LabXAS: a new table-top x-ray spectrometer designed at Politecnico di Milano

3 October 2024

Research Centre for Natural Sciences, Budapest

Dr. Roberto Sant

Politecnico di Milano

Outline

❑ **Introduction**
- The PolimiX group
- Motivations and Objectives
- Design criteria and targets

❑ **LabXAS**
- Overview
- The chamber
- Rowland geometry
- The x-ray source
- The sample
- The crystal analyzer
- The detector
- Motors
- Radioprotection

❑ **Current situation**
- Applications
- Ongoing works
- Program

❑ **Conclusions and Perspectives**

The PolimiX Group

Prof. Giacomo Ghiringhelli

Prof. Marco Moretti

Dr. Roberto Sant

Former Master students:
Matteo Corti & Piero Florio

- **Expertise in synchrotron x-ray techniques and instrumentation design:**
  - Resonant Inelastic X-ray Scattering (RIXS)
  - X-ray Absorption Spectroscopy (XAS)

ID32 and ID20 RIXS spectrometers and
ID32 XMCD station at the ESRF

Motivations & Objectives

*The **technological advanments on x-ray sources** has favoured the rise of **compact table-top** instruments to be hosted **in standard labs**.*

Motivations :

- ❑ **Minimize** costs, size and complexity
- ❑ **Provide fast access** to XAS measurements for local groups :
  - Characterization of freshly synthesized new materials
  - Sample screening prior to synchrotron measurements

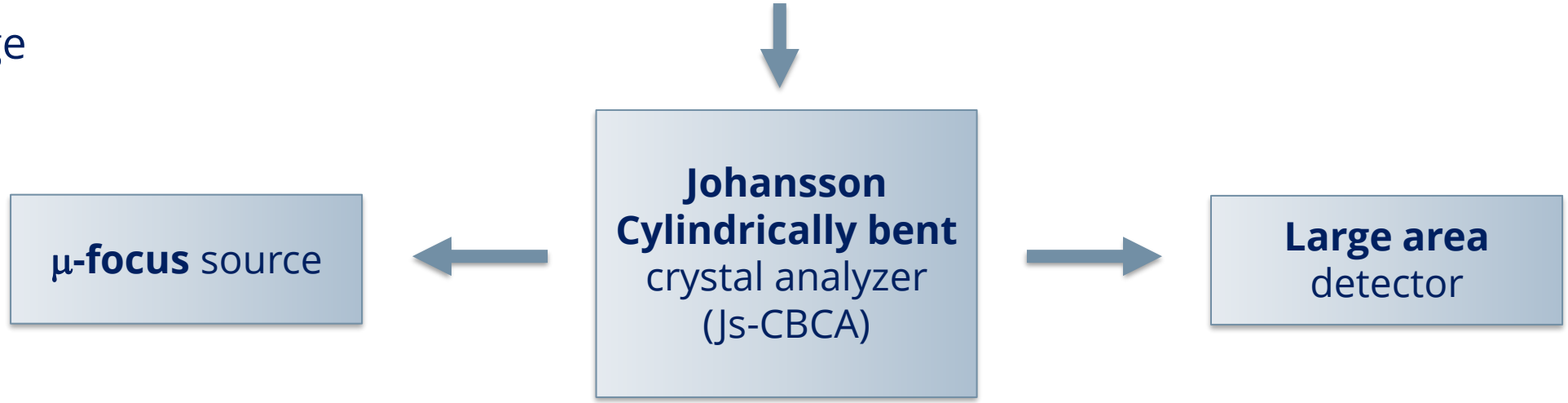
Objectives:

- ❑ Envisaged scientific methods :
  - **XANES and EXAFS** in the energy of 5-15 keV (3d-K, 5d-L edges of TM)

Design criteria and targets

Criterion : "*Minimizing complexity while maximizing the ease of use***"** by :

- **Pumping air** instead of using He
- Eliminate part of the optics by working with a **non-collimated (i.e. divergent) beam**
- exploiting multiple reflection orders from a **single analyzer** to cover the whole desired energy range



Design criteria and targets

Design :

- In transmission
- In vacuum
- μ-focus divergent x-ray source
- Johansson cylindrically bent crystal analyzer (Js-CBCA)
- Large area detector

Targets :

- Energy range: 5-15 keV
- Resolution: < 3 eV
- Counting rate: 10^5 ph/s
- Scan time (1000 pts, ΔE=2 eV) : < 2 hours

Outline

❑ **Introduction**
- The PolimiX group
- Motivations and Objectives
- Design criteria and targets

❑ **LabXAS**
- Overview
- The chamber
- Rowland geometry
- The x-ray source
- The sample
- The crystal analyzer
- The detector
- Motors
- Radioprotection

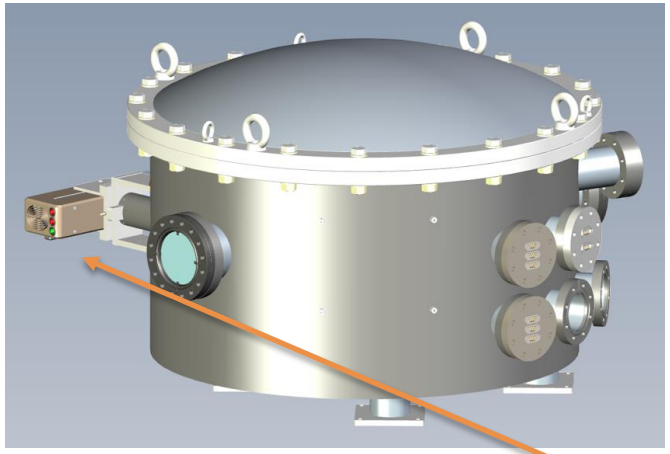
❑ **Current situation**
- Applications
- Ongoing works
- Program

❑ **Conclusions and Perspectives**

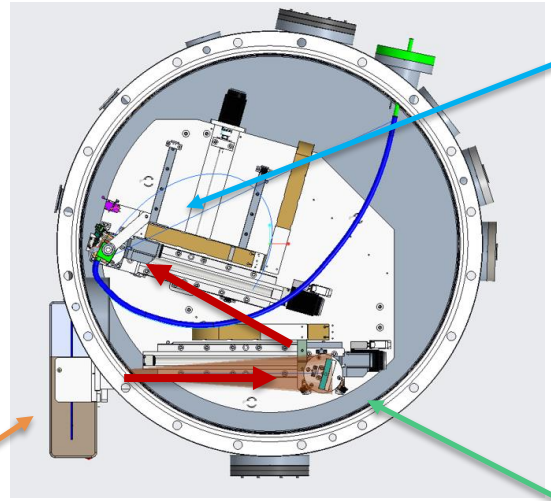
Overview

LabXAS *is a* ***table-top*** *instrument designed and built in collaboration with* ***Cinel s.r.l****. It consists of a vacuum chamber containing an* ***x-ray source****, a* ***crystal analyser*** *and a* ***detector*** *moving on a* ***Rowland circle****.*

Perspective view

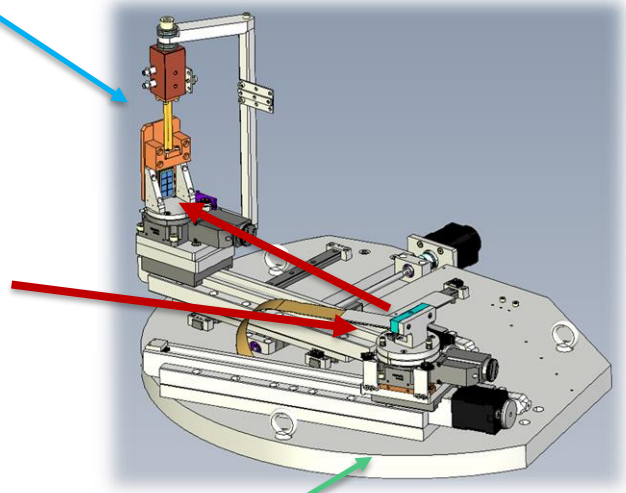


Top view



Detector

Interior view



X-ray source

Crystal analyser

The chamber

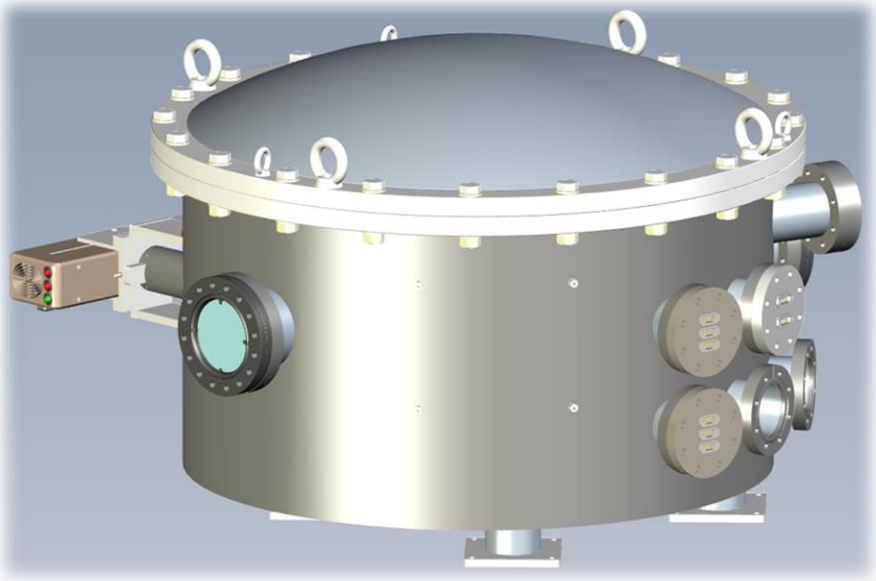
*LABXAS is a **cylindrical vacuum chamber**. The beam travels in **rough vacuum** for most of its path to minimize air absorption. Only the source and the sample are in air.*

Design Specs:

- Stainless steel
- Diameter: 720 mm
- height: 600 mm
- Volume: 240 l
- Weight: 230 kg

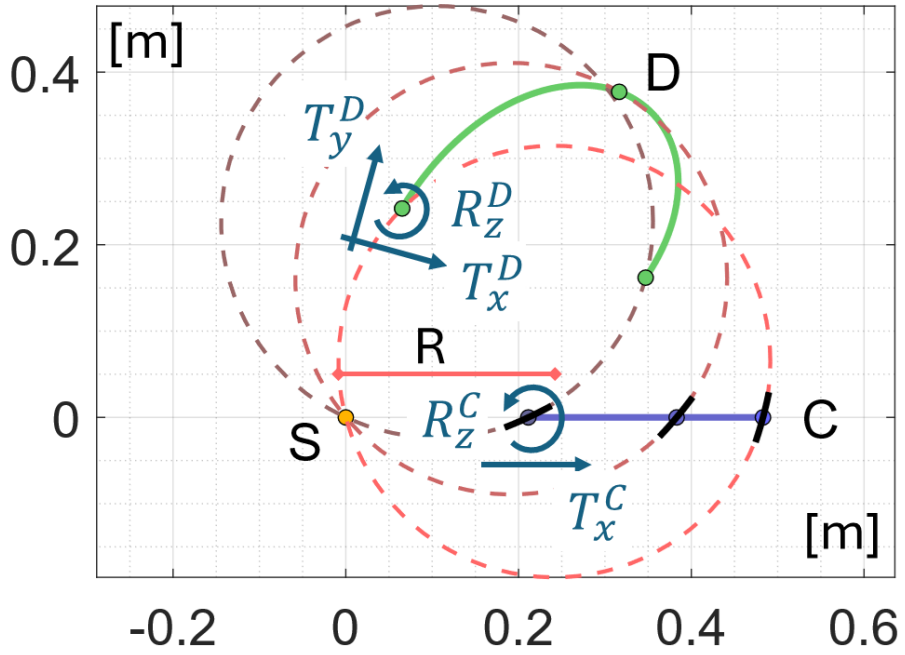
Designed to work:

- In transmission
- In rough vacuum: 10^{-2} mbar



Rowland geometry

*Source (S), crystal analyser (C) and detector (D) stay on a **Rowland circle.** In our setup **S is fixed**.*



- Rowland Radius: **R=0.25m**
- **Fixed source (S)**
- **5 motors* :**
  - Crystal (C): $\boldsymbol{T_x^C}$, $\boldsymbol{R_z^C}$
  - Detector (D): $\boldsymbol{T_x^D}$,$\boldsymbol{T_y^D}$, $\boldsymbol{R_z^D}$

(*) and additional manual movements for adjustments

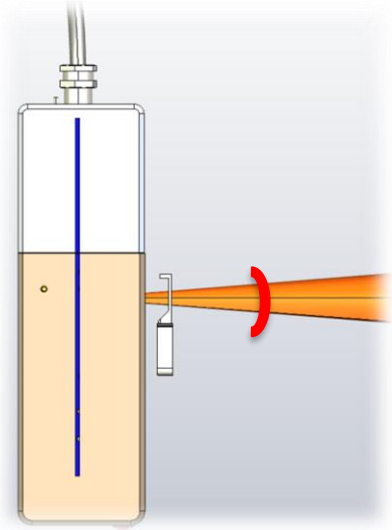
The x-ray source

*We chose a **micro-focus sealed tube** x-ray source. Working in transmission mode with the sample at a few cm from the source, the **beam does not need to be collimated. The source size is 35 μm in the scattering plan.***

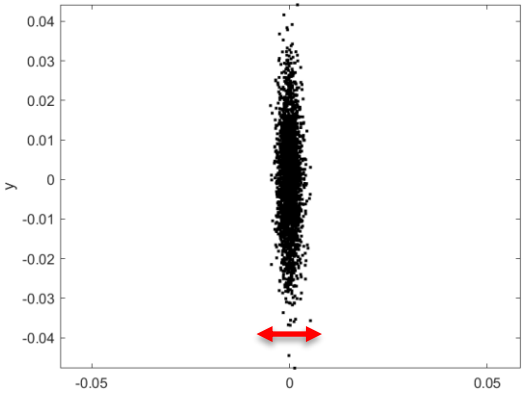
- Bremsstrahlung radiation
- Mo anode
- Air-cooled
- **μ-focus: 35x300 (HxV) μm²**
- **Divergent beam: ~9°**

The **Incoatec IμS 1.0**
(285x90x60 mm³, 3.5kg)

Source divergence : 9°



Source size :
35x300 (HxV) μm²

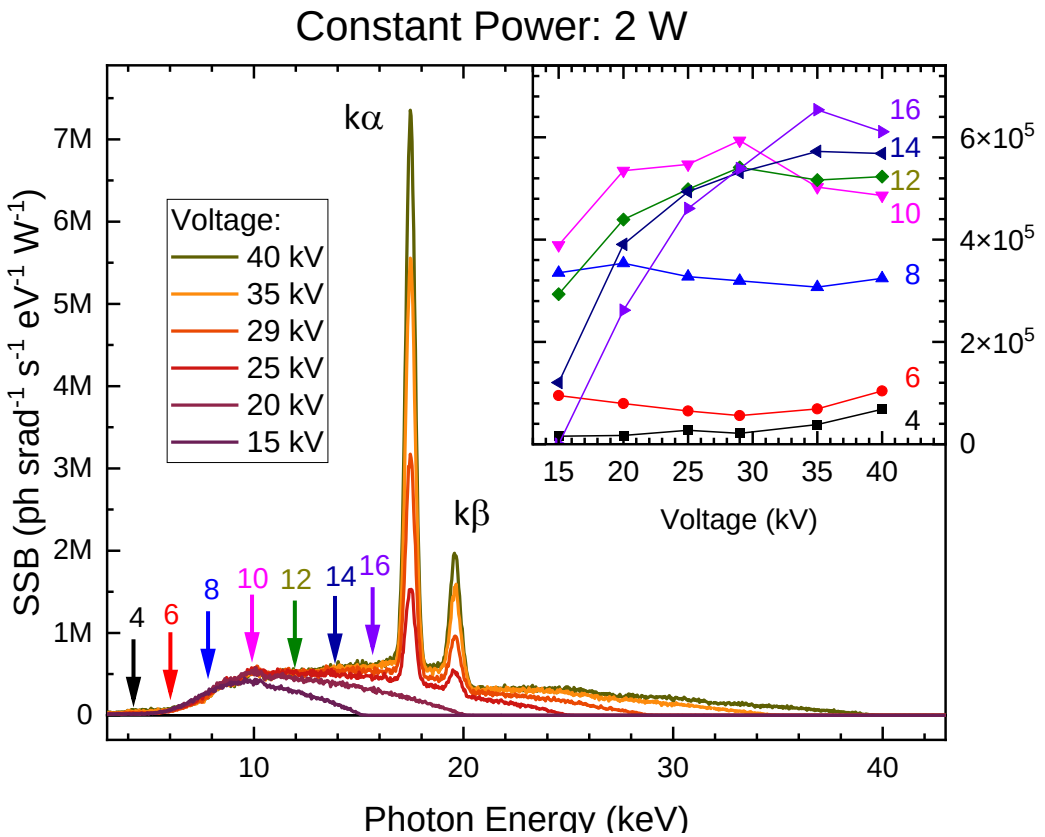


The x-ray source

Low power consumption *and* ***high power density*** *makes the μ-focus source performance comparable to rotating anode. The Intensity* ***spectrum is flat*** *in the TM 3d-K / 5d-L edge region.*

- **Flat emission** for 8-16 keV (25 kV))
- Operating conditions: 650mA, 20-40kV*
- **Low power consumption**: 26W (max)
- **High power density**: 5kW/mm^2

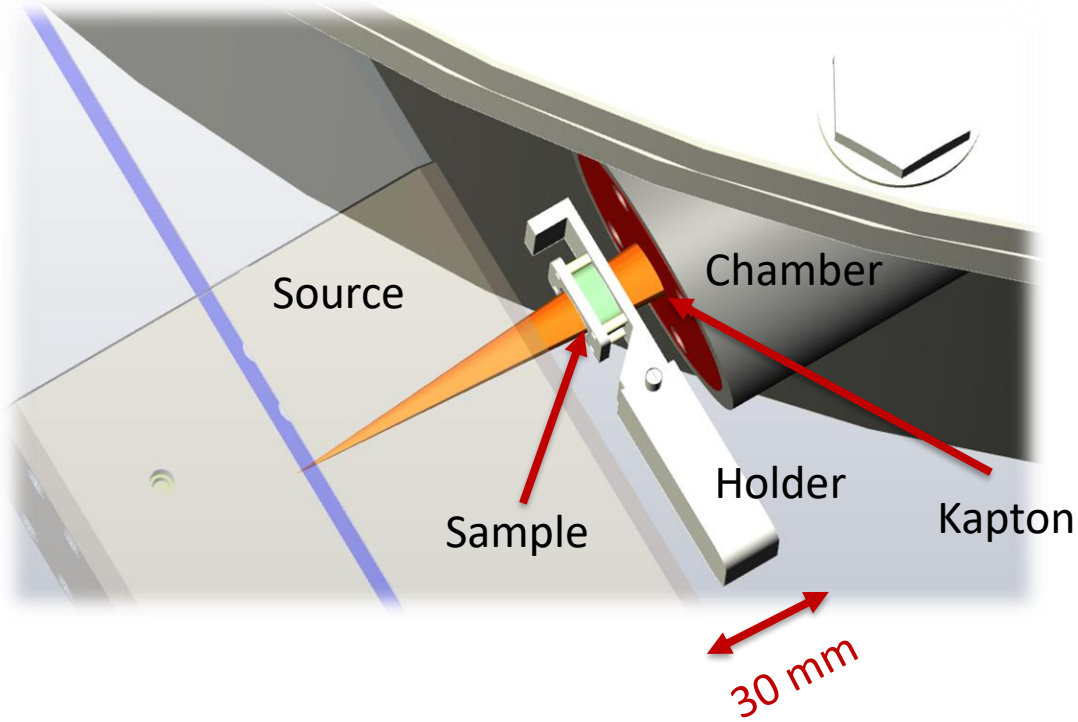
(*) Voltage should be maintained lower to limit the effect of kα and kβ lines



The sample

*The samples are powder pellets to be measured **in transmission**. The **divergent beam** shines the full sample. The beam enters the chamber through a **Kapton window**.*

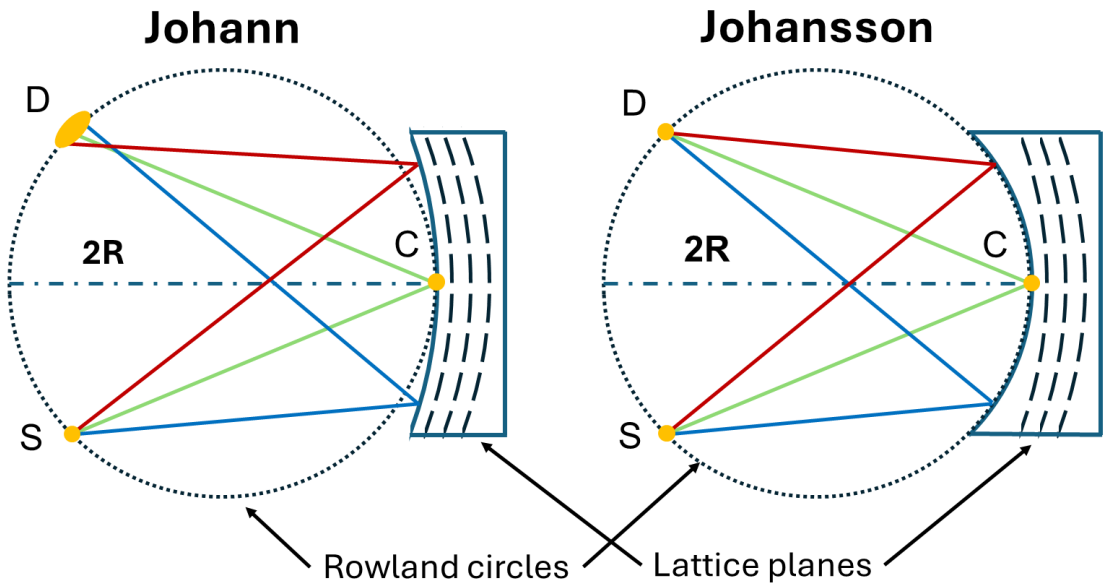
- **Cellulose pellet**
- Disk shaped (D=13 mm, h~1mm)
- Measured **in transmission**
- **In air**
- Single stage holder



The crystal analyzer

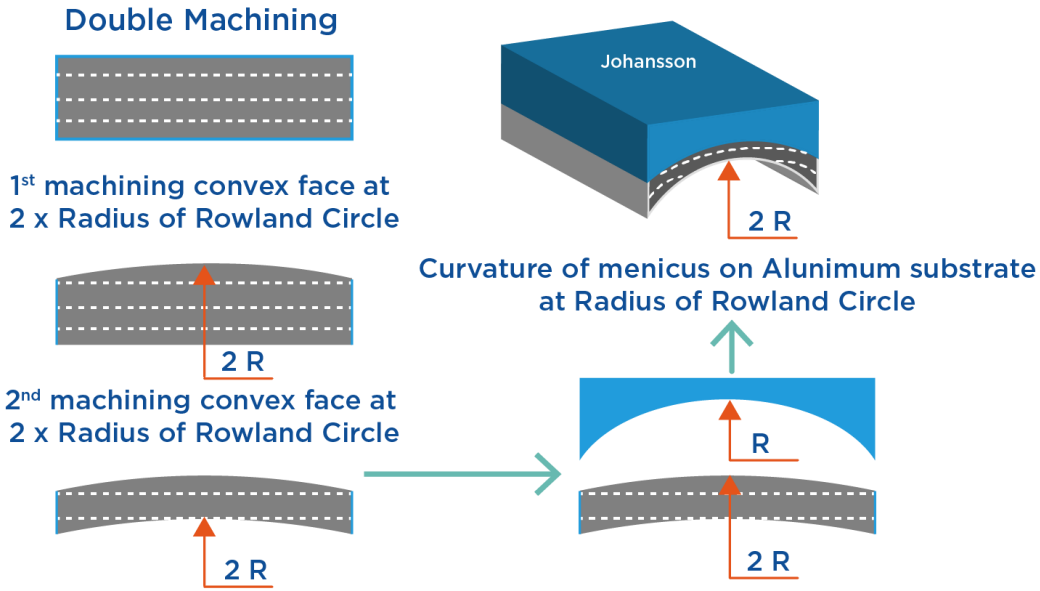
*Johann aberration degrades the focus and the energy resolution. Thanks to a double machinig process the **Johansson** surface lays perfectly on the Rowland circle, allowing for a **perfect focusing**.*

Perfect Focusing



Double Machining process

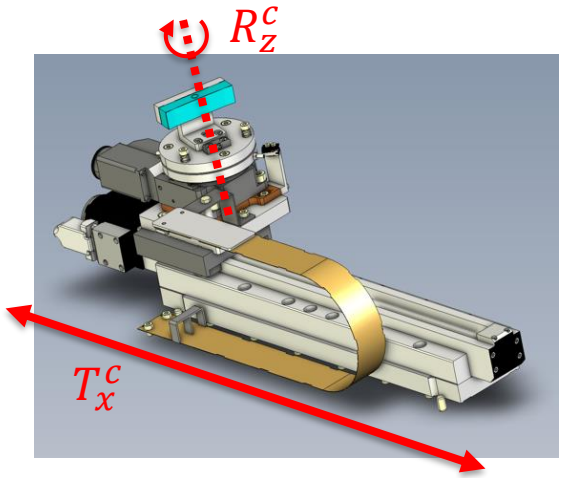
From LuxiumSolutions

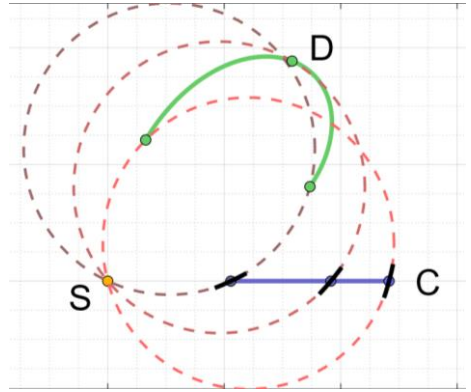


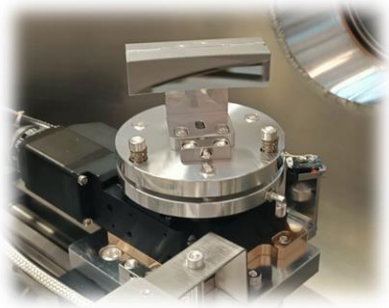
The crystal analyzer

*The transmitted X-ray beam is scanned in energy by a **cylindrical Johansson Ge(nn0)** analyzer and **focused horizontally**.*

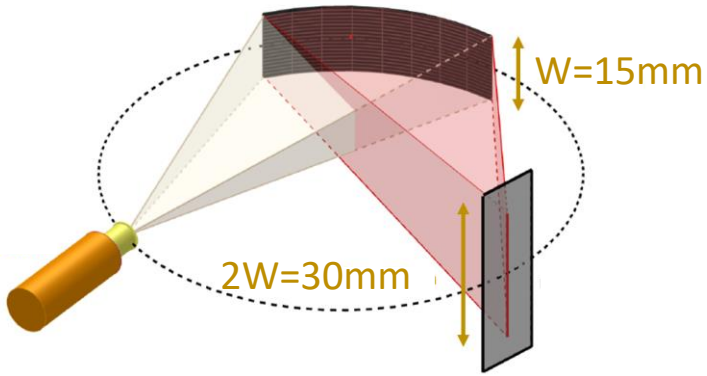
- **Ge(nn0)**
- Size: 60x15 (HxV) mm^2
- 2 motions: T_x^c, R_z^c
- Angular range: 25°-75°
- **Johansson** crystal (R=0.25m)
- **Cylindrically** bent (CBCA)
- **In-plane focusing** (no vertical focusing)





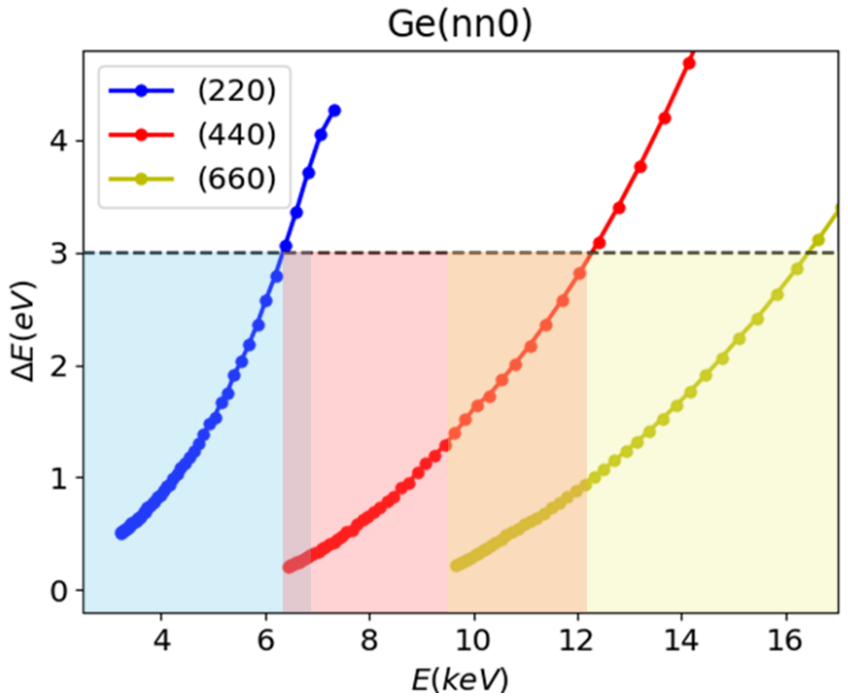


Ge(nn0), Luxium



The crystal analyzer

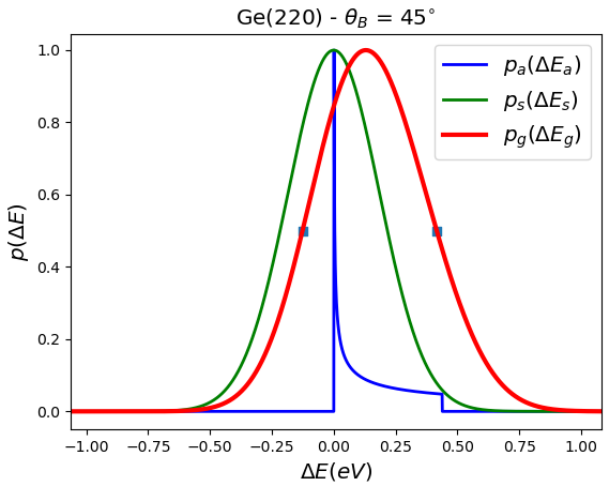
*By exploiting the **n=2,4,6** reflections of the **same Ge(nn0)** crystal, the **whole spectrum** (5-15 keV) can be covered, and the energy resolution maintained **below 3 eV**.*



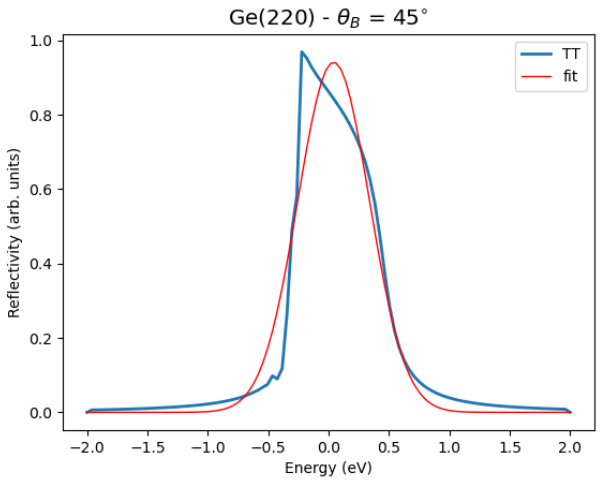
Calculated total resolution for the full bragg angle range (25°-75°)

Contributions to the total resolution:

- Geometric (source and analyzer size)
- Crystal intrinsic (Takagi-Taupin)



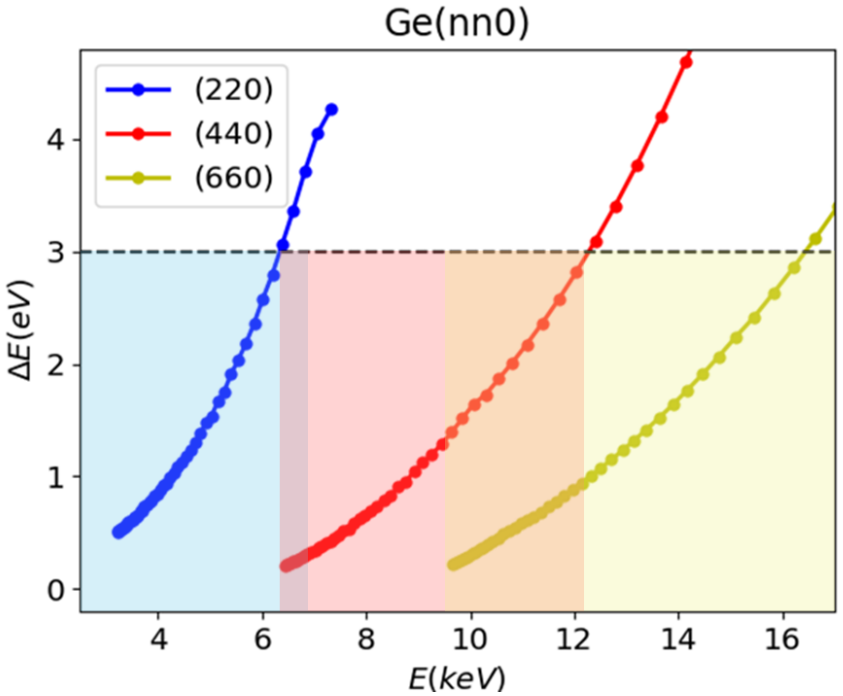
Geometric contribution @ $\boldsymbol{\theta_B = 45°}$ (source+analyzer)



Intrinsic contribution @ $\boldsymbol{\theta_B = 45°}$ (Takaji-Taupin)

The crystal analyzer

*By exploiting the **n=2,4,6** reflections of the **same Ge(nn0)** crystal, the **whole spectrum** (5-15 keV) can be covered, and the energy resolution maintained **below 3 eV**.*



Calculated total resolution for the full bragg angle range (25°-75°)

Source contribution mostly depends on:

- Source in-plane size
- Bragg angle

$$\Delta\theta_s = -\frac{s_y}{R\sin\theta_B} + \tan\theta_B\left(\frac{s_y^2 + s_z^2}{2R^2\sin^2\theta_B}\right)$$

theta	geom	intr	tot
30	2.591	1.105	2.628
45	0.861	0.698	0.861
60	0.409	0.535	0.409
75	0.180	0.485	0.180

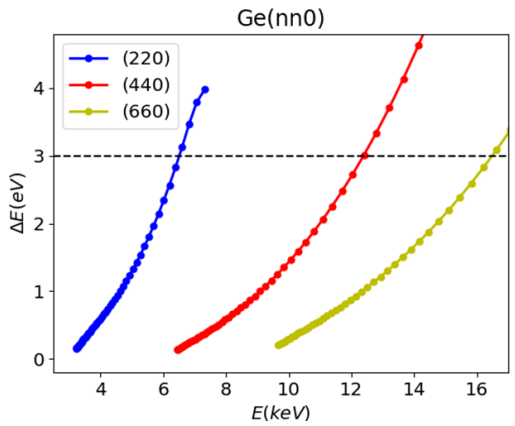
The crystal analyzer

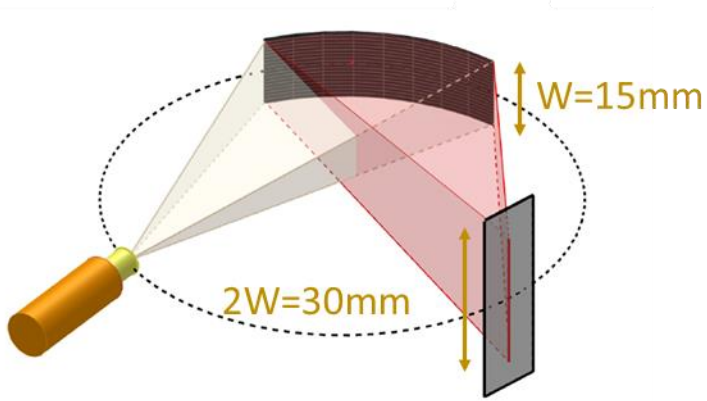
*Using a **single analyzer** influences the **choice of source and detector**, the first due to working mostly at low Bragg angles, the second due focusing only in the scattering plane.*

μ-focus source

Johansson
Cylindrically bent
crystal analyzer
(Js-CBCA)

Large area
detector



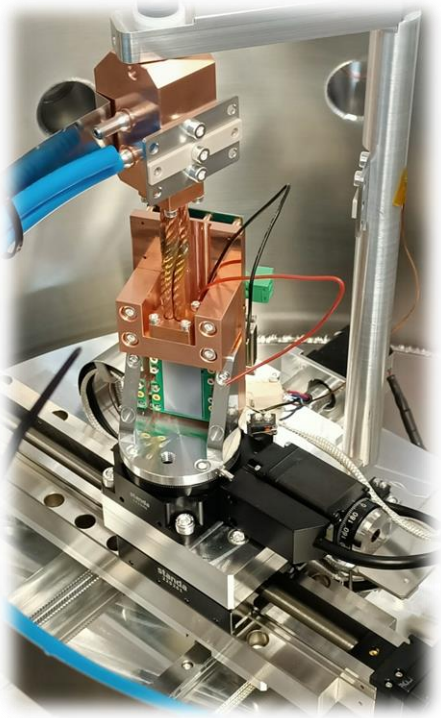


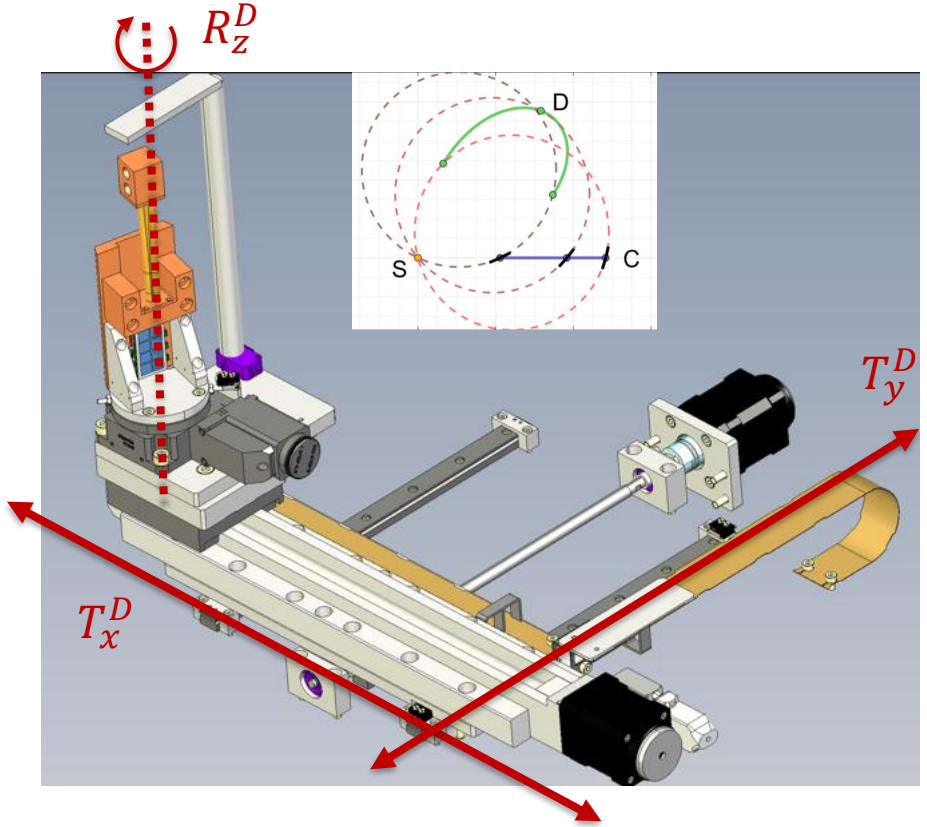
The detector

*The detector was **custom** designed **at Politecnico di Milano (DEIB)**. It is a monolithic **SDD** module, devised with **rectangular** shape and **large active area**.*

- Silicon Drift Detector (**SDD**)
- **2x4** rectangular array : 20x36 (HxV) mm^2
- **Large active area**: **256 mm^2**
- 3 motions: T_x^D ,T_y^D , R_z^D
- Peltier cooled to 5°C *
- High count rate: **10^5 ph/s**

(*) The Cu housing is water cooled to remove heat load from the mechanics.



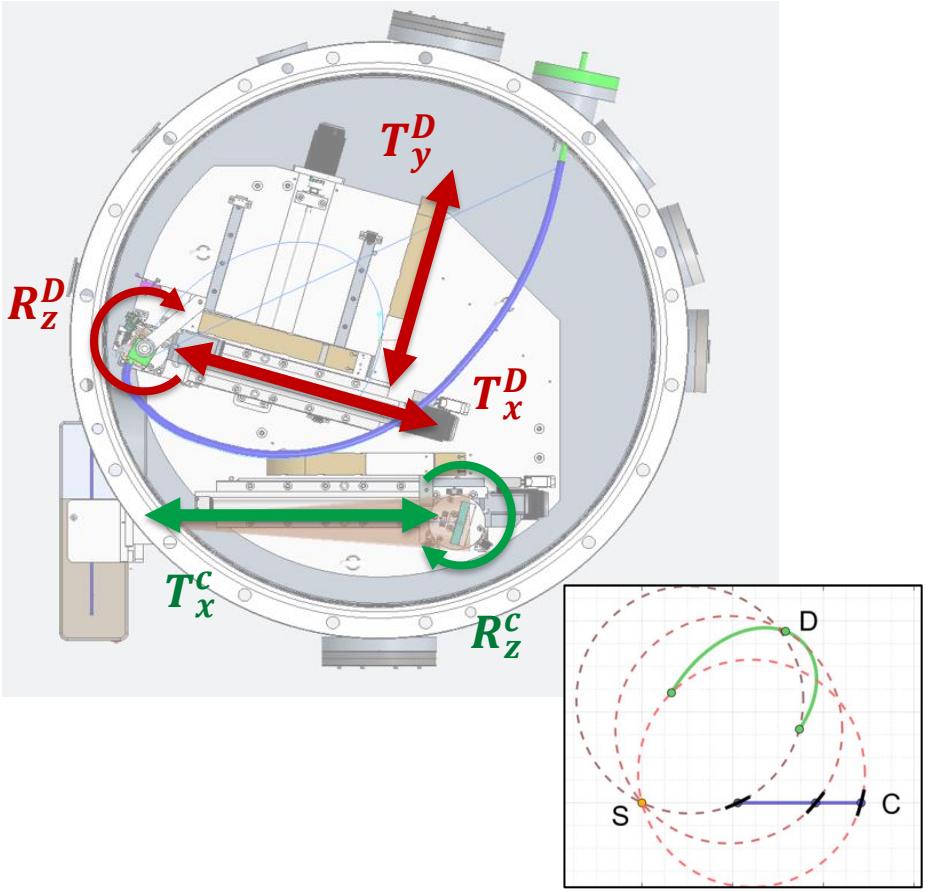


***Quaglia, R.**, et al. (2016). Nuclear Instruments and Methods in Physics Research, Section A, 824(449–451)*

Motors

- **Crystal analyser :**
  - Translation (T_x^C) : 0 – 272 mm, Res: 100 (10) μm
  - Rotation (R_z^C) : 25° - 75°, Res: 0.001 (0.0006) ° (1/16 M.S.)

- **Detector :**
  - Translations (T_x^D,T_y^D) : 0 – 272 mm, Res: 100 (10) μm
  - Rotation (R_z^D) : 30° -130°, Res: 0.1 (0.01) °



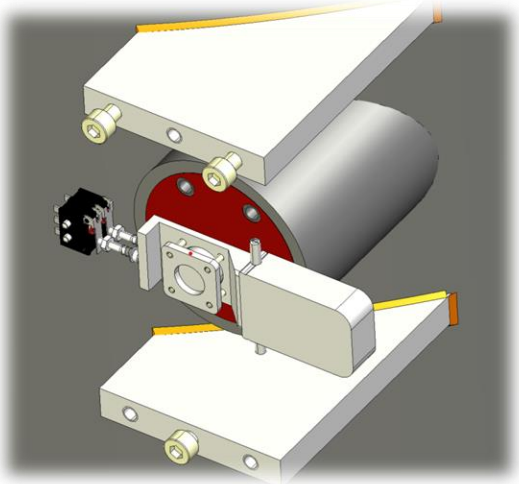
Radioprotection

Radiation shielding :

- Stainless steel chamber walls (thickness: 5 mm)

X-ray source Interlocks :

- Chamber pressure ($p < 10^{-1}$ mbar)
- Inserted sample holder
- x-ray source in place



Outline

- **Introduction**
  - The PolimiX group
  - Motivations and Objectives
  - Design criteria and targets
- **LabXAS**
  - Overview
  - The chamber
  - Rowland geometry
  - The x-ray source
  - The sample
  - The crystal analyzer
  - The detector
  - Motors
  - Radioprotection
- **Current situation**
  - Applications
  - Ongoing works
  - Program
- **Conclusions and Perspectives**

Applications

- **Sample characteristics :**

  - Pure, highly concentrated
  - Homogenous
  - 3d – 5d transition metals

- **Green chemistry and industrial applications :**

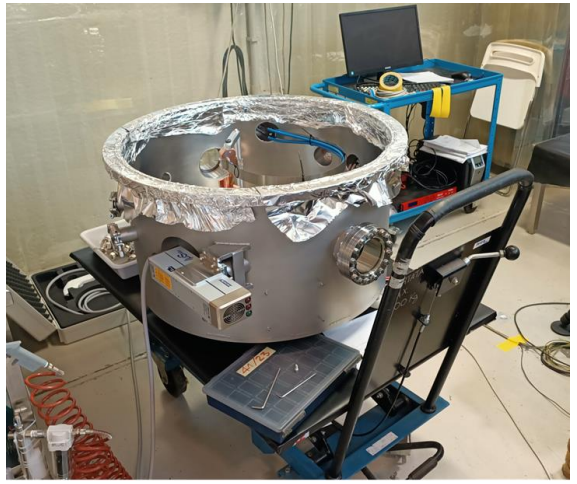
  - Energy: fuel cell and batteries
  - Catalysis: MOFs
  - Industrial chemistry: material recovery in industrial processes

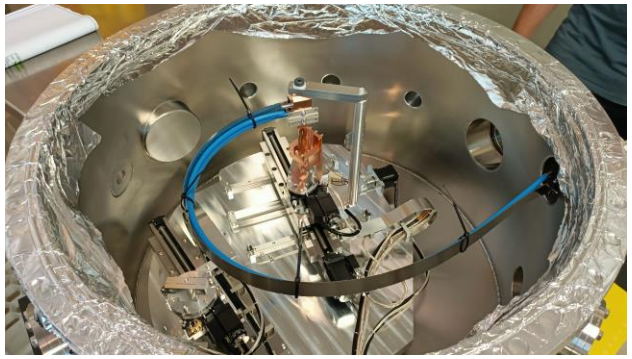
- **Fundamental physics:**

  - valence states and cation coordination

Ongoing works

- **Chamber and mechanics** were tested in July 2024
- The instrument has been **delivered** at the Chemistry Department of the Politecnico di Milano in August 2024
- Soon it will be installed on a custom designed **table with hoist**
- Operation tests are ongoing: **pumping, cooling and motor tests.**





Program

▪ **Ge crystal tests** at the ESRF:	October 2024
▪ Source switch-on and **radioprotection tests**:	November 2024
▪ Spectrometer **commissioning**:	from November 2024
▪ **First measurement** with real samples?	From February 2025 (???)

Conclusions

- A new table-top spectrometer for XANES and EXAFS in-lab measurements will be available at the Politecnico di Milano (hopefully) from Spring 2025
- The design was conceived to minimize costs, size and complexity compared to synchrotron instruments while ensuring minimum performances (10^5 ph/s, < 3eV resolution)
- This is pursued by a combination of choices, e.g.: a single anlyser covering the full energy range, a μ-focus non-collimated x-ray source, a large SDD detector
- Samples candidates are powder compounds containing 3d and 5d transition metals (good homogeneity is required)

Perspectives

- In situ measurements (sample in vacuum)
- Multiple samples in a raw
- Upgrade for X-ray emission spectroscopy (XES)

Acknowledgements

❑ **The PolimiX group**
- Prof. Giacomo Ghiringhelli
- Prof. Marco Moretti
- Dr. Matteo Corti
- Dr. Piero Florio

❑ **Fundings:**
- **MUSA**: Multilayer Urban Sustainability Action, NextGenerationUE, PNRR
- **Regione Lombardia**
- **NEXTGAME projetc**

❑ **Politecnico di Milano – Physics Department**
- Antonio Di Natale
- Dr. Giuseppe Romano
- Dr. Francesco Sangalli

❑ **Politecnico di Milano – Chemistry Department**
- Prof. Piero Macchi
- Prof. Giancarlo Terraneo
- Prof. Pierangelo Metrangolo
- Dr. Daniele Lucchini

❑ **Politecnico di Milano – Electronics Department**
- Prof. Carlo Fiorini
- Dr. Giacomo Borghi
- Dr. Lorenzo Toscano

❑ **Cinel s.r.l.**
- Dr. Riccardo Signorato
- Dr. Roberto Baruzzo
- Dr. Giulio Terracina
- Dr. Giuseppe Lamanna

Thank you!

Thanks for your attention!