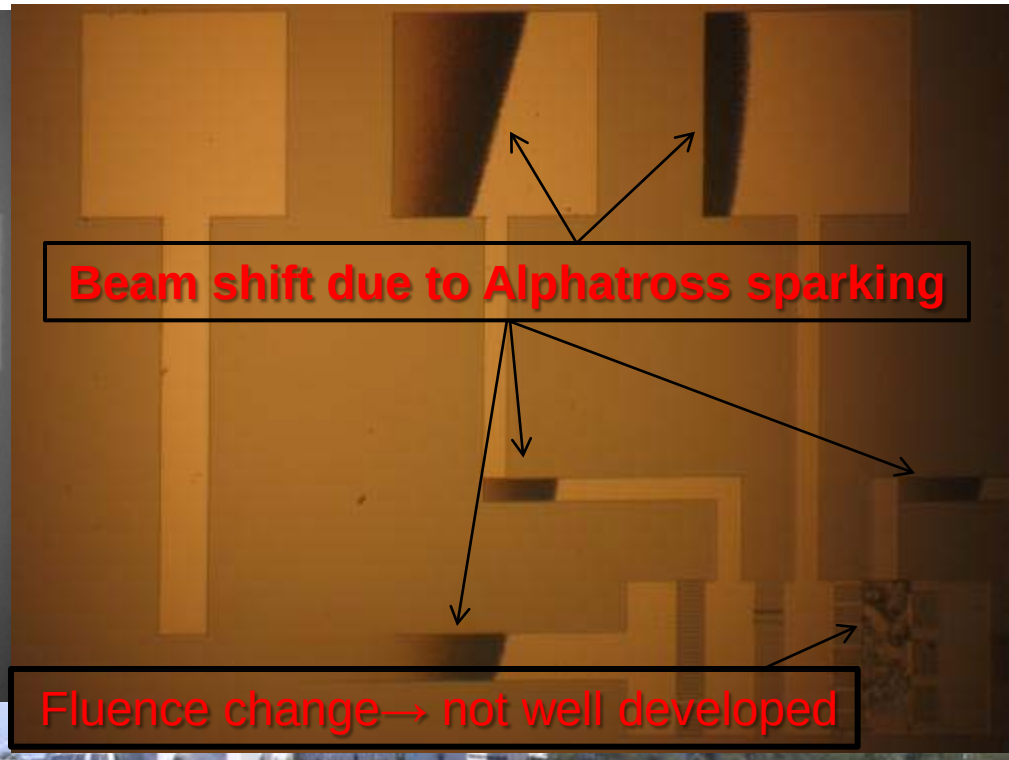
Example which has OK parameters



Example with parameters NOT OK



Need for negative helium

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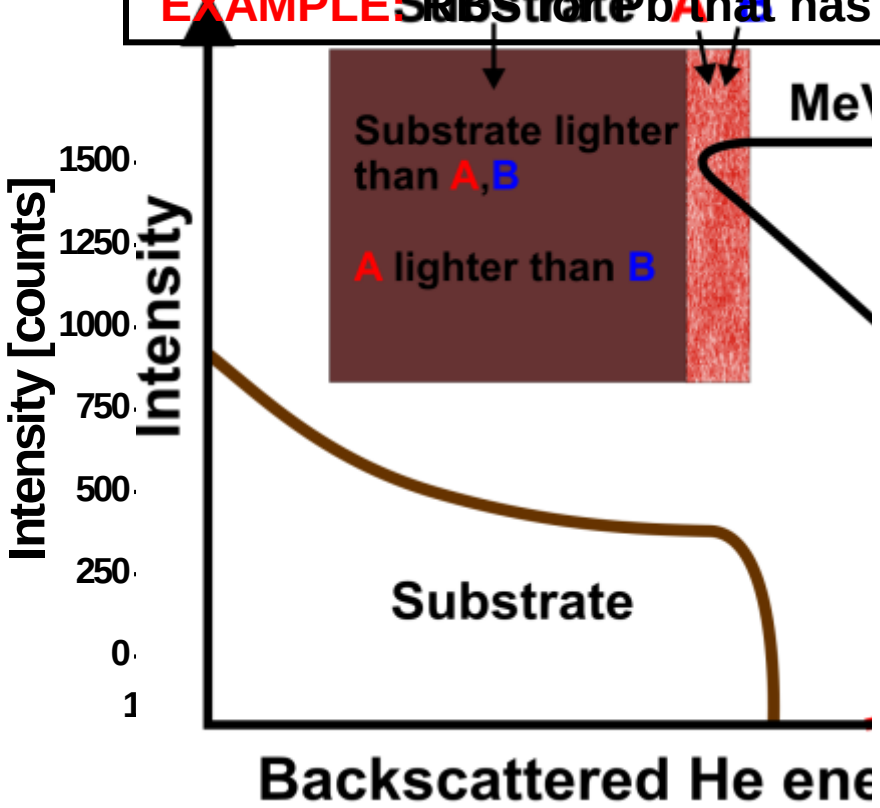
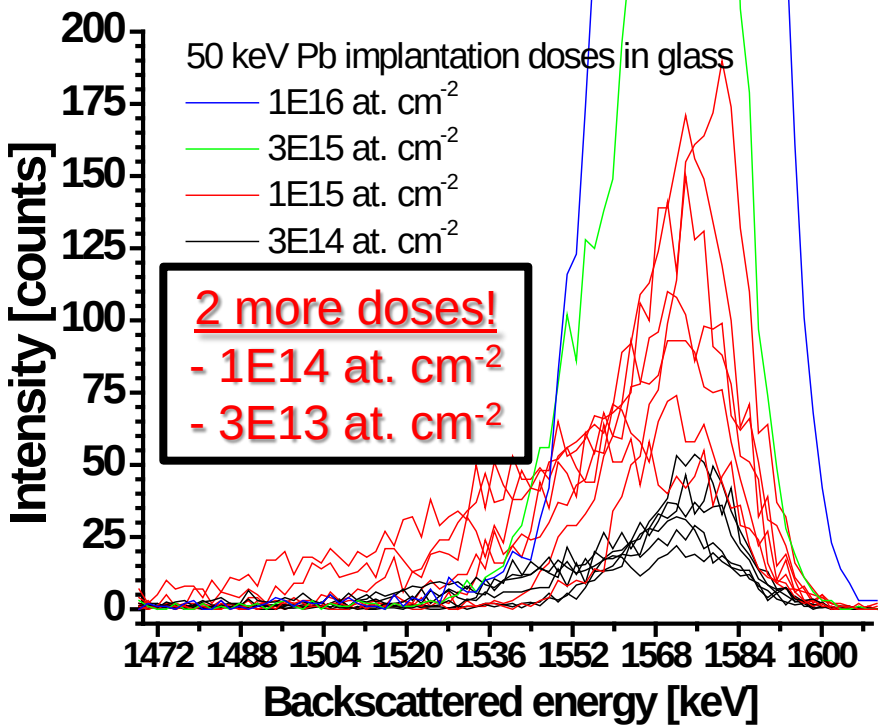
- Rutherford Backscattering Spectrometry (RBS) uses often He to probe the sample from the surface (few 10's of nm to few µm).
- Higher the energy, lower the backscattering yield, but better mass resolution (and relative energy resolution at silicon detector).
- Lower the energy, better the depth resolution, but worse transmission through the accelerator → High He input current needed from ion source.

EXAMPLES: RBS for Pb that has diffused in glass at elevated temperatures





What is needed: Conclusions I

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- ToF-ERDA: Selection of heavy ions that are fast to switch, stability in intensity not that important but higher charge states from small accelerator is needed for reasonable count rates.
- Lithography: Stability in both energy and intensity most important. Light ions can create deep ’open’ structures where heavy ions can create closed channels directly (Bragg peak).
- RBS: Helium beam most used beam as it can separate heavy target/sample masses from each other
- PIXE (particle induced x-ray emission): Protons or helium most often used. Data can be collected often together with other methods easily.

What is needed: Conclusions II

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- For ion beam applications, for both characterization and modification variety of negative ion beams is needed.
- Stable in energy and fluence, easy to maintain ion sources are priority parameters over higher beam intensities.
- Protons: PELLIS H^- source performs very well with long life times and is easy to operate by users.
- Helium: Alphatross currently has poor to worse performance. Upgrades coming: helical RF-plasma coupling and temperature stabilized Rb charge exchange chamber for stable Rb backflow to oven.
- Heavy ions: SNICS ’1’ has moderate performance. ”New” 40 MC-SNICS to be installed still in this year.

Growth of Al_2O_3 on TiO_2

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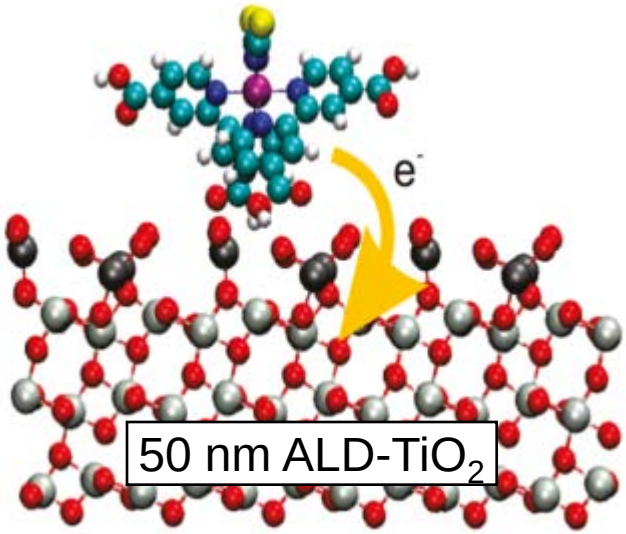
- In dye sensitized solar cells even single ALD cycles of Al_2O_3 were found to reduce the interfacial electron transfer between semiconductor TiO_2 and dye molecule. This improves the performance of the cell.
- How thick films of Al_2O_3 were deposited during first cycles of ALD growth?



Liisa Antila, Mikko Heikkilä, Viivi Aumanen, Marianna Kemell, Pasi Myllyperkiö, Markku Leskelä, and Jouko E. I. Korppi-Tommola
J. Phys. Chem. Lett. 1, 536 (2010).

Minimizing background

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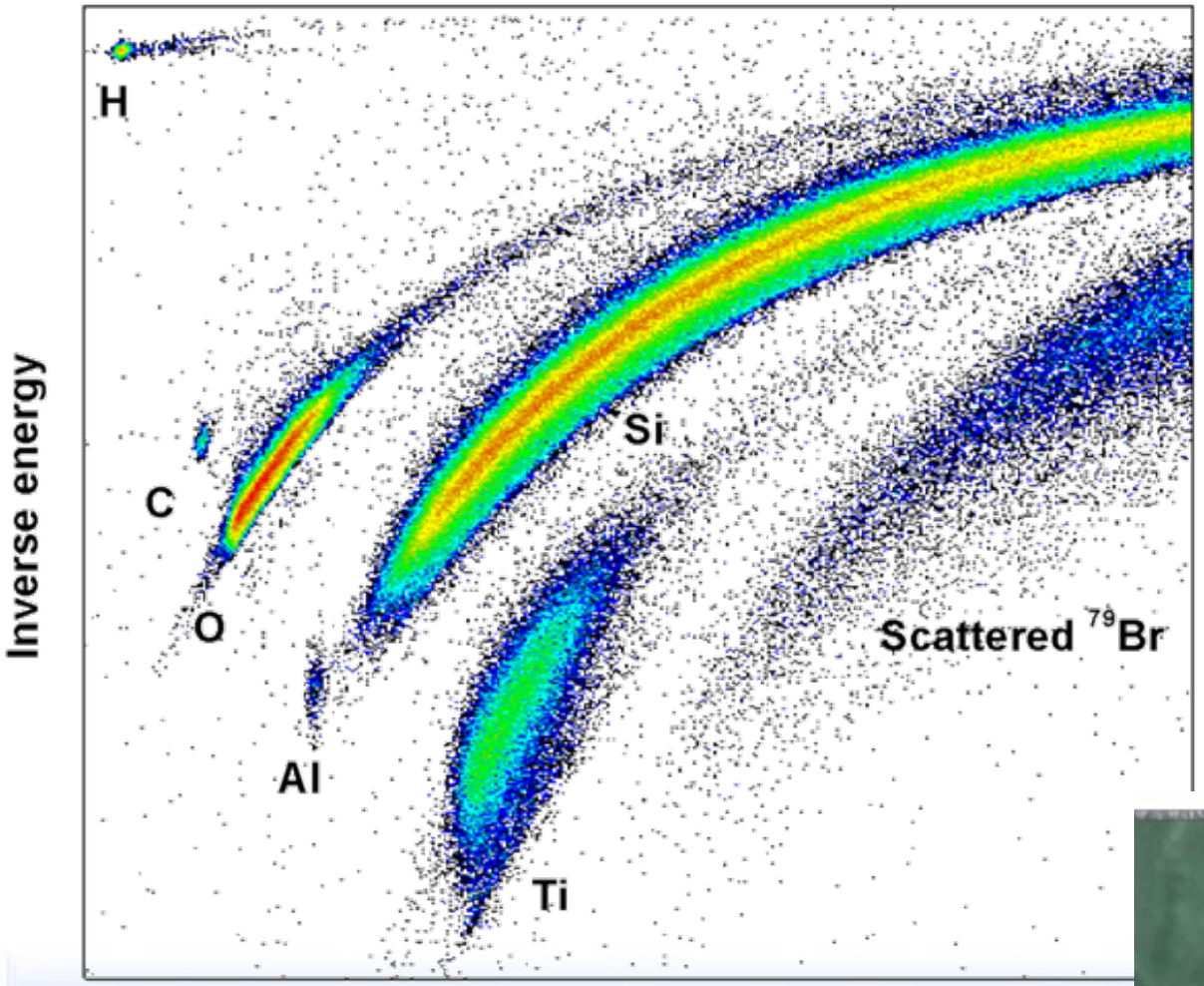
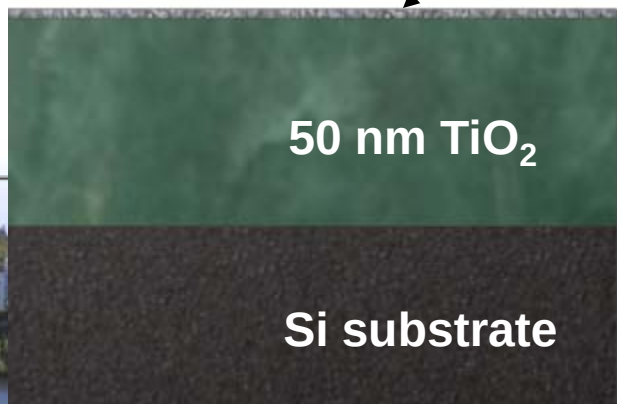


Sample:
50 nm TiO_2 on Si substrate where 5 ALD cycles of Al_2O_3 has been grow on to (corresponding 0.5 nm thickness)

All counts visible on log intensity scale

5 ALD cycles of Al_2O_3



Growth of Al_2O_3 on TiO_2

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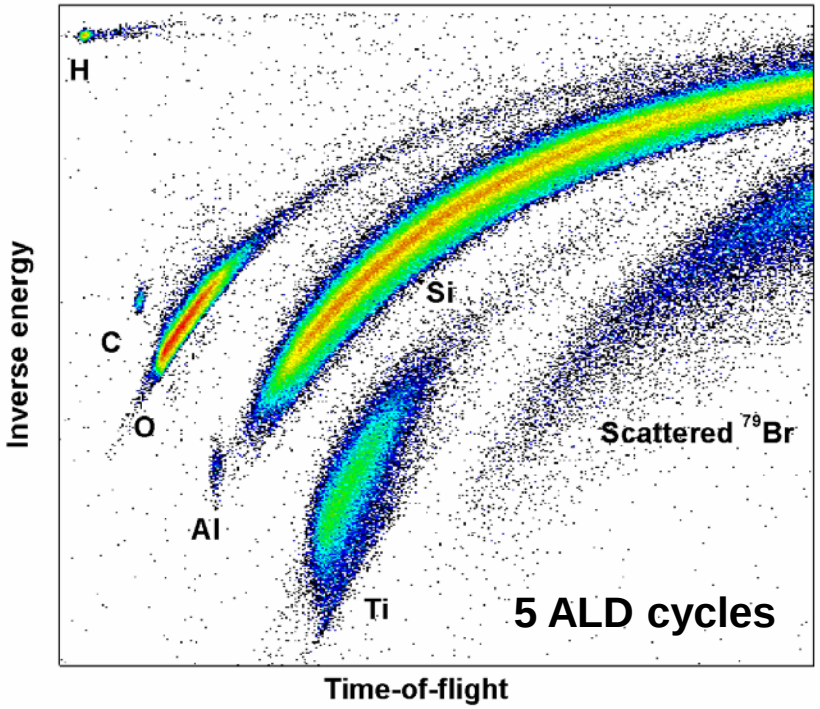
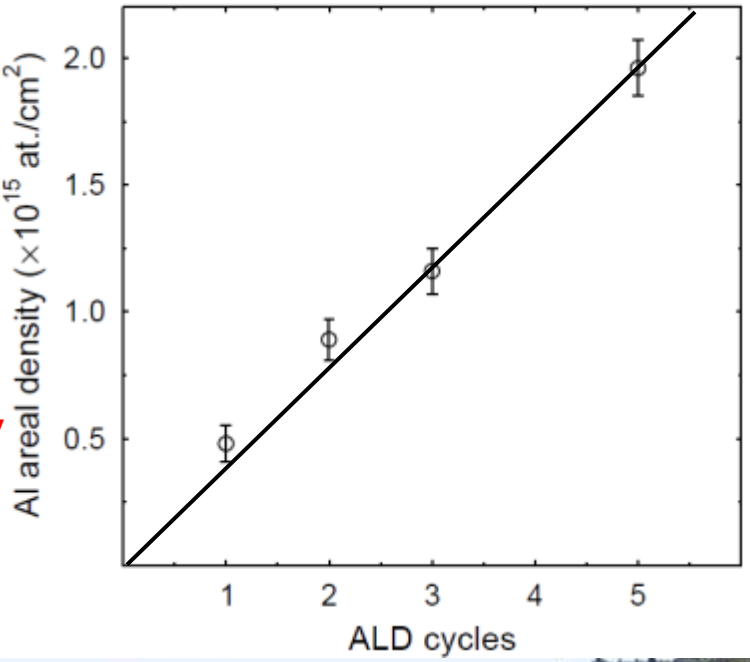
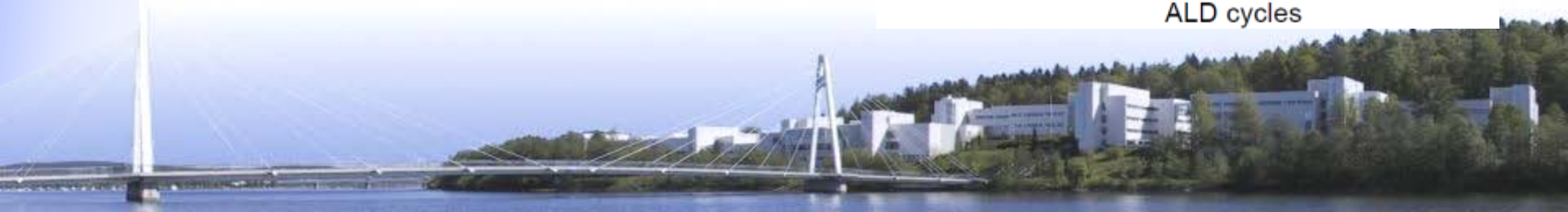


- Samples with 1, 2, 3 and 5 cycles of ALD Al_2O_3 on 50 nm ALD TiO_2 were studied
- Measurements using 8.26 MeV ^{79}Br beam



Conclusion: Al_2O_3 film growth starts rapidly from the first cycles and then the growth rate per cycle reduces



Timing gates: construction

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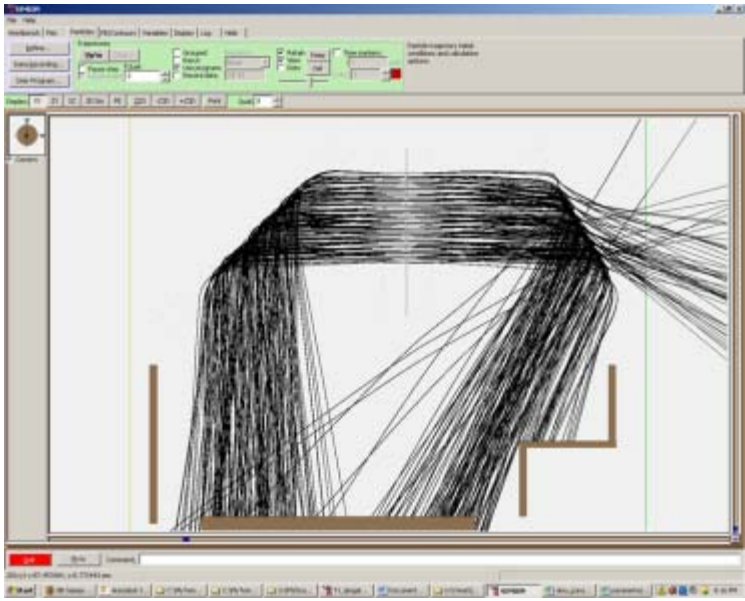
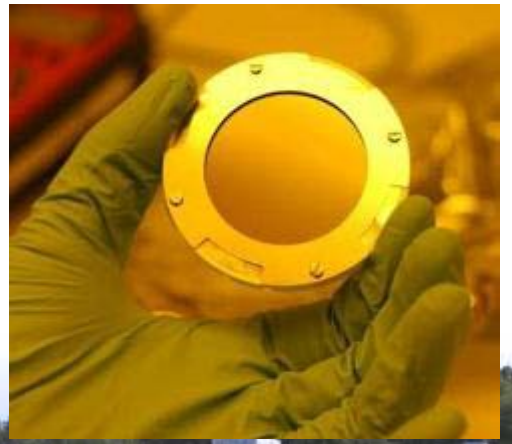


- Carbon foils
  - T1 foil 3 μg/cm^2 (diameter 9 mm)
  - T2 foil 10 μg/cm^2 (diameter 18 mm)
    - Determines solid angle of 0.29 msr
  - Distance between foils 633 mm
- Wires in the electrostatic mirror grids
  - Spot-welded 25 μm diameter Au wire
  - Wire-to-wire distance 1 mm, 97.5% transmission, telescope transmission 87%
- Micro-channel plates
  - Low-cost chevron-type MCP assembly from TECTRA
  - Active diameter >40 mm, pore size 12 μm, channel length/diameter 40:1
  - Specified only for 10 ns timing resolution!



TOF detection efficiency

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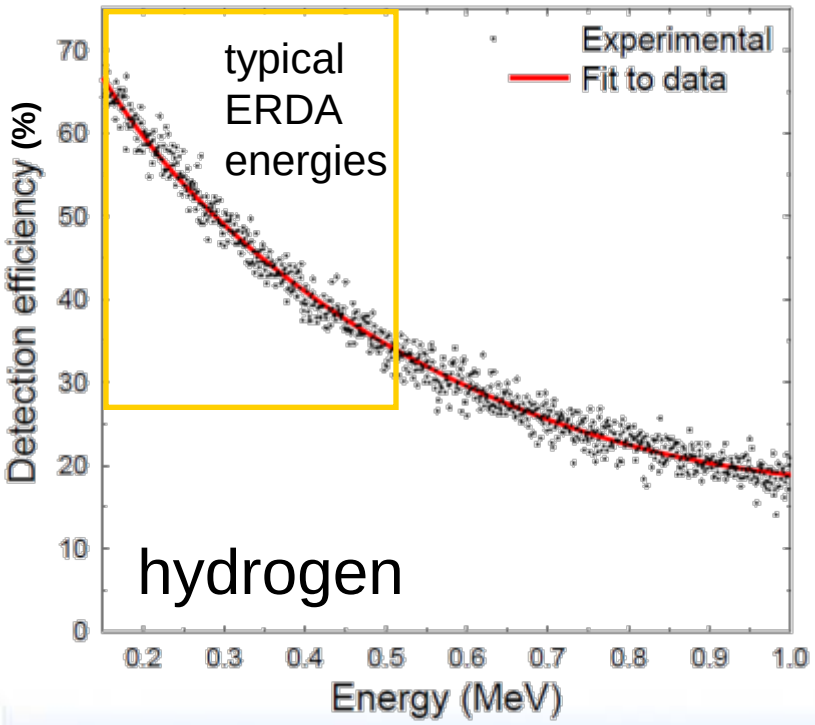
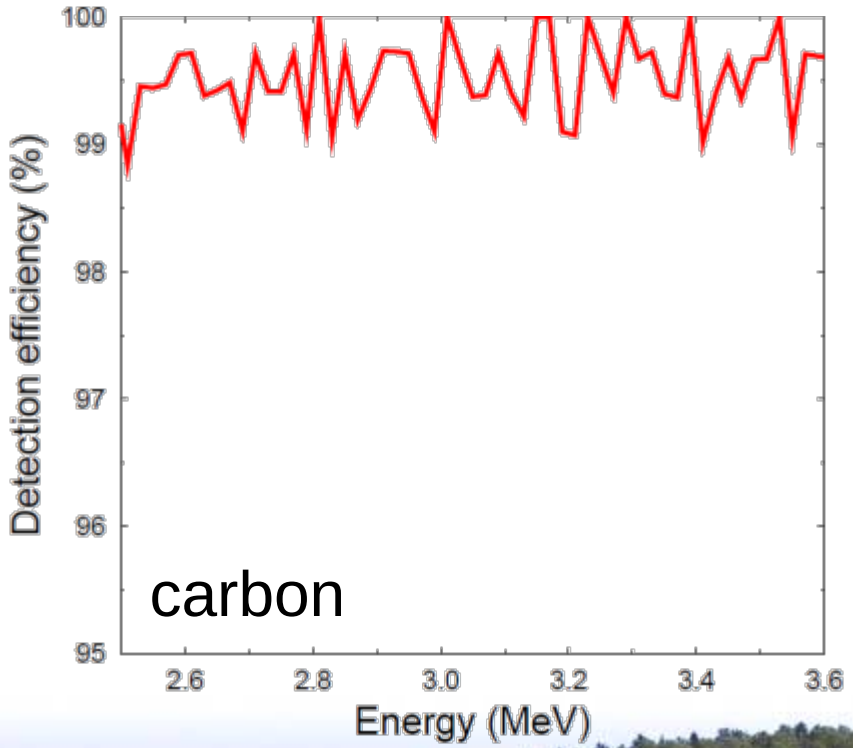
- Detection efficiency against energy detector
- All sample elements, including H, can be detected and quantified
- No pinholes in C foils





Timing resolution for He and H

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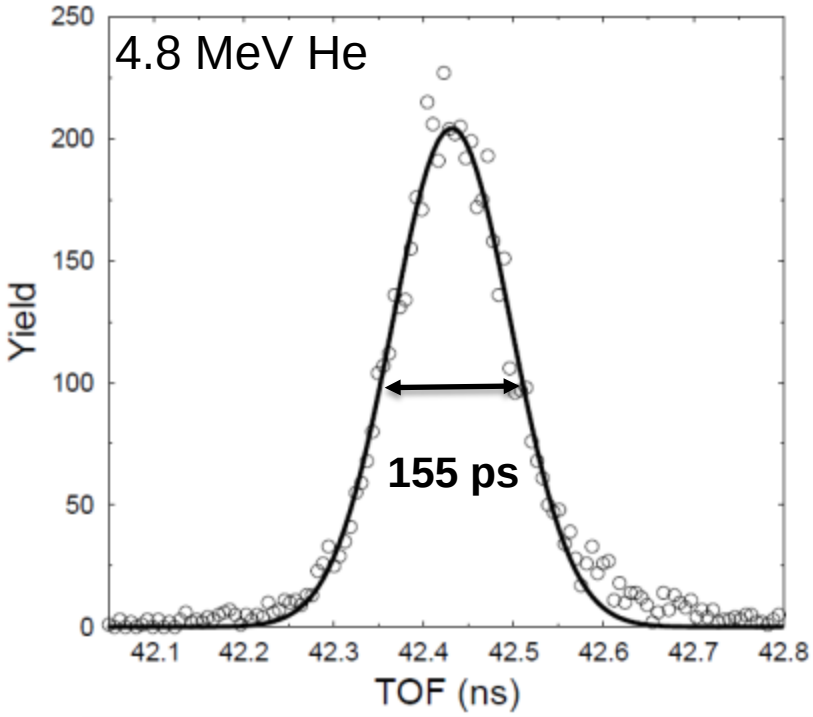
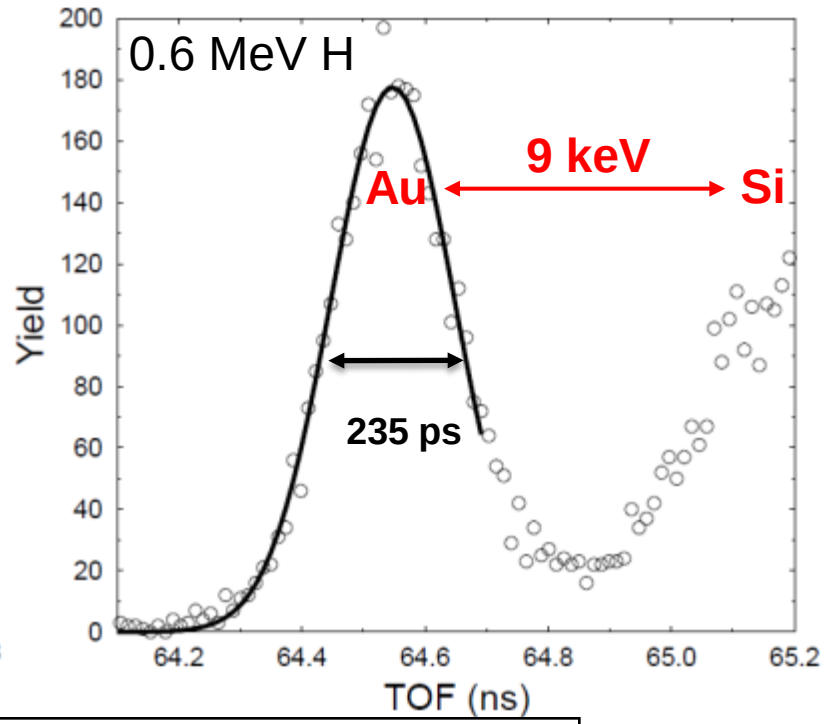
- Current timing resolution 155 ps (FWHM) for 4.8 MeV incident He ions and 235 ps for 0.6 MeV incident H ions scattered from 1-2 nm Au film on SiO_2/Si substrate





For He 155 ps timing resolution gives 32 keV resolution at 4.8 MeV but 0.8 keV resolution at 400 keV!

Example: Diamond-like carbon films

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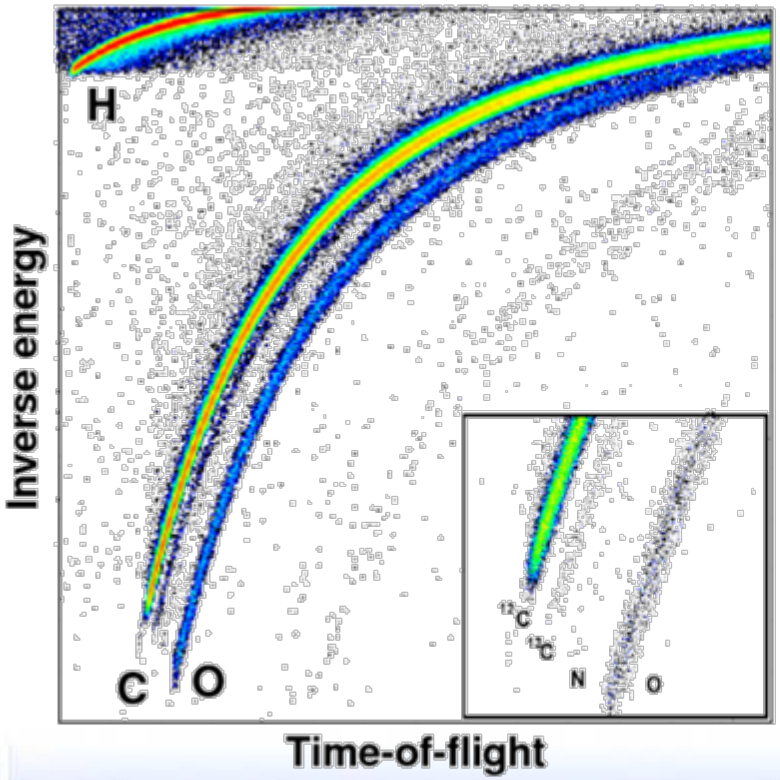
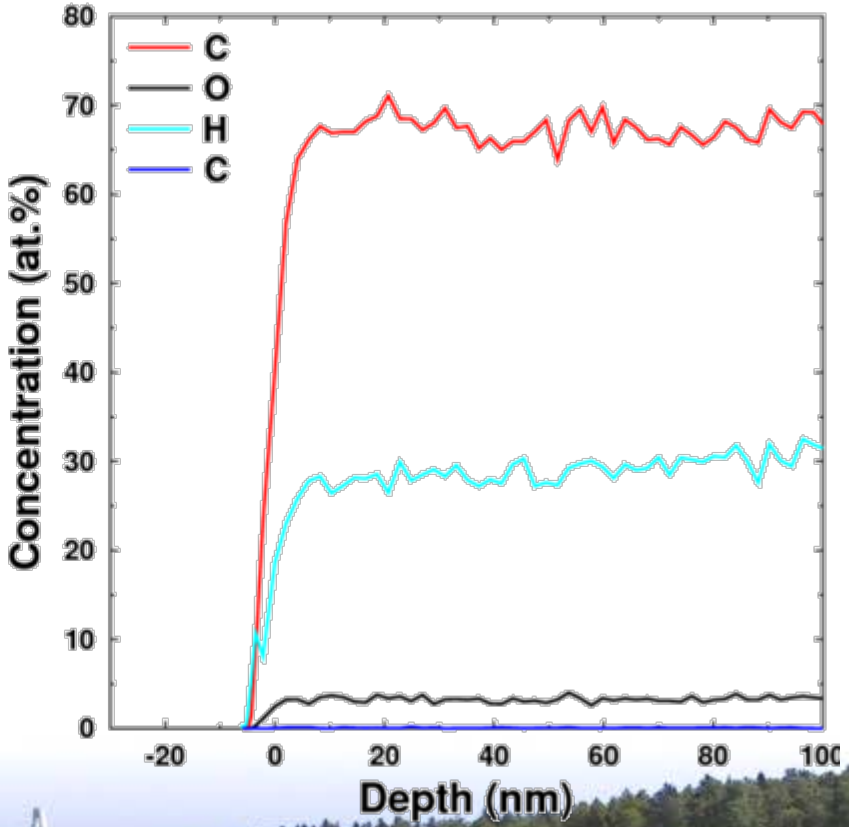
- 2.3 µm thick diamond-like-carbon film on Si, measured with 9 MeV ^{35}Cl
- All isotopes can be determined for light masses
- Light elements can be well quantified (N content 0.05 0.02 at.%)





Low energy heavy ion ERDA

Selection of coincident events

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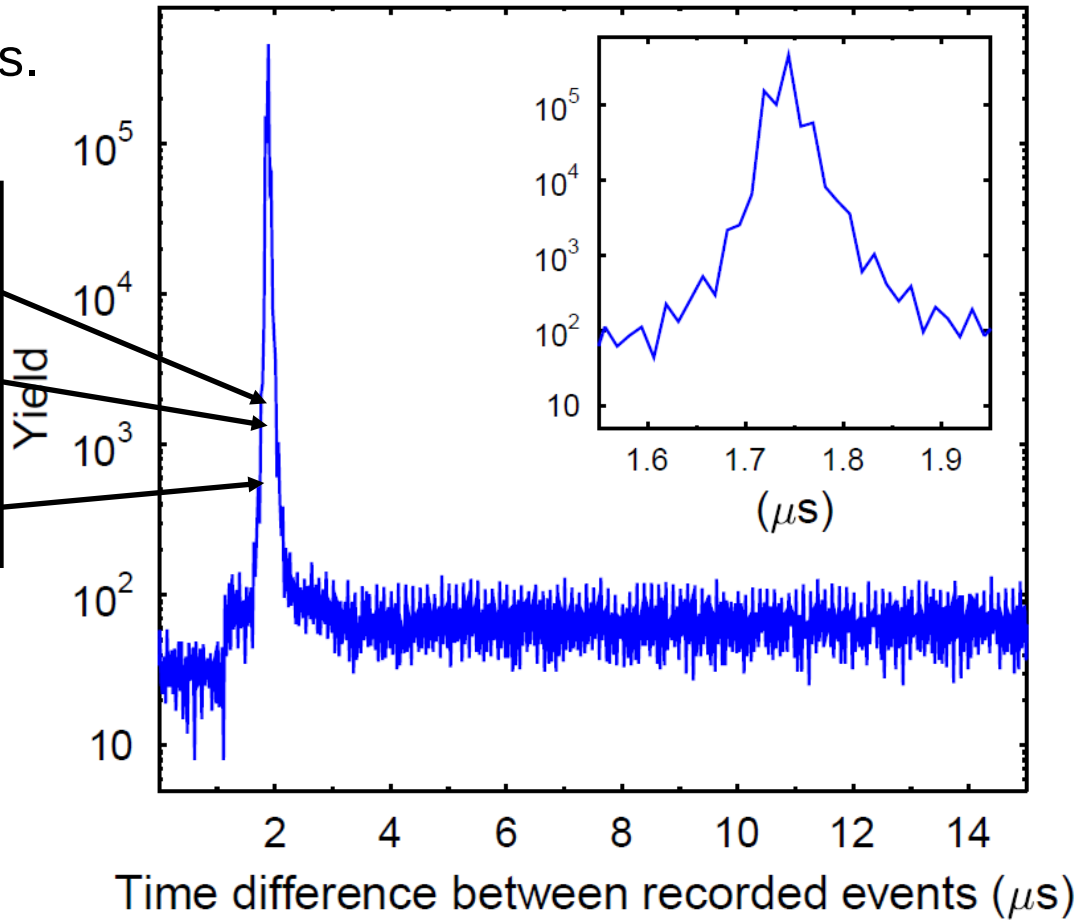
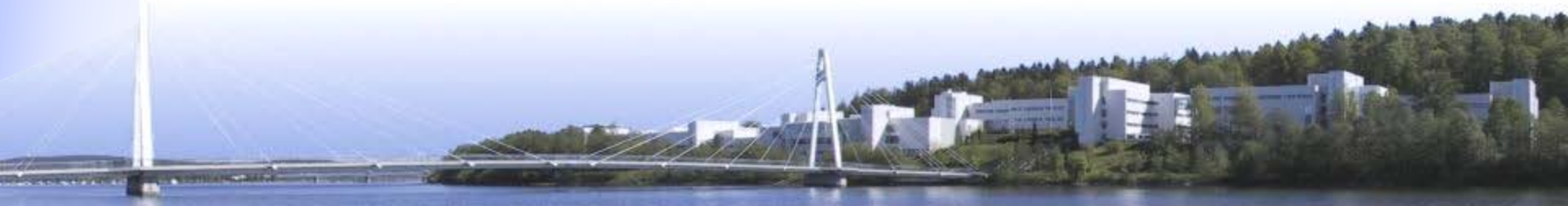
Time-stamp resolution 25 ns.

	ADC n:o	Ch	Timestamp
TOF	0	2675	457669383399
energy	1	2624	457669383471
	0	3756	457669459909
	1	1332	457669459982
	0	6688	457669499237
	0	3044	457669862394
	1	1987	457669862467

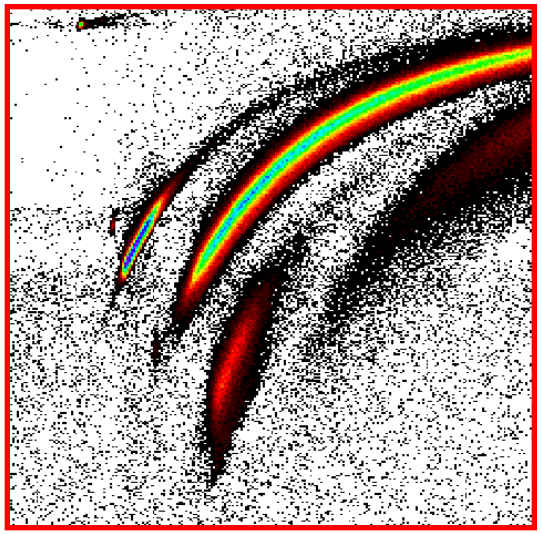


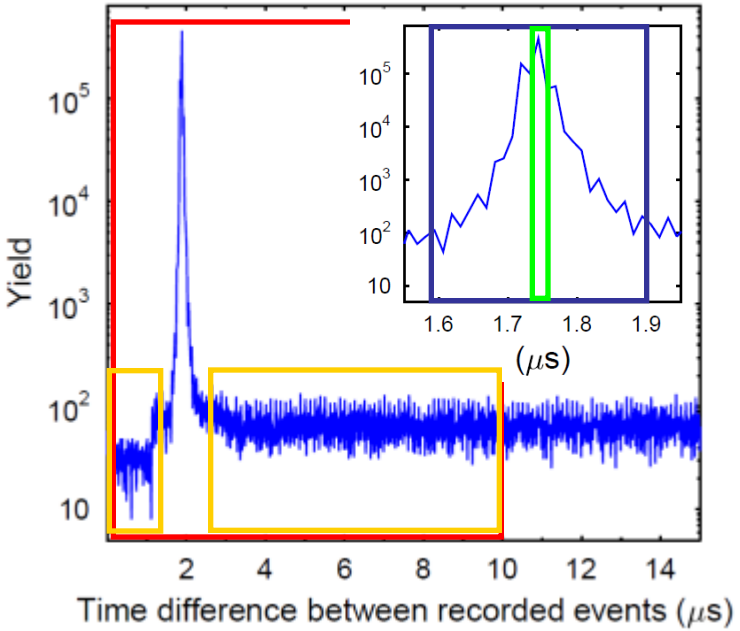
Lonely TOF-event, has probably hit T2 detector C-foil frame



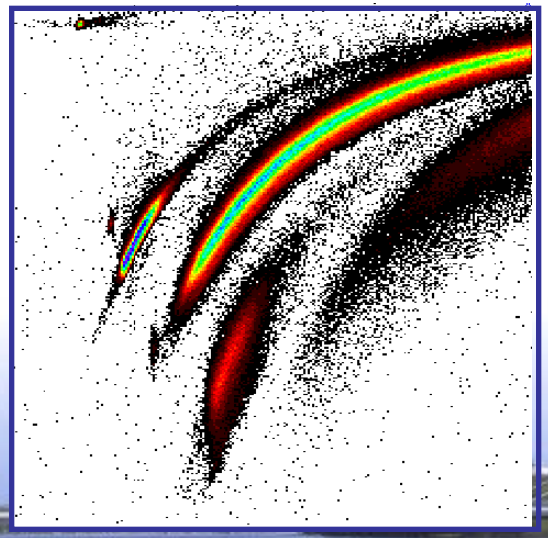
Selection of coincident events

Coincidence <10μs, 585117 counts

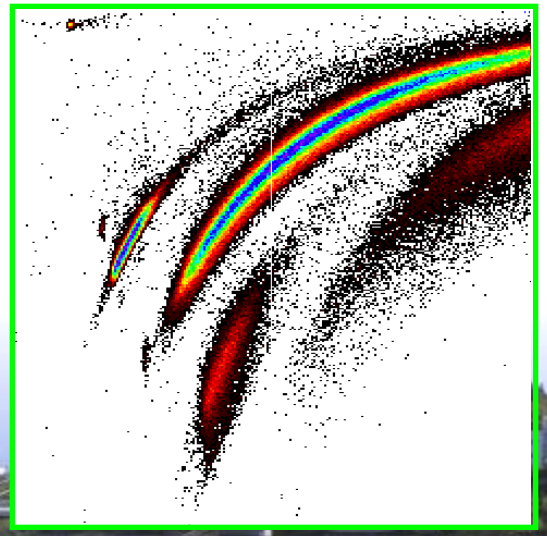




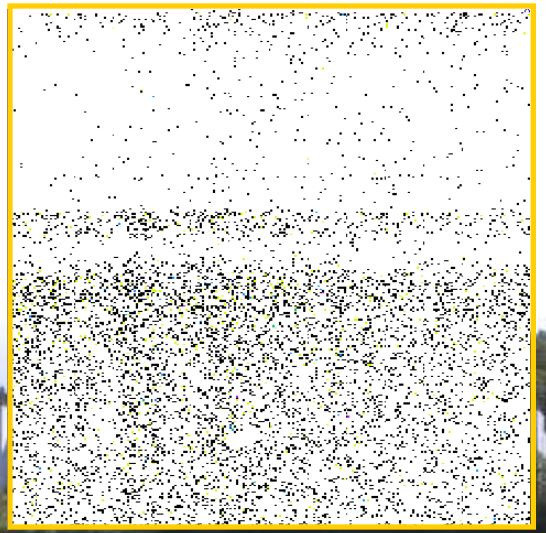
1.6-1.9 μs, 576741 counts



1725-1750 ns, 329445 counts



outside peak, <10μs



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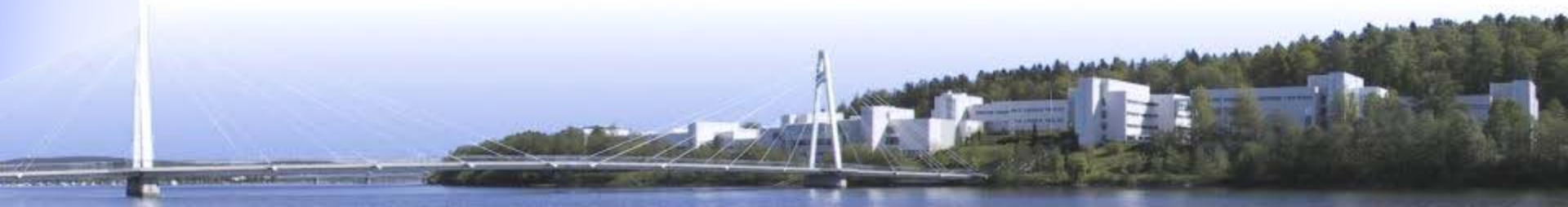
ANALYSIS OF THE Al_2O_3/TiO_2 NANOLAMINATES



Measurements

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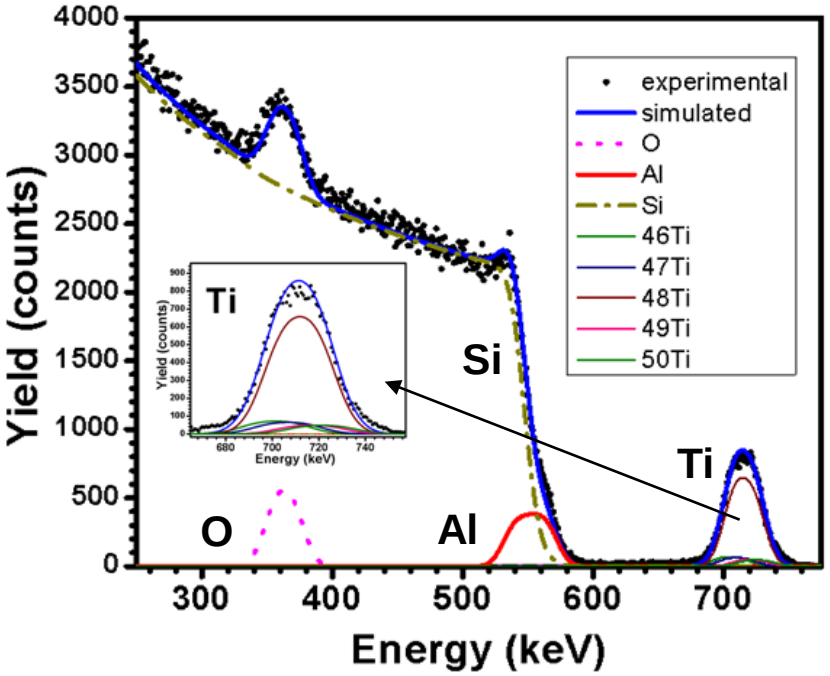
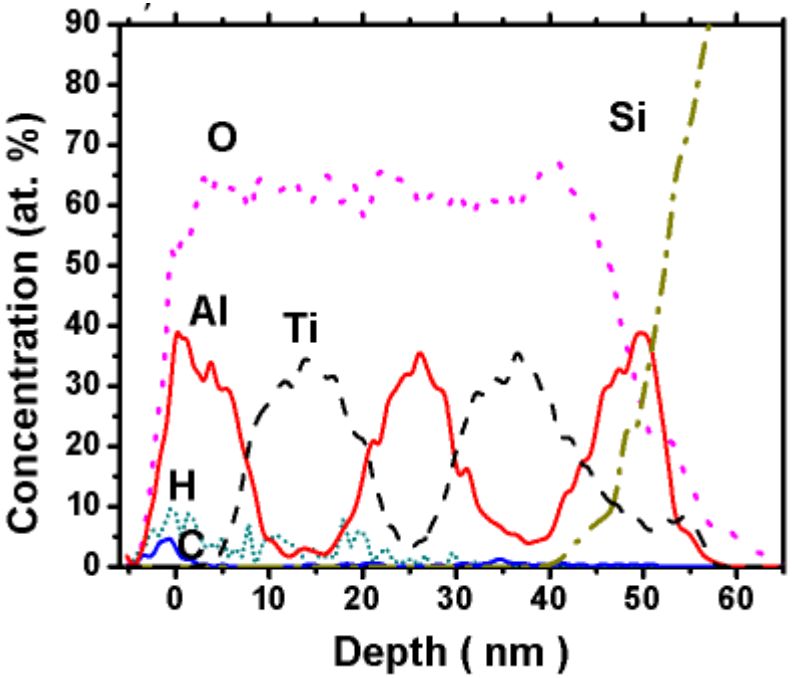
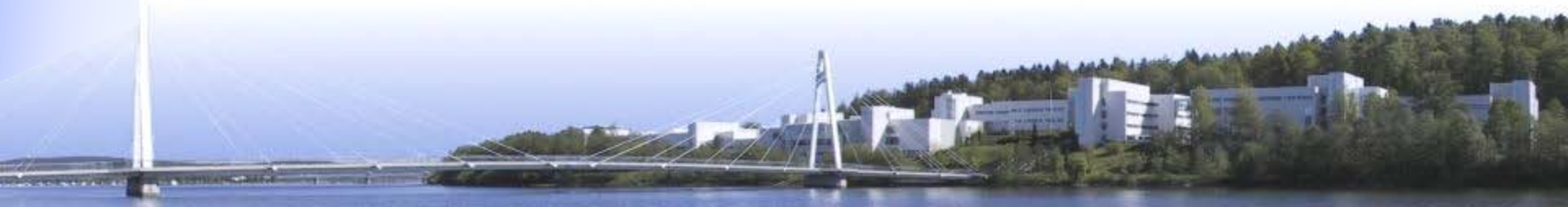
- 9.9 MeV $^{35}Cl^{5+}$ - all samples, reflectance 69.5 geometry and 84
- Quick test to all samples with standard 1 MeV $^{4}He^{1+}$ RBS at 168

RBS - R2, 10 nm layers



ToF-E, R2, 10 nm layers





Measurements

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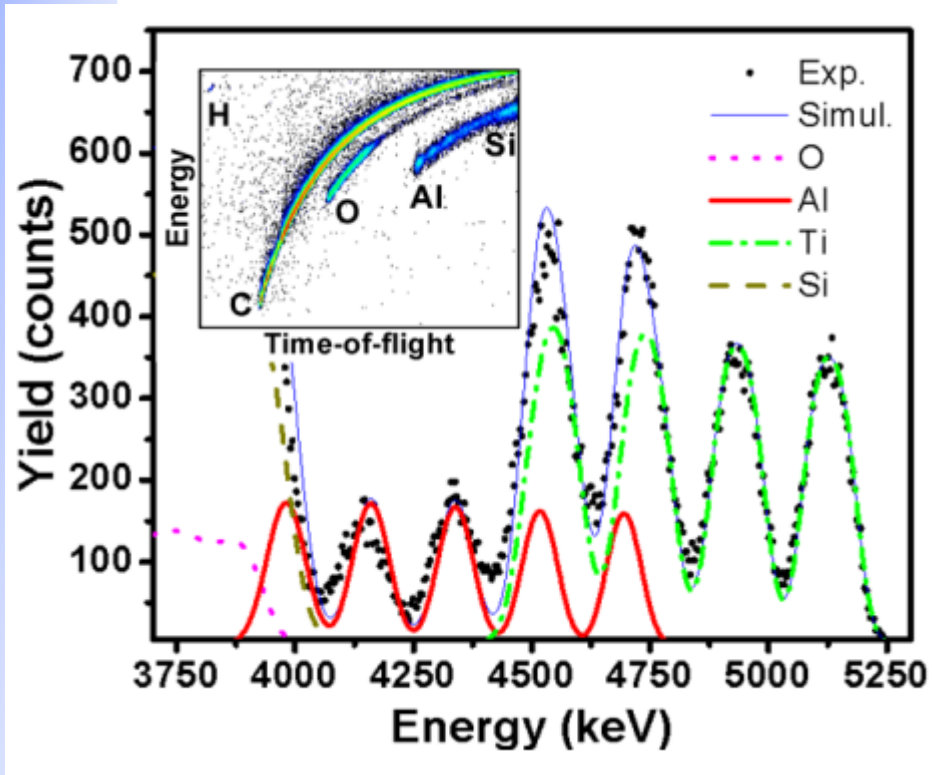
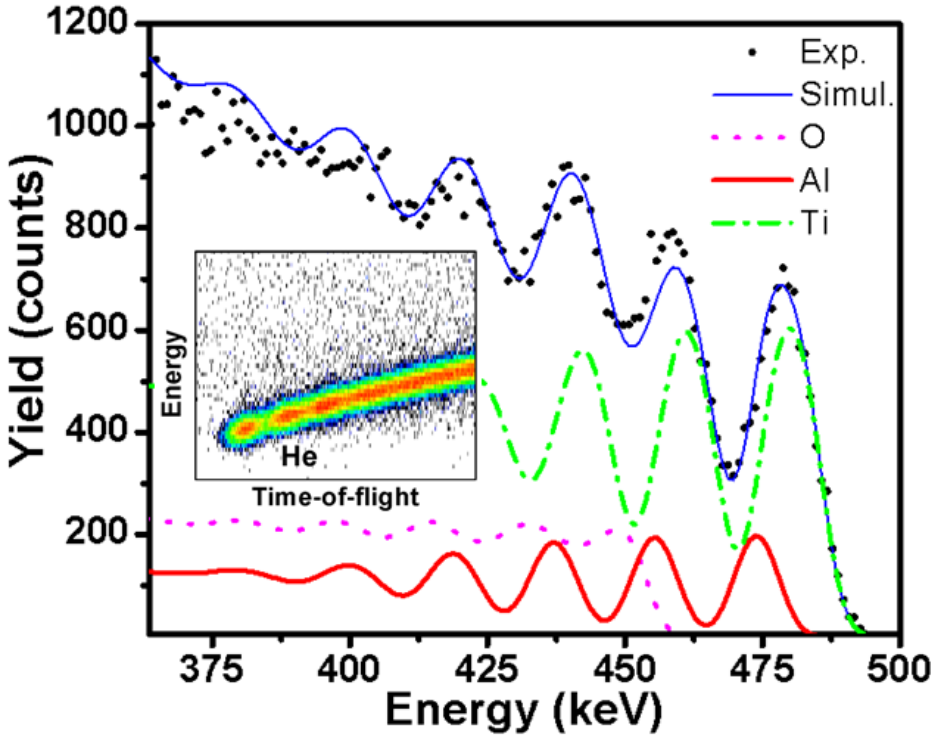
- 6.0 MeV $^{12}C^{3+}$ - 3 thinnest layered samples, 84 and 86
- 0.5 MeV $^{4}He^{1+}$ - Rutherford Scattering to forward angles up to 88

Sample R3, 5 nm layers



Sample R4, 2 nm layers



Monte Carlo -simulations

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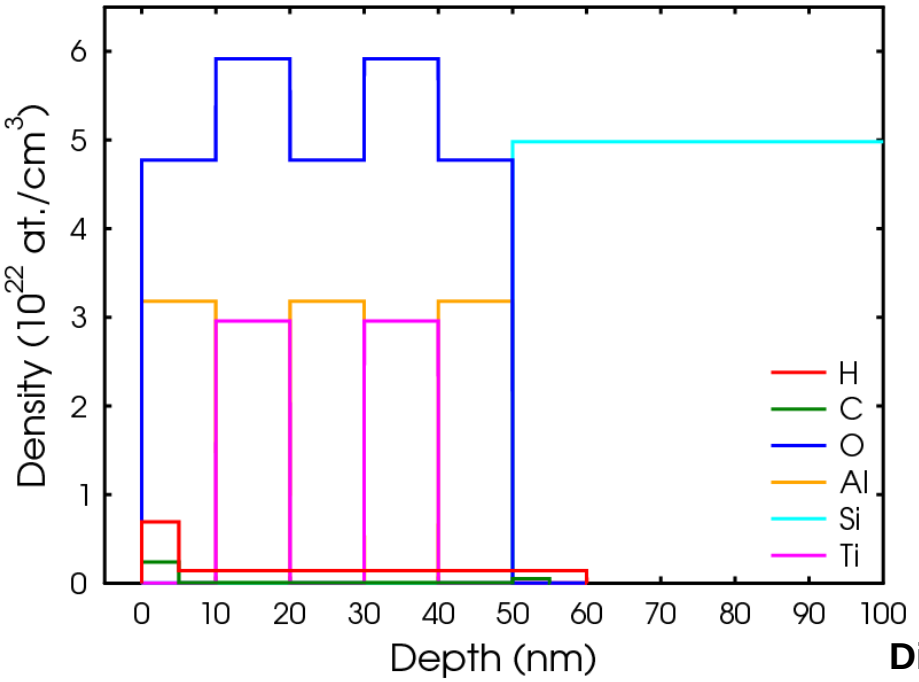
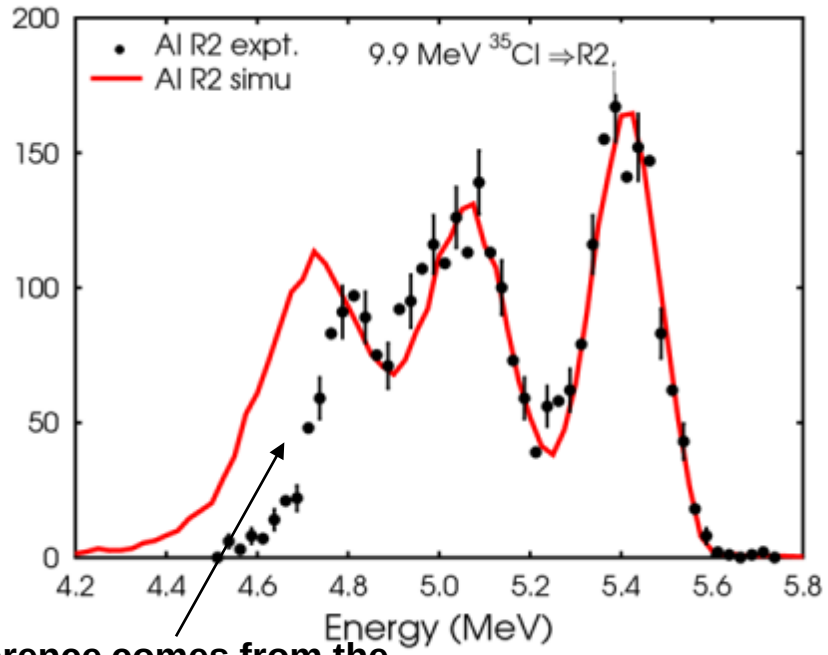
- MC-simulations made for the spectra in reflectance geometry
- Better undertanding of the composition and thicknesses

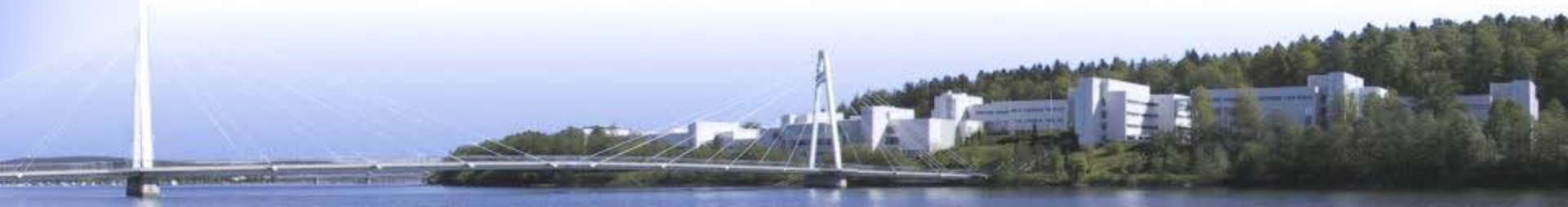
Sample R2, 10 nm layers, start profile





Difference comes from the incomplete selection of Al counts from Si substrate (does not effect MC-result)

FURTHER IMPROVEMENTS



Gas ionization detector

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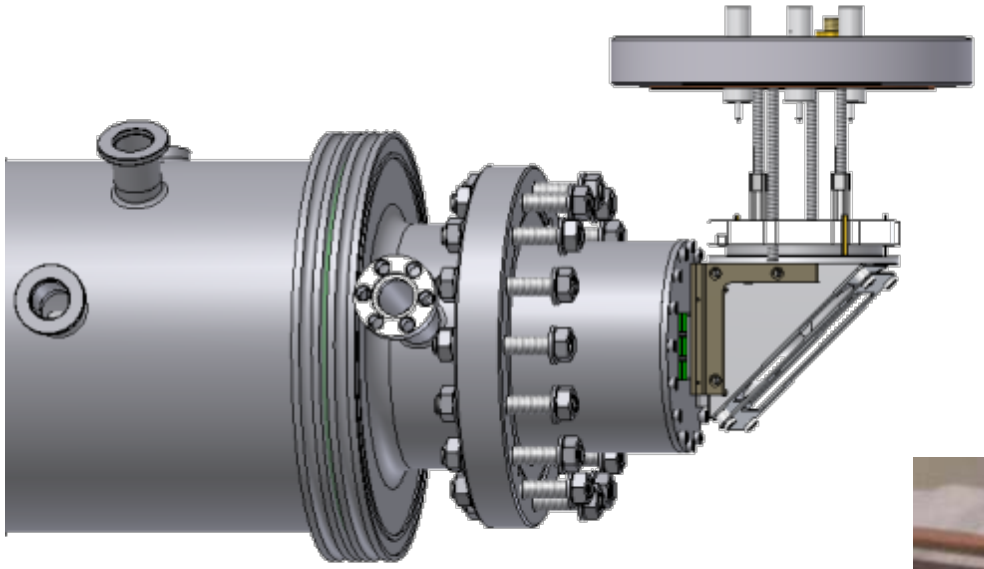

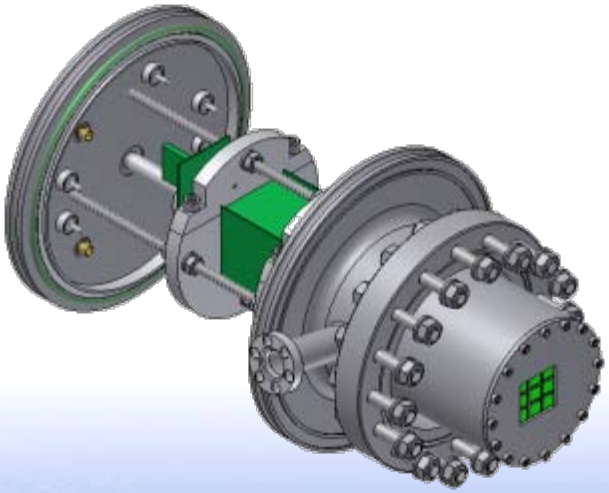

- Thin (~100 nm) SiN window
- Electrons for T2 timing signal emitted from the membrane

Conclusions

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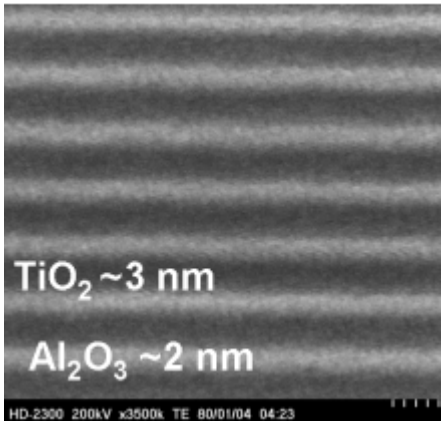
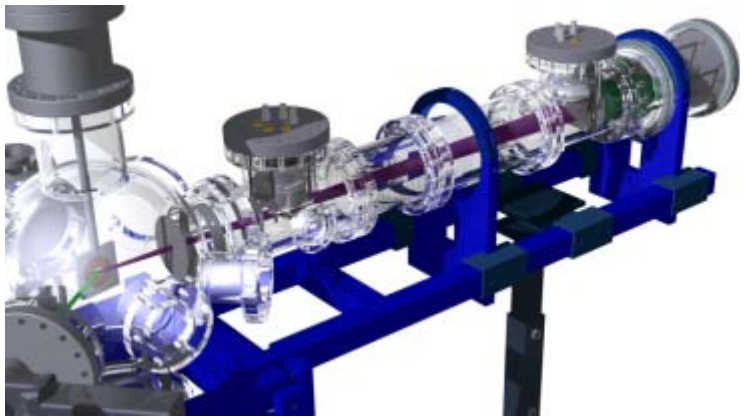
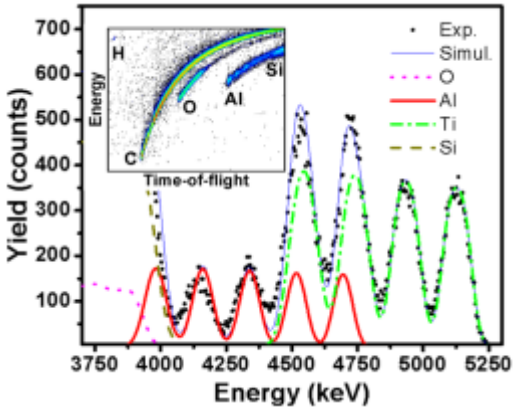
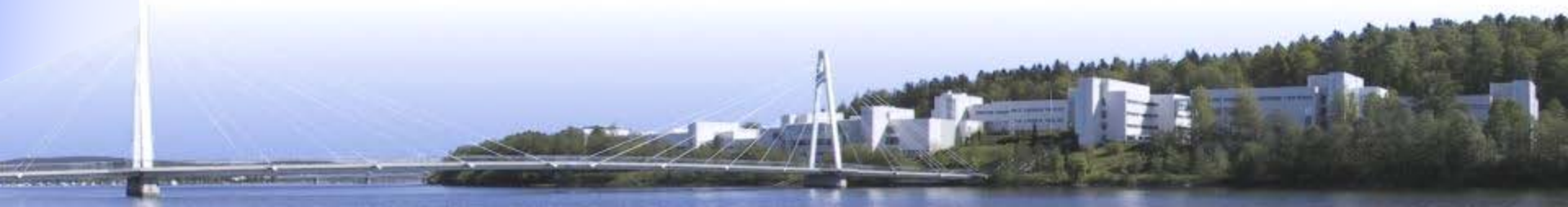
- All Al_2O_3/TiO_2 nanolaminates could be depth profiled and impurities, including hydrogen were analyzed
- Nanolaminates with individual layers of 5 nm could be resolved
- Depth resolution of <2 nm at the surface was reached
- Gas ionization detector as an energy detector and yet coming position sensitivity will push the performance to even higher level









Future improvements: Gas ionization detector

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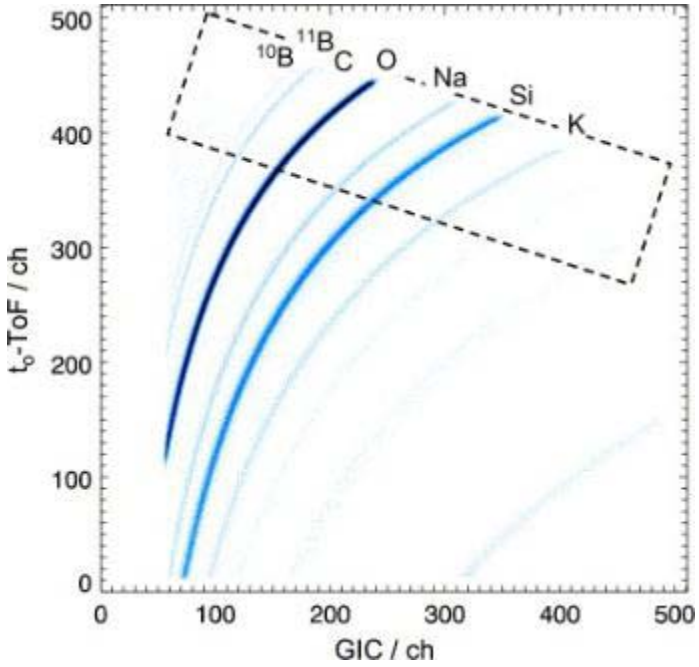
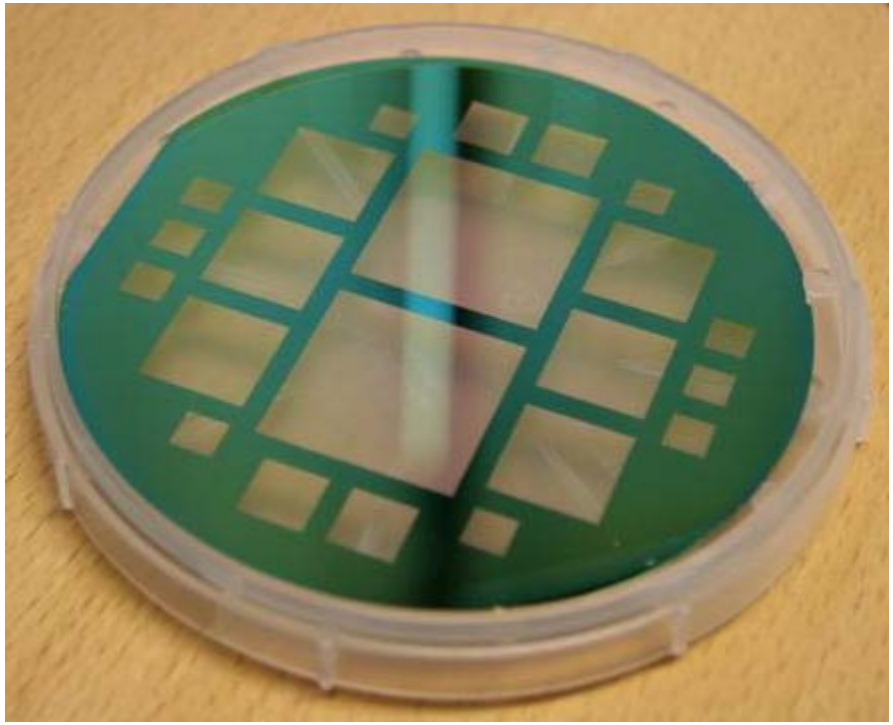


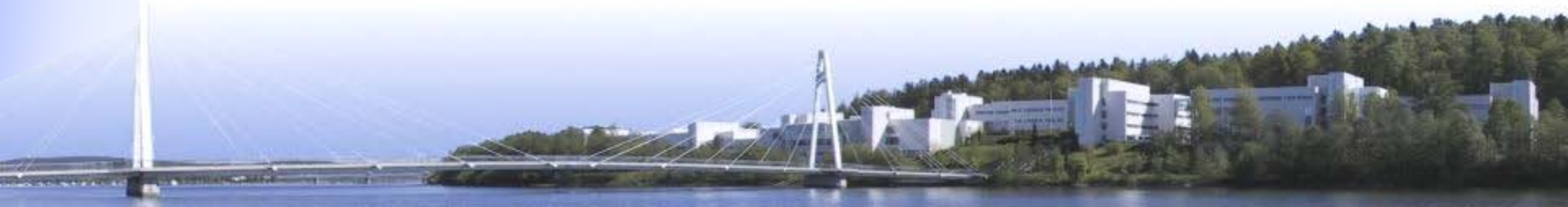
TOF-E results from ETH Zürich

Incident ion 12 MeV ^{127}I and borosilicate glass target

***Nucl. Instr. and Meth. B* 248 (2006) 155-162**



200 nm thick SiN membrane from Aalto University, Finland, on 100 mm wafer



ALD 8.6 nm Al_2O_3/Si

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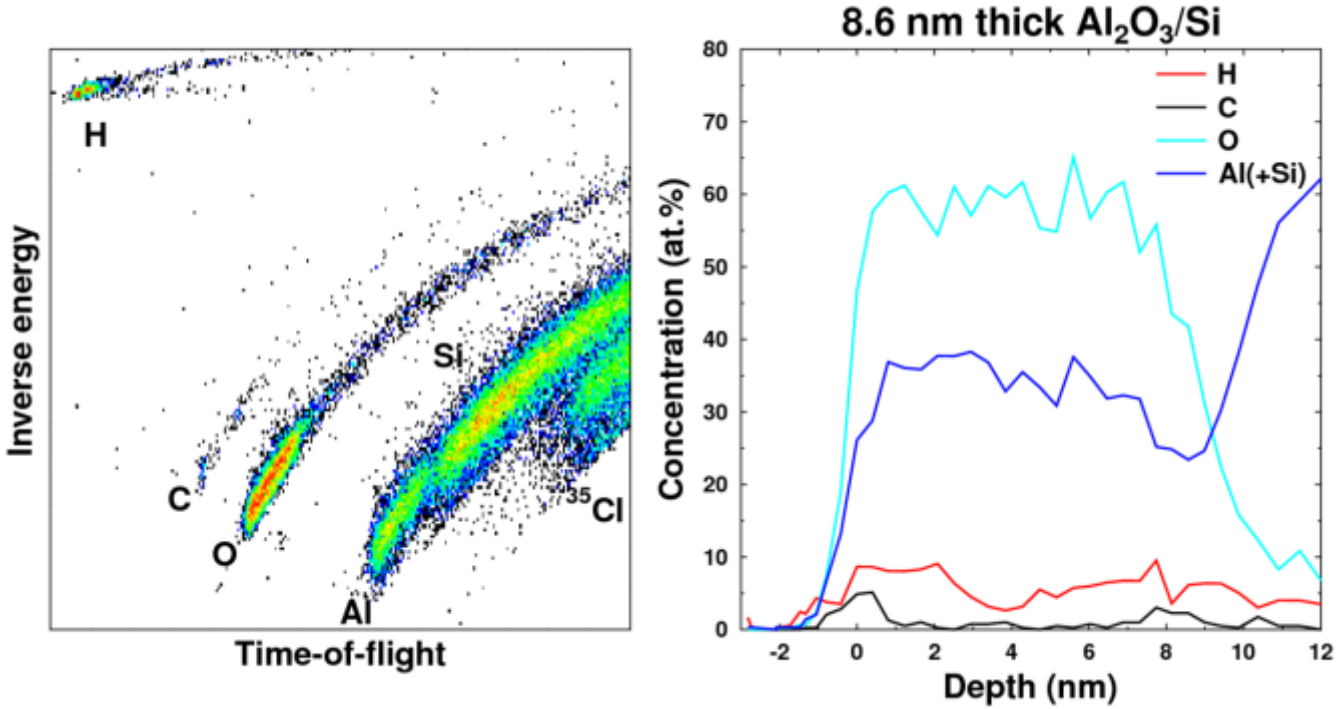
- Atomic layer deposited Al_2O_3 film on silicon (Prof. Ritala, U. of Helsinki)
- Density of 2.9 g/cm^3 and thickness of 8.6 nm determined with XRR (Ritala)
- Elemental concentrations in the film bulk as determined with TOF ERDA are O 60 3 at.%, Al 35 2 at.%, H 4 1 at.%. and C 0.5 0.2 at.%



10 nm CN_x on silicon

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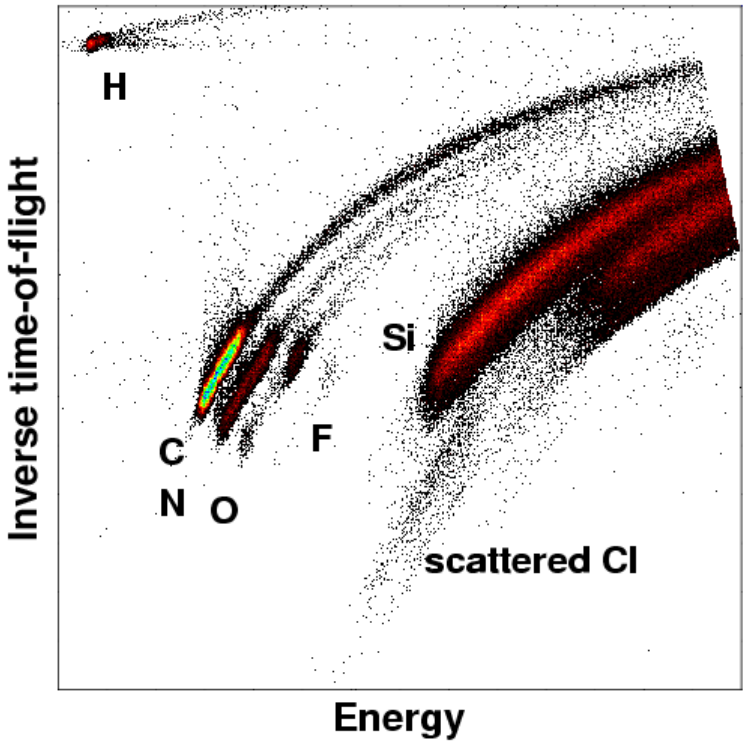
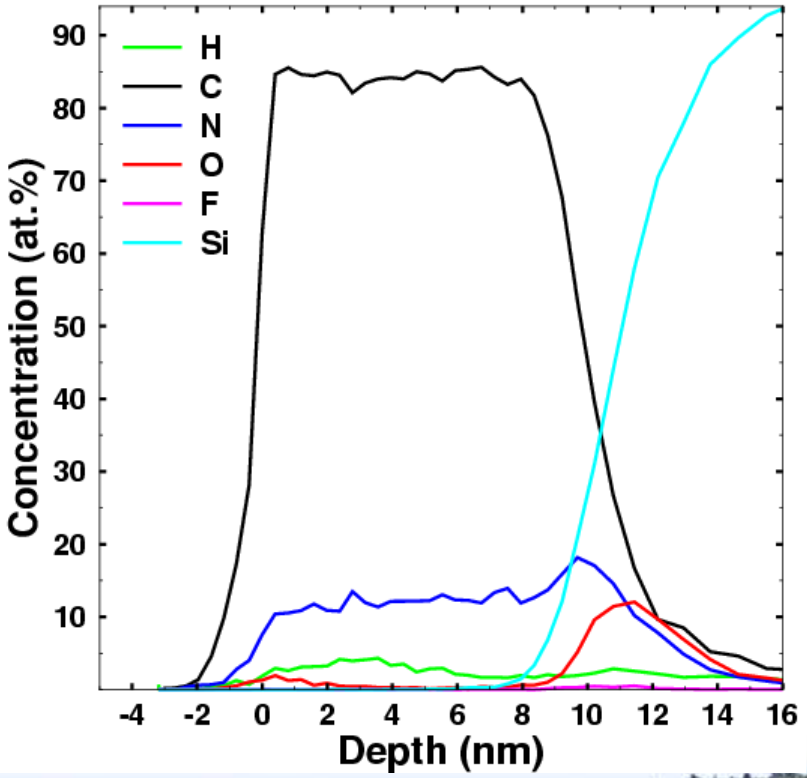
- TOF-ERDA results from sputter deposited 10 nm thick CN_x hard coating on Si. Measured with 6 MeV ^{35}Cl beam and extreme glancing angle of 3
- A density of 2.0 g/cm^3 was used in converting areal densities to nm





Effect of stripper gas pressure

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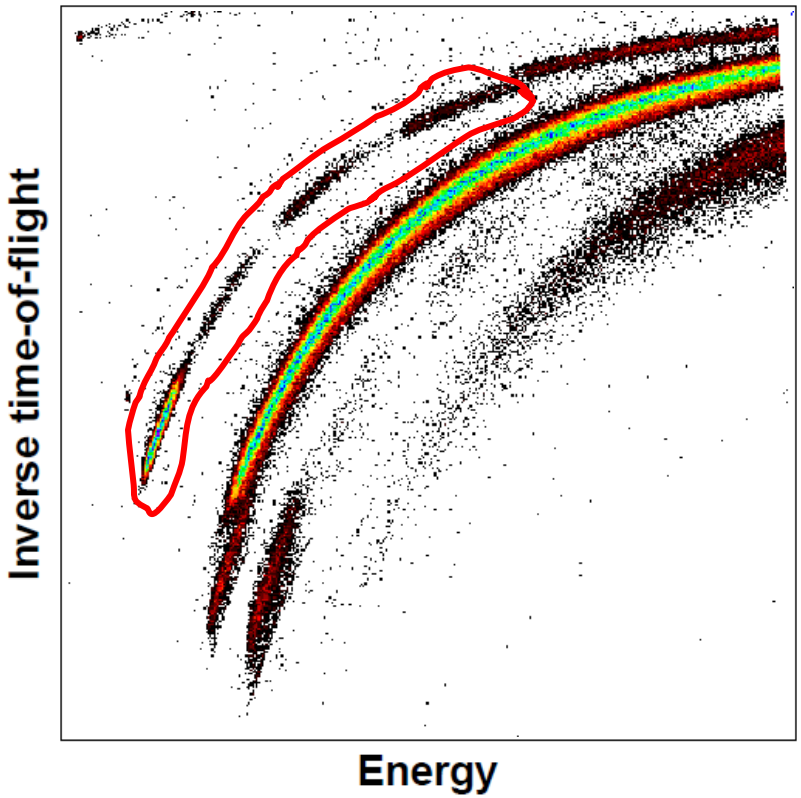
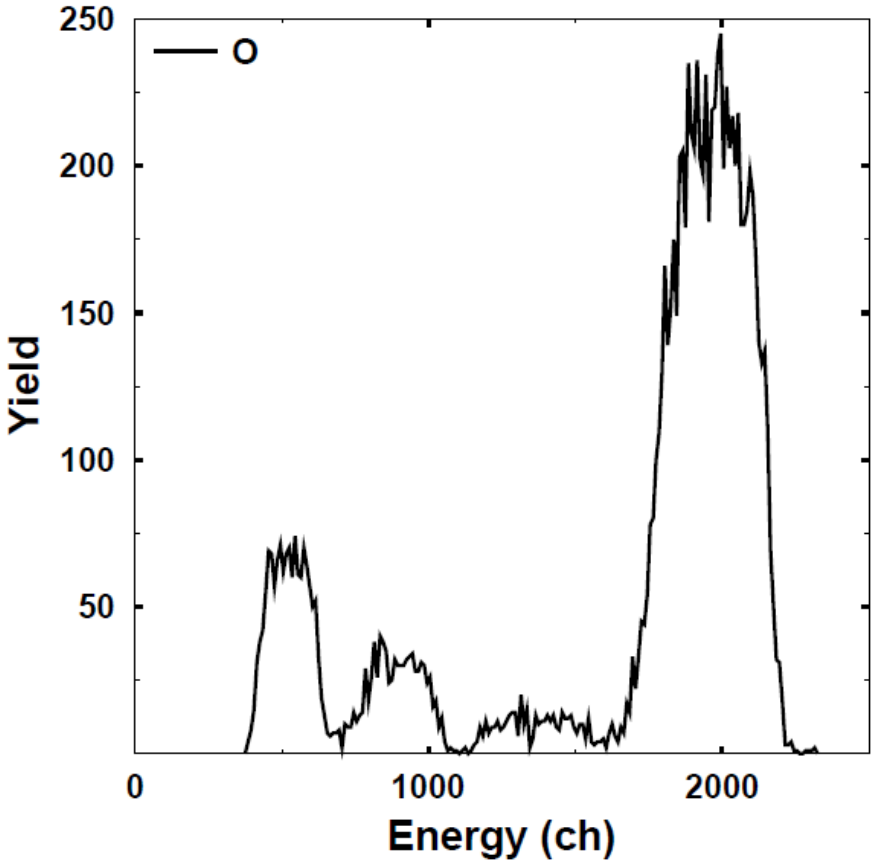
- 13.6 MeV $^{63}Cu^{7+}$ CaPO (hydroxyapatite)





Timing resolution

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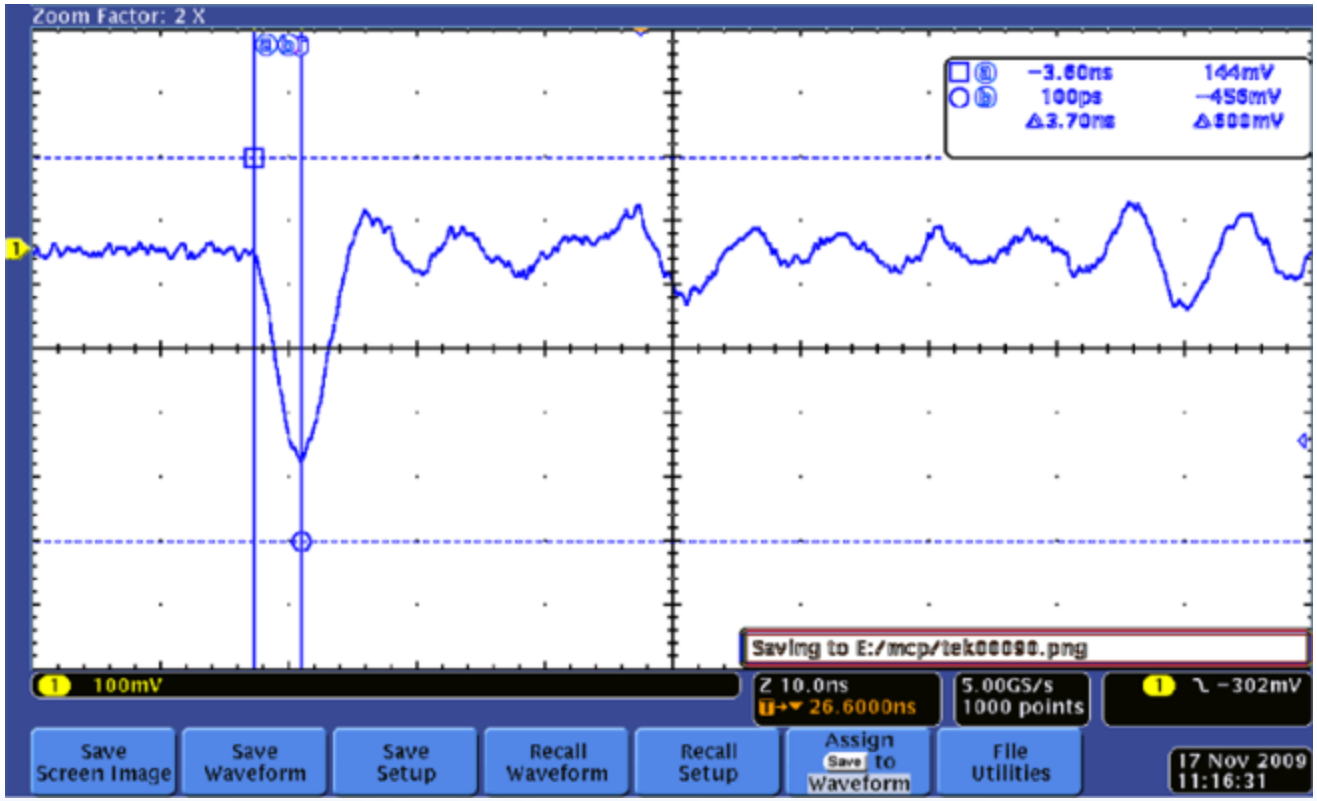
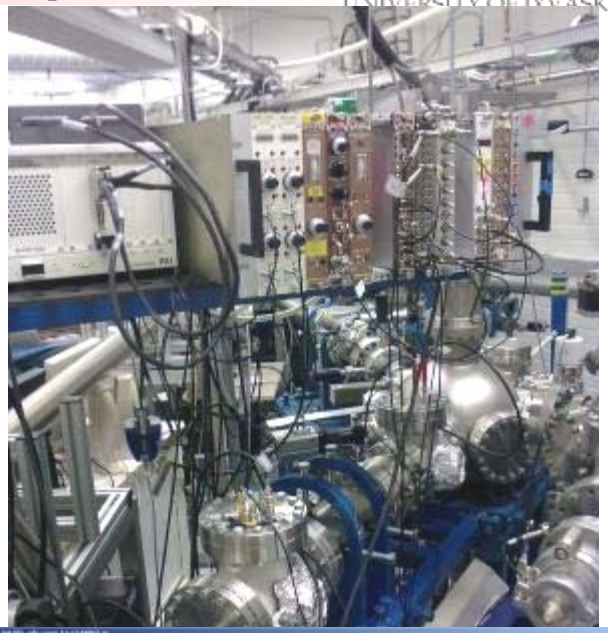
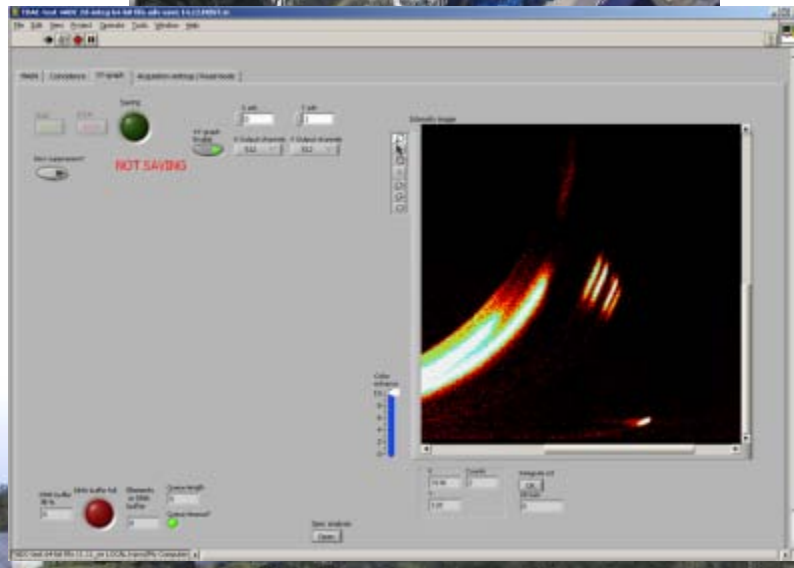
- Shape of the MCP signal with the original anode
  - **Risetime ~3 ns for He ions**



Electronics and data acquisition

- Timing gates
  - Fast Phillips Scientific 776 preamplifier (10)
  - Ortec 935 quad CFD
  - FAST 7072T TDC/ADC, no delay used
- Energy detector
  - Implanted 450 mm^2 ORTEC detector (ULTRA series)
  - Ortec 142 preamplifier
  - Ortec 571 amplifier
  - FAST 7072T TDC/ADC
- Data acquisition with LabVIEW
  - National Instruments FPGA card based multiparameter data acquisition system, programmed in Jyväskylä
  - 40 MHz (25 ns) time stamping
  - Can host up to 8 ADCs, easily expandable
  - **List-mode data – coincident events determined off-line!**





Depth resolution optimization

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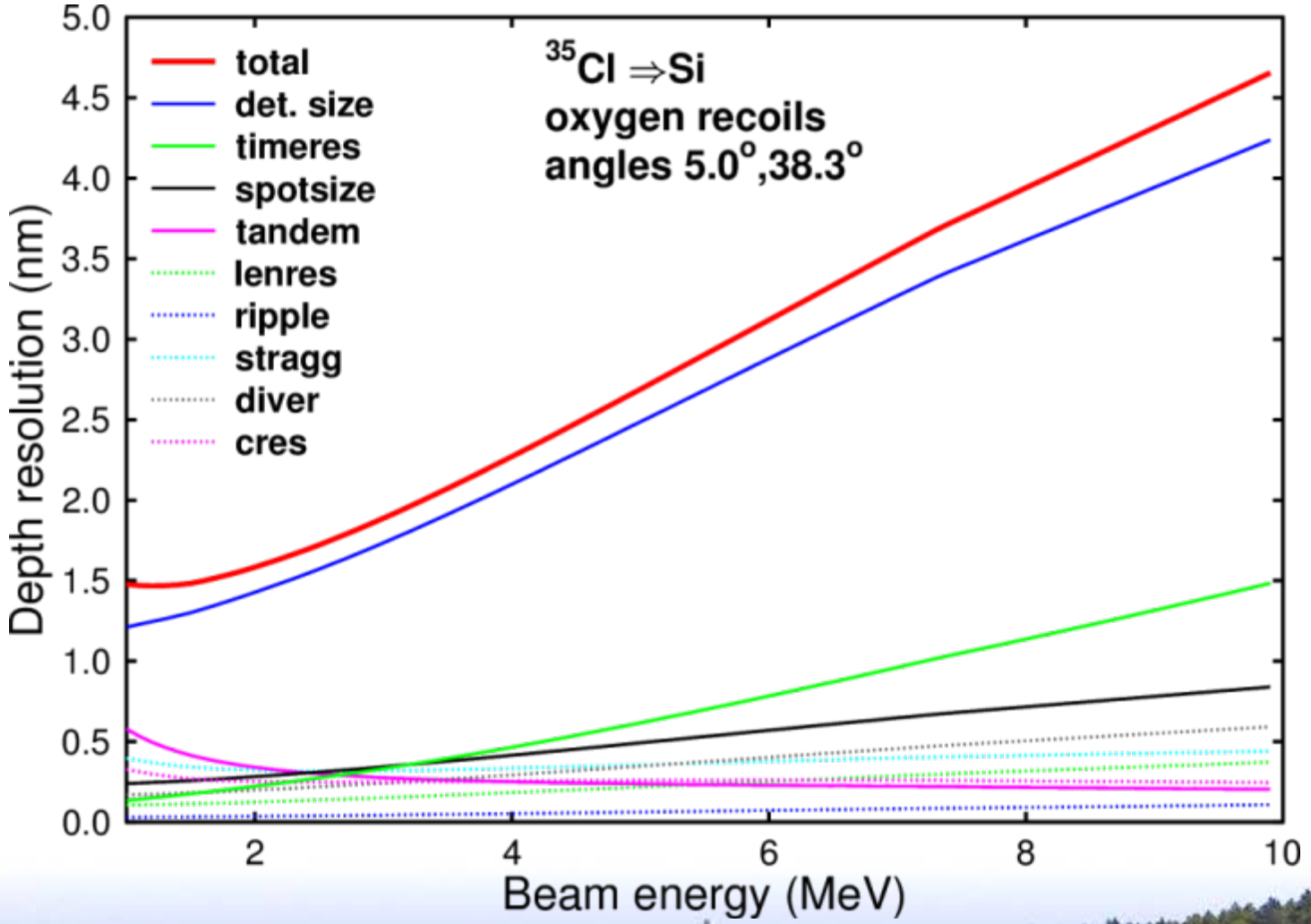

TOF-ERDA beamline and chamber

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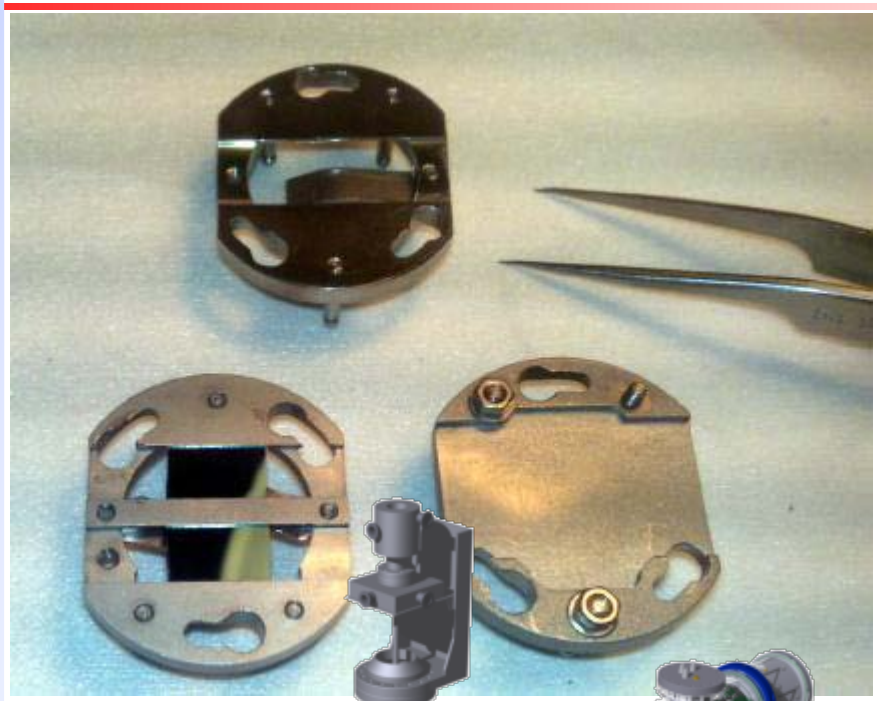
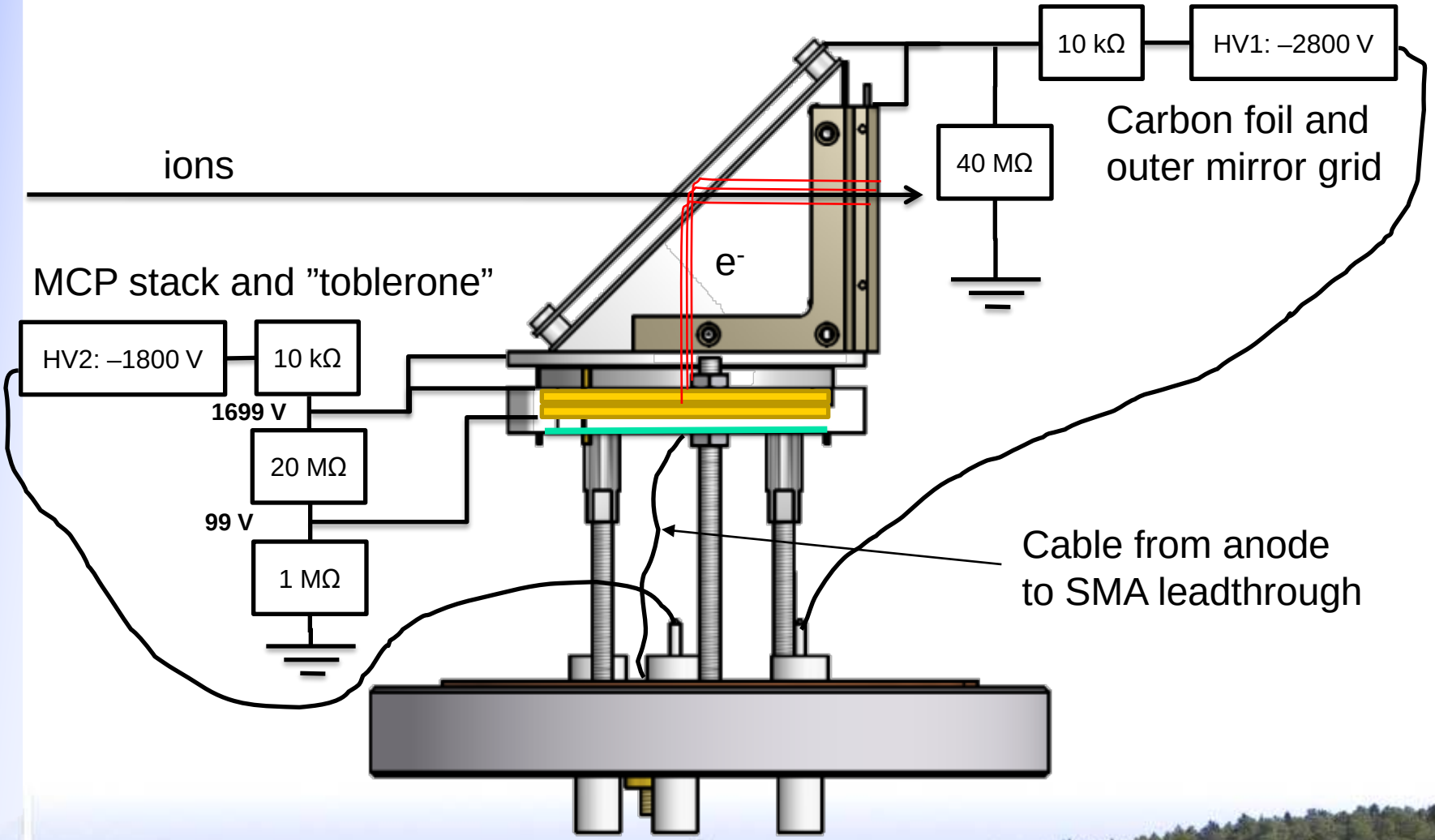


- UHV compatible chamber with a load lock
- LabVIEW controlled stepping motor driven 6-axis goniometer (Panmure instruments)
- Currently sample holder for two samples, holder for 7 samples in design
- Beamline equipped with high precision slits and NEC beam profile monitor
- Telescope angle 41º

Timing gates: voltages



Towards position sensitivity

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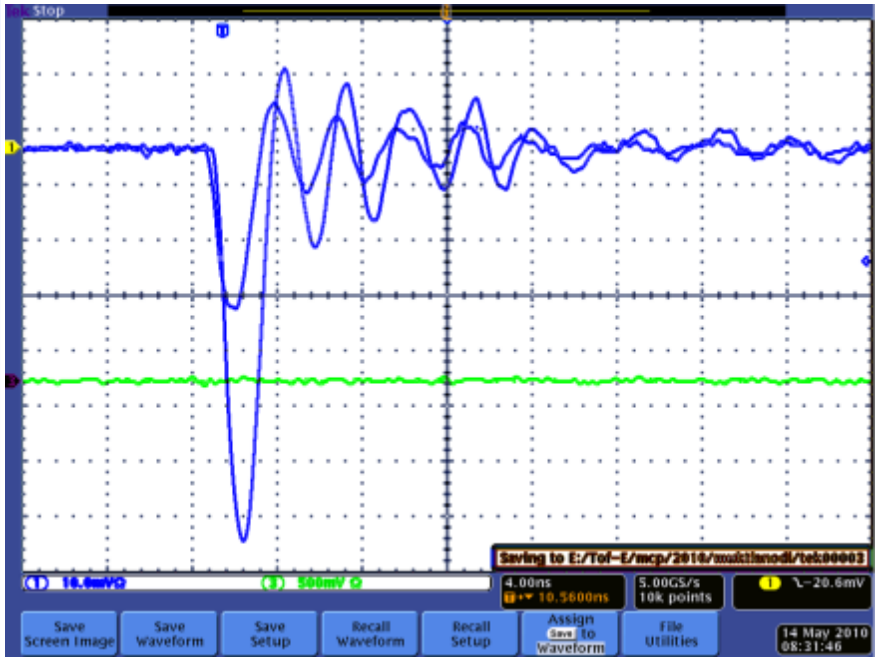
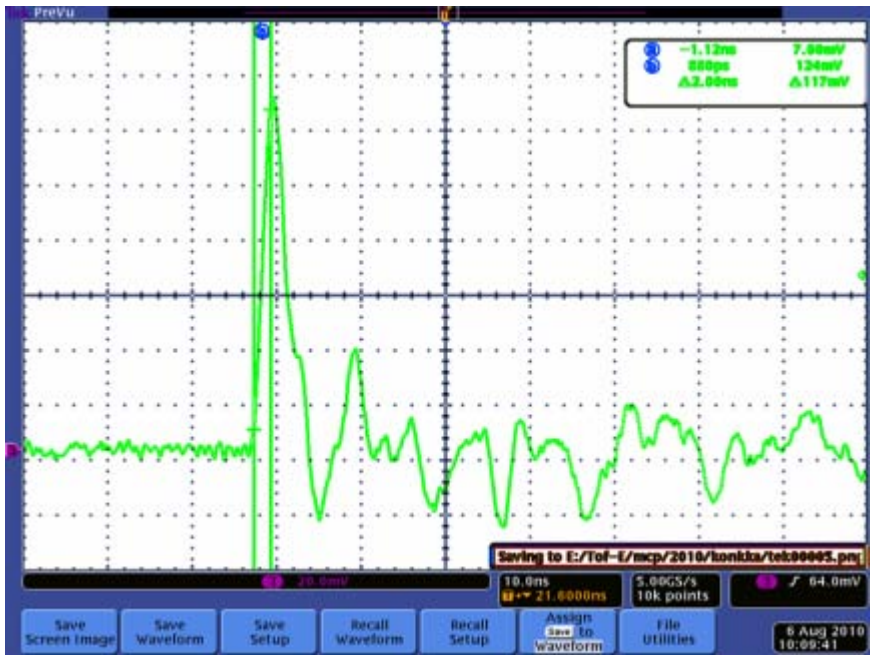
- Risetime of 1 ns with PCB anode achieved with the test detector
- Risetime of 2 ns from lower MCP electrode through 1 nF capacitor achieved with the test detector





Also ALD-Al_2O_3 coated carbon foils have been fabricated, much higher electron yields expected

Conclusions

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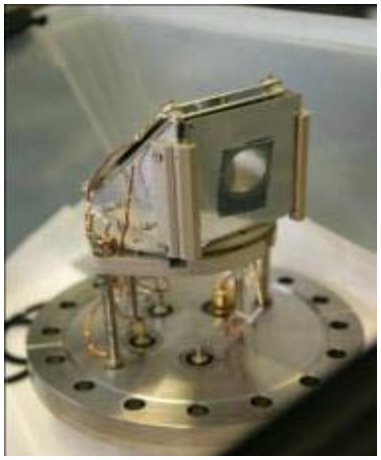
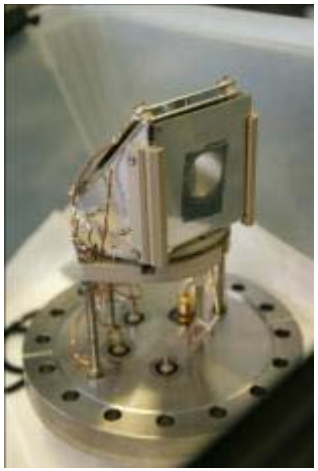
- New high performance spectrometer has been built in Jyväskylä
- Detector telescope has high detection efficiency and good timing resolution
- Depth resolution of <2 nm at the surface has been reached
- Position sensitivity and gas ionization detector as an energy detector will push the performance to even higher level



1st timing detector,
3 μg/cm^2 C-foil



2nd timing detector,
10 μg/cm^2 C-foil