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Combined XRR-GIXRF analysis at LNHB: technical and analytical developments

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- The **XRR** ("X-Ray Reflectivity") and **GIXRF** ("Grazing Incidence X-Ray Fluorescence") techniques make it possible to characterize the properties at the interfaces and in depth of materials deposited in very thin layers (thickness, roughness, composition, densities), CASTOR is a goniometer dedicated to these techniques.
- At LNE-LNHB we are developing the combined XRR-GIXRF analysis in a **reference-free** mode using calibrated instrumentation (SDD, photodiodes) and a user-friendly software dedicated to the complete analysis.

CASTOR goniometer

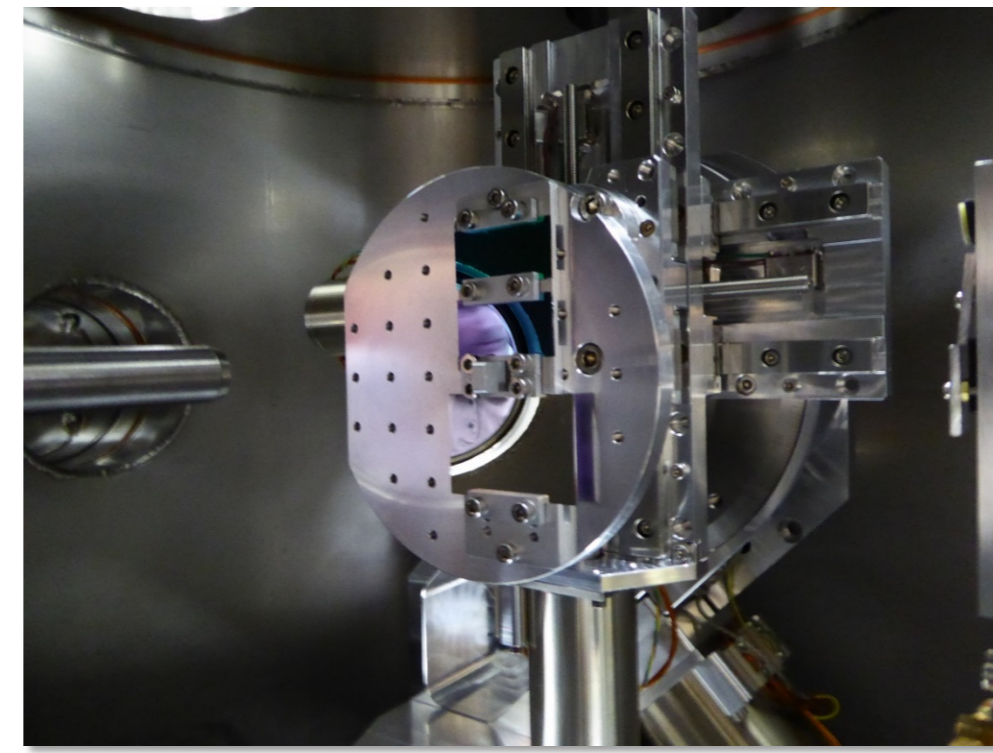
The **METROLOGIE** beamline (bending magnet) at SOLEIL has 2 branches equipped with monochromators:

- from 45 eV to 1,9 keV → "XUV", 3 VLS gratings
- from 3 keV to 35 keV → "XDURS", 2 Si(111) crystals
- Several filters and adjustable slits

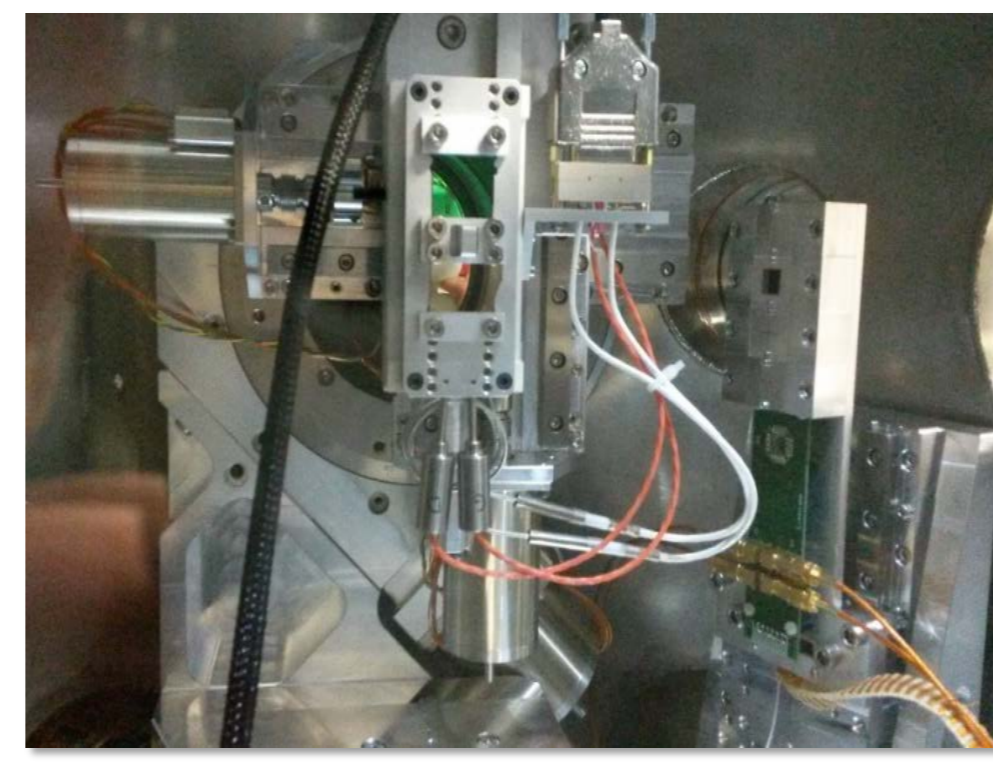
CASTOR ("Chambre d'Analyse Spectrométrique en Transmission Ou en Réflexion") :

- HV chamber (10⁻⁶ hPa)
- 7 axes manipulator (2 rotations and 3 translations for the sample & 1 rotation and 1 translation for the photodiode)
- 3 axes frame
- Up to 4 photodiodes
- Spectrometer (SDD) on a translation stage
- Heating sample holder (up to 300 °C)
- dedicated LabVIEW® acquisition program (variable scanning steps, results in ASCII format)

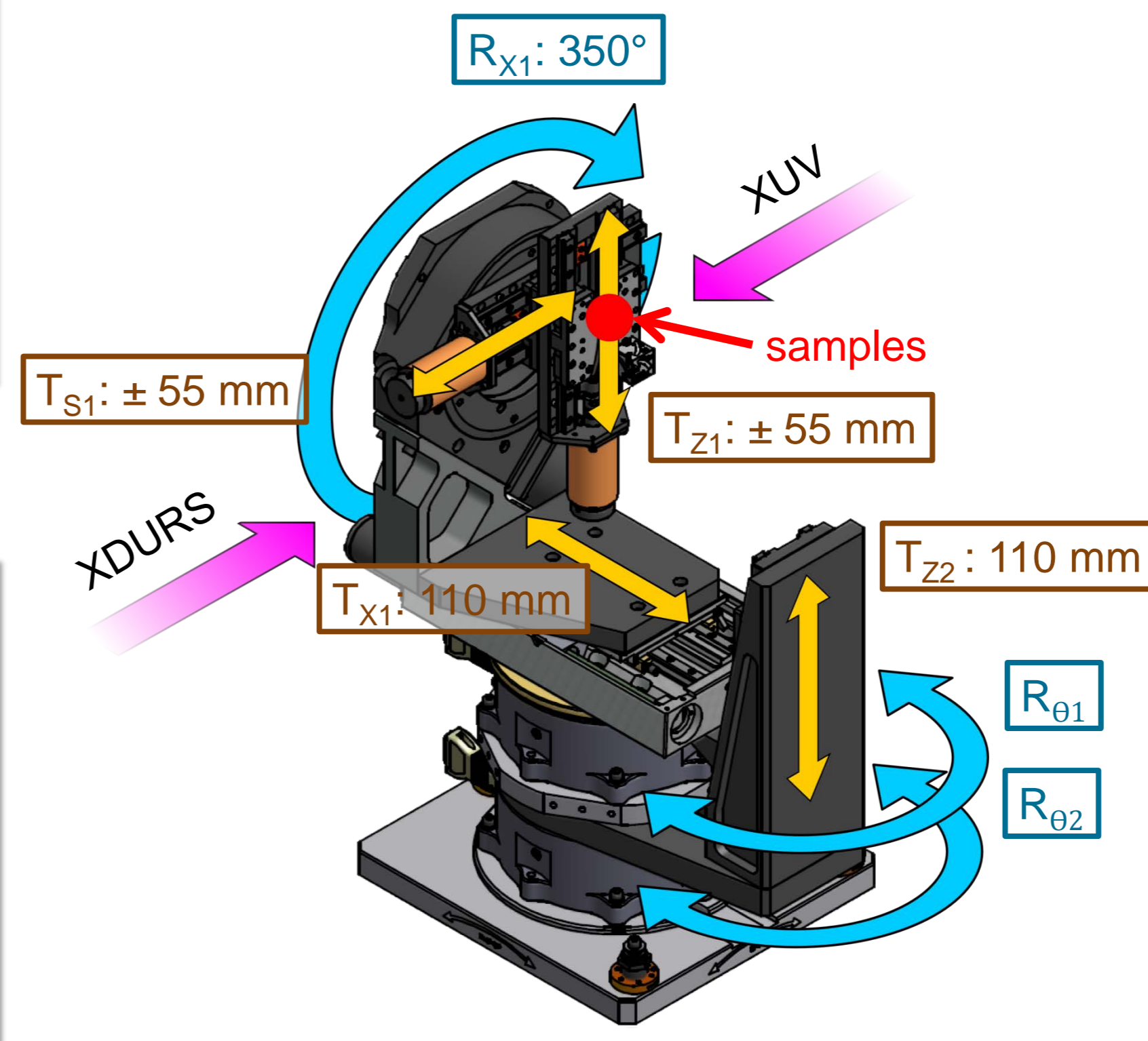
→ Ménesguen, Y. *et al.*, X-Ray Spectrometry, 46, 303-308 (2017)



Sample holder



Heating sample holder

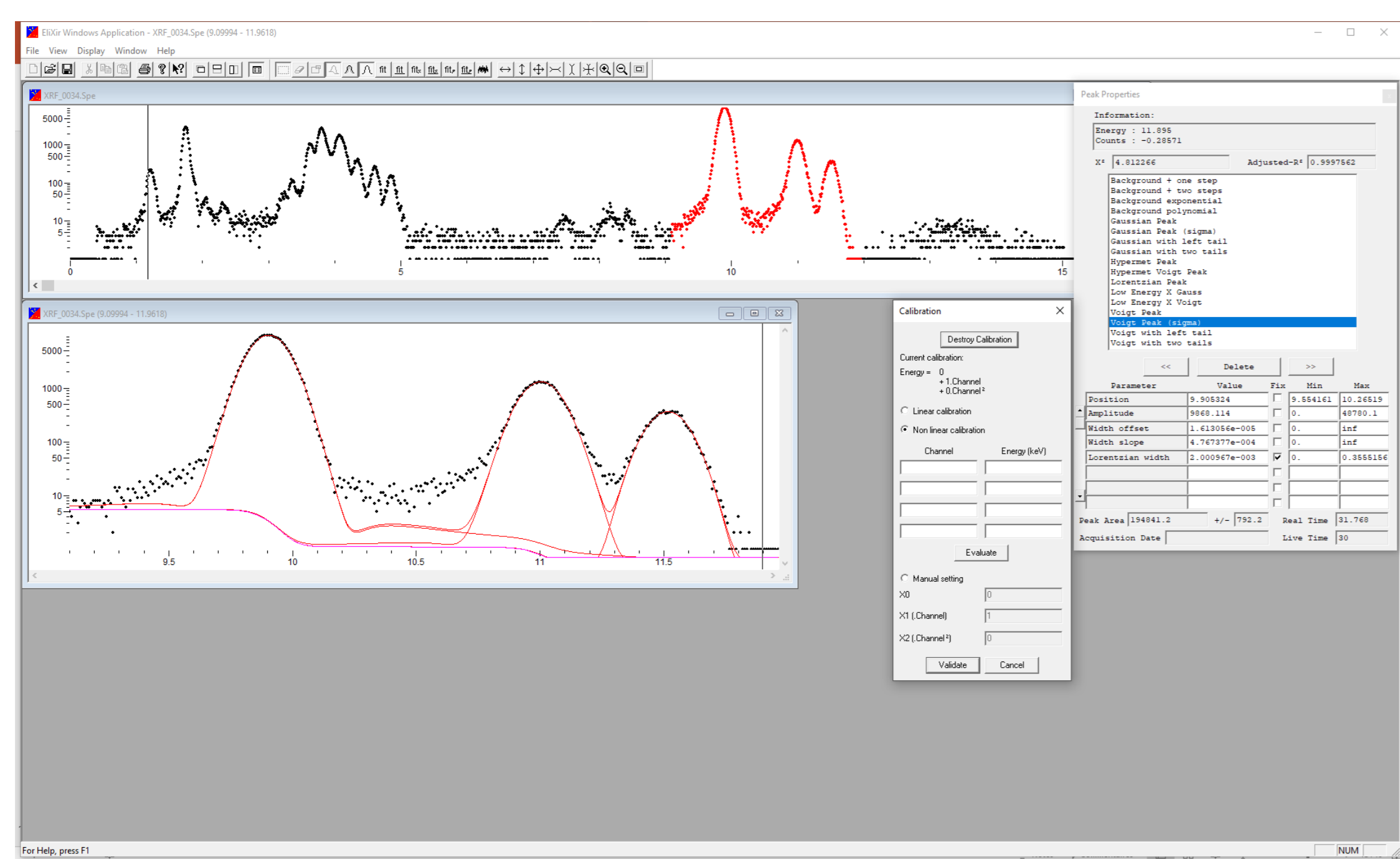


7 axes manipulator



CASTOR on XDURS

Reference-free combined XRR-GIXRF analysis with EliXir

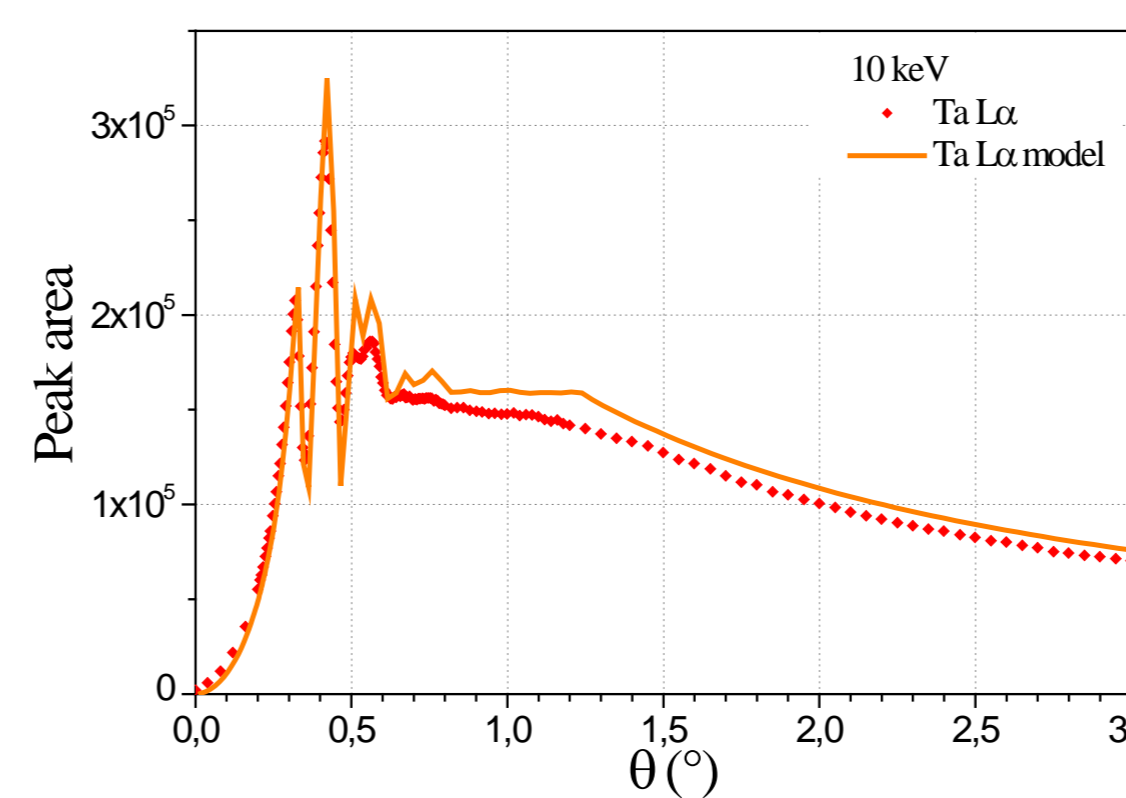
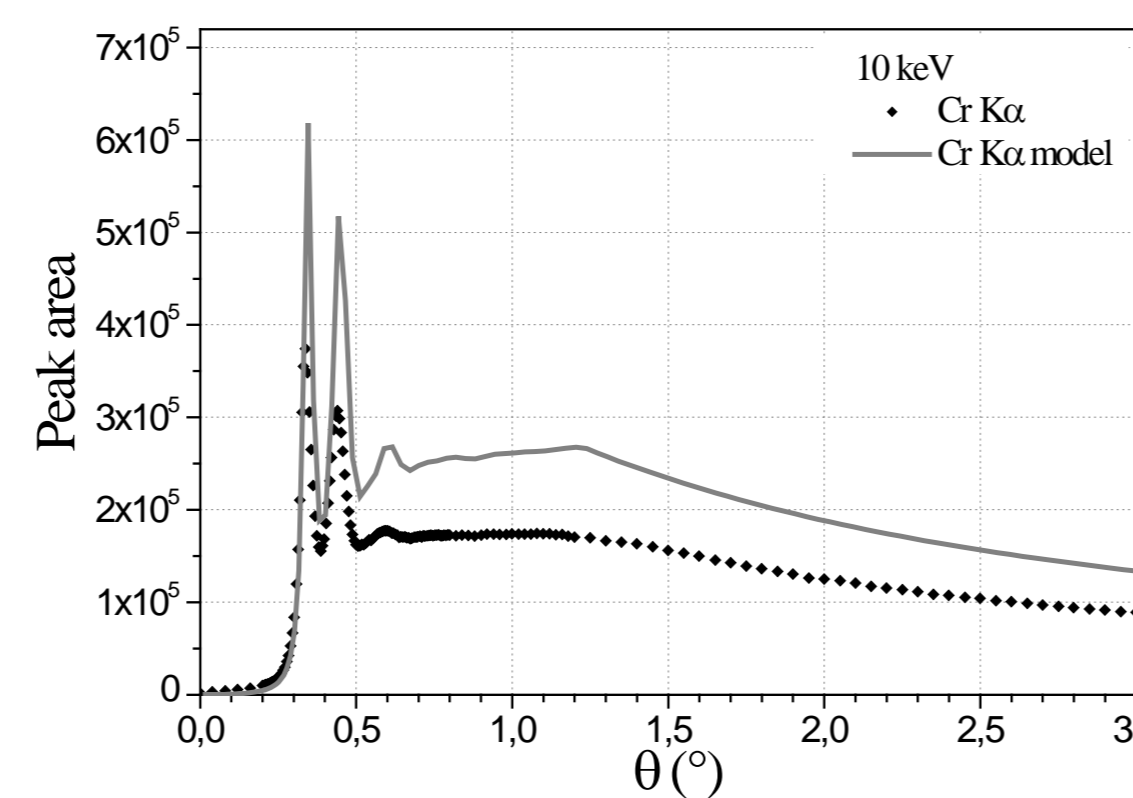
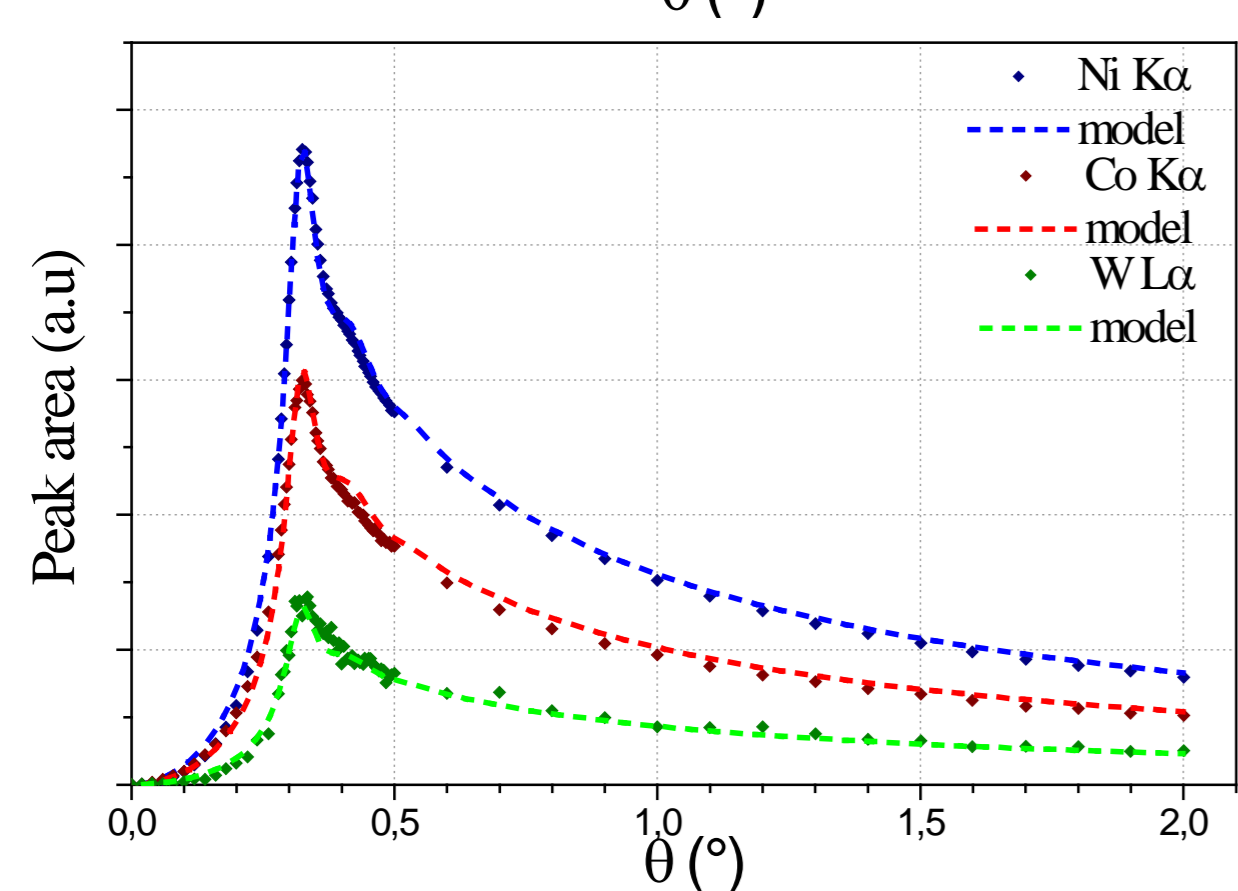
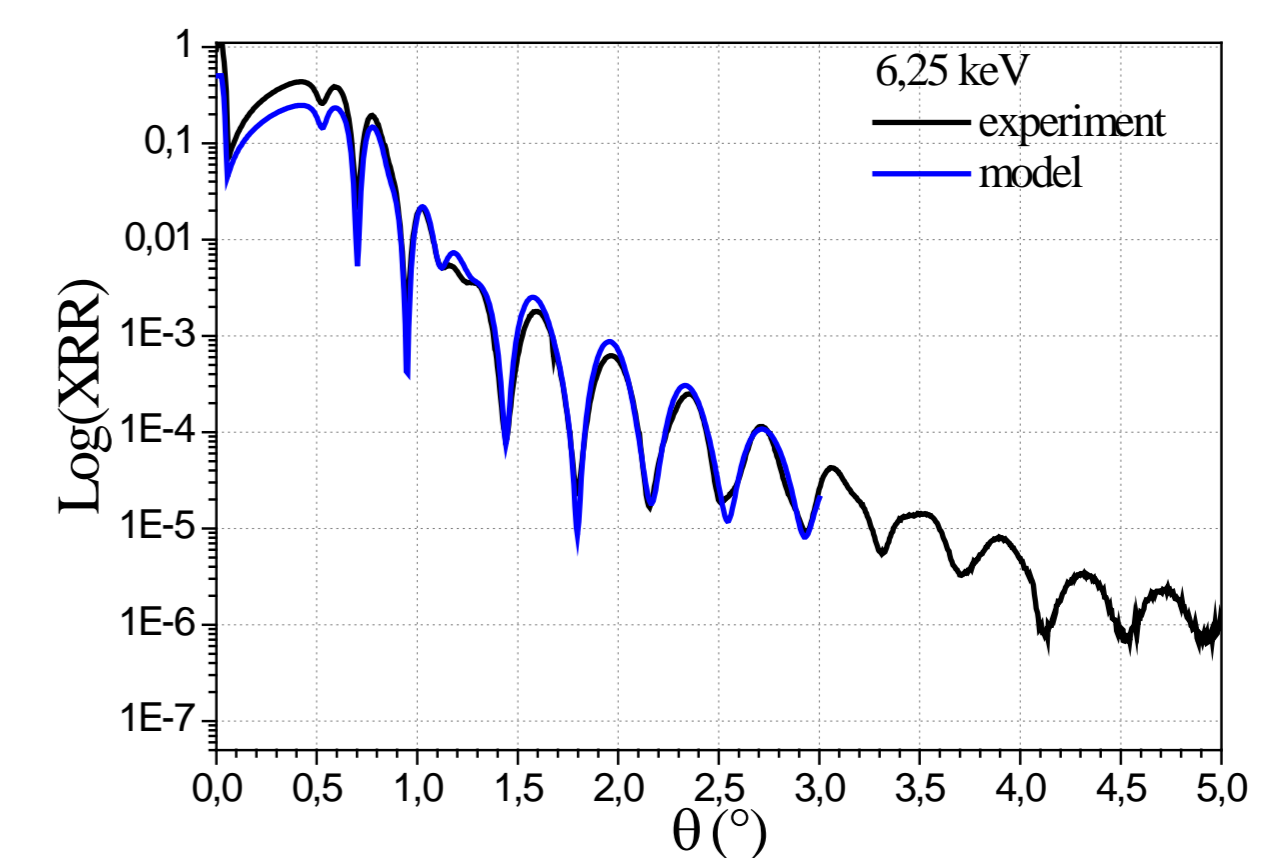
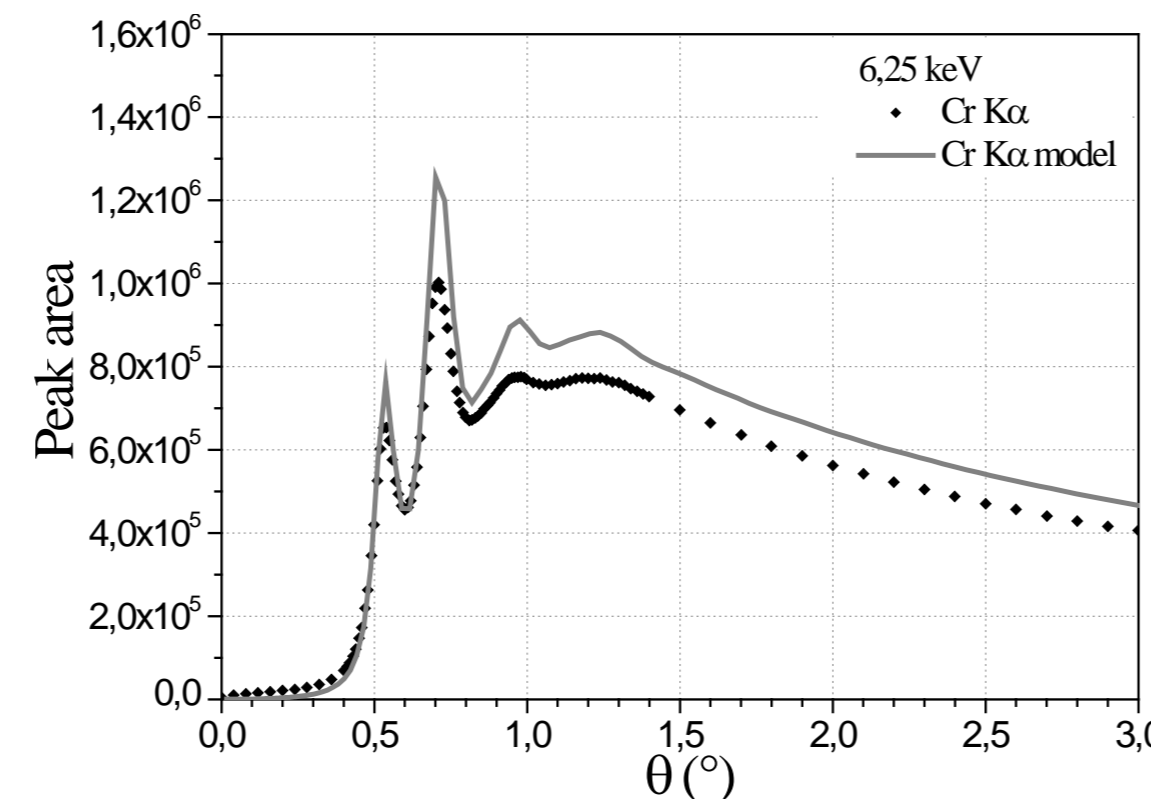
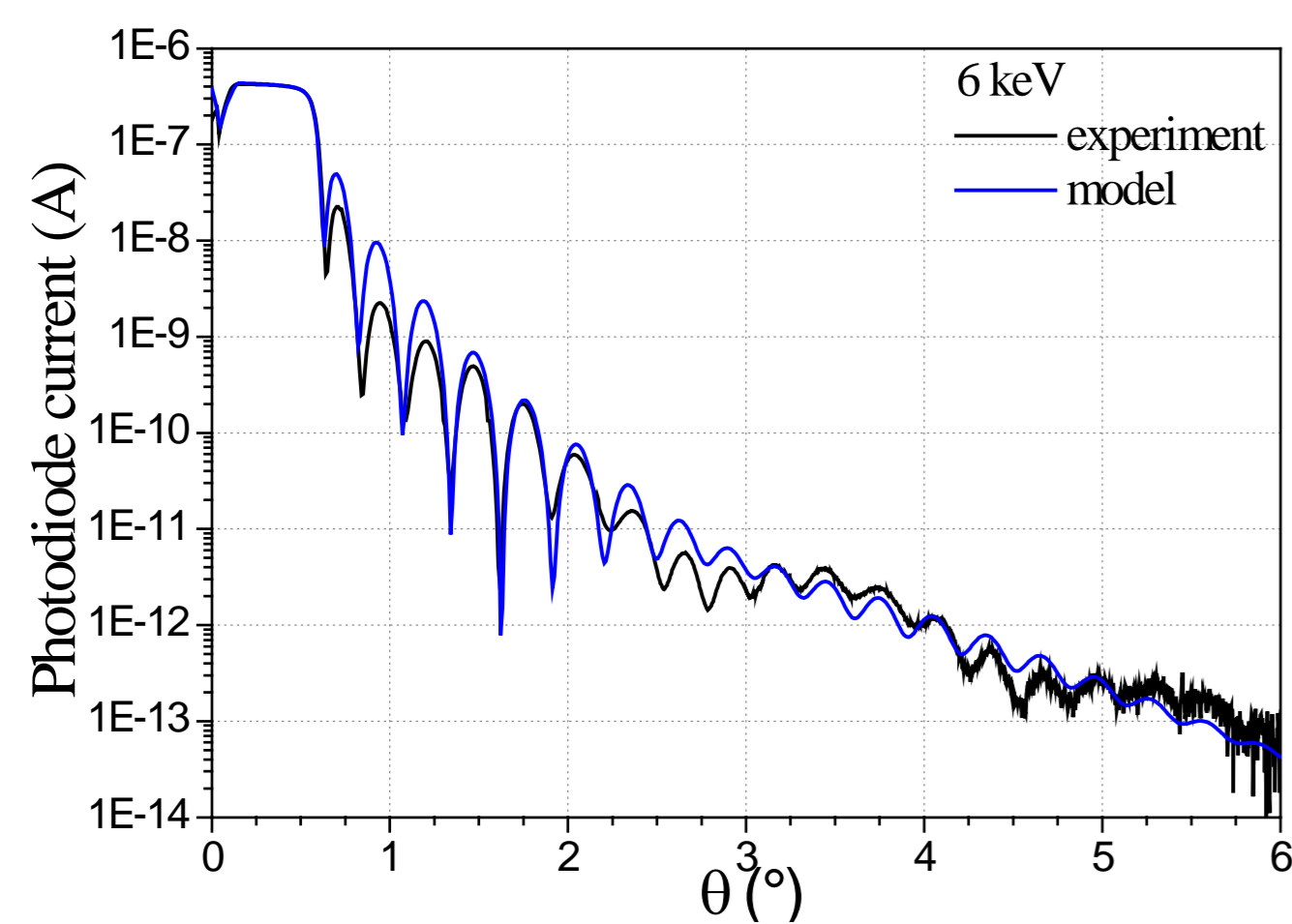


- Same XRF spectra analysis as **COLEGRAM** (Ménesguen, Y. *et al.*, Nuclear Instruments & Methods In Physics Research A, 1003, 165341 (2021))
- Atomic fundamental parameters (μ , τ , ω) (Ménesguen, Y. *et al.*, Metrologia, 53, 7-17 (2016))
- Beam geometry (Ménesguen, Y. *et al.*, Physica Status Solidi A, 2100423 (2021))

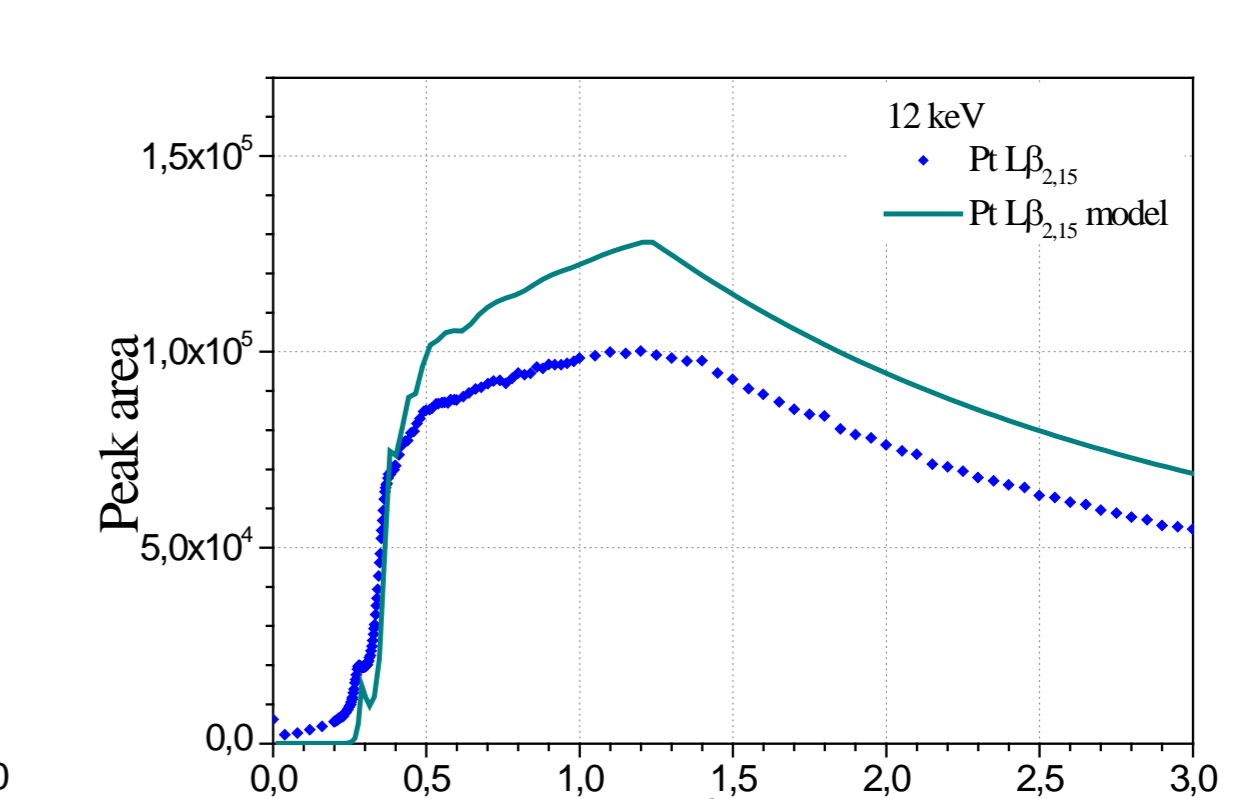
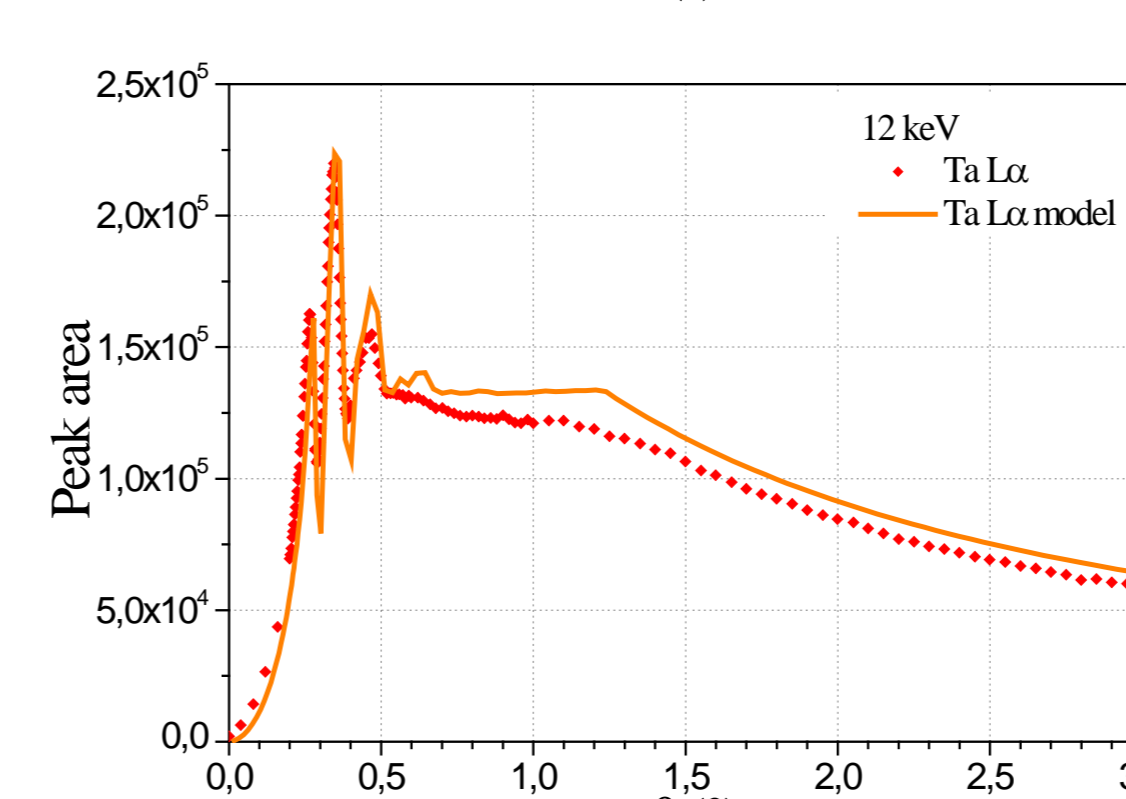
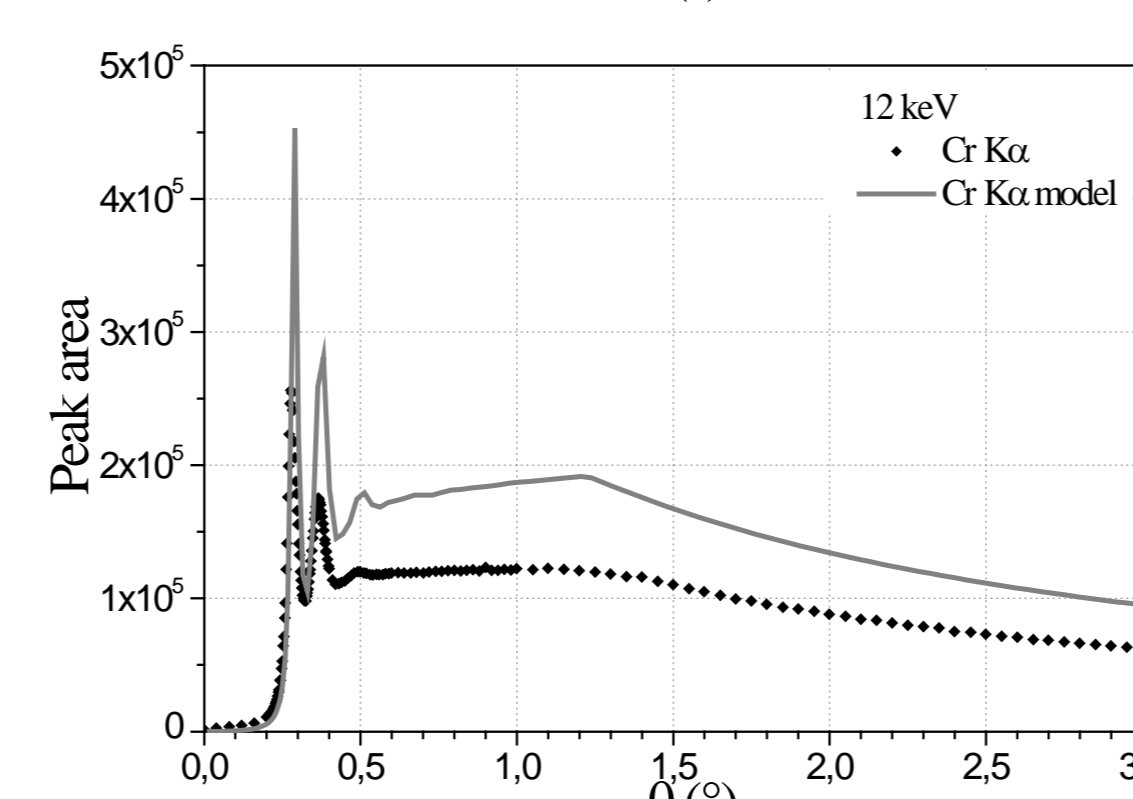
Software interface for multilayer model and optimization parameters. Includes sections for:

- Multilayer model
- Optimization parameters
- Solid angle calculation for the XRF and the XRR
- Layer definition: (thickness, roughness, density, composition)
- Excitation energy (XRR, XRF), acquisition time and flux (XRF)
- Optimization: XRR only, XRF only, both or none
- Parameters constraints
- Elements to compute the GIXRF

Examples of results



Ta ₂ O ₅	d = 1,88 nm	ρ = 6,16 g.cm ⁻³	σ = 1,11 nm
Ta	d = 3,97 nm	ρ = 16,14 g.cm ⁻³	σ = 1,12 nm
Cr	d = 13,65 nm	ρ = 7,00 g.cm ⁻³	σ = 0,94 nm
Pt	d = 13,9 nm	ρ = 21,5 g.cm ⁻³	σ = 0,53 nm
PtSi	d = 0,19 nm	ρ = 18,95 g.cm ⁻³	σ = 0,75 nm
Si Substrate			



NiCo _{0,11} W _{0,01}	d = 1,02 nm	ρ = 5,76 g.cm ⁻³	σ = 0,53 nm
NiCo _{0,11} W _{0,01}	d = 17,52 nm	ρ = 8,74 g.cm ⁻³	σ = 0,47 nm
NiCo _{0,11} W _{0,01}	d = 1,15 nm	ρ = 8,12 g.cm ⁻³	σ = 0,47 nm
SiO ₂	d = 1,05 nm	ρ = 2,65 g.cm ⁻³	σ = 0,26 nm
Si Substrate			