X-ray and IR spectrometry

IAEA International Atomic Energy Agency

Elettra Sincrotrone Trieste

A multipurpose experimental facility for advanced X-ray Spectrometry applications

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Outline

- The IAEA project: Motivation & Partnerships
- XRF beamline
- Instrumentation of the beamline endstation
- Commissioning work and preliminary results on the set-up characterization
- Instruments to facilitate access/utilization of the end-station@ Elettra XRF beamline
- Outlook to the research foreseen

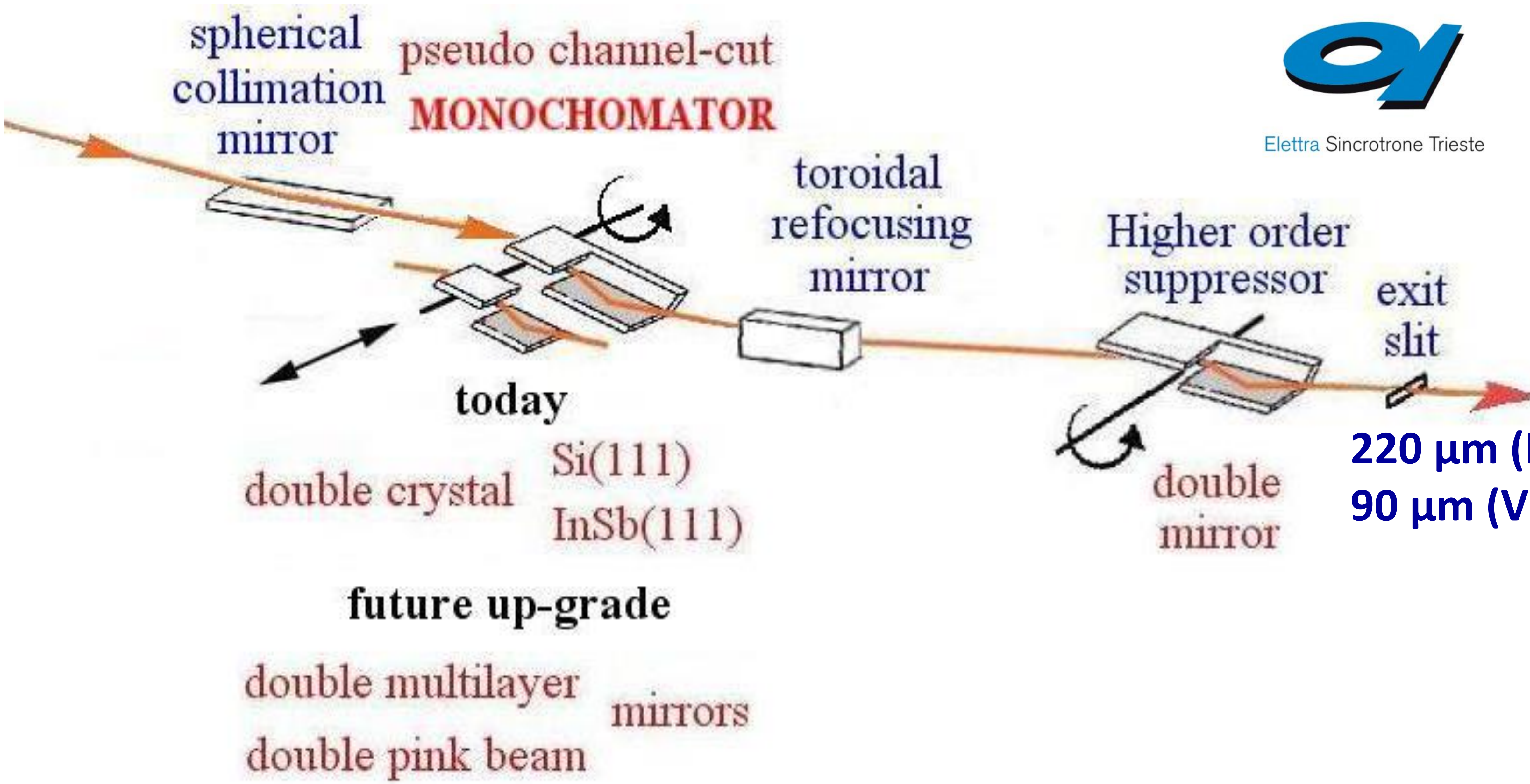
Motivation: Ultra High Vacuum Chamber (UHVC) project

- To support/enhance the training of scientists/engineers from developing countries in the operation of synchrotron radiation instrumentation;
- To provide beam time access for R&D projects and hands-on training in SR-XRS based techniques;
- To promote networking and knowledge sharing;
- To increase the quality and the competitiveness of the developing countries to apply beam time proposals at SR facilities;
- To contribute in the further development of XRS techniques in applications with socioeconomical relevance (characterization of energy storage/conversion materials, environmental, biological and biomedical applications)

Roadmap of the UHVC development

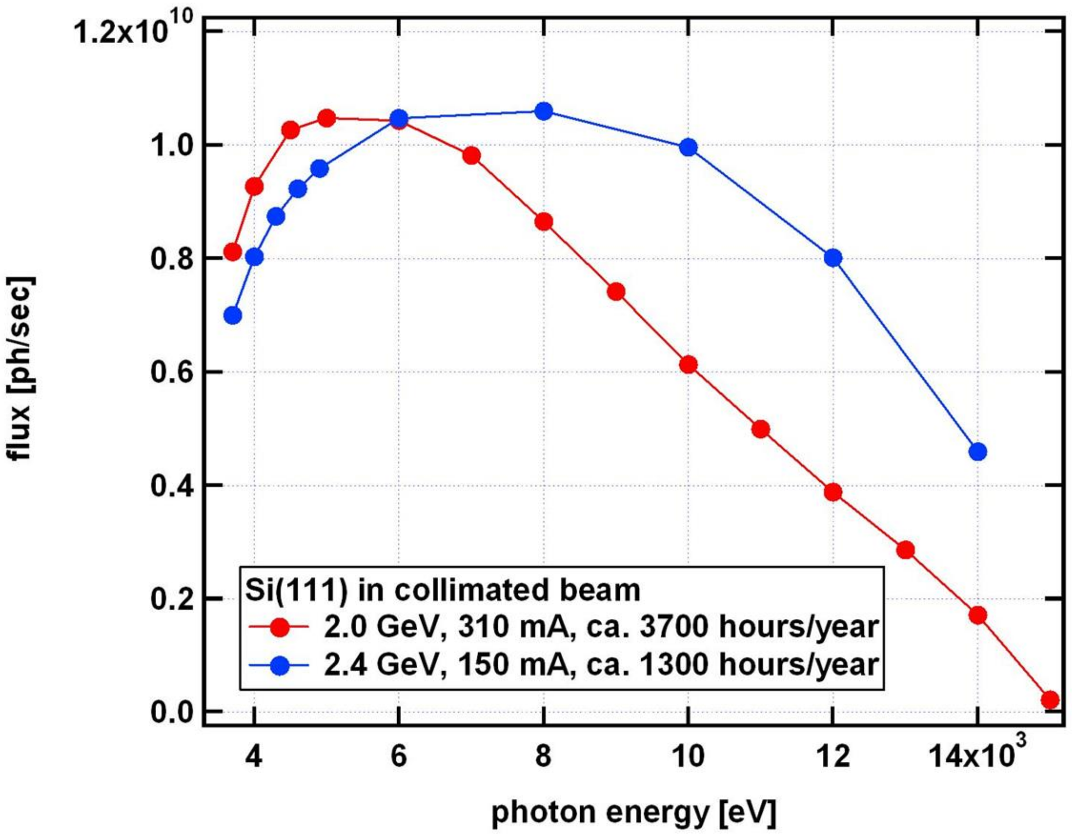
Time	Activities
September 2011:	First discussions with Elettra, Establishment of cooperation with **PTB/TUB**, Start of the procurement of the instrumentation components
September 2012	Data Acquisition software development
May-August 2013	**5 months testing/evaluation work at BESSY II, PTB**
June 2013	Formal agreement with **Elettra Sincrotrone Trieste** for commissioning, beamtime access etc., **40%** was granted to the IAEA
October 2013	Installation at Elettra
April 10 2014	First Synchrotron light at the end station
April-May 2014	First commissioning experiments
2nd semester 2014	Continuation of the commissioning work, Feasibility experiments
January 2015	Open to end users

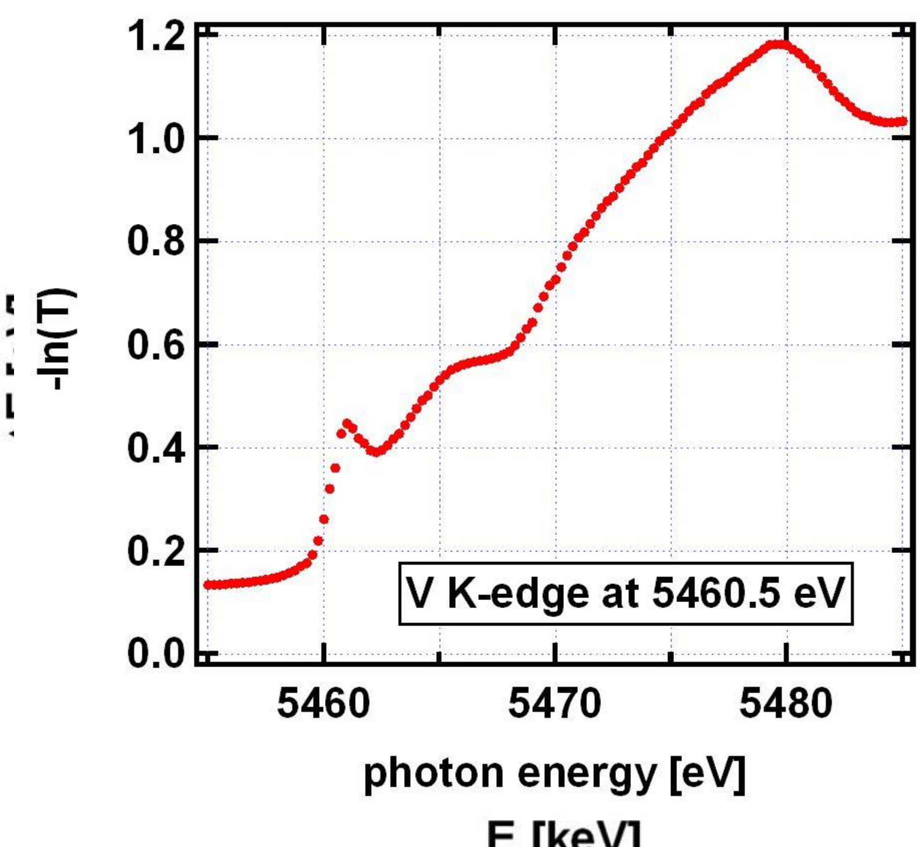
XRF Beamline@ Elettra Sincrotrone, Trieste



220 µm (H) X 90 µm (V)

XRF Beamline@ Elettra Sincrotrone, Trieste





Energy Range (excitation)	2000 - 14000 eV
Beam size (at exit slits)	220 µm (hor) X 90 µm (vert)
Beam divergence (at exit slits)	0.15 mrad
Flux @5.5 keV (2 GeV) or 7 keV (2.4 GeV)	5 10^9 ph/s (theory)
Monochromator	Si (111), InSb
Resolving power	DE/E: 1.5 10^{-4} (Si(111))
Source	Bending Magnet

Elettra Sincrotrone Trieste

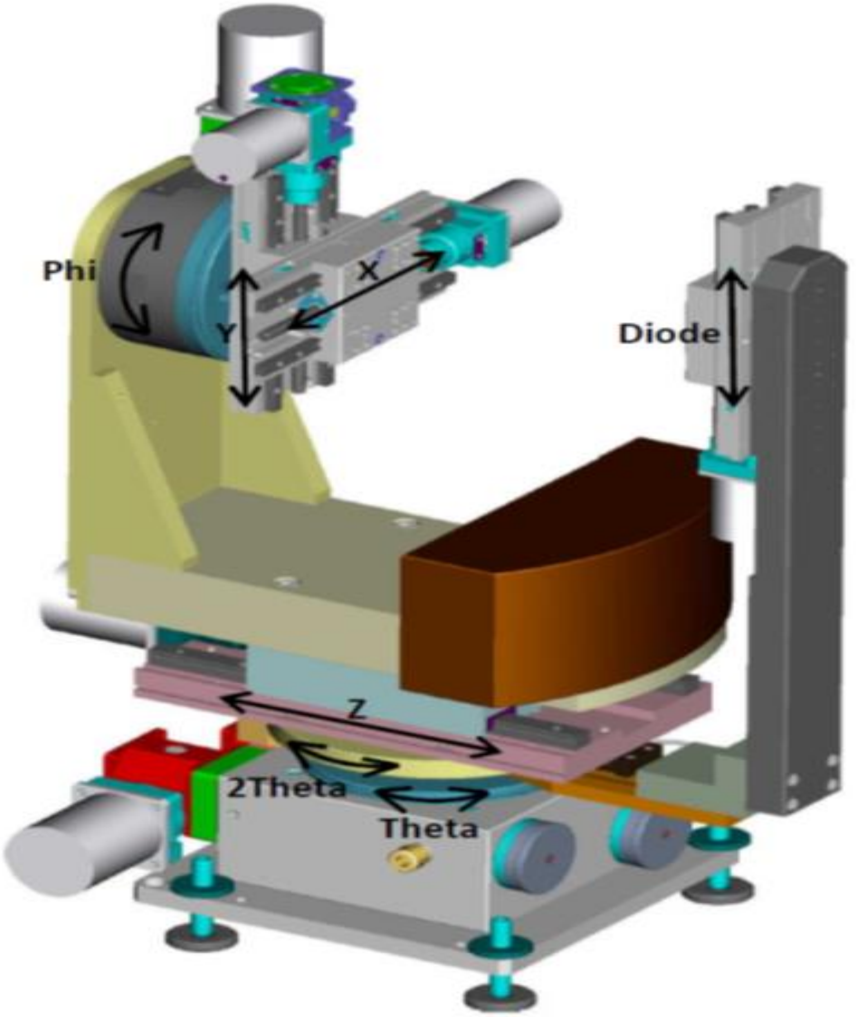
The IAEA integrated XRS end station

The IAEA Ultra High Vacuum Chamber (UHVC) is based on a prototype design by Physikalisch - Technische Bundesanstalt (PTB, Berlin)* and Technical University of Berlin (TUB)

***J. Lubeck et *al*, A novel instrument for quantitative nanoanalytics involving complementary X-ray methodologies, Rev. Scientif. Instrum. 84 (2013) 045106-7**

UHVC Instrumentation: 7-Axis Manipulator

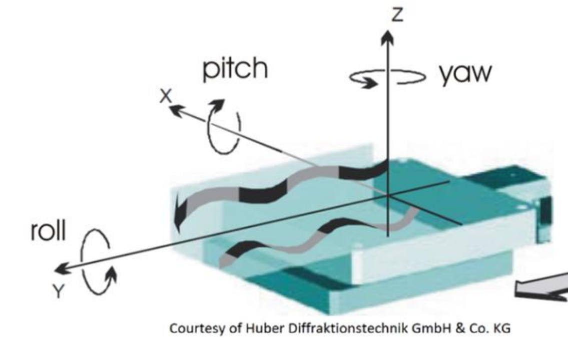
Sample alignment:
Three (3) linear stages ('X', 'Y', 'Z',)
Two (2) goniometers ('Theta', 'Phi')
Photodiodes alignment:
One (1) linear stages ('diode')
One (1) goniometer ('2Theta')



Sample can be moved in various directions/ orientations with respect to the exciting X-ray beam or with respect to the detectors.

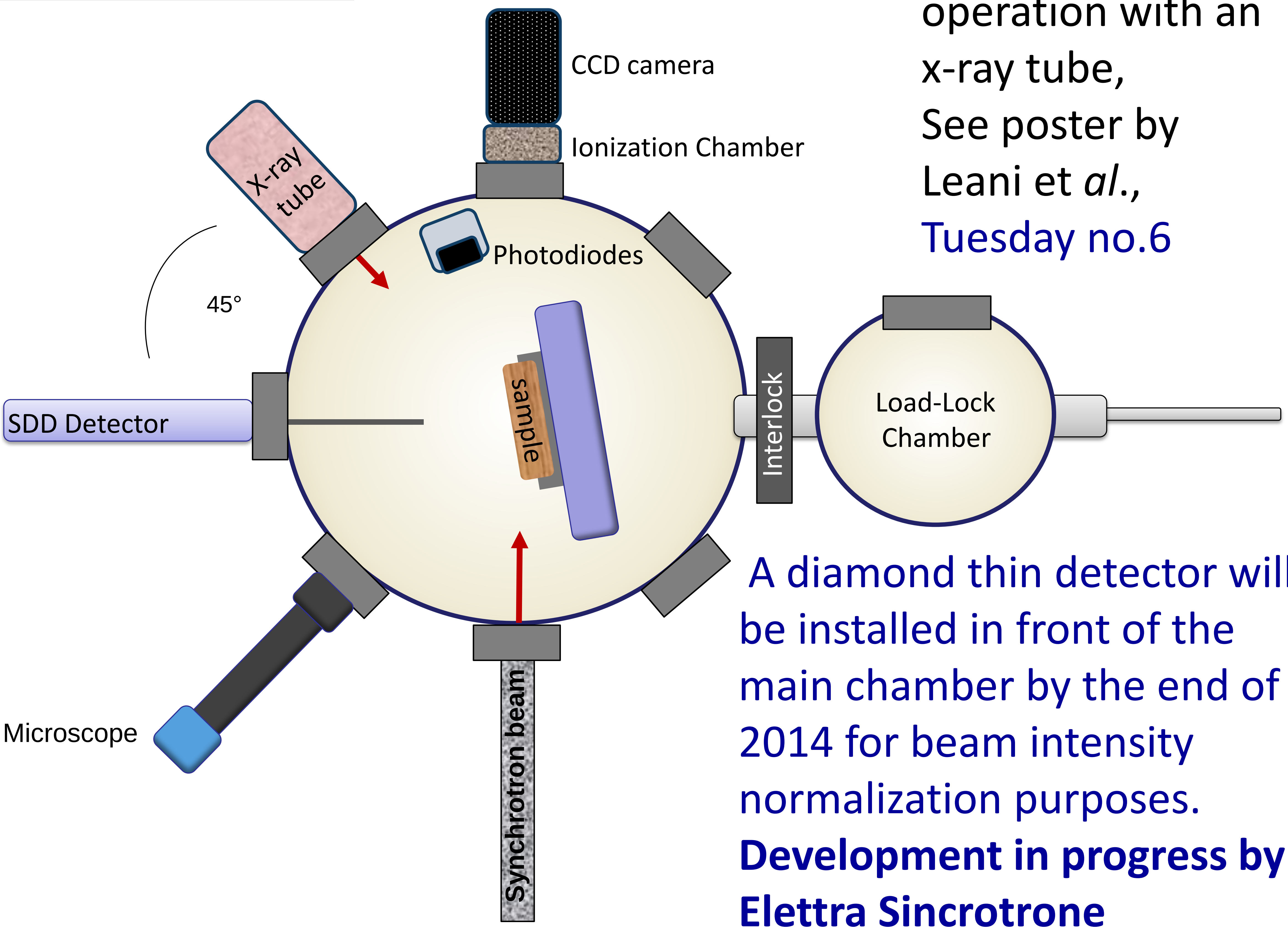
Evaluation results @ PTB
Linear axes <150 micro-radians, 11cm distance
Theta, 2theta, phi: Accuracy, reversal error<40 micro-radians

X-ray Detectors:
Ultra Thin Window (UTW) Bruker Silicon Drift detector (30 mm^2, FWHM 131 eV @ Mn-Ka), Si/GaAs several photodiodes



UHVC general geometry and devices

UHV Chamber (Top view)

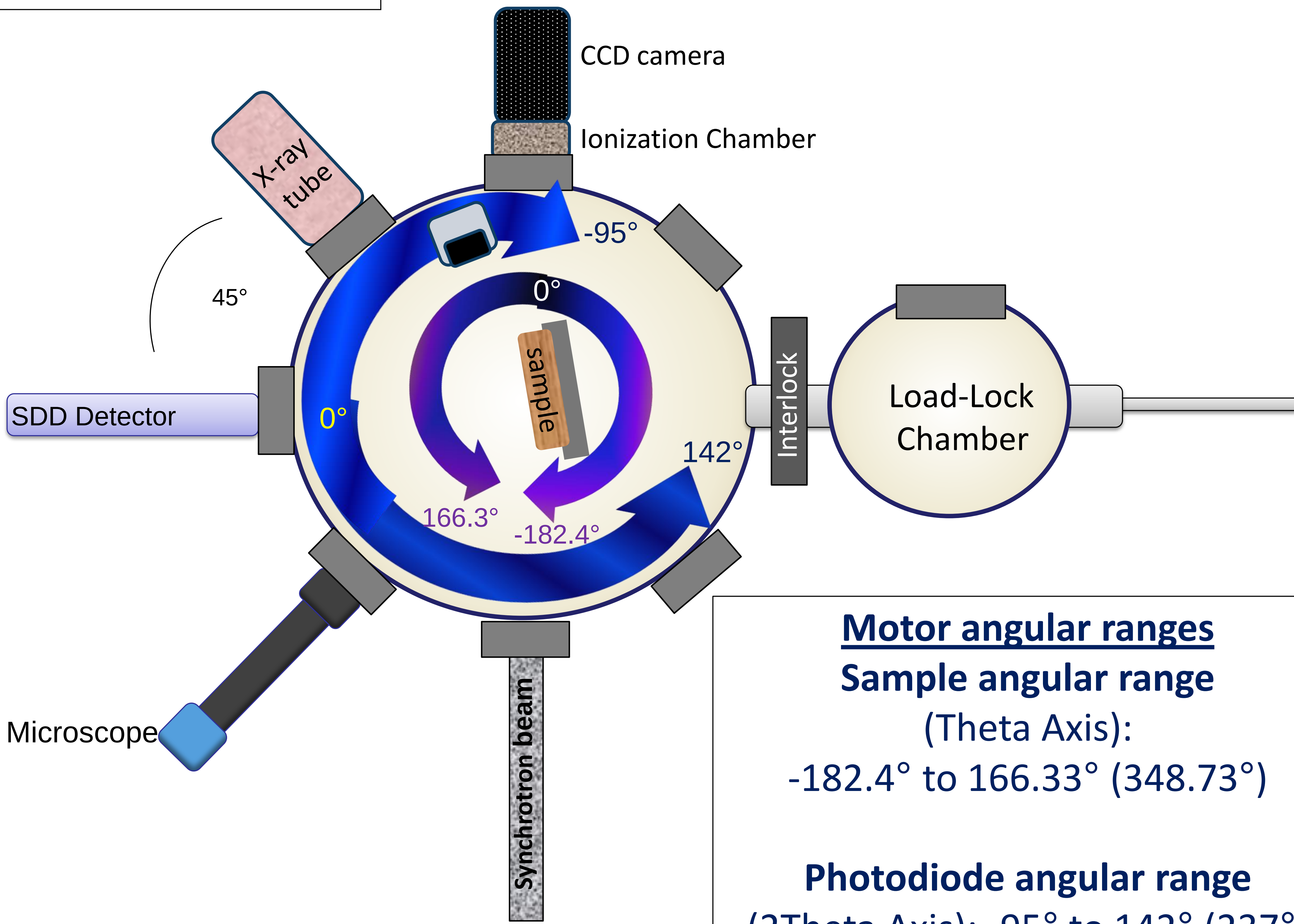


Standalone operation with an x-ray tube, See poster by Leani et *al*., Tuesday no.6

A diamond thin detector will be installed in front of the main chamber by the end of 2014 for beam intensity normalization purposes. **Development in progress by Elettra Sincrotrone**

UHVC general geometry and devices

UHV Chamber (Top view)



Motor angular ranges

Sample angular range

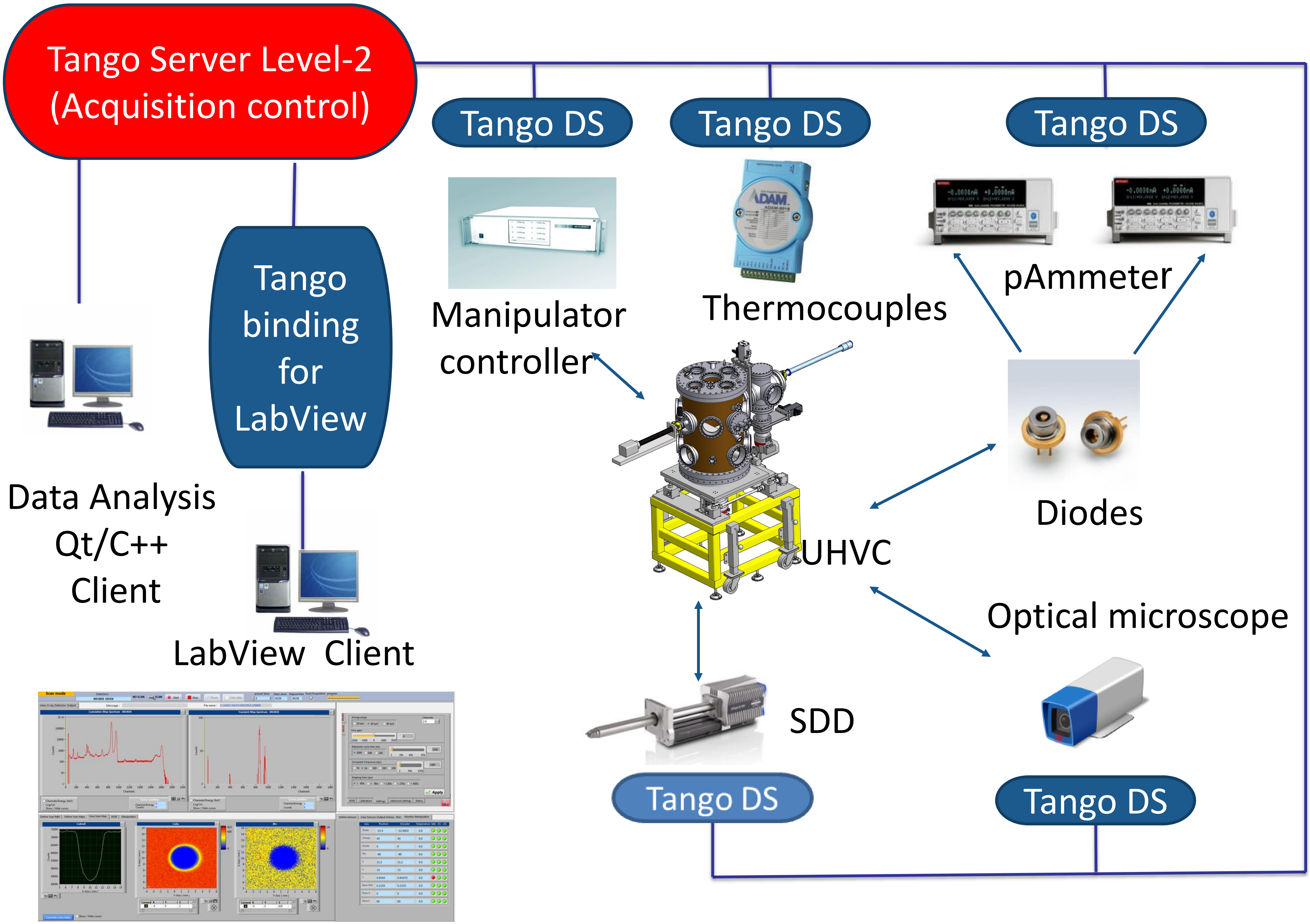
(Theta Axis):

-182.4° to 166.33° (348.73°)

Photodiode angular range

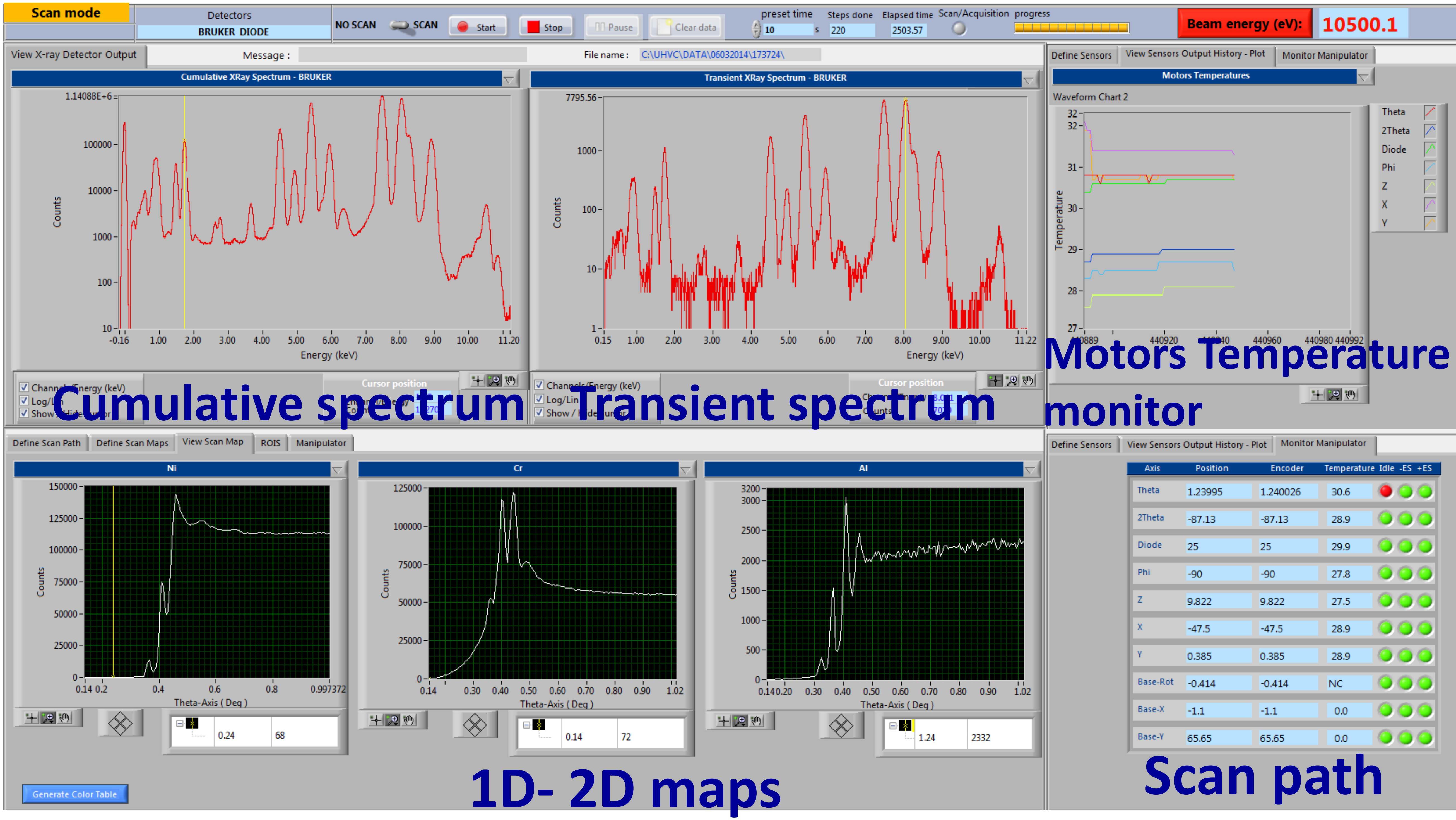
(2Theta Axis): -95° to 142° (237°)

UHVC Data Acquisition System - Architecture



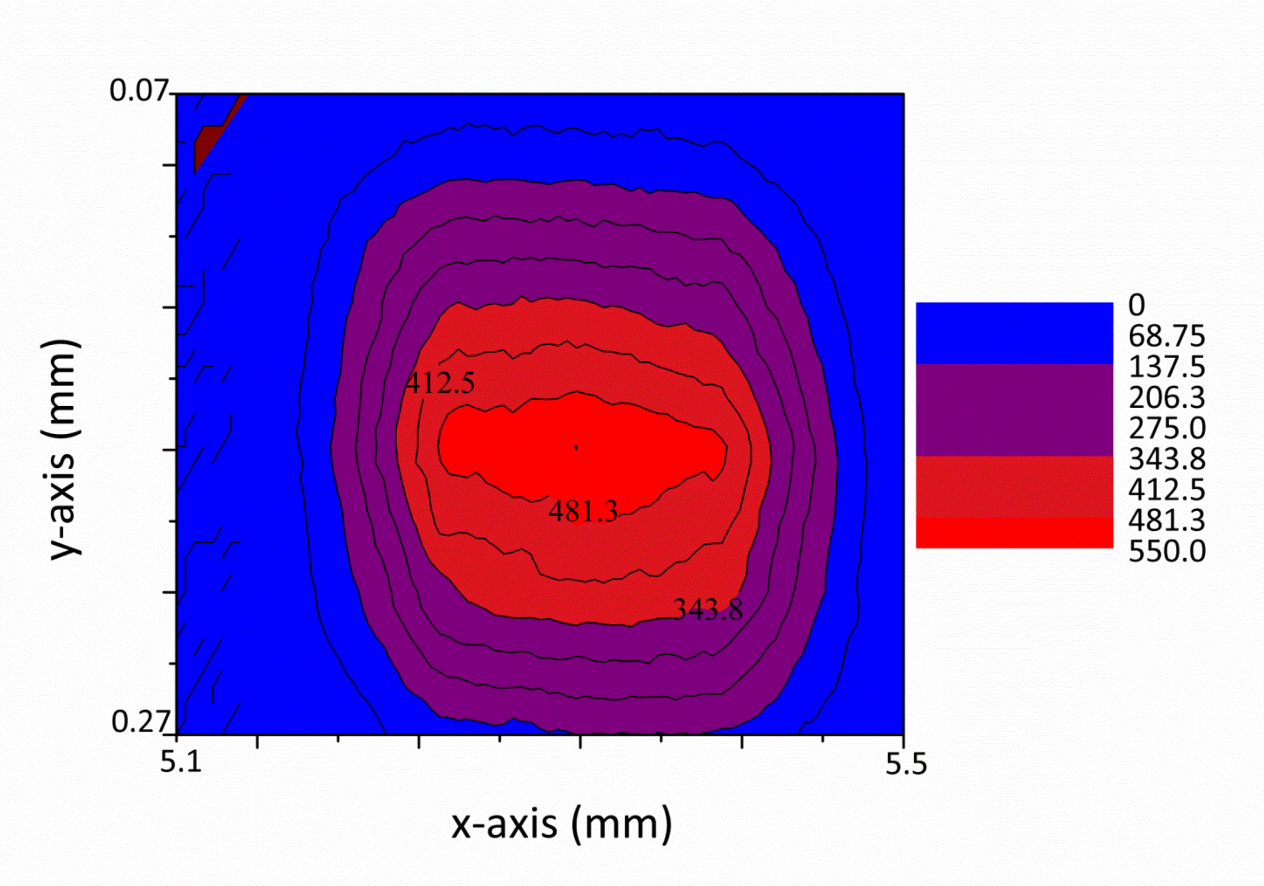
Developed Acquisition software - DAUHVC

GIXRF/XRR experiment: Analysis of thin multi-elemental structure on silicon nitride film

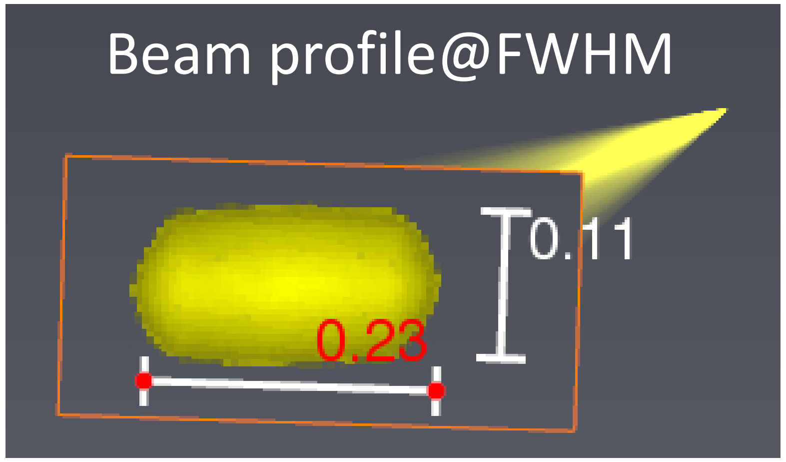


LabView GUI platform

Beam profile/divergence measurements

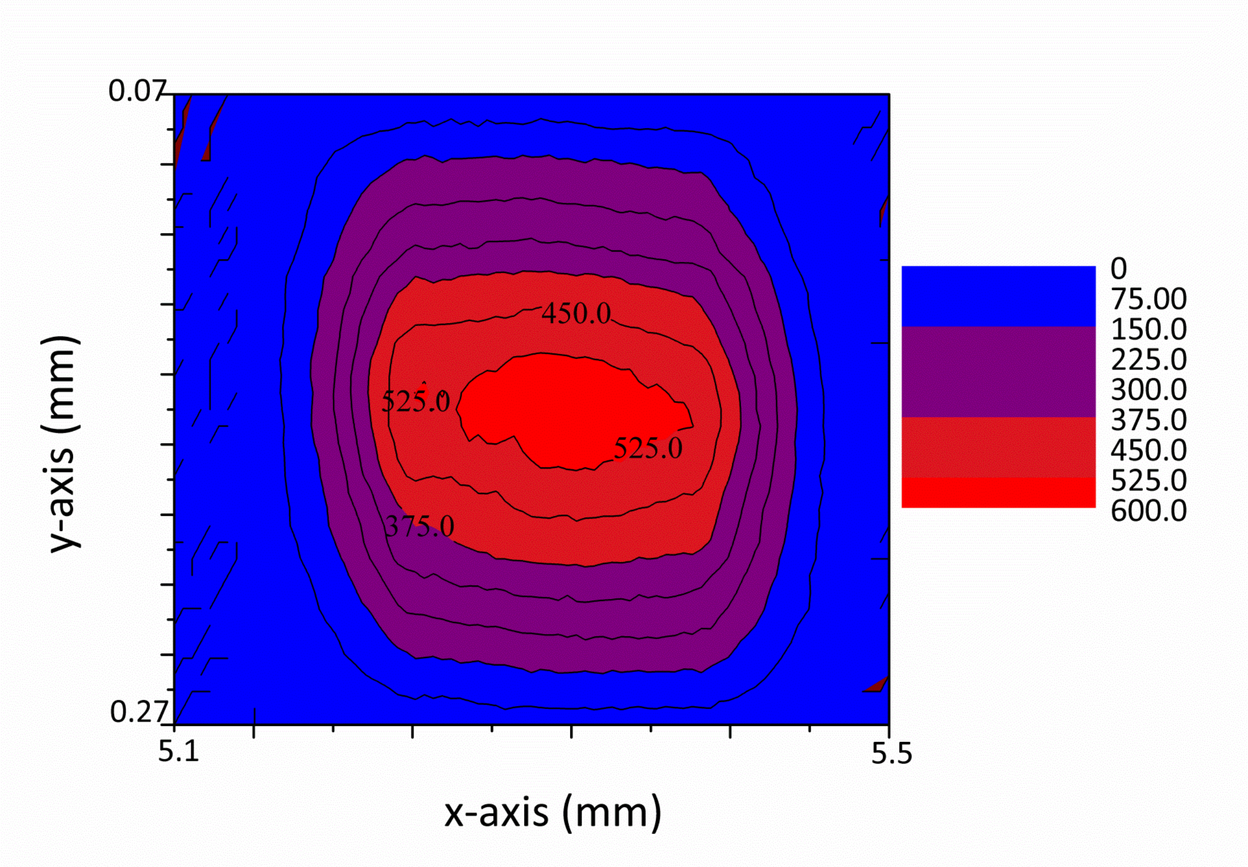


Incident Energy: 3600 eV

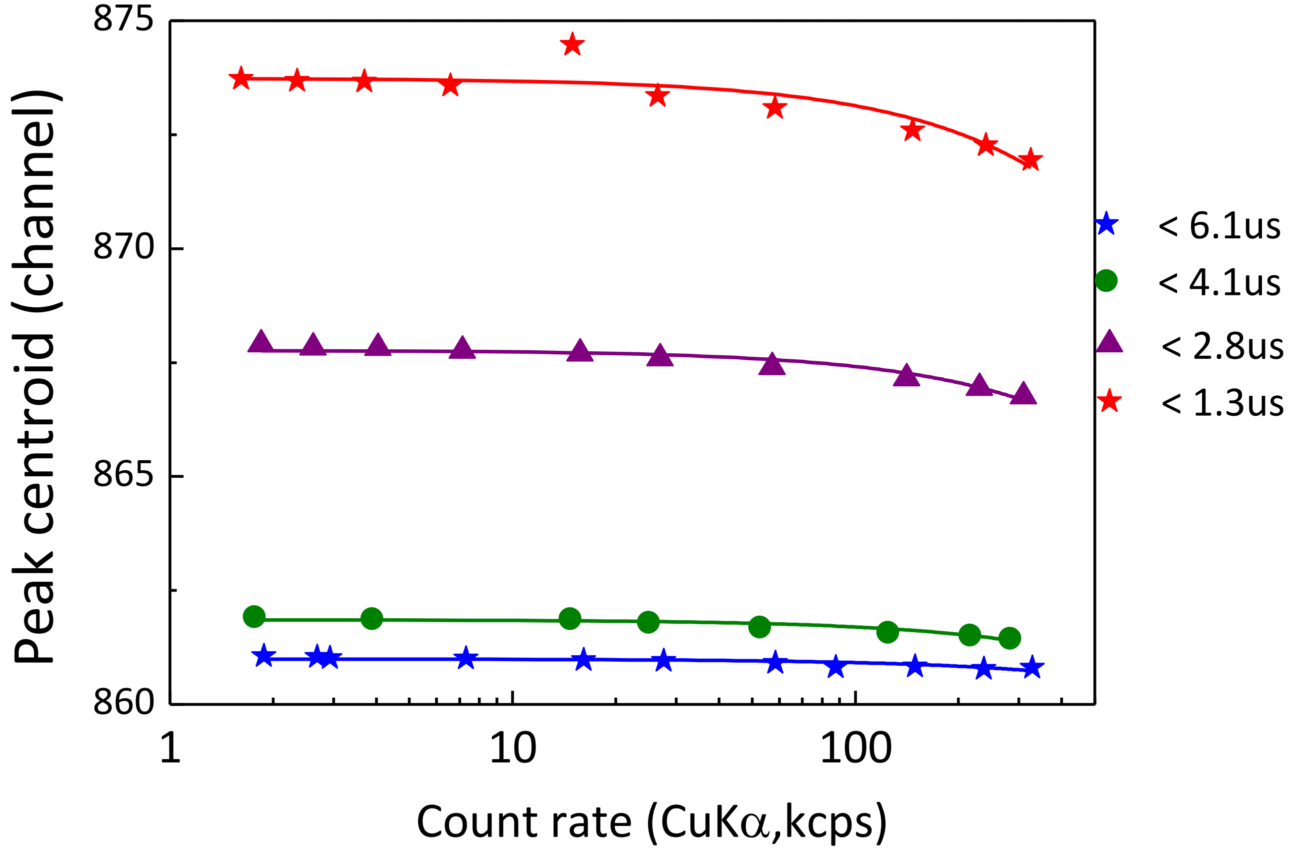


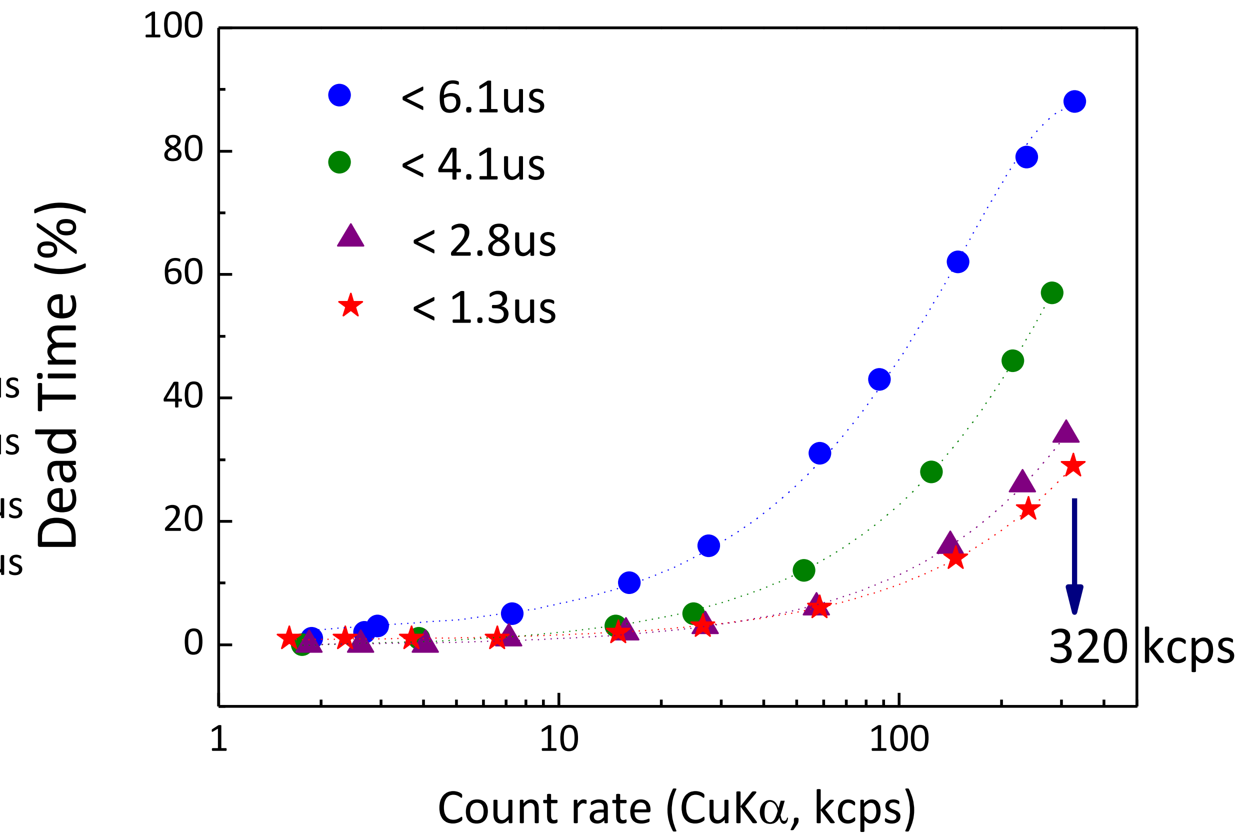
10um pinhole/5 um step

Beam profile measurements at the two switch limits (distance of 11cm) have shown no measurable difference in the beam dimensions (measurement sensitivity corresponds to less than 50 micro-radians

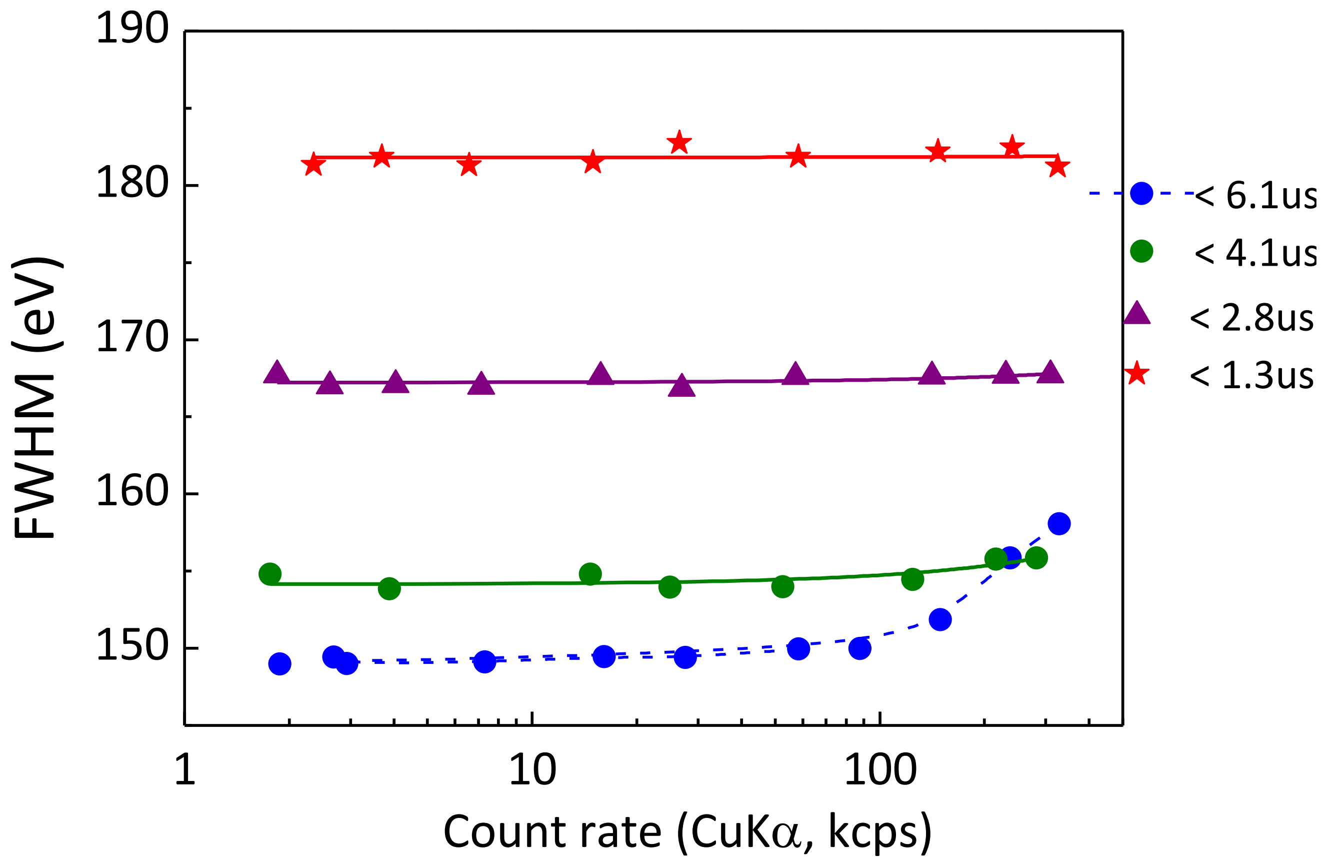


Silicon Drift Detector: Count rate performance

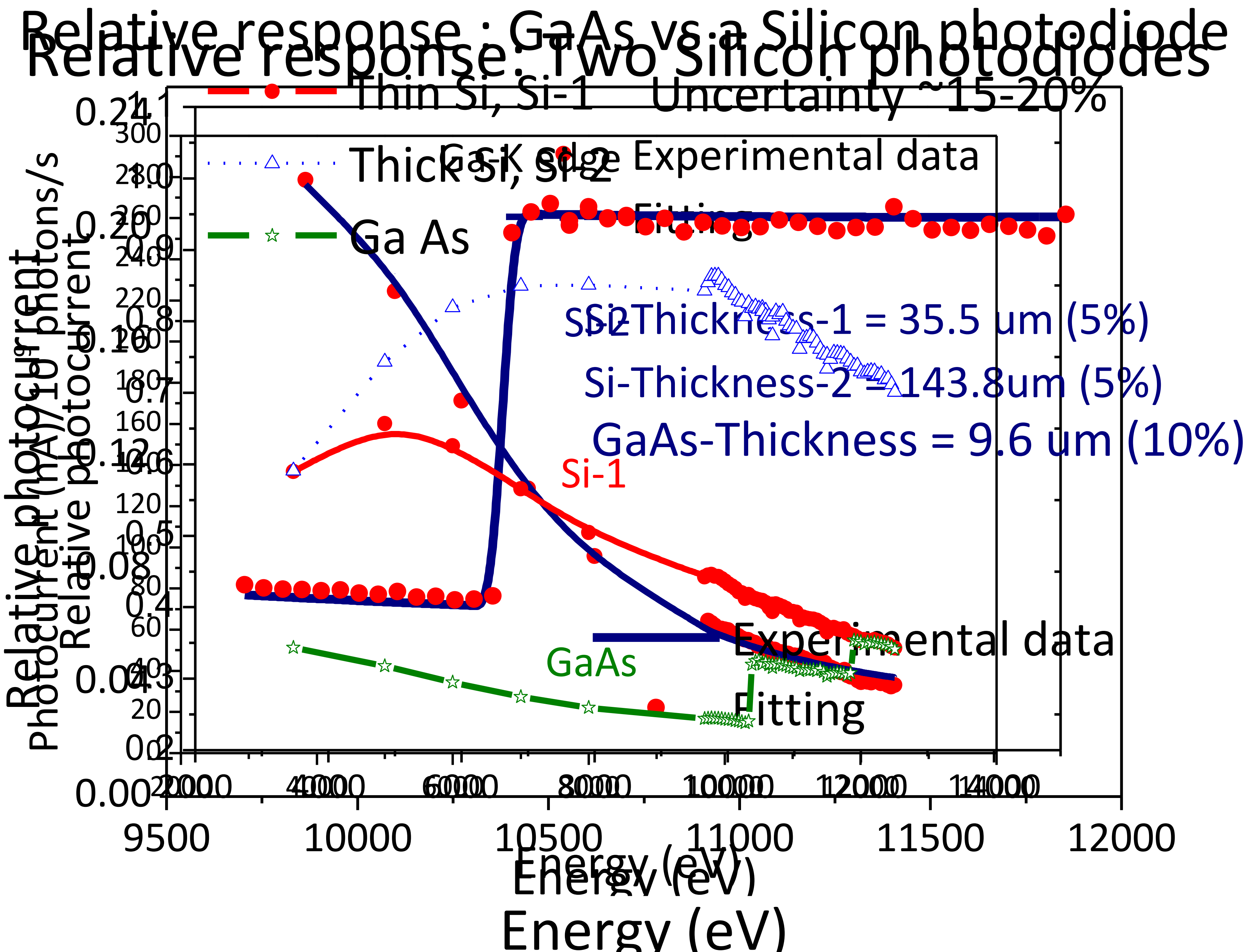




Measurements for Cu-Kα

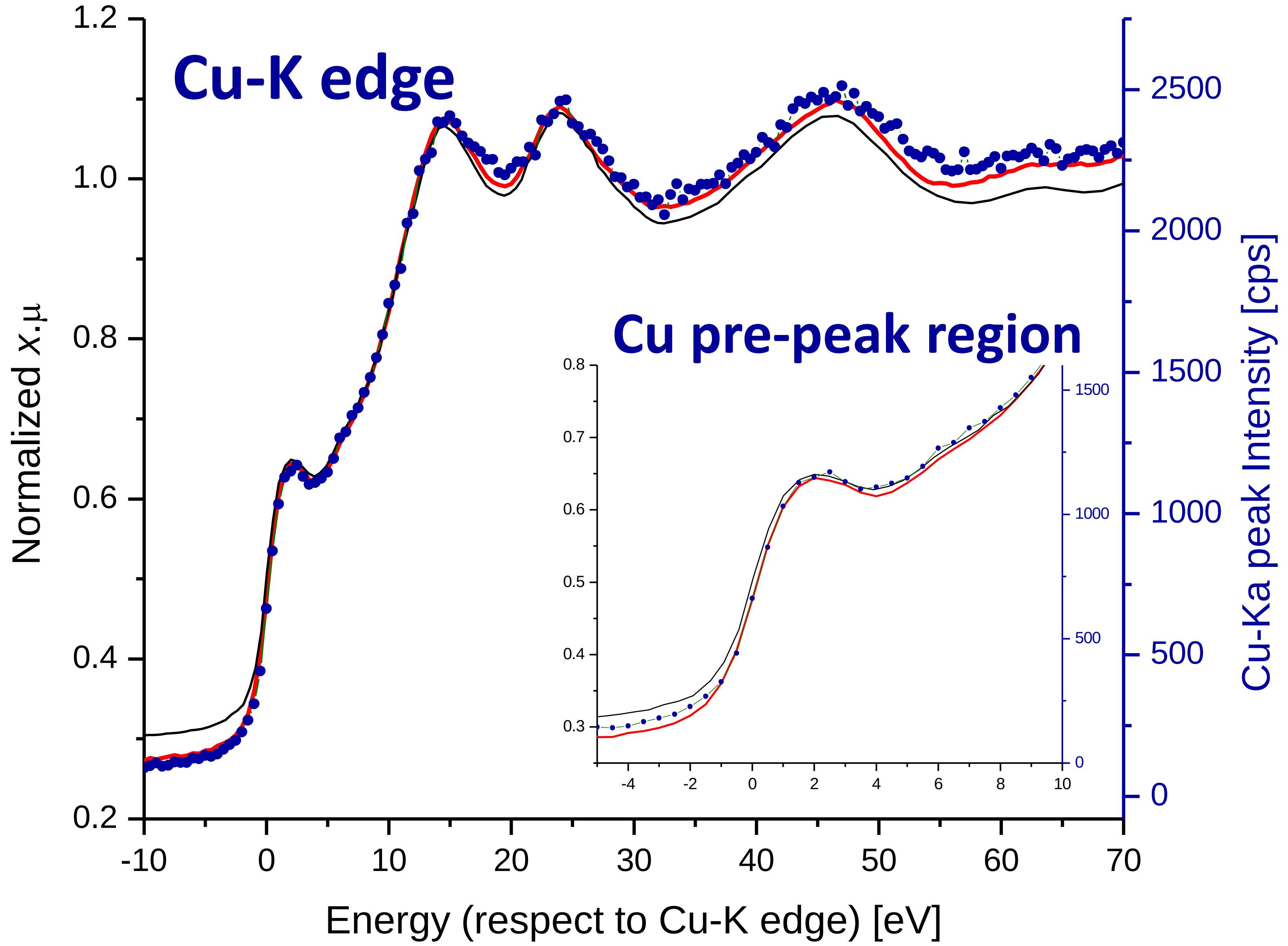


Photodiodes characterization: In progress



By combining the photodiodes current with the one registered by an Ionization Chamber

XANES: First measurements@endstation



XANES measurements on transmission mode carried out using a Si photodiode

XANES measurements on fluorescence mode distance sample-detector: 15 mm)

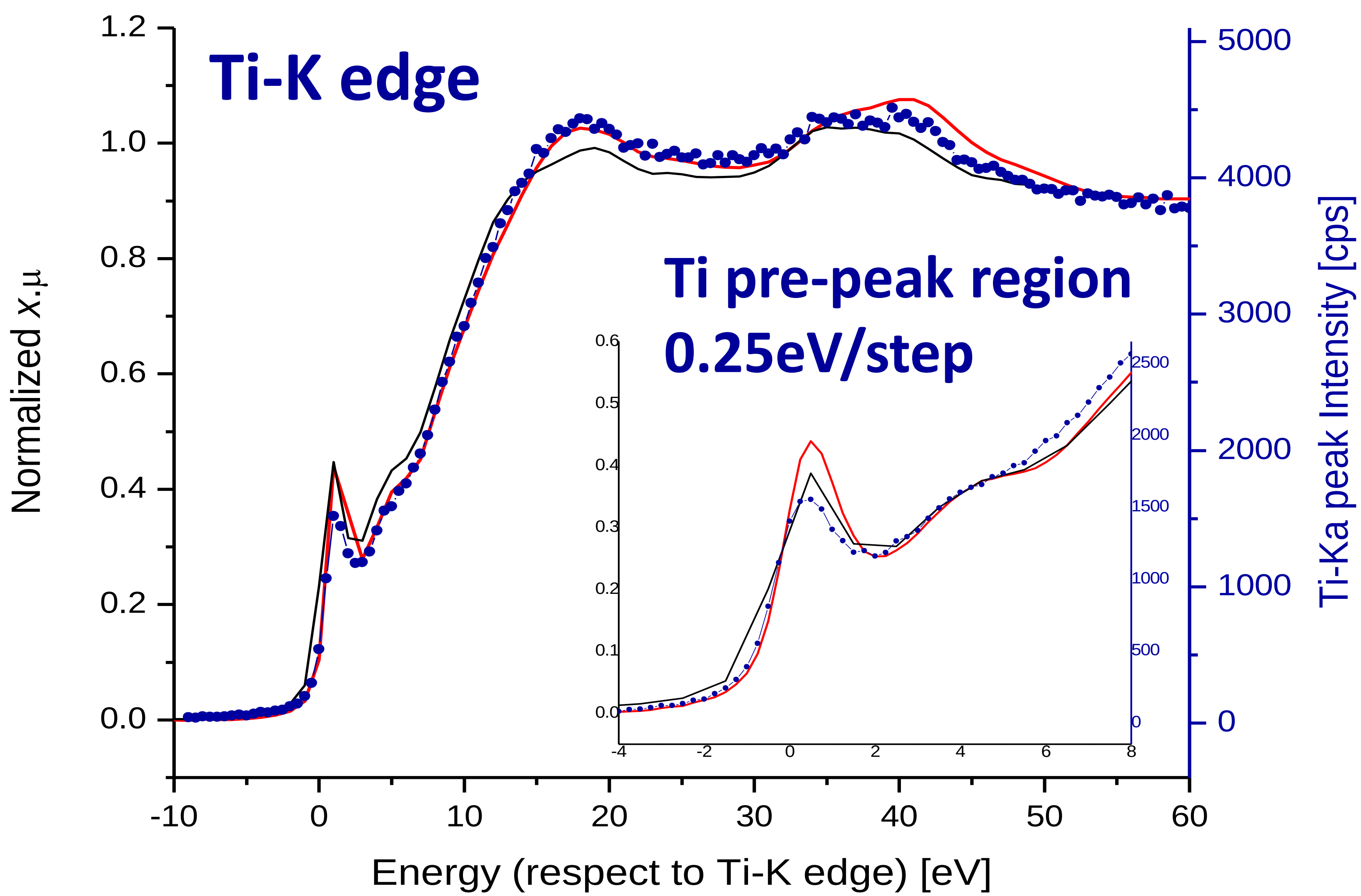
— Transmission mode on 7 um Cu-foil (0.5 eV step, 1 sec/step).

••• Fluorescence mode on thin, 9 ug/cm^2 Cu target (0.5 eV step, 5 s/step).

— Transmission mode on 12 um Cu-foil (1 eV step) from CARS database (http://cars.uchicago.edu/~newville/ModelLib/search.htm)

Inset: zoom of the pre-peak area

XANES: First measurements@endstation



XANES measurements on transmission mode carried out using a Si photodiode

XANES measurements on fluorescence mode performed (distance sample-detector: 15 mm)

– Transmission mode on 6 um Ti-foil (1 eV step, 1sec/step).

•• Fluorescence mode/thin 9 ug/cm^{2}l Ti target (0.5 eV step, 5sec/step)

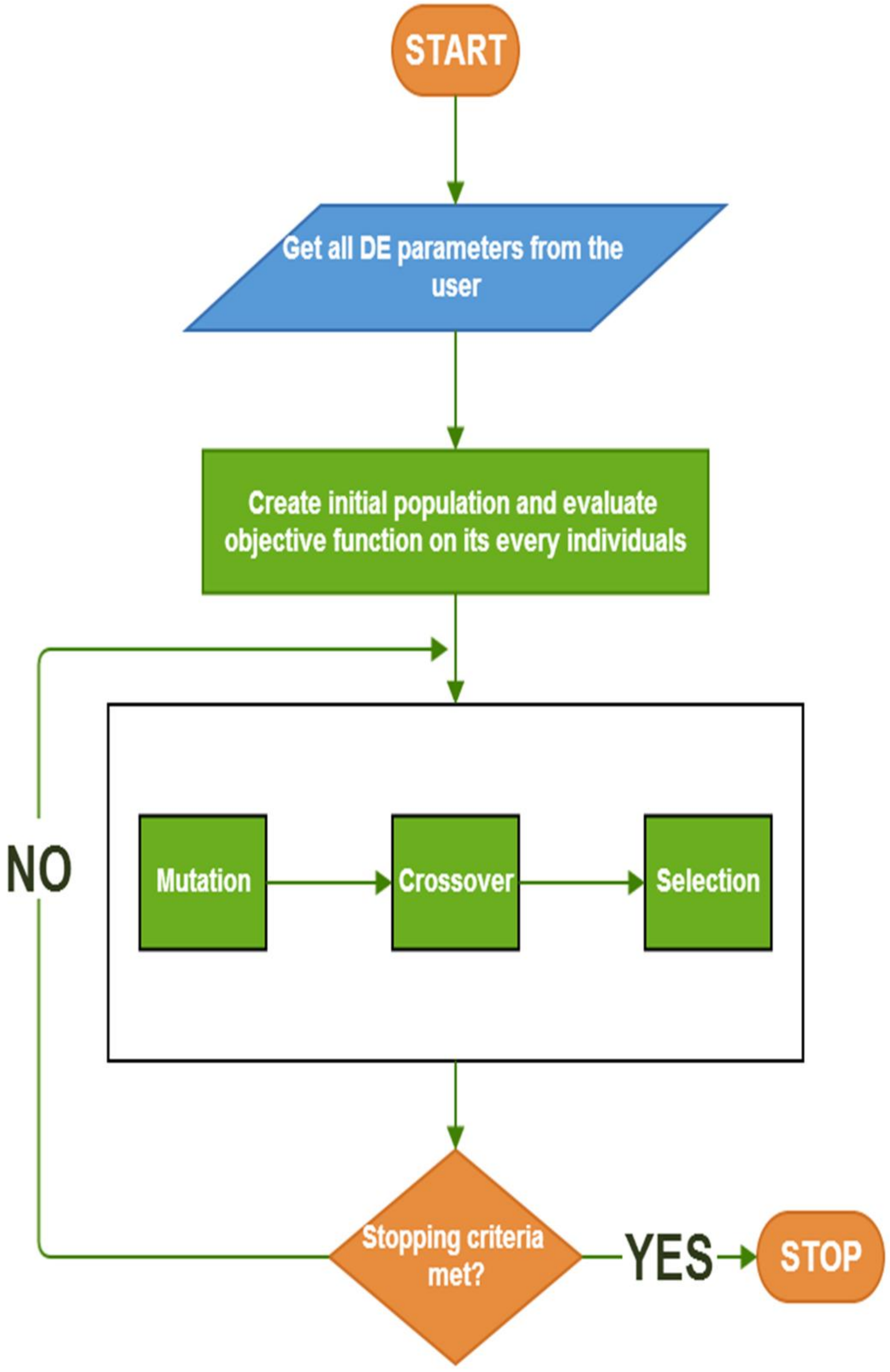
— Transmission mode on 5 um Ti-foil (1 eV step) from IXAS database, http://ixs.iit.edu/data/Farrel_Lytle_data

— Transmission mode on 6 um Ti-foil (0.25 eV step, 1sec/step).

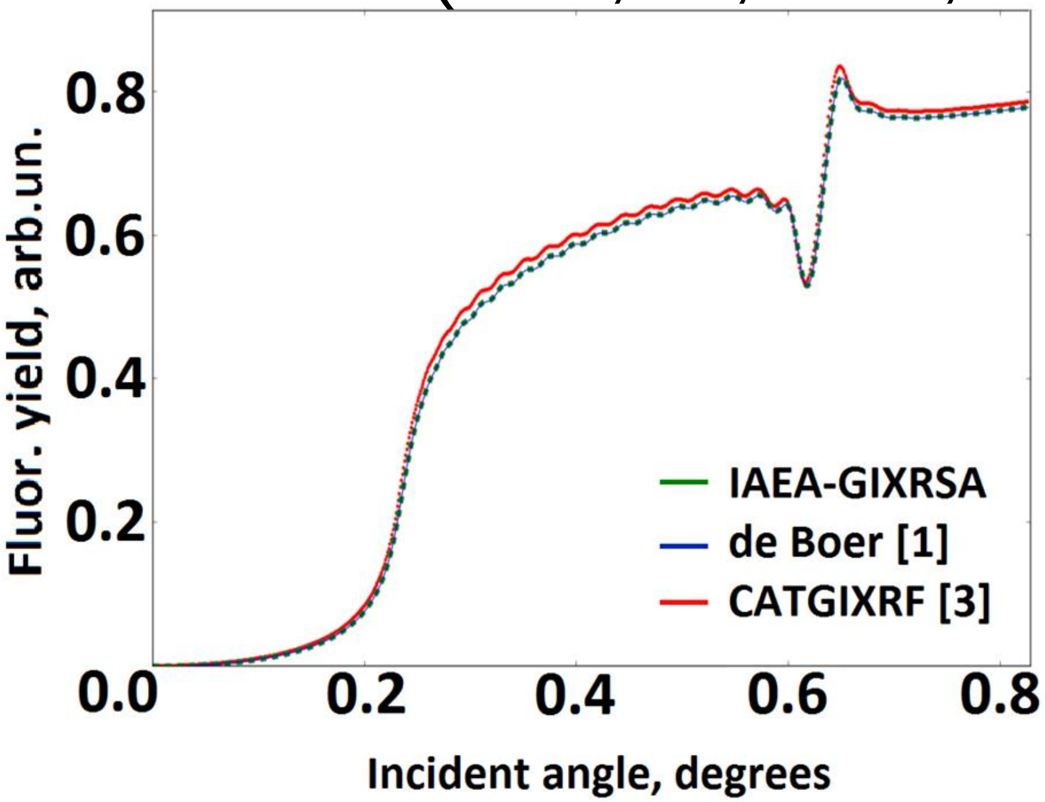
•• Inset: Fluorescence mode/(0.25 eV step, 5 sec/step).

Development of the IAEA-GIXSA package

Fitting Based on Differential Evolution Algorithm



XSW based on Parrat's recursive formalism, GIXRF profiles by De Boer (PRB, 44, 1991, 498)



Theoretical Simulation 20 periods 1.5nm **Co/** 2nm **Au** on Si

GIXRF/XRR Data

- Many parameters to fit
- Many local minima (multimodal objective functions)

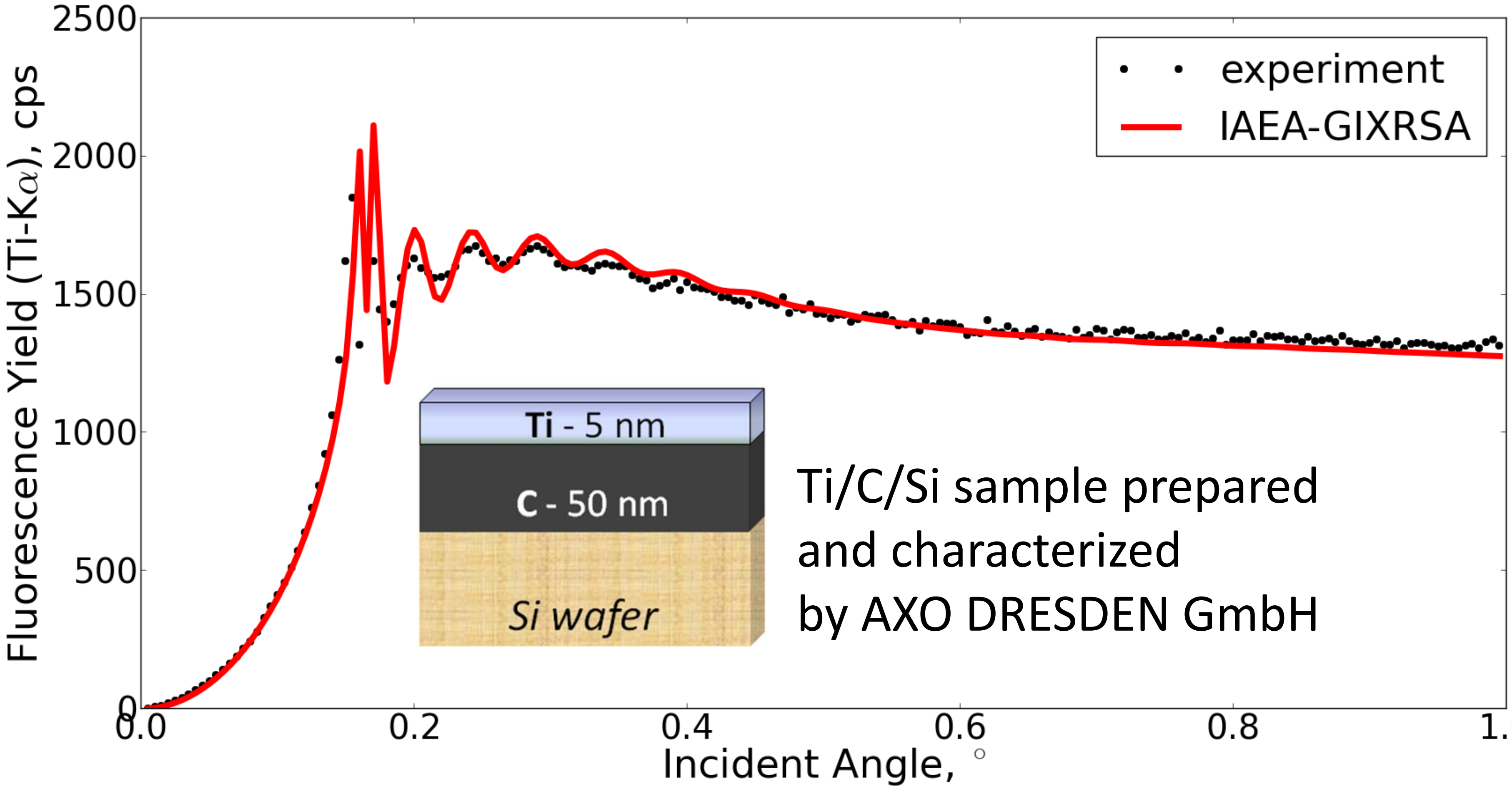
Genetic Algorithms:

- Allow to find the global minimum
- No need to know derivatives
- The convergence could be unstable

Fitting GIXRF profiles using the IAEA-GIXRSA package

Fluorescence data corrected for dead time and geometrical effects due to the beam footprint and sample-detector geometry [Wenbin et al., *Rev. Scientific Instruments, 83, 053114 (2012)*]

Incident X-ray beam Energy: 10500 eV, step: 0.005 degrees



Nominal values
d(Ti) = 5 nm
d(C) = 50 nm

Fitted values
d(Ti) = 6.9 nm
d(C) = 50.2nm

Absolute intensity **was not** fitted, in progress!

IAEA Coordinated Research Project (CRP)@Elettra

CRP Title: Experiments with Synchrotron Radiation for Modern Environmental and Industrial Applications, 2014-2017

Expected results

- To improve the operating and analytical protocols at the UHVC end-station at the EST XRF beamline
- To develop new, optimized and combined GIXRF/TXRF/ XRR/XRF methodologies, instrumentation and interdisciplinary applications
- To enhance research links and cooperative schemes in the field of SR based XRF techniques
- To provide advanced hands-on training in SR techniques and instrumentation

Eighteen (18) Countries with Eight (8) Synchrotrons involved

- Elettra Sincrotrone Trieste, Italy
- BESSY II, PTB, Berlin
- Indus-II, RRCAT (Raja Ramanna Centre for Advanced Technology, India
- Stanford Synchrotron Radiation Lightsource, USA
- Brazilian Synchrotron Radiation source, Brazil
- SESAME, Jordan
- PETRA III, Germany
- Synchrotron Light Research Institute - Thailand

IAEA Coordinated Research Project (CRP)@Elettra

Materials Science: Structured materials for energy storage and conversion technologies, (Thin-film solar cells, TCOs, Li ion batteries, TELs). Characterization of implants

Biomedicine: Biosensing technologies and nanomedicine design. Oxidation state analysis at trace/ultra-trace levels in tissue and cell samples.

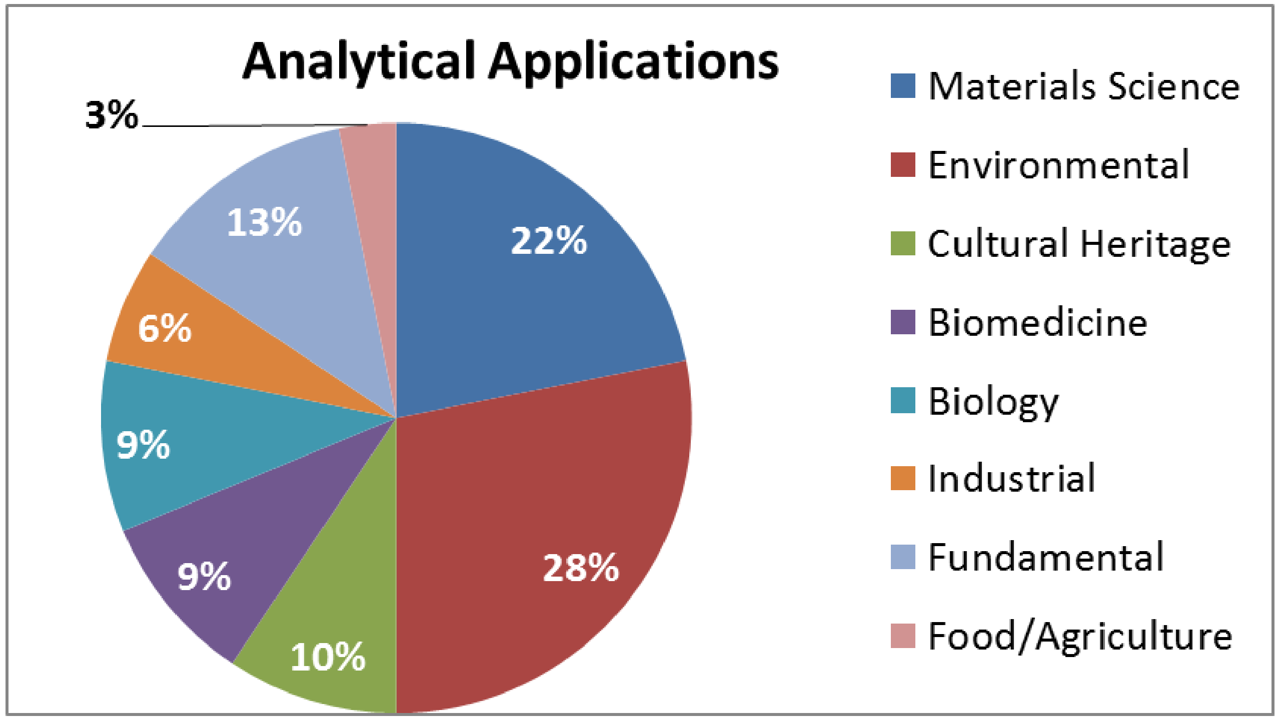
Environmental: Oxidation state analysis at trace/ultra-trace levels in aerosols, water samples, suspensions

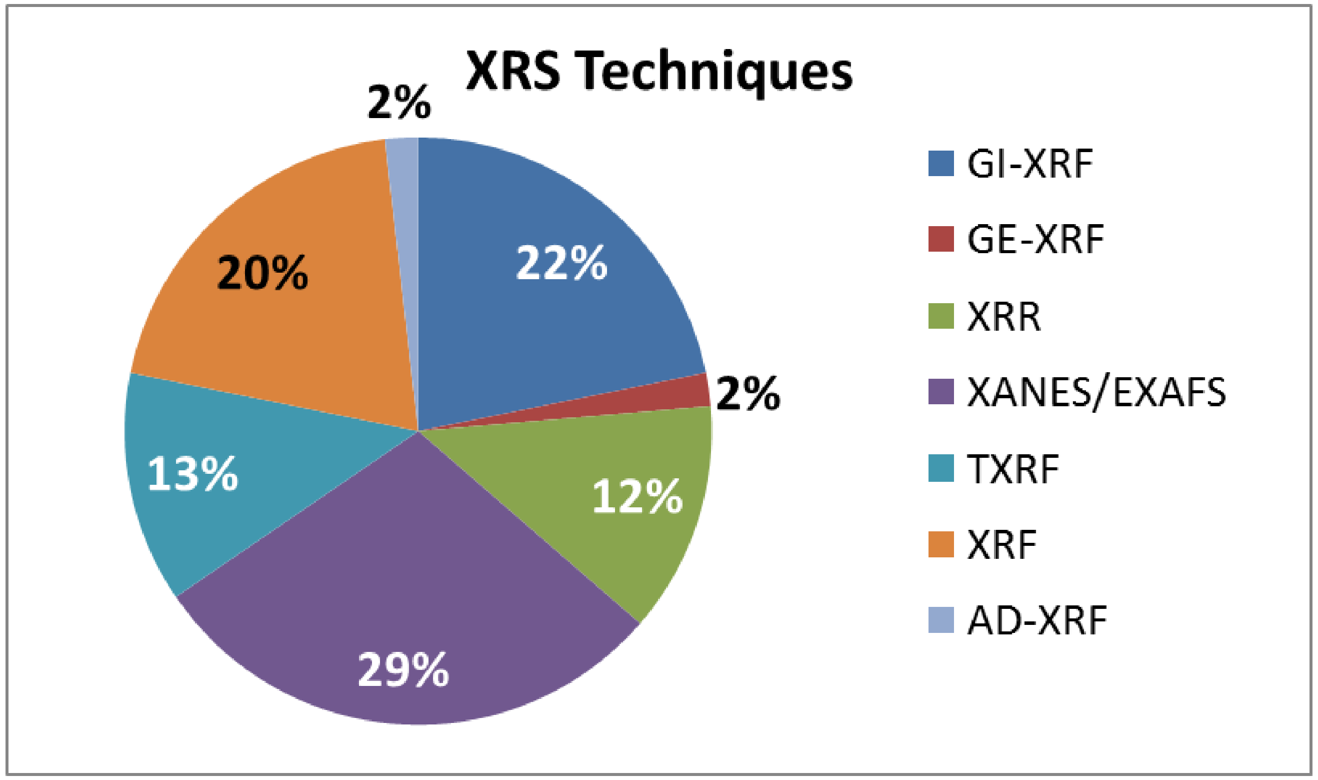
Biological: Study of metal metabolism in plants to develop/improve phytoremediation, biofortification and phytomining techniques. Elemental distribution/speciation on plant organ (leaves, roots, shoots, seeds, etc.)

Cultural Heritage: Painted decorations of archaeological pottery. Characterization of novel materials for preventive conservation of outdoor monuments/objects of Cultural Heritage

Food products security: Authenticity /contamination with hazardous metals

Fundamental: Fundamental parameters (Relative intensity ratios, RRS CS), Methodology in GE-XRF





Joint ICTP-IAEA School, 17-28 November 2014

The Abdus Salam
International Centre for Theoretical Physics
50th Anniversary 1964–2014

Joint ICTP-IAEA School on Novel Experimental Methodologies for Synchrotron Radiation Applications in Nano-Science and Environmental Monitoring

17 - 28 November 2014
Trieste, Italy

The Abdus Salam International Centre for Theoretical Physics (ICTP), Trieste, Italy, and the International Atomic Energy Agency (IAEA), Vienna, Austria, are jointly organizing a **School on Novel Experimental Methodologies for Synchrotron Radiation Applications in Nano-Science and Environmental Monitoring**, to be held in Trieste from 17 to 28 November 2014.

The purpose of the Synchrotron Radiation School is to introduce and demonstrate advanced analytical methodologies based on X-ray spectrometry with application in the fields of Nano-Science and Environmental monitoring. The introduction of environment-friendly energy sources and the diagnosis, monitoring and understanding of environmental problems are issues of high concern for developing countries due to the destructive impact of the growing industrialization. These problems in many cases can be tackled by novel technologies based on the development of nano-structured materials that can be fully characterized due to the remarkable properties of synchrotron radiation.

This two-week School is addressed to young motivated scientists with a Ph.D., or at least several years of research activity. It will offer a balanced program composed by lectures, tutorials and practical hands-on training sessions at different Elettra beamlines including the newly developed XRF beamline and the IAEA multipurpose endstation. The School aims to enhance the skills of participants in applying synchrotron radiation based on X-ray spectrometry techniques. It will also represent an opportunity for young researchers from developing countries to utilize in a competitive manner the access mechanisms offered by Elettra, the IAEA and the ICTP.

The following specific topics will be included:

- Synchrotron Radiation Instrumentation: X-ray optics, UHV instrumentation, Advanced sample manipulator systems, trends and developments
- Theory and analytical applications of X-ray Absorption Fine Structure (XAFS) techniques
- Synchrotron Radiation based X-Ray Fluorescence (XRF) analysis Methodologies: Grazing Incidence XRF (GI-XRF), Total Reflection XRF (TR-XRF), 2D-3D Micro-XRF analysis, Quantification methodology and Monte Carlo Methods
- Tutorials on XRF/GIXRF and XAFS data analysis and on Monte Carlo Methods
- Experimental hands on sessions at Elettra XRF, TwinMic and XAFS beamlines

PARTICIPATION

Young scientists with a Ph.D., or at least several years of research activity, who are from countries that are members of the UN, UNESCO or IAEA, may attend the School. The Organizers will select participants upon evaluation of the application forms. The main purpose of the Centre is to help researchers from developing countries through a programme of training activities within the framework of international cooperation, however, scientists from developed countries are also most welcome to participate. As the School will be conducted in English, participants must have an adequate working knowledge of that language.

There is no registration fee to attend this School. As a rule, travel and subsistence expenses of the participants are borne by their home institutions. Limited funds are available for a limited number of applicants who are nationals of, and working in, developing countries, and who will be attending the entire School. As scarcity of funds allows travel to be granted only in a few exceptional cases, every effort should be made by candidates to secure support for their fare from their home country. Funds from external co-sponsors will allow limited support of a few participants from developed countries, to be selected by Organizers.

How to apply for participation: until 10 July 2014, candidates can access the Online Application Form through the activity webpage: http://agenda.ictp.it/smr.php?2611. Once in the website, comprehensive instructions will guide you step-by-step, on how to fill out and submit online the application.

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Trieste, April 2014

Co-sponsors:

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Luca Gregoratti
ELETTRA, Trieste

Andreas Karydas
IAEA, Vienna

DEADLINE
10 July 2014

School Webpage:
http://agenda.ictp.it/smr.php?2611

Elettra Sincrotrone Trieste

PTB
X-ray and IR spectrometry

Joint ICTP-IAEA School on Novel Experimental Methodologies for Synchrotron Radiation Applications in Nano-science and Environmental monitoring
17-28 November 2014

Co-directors:
B. Beckhoff (PTB)
Nadia Bingeli (ICTP)
L. Gregoratti (Elettra)
Andreas Karydas (IAEA)

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- ✓ **Elettra Sincrotrone Trieste** and in particular of the **Informatics, Technical, Radiation Safety and Administrative staff**
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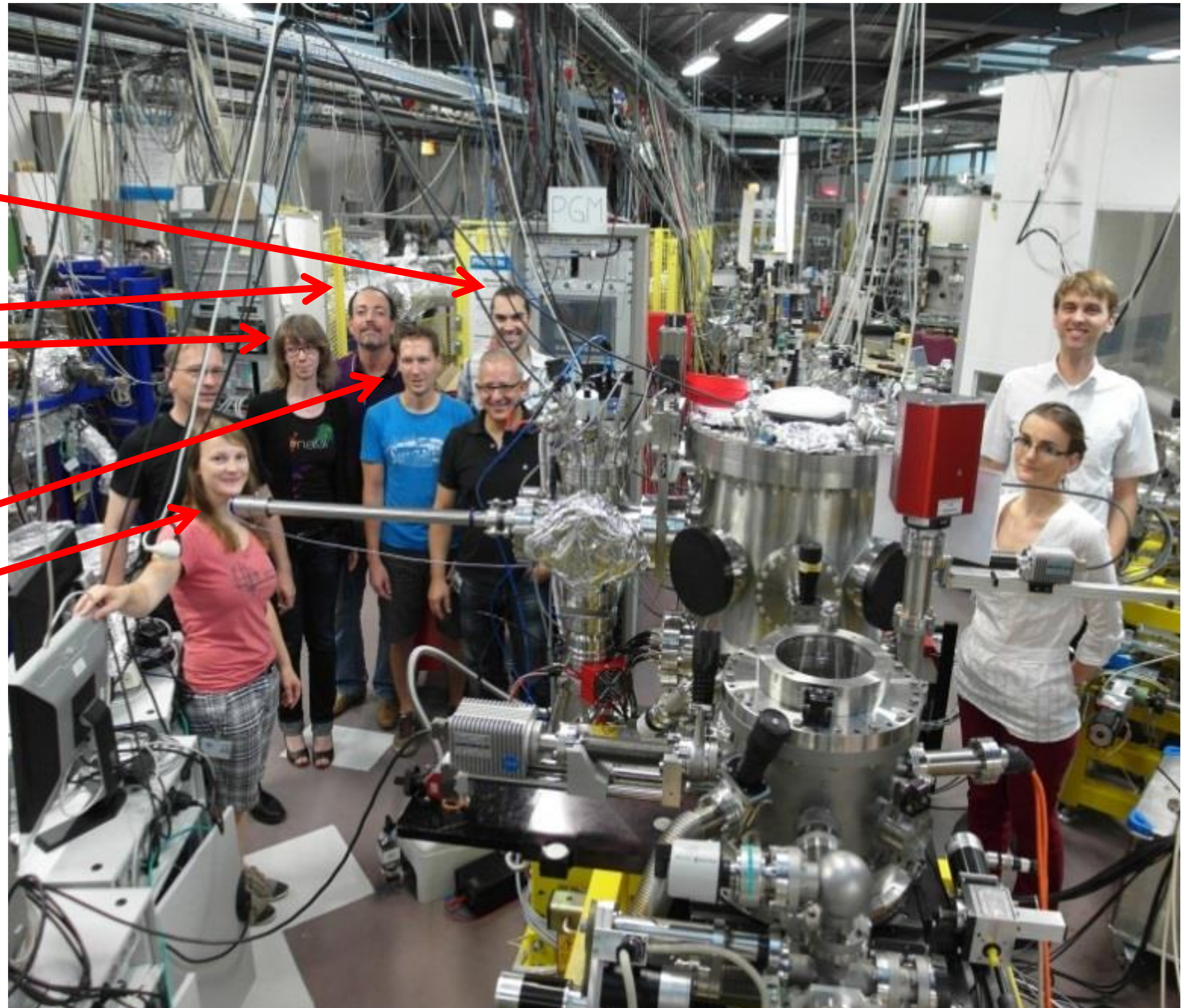
- ✓ **Bruker Nano**

Thank you for your attention!!

J. Leani
A. Karydas
A. Migliori
B. Pollakowski
B. Beckhoff
D. Grötzsch
J. Lubeck



L. Luhl
D. Eichert

2nd of August 2013

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