FROM RESEARCH TO INDUSTRY

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COMPARISON OF FOUR DATA ANALYSIS SOFTWARE FOR COMBINED X-RAY REFLECTIVITY AND GRAZING INCIDENCE X-RAY FLUORESCENCE MEASUREMENTS

Bérenger Caby (1), Fabio Brigidi (2), Dieter Ingerle (3), Blanka Detlefs (1), Gaël Picot (1), Luca Lutterotti (2), Emmanuel Nolot (1), Giancarlo Pepponi (2), Christina Streli (3), Magali Morales (4), Daniel Chateigner (5)

(1) CEA, LETI, MINATEC Campus, Grenoble, France
(2) Fondazione Bruno Kessler, Trento, Italy
(3) Atominstitut, Vienna University of Technology, Vienna , Austria
(4) CIMAP , Caen , France
(5) CRISMAT-Ensicaen, IUT-Caen UCBN, Caen, France

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OUTLINE

- GiXRF – XRR combined analysis
- Comparison of 4 data analysis software
  - GIMPY, JGIXA, MAUD, MEDEPY
  - Main features
  - Key differences
    - Material Database
    - Sample definition
    - Instrumental function
  - XRR simulation
  - GiXRF simulation
  - Fitting capabilities
- Summary and outlook

NON-DESTRUCTIVE ELEMENTAL DEPTH PROFILING WITH X-RAYS

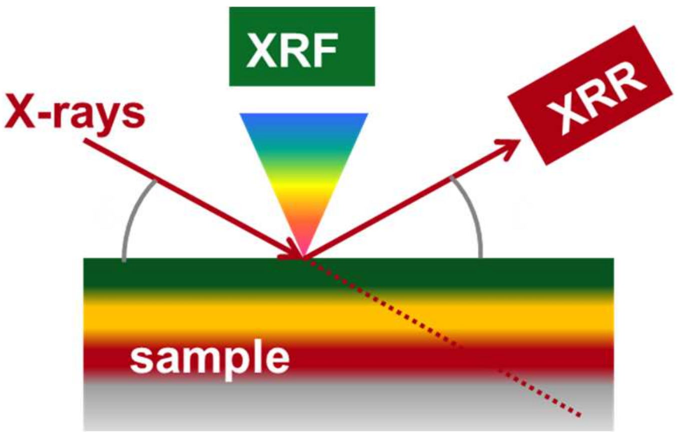
- Analysis of (ultra)thin layered films for advanced applications (micro/nano electronics, memory, photonics, PV, …)

- **Analytical challenges**
  - Reduced material quantities ⇨ limits of detection
  - Material properties different from bulk ⇨ non-existent standards
  - Analysis of interfaces and buried layers ⇨ destructive or indirect methods
  - Accuracy, standardization

- **Need for non-destructive depth-profiling method**
  - Avoid artifacts (preferential sputtering, atom mixing, implantation)
  - Limited (if any) degradation of the sample
  - On beamlines, in the Labs, … in R&D cleanrooms, in industry

- **Combined GIXRF/XRR ?**

GIXRF+XRR ANALYSIS



- XRR : FT(electron density), $\theta \geq \theta_c$
- GiXRF: FT(atomic density), $\theta \leq \theta_c$
- Combined XRR-GiXRF: depth-dependent characterization

GIXRF+XRR DEPTH PROFILING

$$I_x(\theta,\alpha,E_0) = I_0\, G(\theta,\alpha,E_0) \sum_{j=1}^{n} S_{x,E_0} \exp\left[-\sum_{n=1}^{j-1} (\mu/\rho)_{n,E}\, \rho_n d_n\right] \int_0^{d_j} C_{j,x}(z)\, A_j^{XSW}(E_x,\theta,z) \exp\left[-(\mu/\rho)_{j,E}\, \rho_j z\right] dz$$

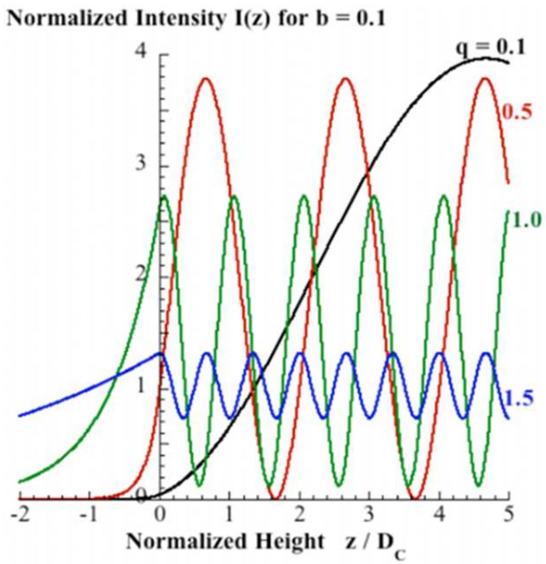
Propagation of GiXRF-XRR requires :

⇨ X-ray dedicated tools

⇨ Optimized protocols

⇨ **Data reduction software**

- Fundamental parameters (cross sections, absorption coefficients), densities, XSW enhancement
- Thicknesses of layers to fit
- Quantification of the XRF dose (geometrical factors)
- Same model for XRR and GiXRF : increase the level of information. Add constraints & reduce uncertainties



ANALYSIS SOFTWARE

SOFTWARE	AUTHORS	KEY FEATURES	REFERENCES
GIMPY *Grazing Incidence Material analysis with Python*	G. Pepponi, F. Brigidi	XRR, XRF, GiXRF *Integrated intensities*	• **TXRF'15 : Frid. 10.10 am**
JGIXA	D. Ingerle	XRR, GiXRF *Integrated intensities*	• Spectrochimica Acta Part B 99 (2014) 121–128 • **TXRF'15 : Wed. 3.30 pm**
MAUD *Material Analysis Using Diffraction*	L. Lutterotti	XRR, XRF, GiXRF, XRD ***Full spectrum***	• Nuclear Inst. and Methods in Physics Research, B, 268, 334-340, 2010 • http://maud.radiographema.com/
MEDEPY *Material Elemental DEpth profiling using PYthon*	B. Detlefs, G. Picot, E. Nolot, H. Rotella …	XRR, GIXRF, XSW *Integrated intensities*	• **TXRF'15 : Frid. 9.30 am**

OVERVIEW

- **Common points**
  - XRR based on Parrat formalism (L. G. Parratt, Phys. Rev., vol. 95, no. 2, p.359, 1954)
  - GiXRF based on De Boer formalism (D. K. G. de Boer, http://dx.doi.org/10.1103/PhysRevB.44.498)

- **Key differences**
  - XRF : full spectrum *vs* integrated intensity
    - *Additional SW (e.g PyMCA) is required to extract the integrated XRF intensities for each angle / each XRF line*
  - Material database
  - Sample definition
  - Instrumental function
  - Other features (simulation & fitting modules)

MATERIAL DATABASE

The values of parameters such as:

Fluorescence yield, Atomic scattering factors, Photoelectric, elastic and inelastic scattering cross sections …

may not be constant over publications / material database

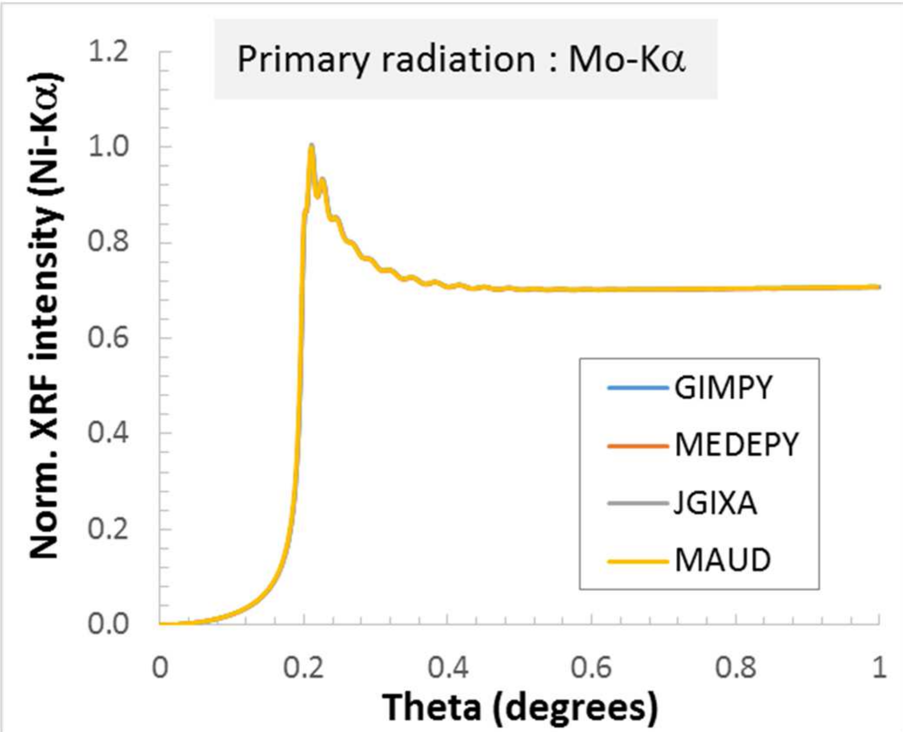
SOFTWARE	MATERIAL DATABASE
GIMPY, JGIXA, MAUD	• https://data-minalab.fbk.eu/txrf/xraydata/element/
MEDEPY	• User defined • Xray Lib (http://ftp.esrf.eu/pub/scisoft/xraylib/readme.html)

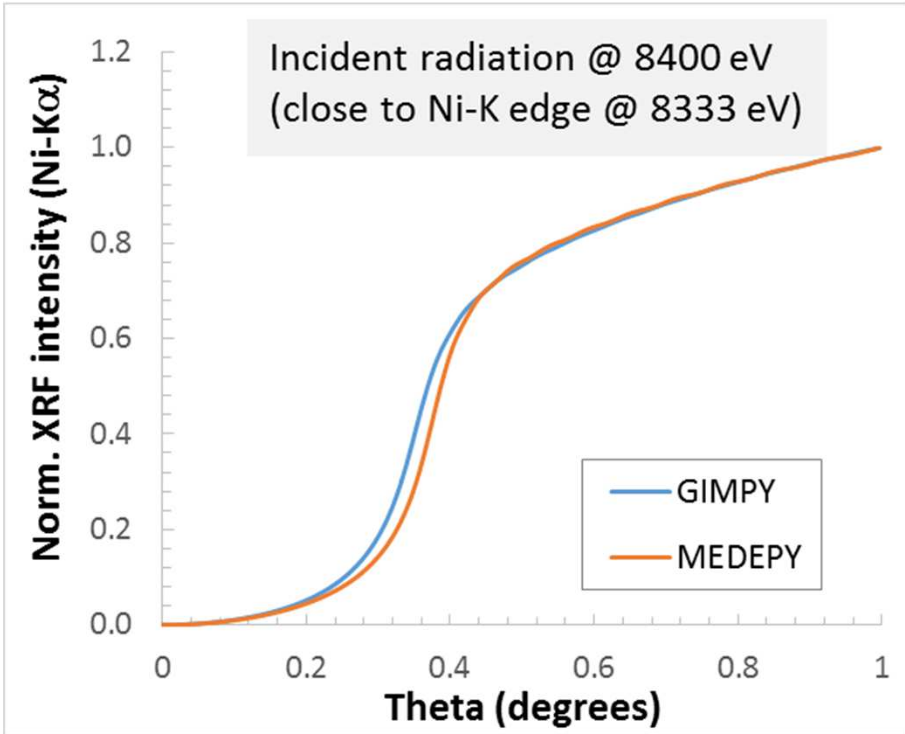
MATERIAL DATABASE

NiO_2 (5nm, d=6.0g/cc)

Ni (50 nm, d=8.9 g/cc)

Si (sub, d=2.33 g/cc)





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SAMPLE DEFINITION

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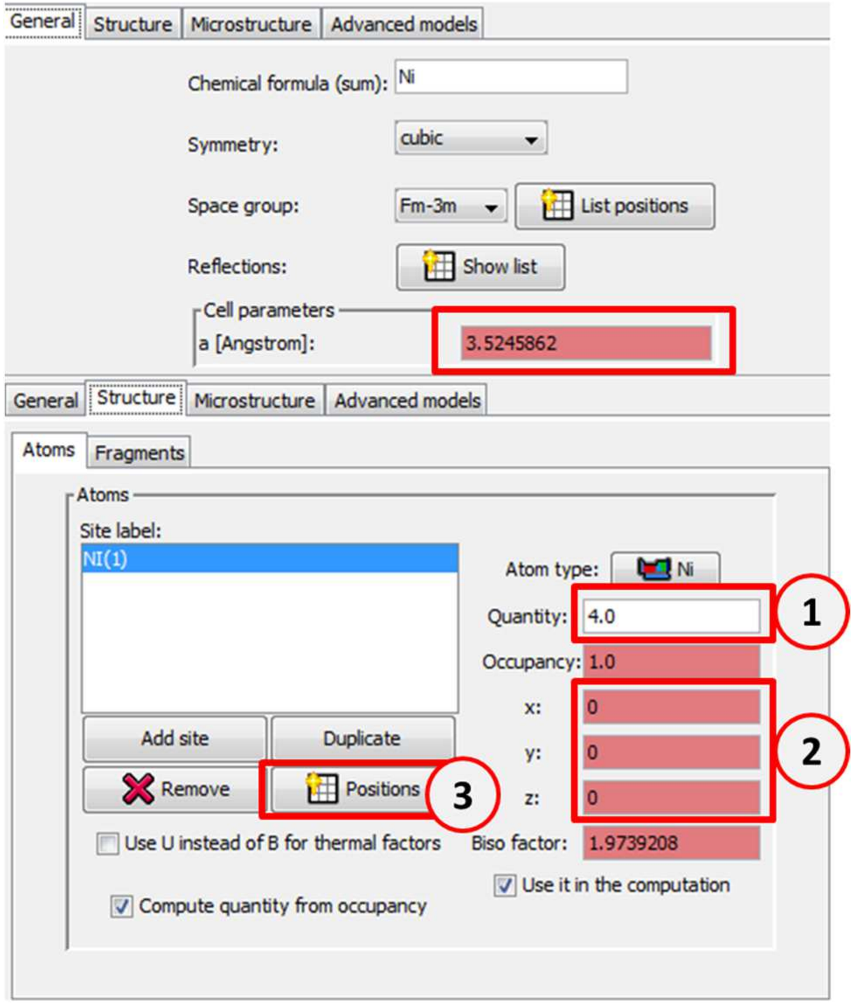
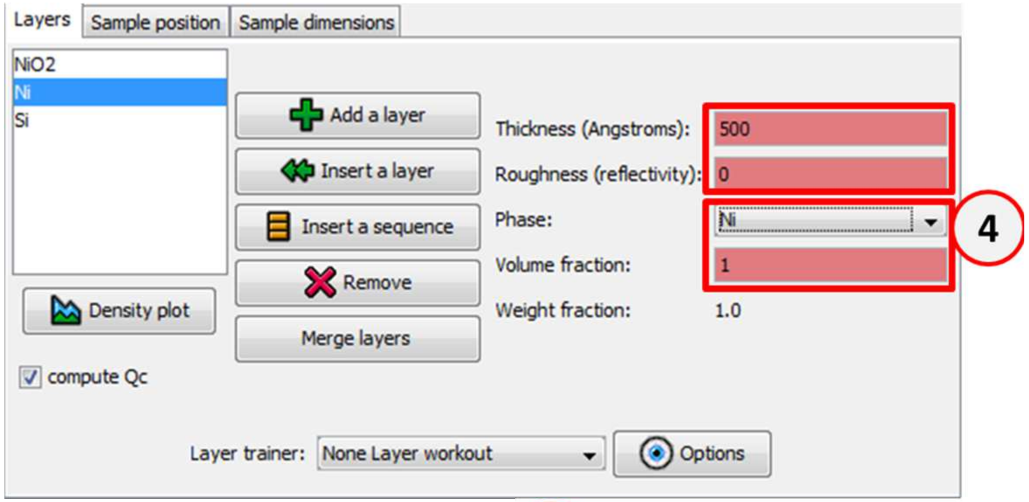
SOFTWARE	PARAMETERS	REMARKS
GIMPY, JGIXA	Thickness Roughness Mass density Stoichiometry	• No correlation between mass density and stoichiometry
MAUD	Thickness Roughness Phase Stoichiometry	• XRD-based definition of the sample structure • Compatible with XRR-GiXRF-XRD combined analysis
MEDEPY	Thickness Roughness Mass density or atomic density Stoichiometry	• Mass density and stoichiometry are correlated • GENX-based definition

SAMPLE DEFINITION (MAUD)





4 Density / Stoechio described by phase

1 Atoms per cell

2 Position of the first atom

3 Position of all the atoms

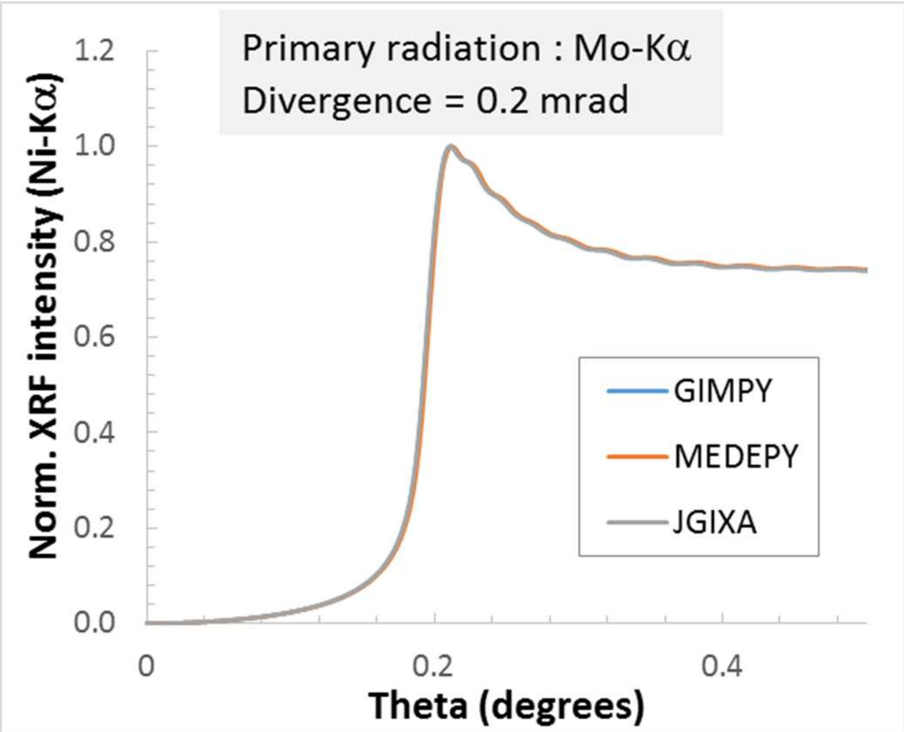
INSTRUMENTAL FUNCTION

- **XRR**
  - Divergence ~ overall resolution

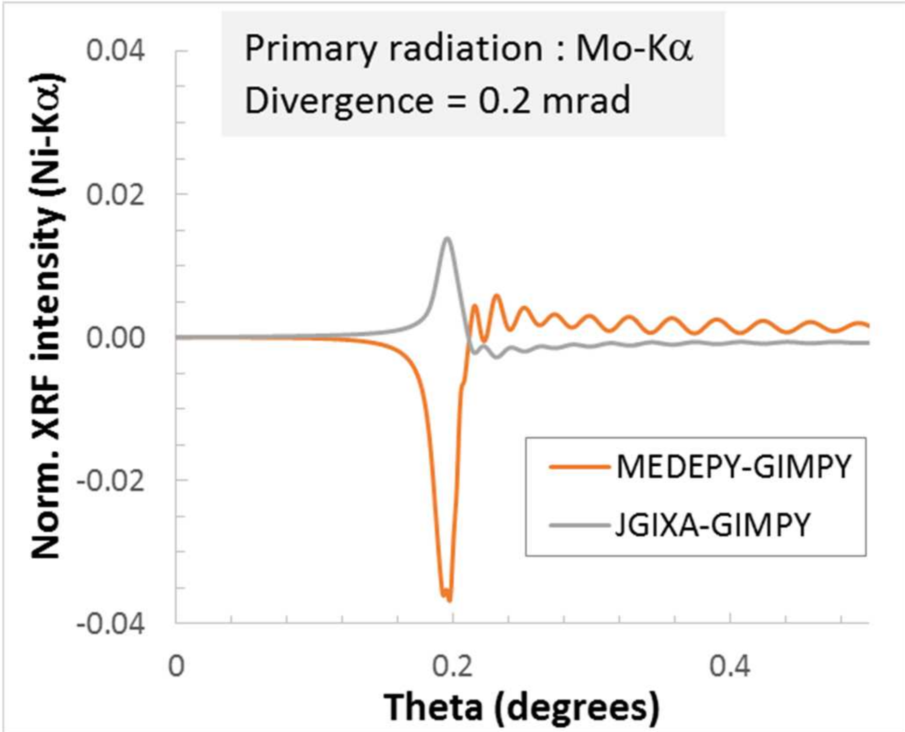
NiO_2 (5nm, d=6.0g/cc)

Ni (50 nm, d=8.9 g/cc)

Si (sub, d=2.33 g/cc)

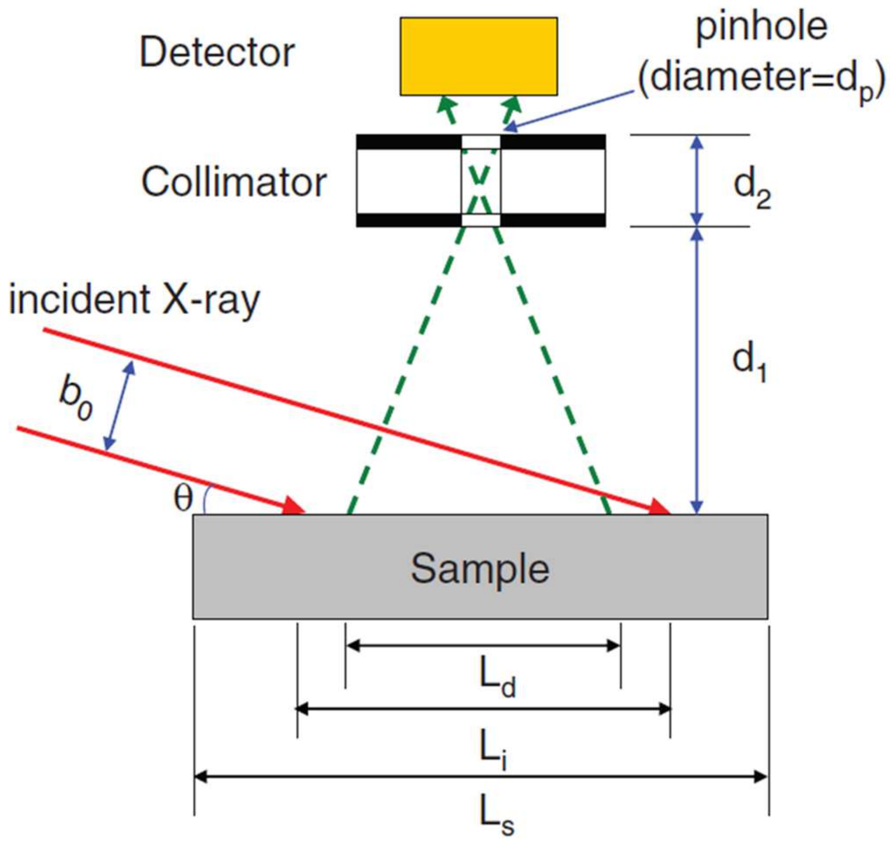


- **GiXRF**
  - Divergence (convolution ~ approximation …)
  - Geometrical correction



GEOMETRICAL CORRECTION

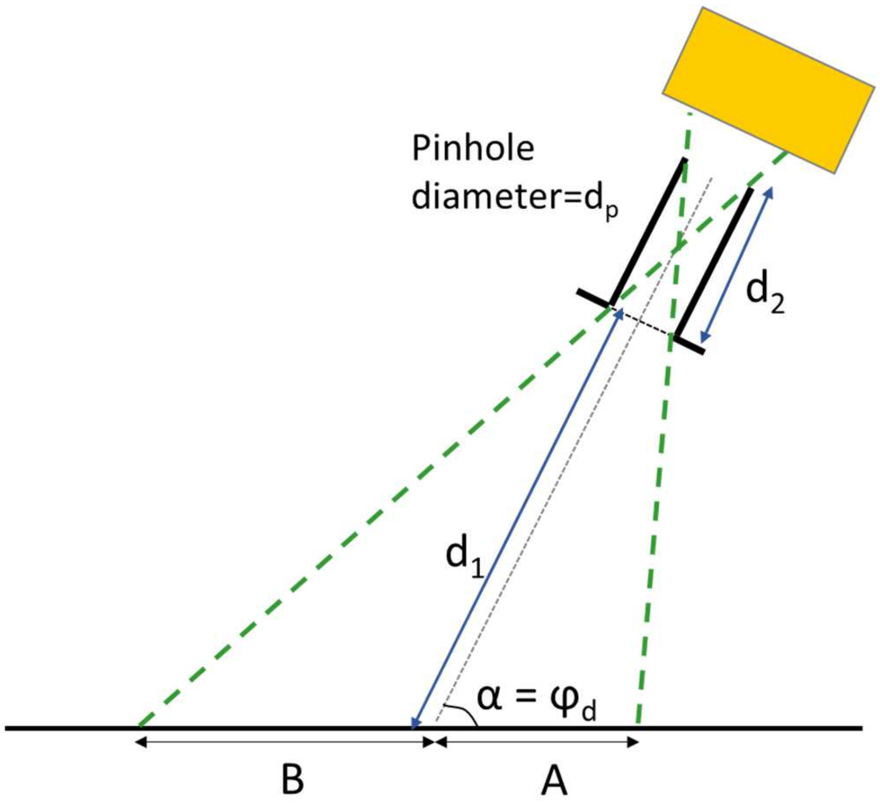
- **Geometrical correction**
  - Acceptance function (detected area corrected by solid angle of detection)
  - Spatial intensity distribution of the incident beam (*e.g* gaussian)

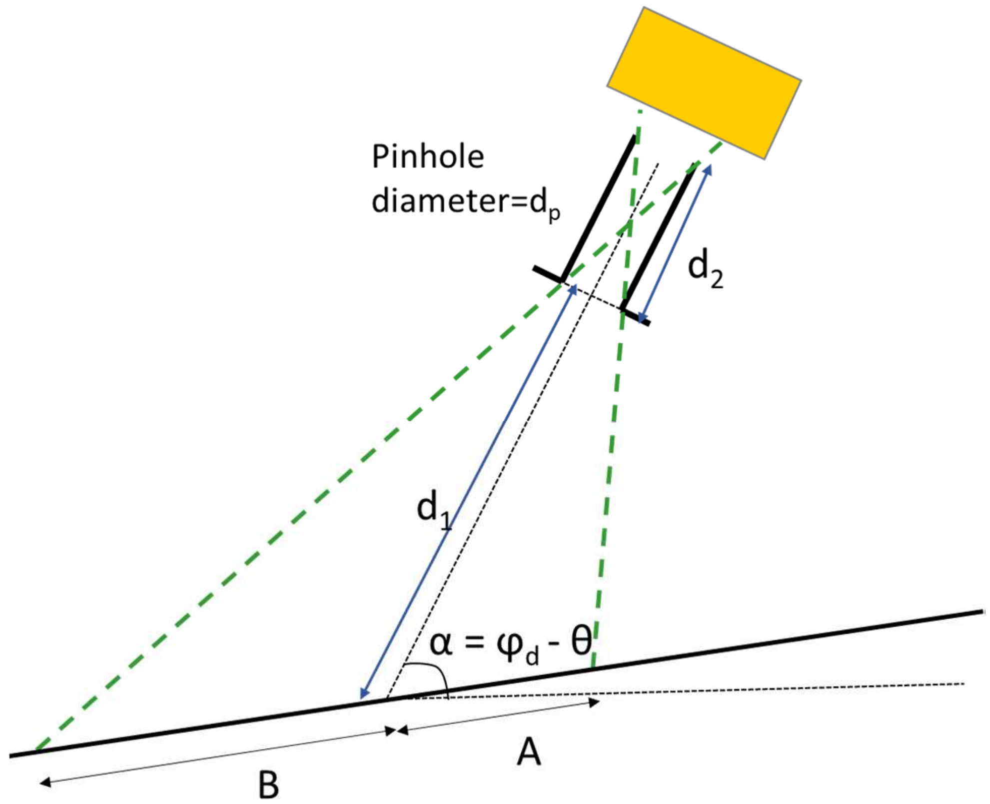


theta-theta configuration
Detector angle = 90°

W. Li et al, Review of Scientific Instruments ***83****, 053114 (2012)*

GEOMETRICAL CORRECTION





theta-theta configuration
Detector angle ≠ 90°

theta-2theta configuration
Detector angle ≠ 90°

ACCEPTANCE FUNCTION

- **JGIXA**
  - Rectangular function (width L_d)
  - Parameter = L_d
  - 1/cos(θ) correction for θ-2θ geometry

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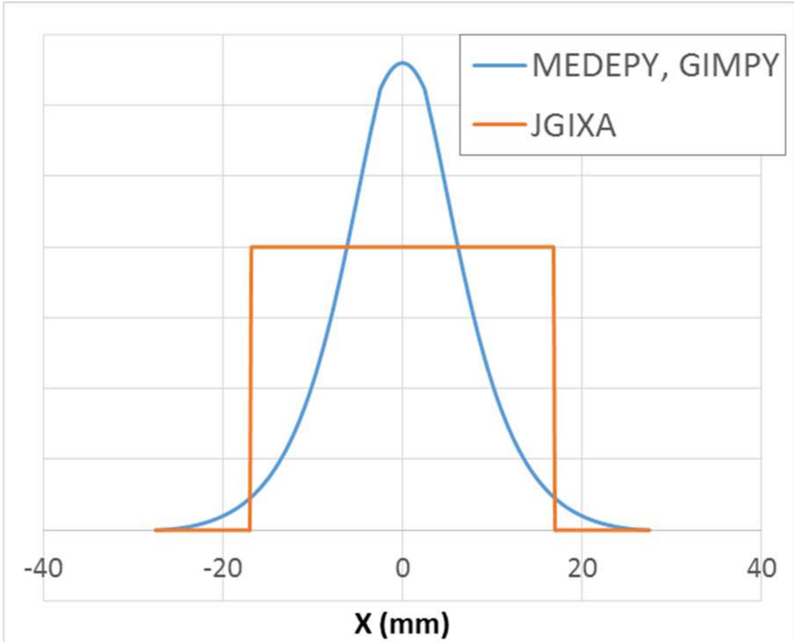
$$G(\theta) \propto \frac{\Delta\Omega}{4\pi} \int_{-L_S/2}^{L_S/2} g(\theta, t) \prod\nolimits_{L_d}(t)\, dt$$

Spatial intensity distribution of the incident beam

Rectangular function of the detectable area with a width of Ld

- **GIMPY, MEDEPY**
  - Parameters d_1, d_2, d_p
  - Heumans lambda function (solid angle of detection)
  - Independent (resp. dependent) of theta in θ-θ (resp. θ-2θ) geometry



GEOMETRICAL CORRECTION

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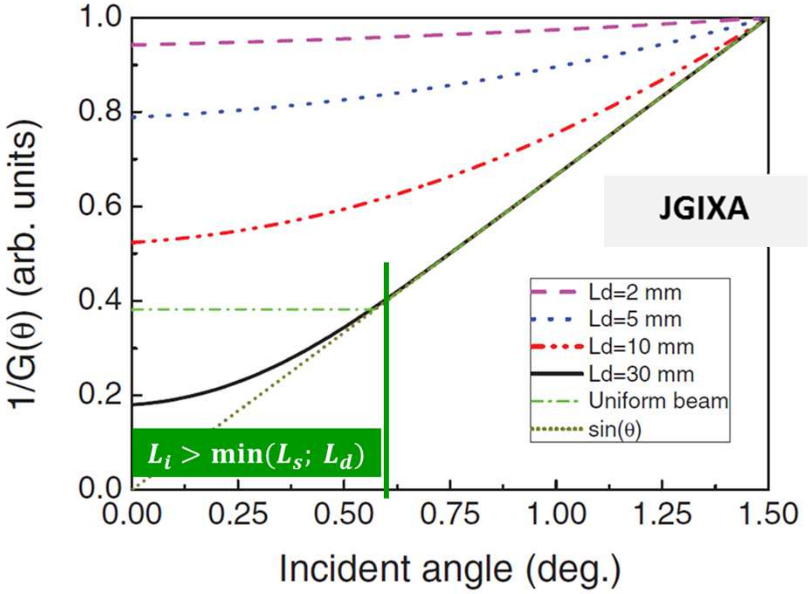
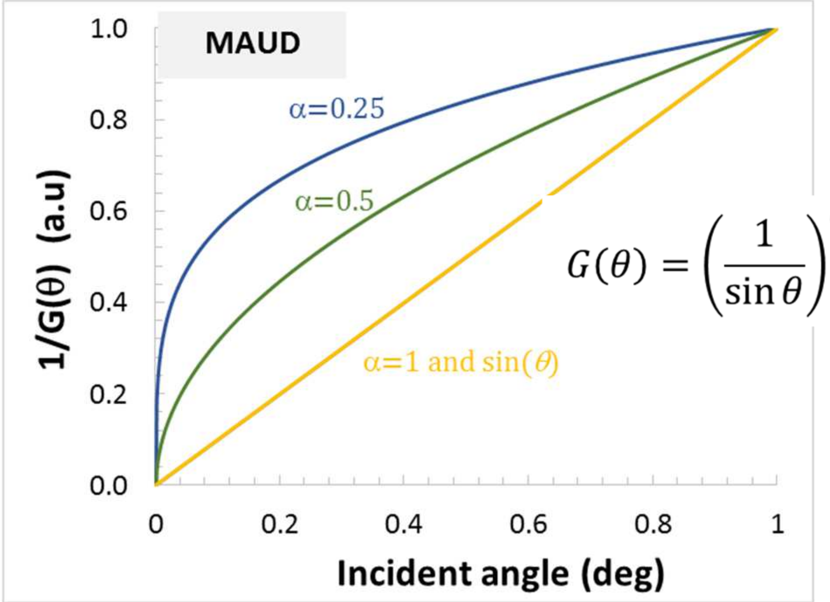
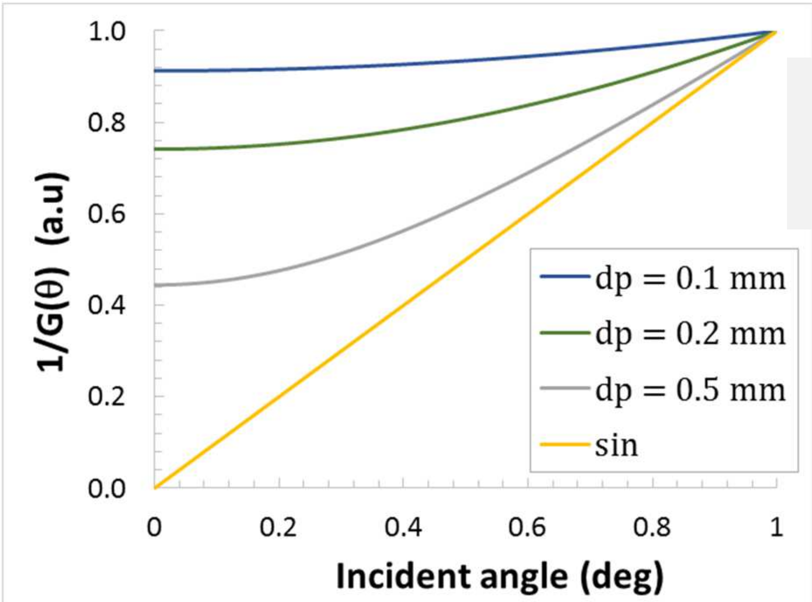
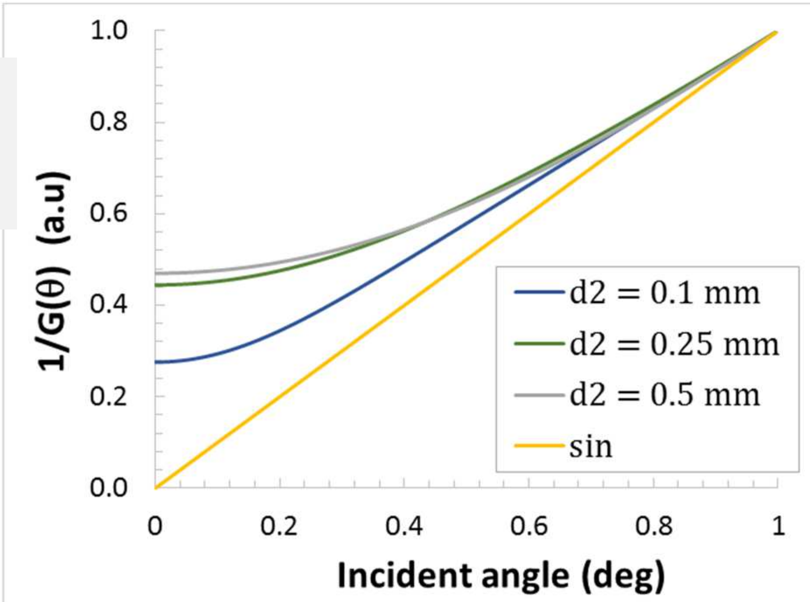




GIMPY

MEDEPY





SIMULATION

NiO_2 (5nm, d=6.0g/cc)
Ni (50 nm, d=8.9 g/cc)
Si (sub, d=2.33 g/cc)

XRR simulation	GiXRF simulation
For NiO_2/Ni/Si sub case study where thicknesses, densities and roughness were varied and when using the same database : • the simulated XRR data obtained with the 4 different software were found almost perfectly identical • the simulated GiXRF data obtained with the 4 different software on a « perfect » tool (no divergence, no instrumental function) were found almost perfectly identical	
Impact of the instrumental function (overall divergence) is almost perfectly identical for the different software	• Limited discrepancy induced by divergence • Significant impact of the geometrical correction
	• Only GIMPY includes secondary fluorescence…

FITTING CAPABILITIES

SOFTWARE	CAPABILITIES	REMARKS
GIMPY		• Fitting module under development
JGIXA	• Combined fitting of XRR and GiXRF datasets acquired at the same energy	• Fast and user friendly • Monochromatic primary radiation
MAUD	• Unique capability for XRR-XRD-GiXRF combined analysis • Stoichiometry	• Full spectrum only • GiXRF instrumental function to be corrected ! • Monochromatic and polychromatic primary radiation
MEDEPY	• Combined fitting of various XRR and GiXRF datasets acquired at various energies • Stoichiometry	• Monochromatic primary radiation • Still under optimization (definition of FOM for combined analysis …)

SUMMARY AND OUTLOOK

- Analysis of (ultra)thin layered films for advanced applications (micro/nano electronics, memory, photonics, PV, …)

- **Need for combined GIXRF/XRR as a non-destructive depth-profiling method**
  - On beamlines, in the Labs, … in R&D cleanrooms, in industry

- **GiXRF/XRR software**
  - 4 powerful software have been tested

- **Need for standardization** (reduced instrumental function …) in order to meet the needs for depth-dependent quantitative analysis in Labs, R&D facilities and industry

Thank you

for your attention!