In-house X-ray Standing Wave study of LaN/B multilayer mirrors

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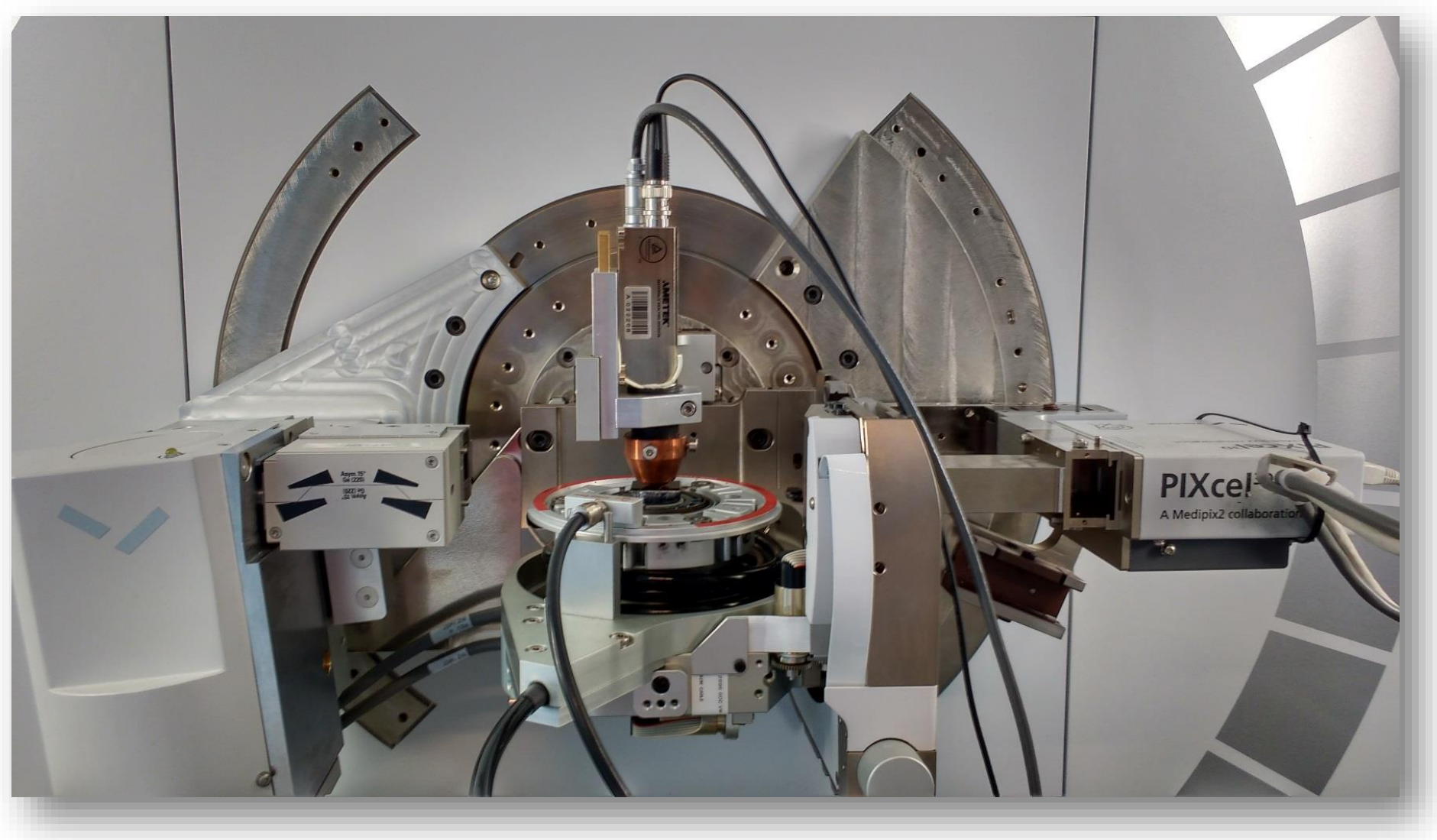
Introduction

→ X-ray Fluorescence (XRF) used for non-destructive characterization of atomic concentration of thin film.

→ The X-ray standing wave (XSW) combines Grazing Incidence X-ray Reflectivity(GIXR) and XRF to reconstruct the atomic profile of thin films and multilayers.

→ Measurements done on PANalytical Empyrean XRD setup equipped with Amptek XR-100 Silicon Drift Detector.



Goal:
→ Demonstrate applicability of XSW on in-house XRD setup.

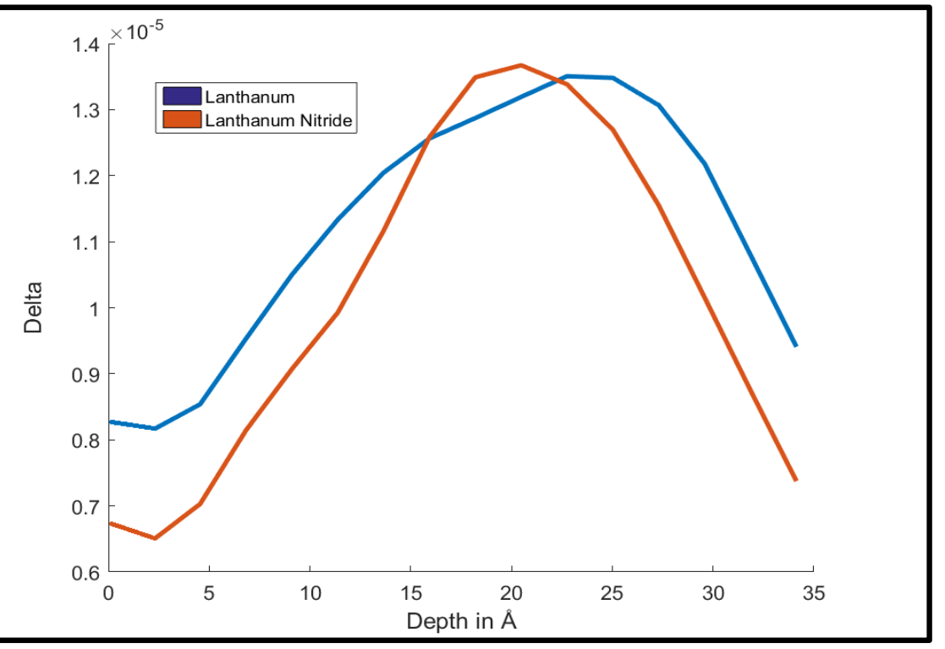
Benefits:
- Non-destructive
- Suitable for ambient
- Realizable on standard XRD equipment

The reconstruction of the multilayer period

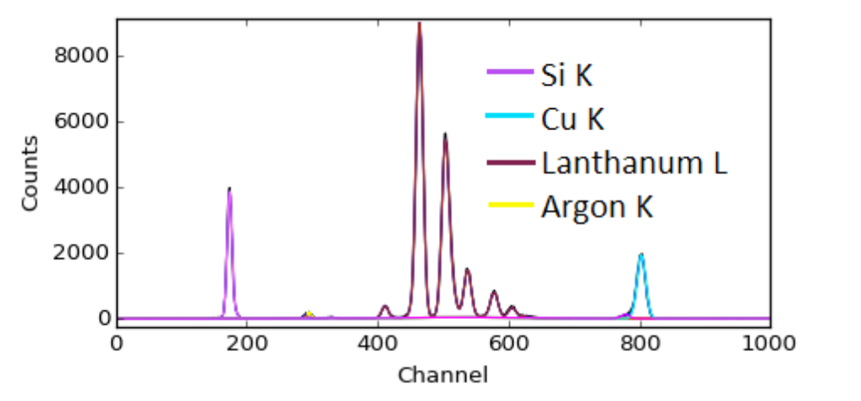
Samples: 2 periodic La/B and LaN/B multilayer mirrors:
- One baseline sample with clear intermixing between the 2 layers;
- Passivation of La with nitrogen reduces intermixing between the La and B layers;
  - → XSW was used to study the width of La distribution.

GIXR Measurements

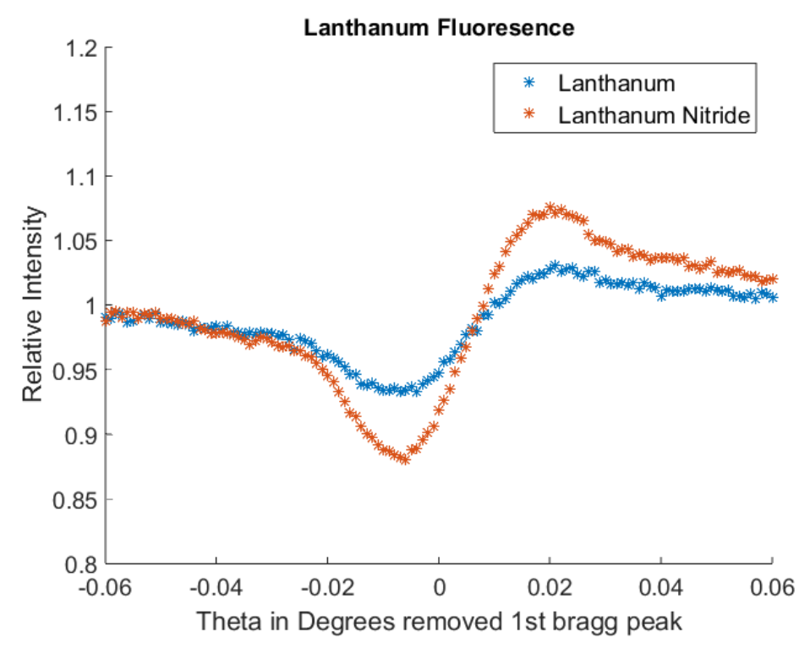
The optical constant profiles are obtained from the GIXR data analysis;
→ The passivated sample shows a more centered distribution.



Fluorescence Measurements



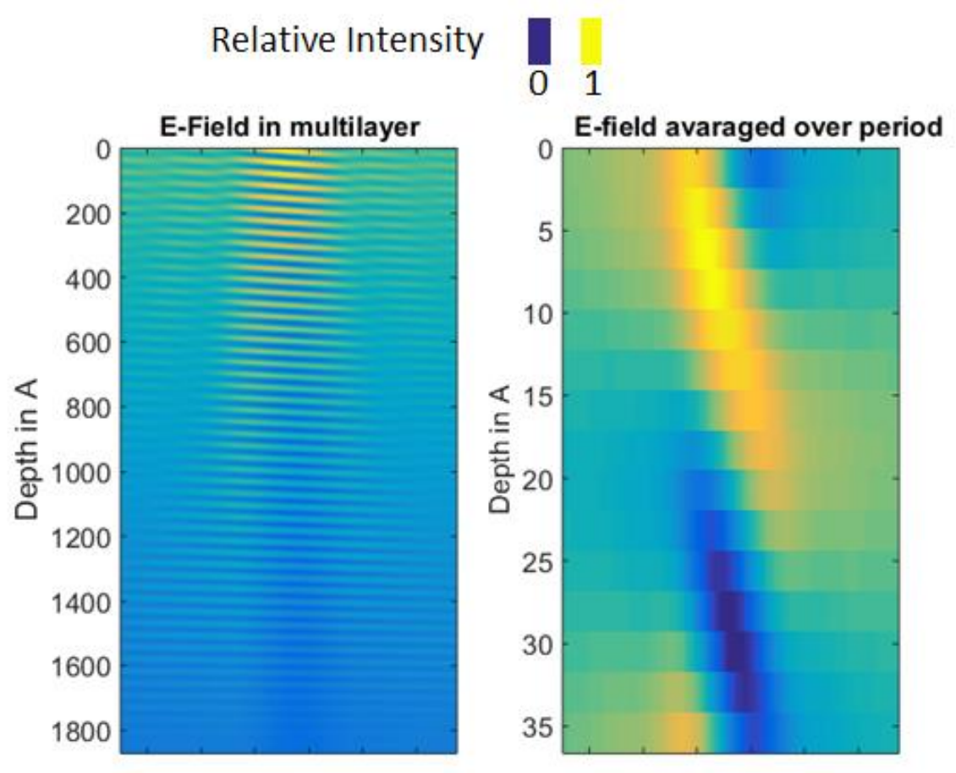
The fluorescence intensity is measured for different angles and the intensity per peak is obtained.



The angular fluorescence intensity shows a modulation by the standing wave

Electric Field Calculations

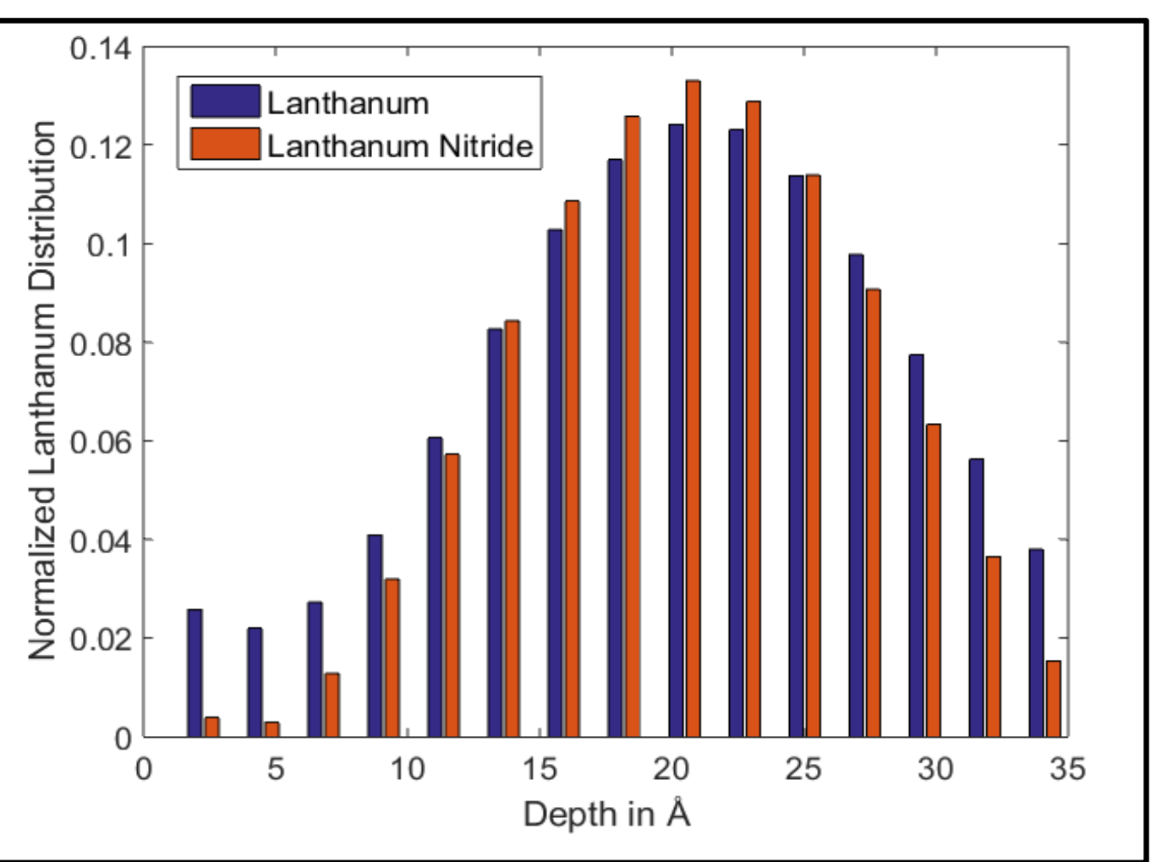
- Field Intensity is calculated using dynamical theory and the obtained optical profiles from GIXR data;
- The field is summarized to create an effective field for one effective period.
  → A clear effective standing wave is visible in the first Bragg peak.



Calculation of Profiles

The profiles are calculated by solving the linear system (1);

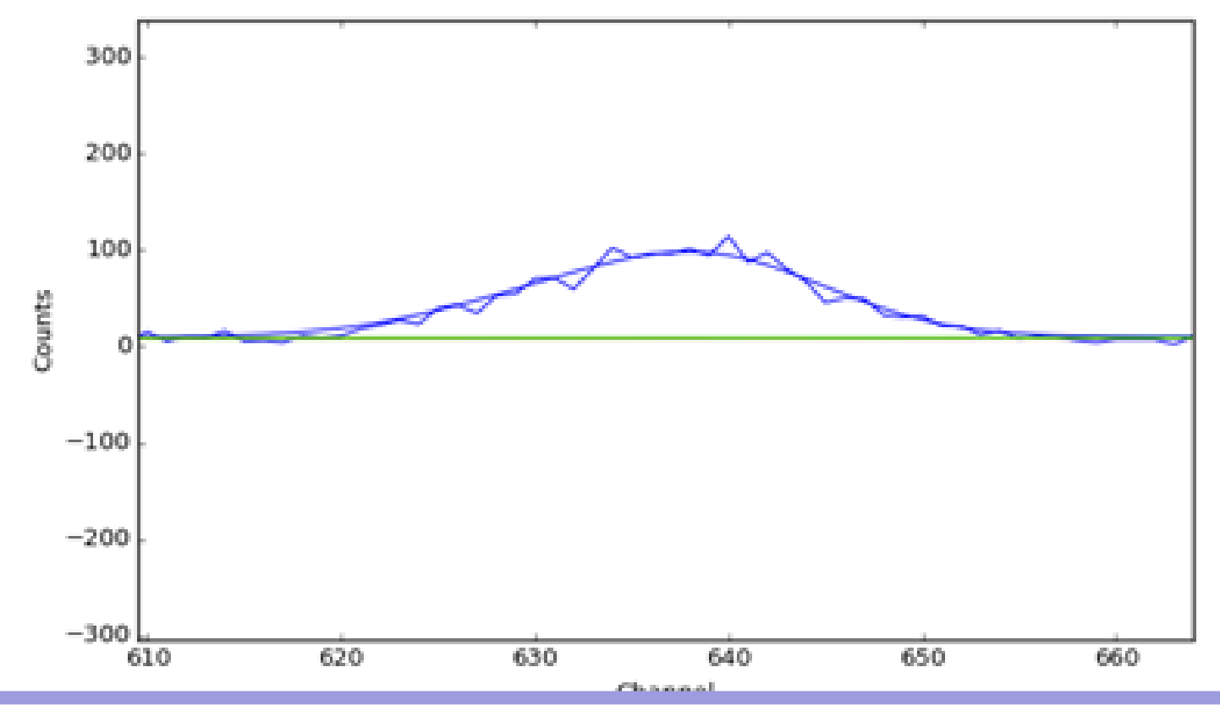
→The passivated sample shows a more centered La distribution and more La is present in the B layer in the baseline sample;



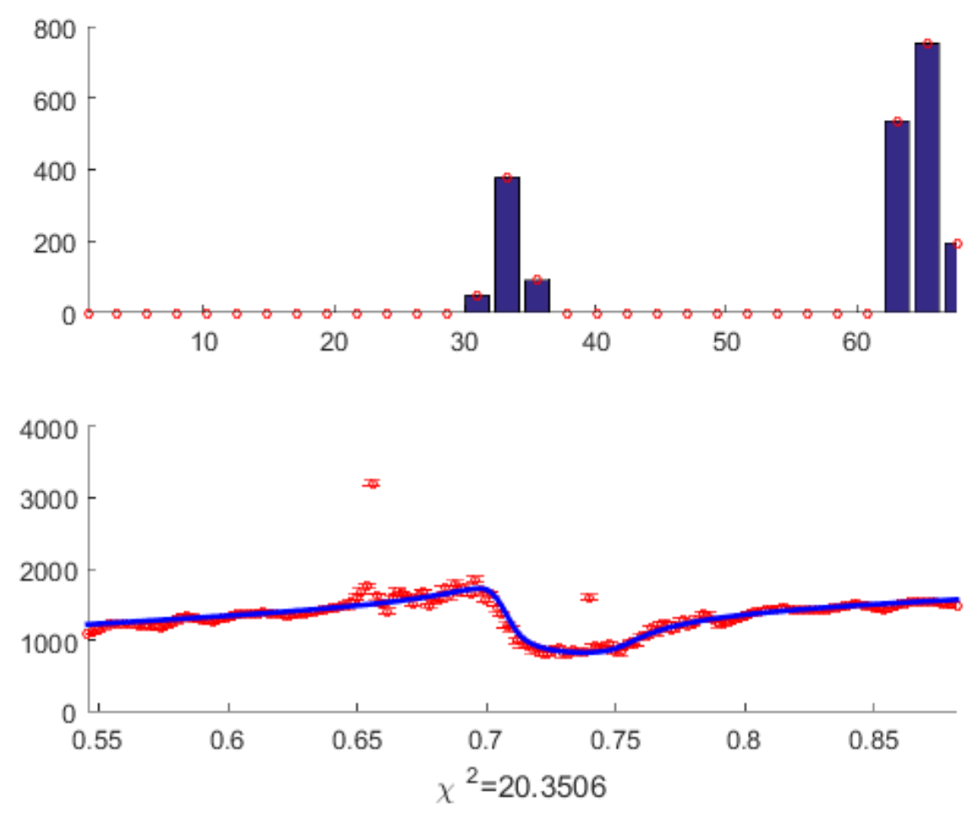
→ The La barrier in the passivated sample has less intermixing with the B barrier than for the baseline sample → Expected result is a good confirmation of the effectiveness of the XSW technique.

Application example: analysis of multilayer contamination

XSW analysis of small Iron traces
→ Iron can enter the system during deposition
→ This can lead to a very small periodical contamination which is invisible to any non-atomic sensitive technique
→ Sensitivity higher than a promille



Iron atoms in this multilayer were found in the interfaces.



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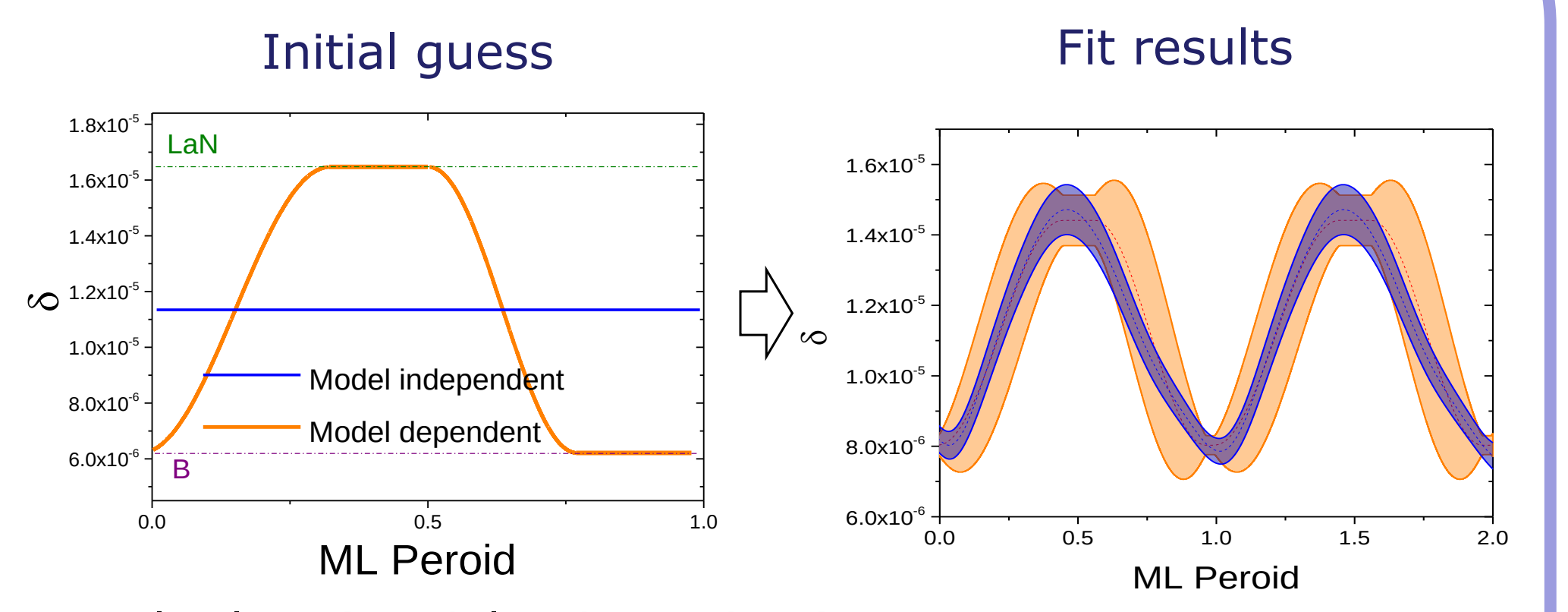
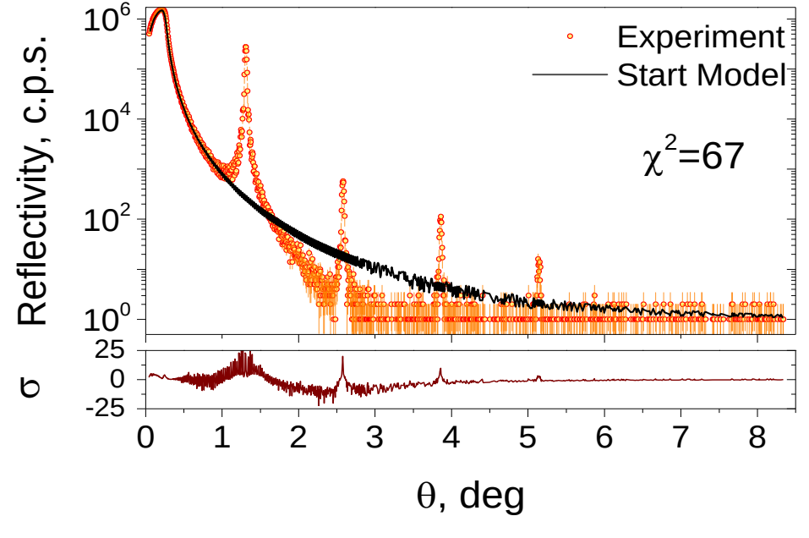
Project: model independent approach to the GIXR data analysis

→ Problem of classical of GIXR analysis:
initial guess (typically bi-layer model) required
→ Outcome of the fit depends on the initial model
→ Time consuming manual procedure

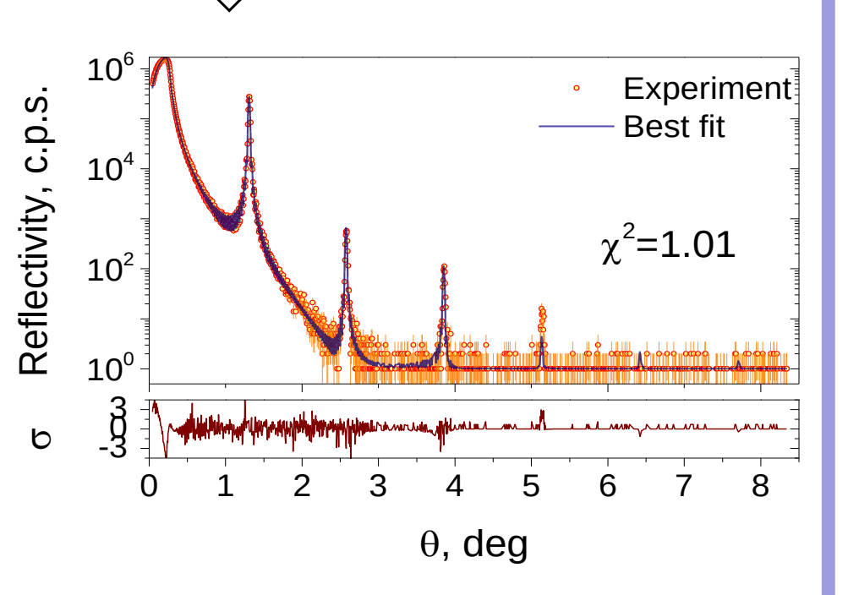
Solution:
Present a multilayer period as a set of "independent bins" and vary only the optical constants of these bins.
→ No pre-defined number of sub-layers in ML period
→ No pre-defined shape of the interfaces
→ The same initial model for all analyzed multilayers
→ Even useful in more complex systems.



Flat line (model independent)



30 min, 1 start



Result:
Fast and reliable fit!

Model independent approach to XSW data analysis

$I(\theta)$-Measured fluorescence intensity:

$$I(\theta) = \sum_{j=1}^{m} P(z_j)\ |E(\theta, z_j)|^2$$

E – 2D Electromagnetic field
P(z)-unknown atomic profile

To find *P(z)*: the system of linear equations to be solved:

$$\hat{A}\cdot\mathbf{x} = \mathbf{b} \qquad (1)$$

Where: $\mathbf{x} = \begin{Bmatrix} P_1 \\ P_2 \\ \ldots \\ P_m \end{Bmatrix}$

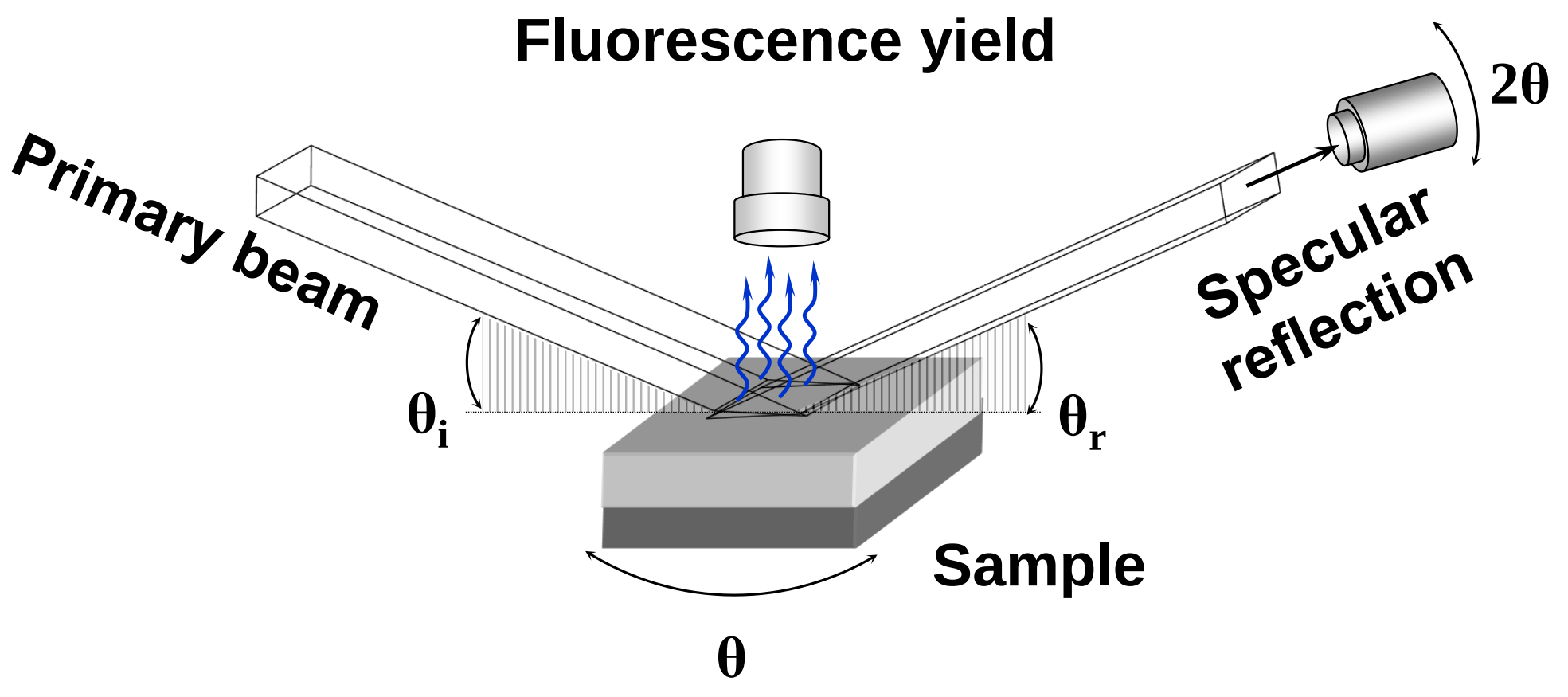
A= Electric field intensity for different angles and depths
b = experimentally measured fluorescence

Measurements have errors and the problem is over-defined→ ill-posed problem
Solution → regularization required!

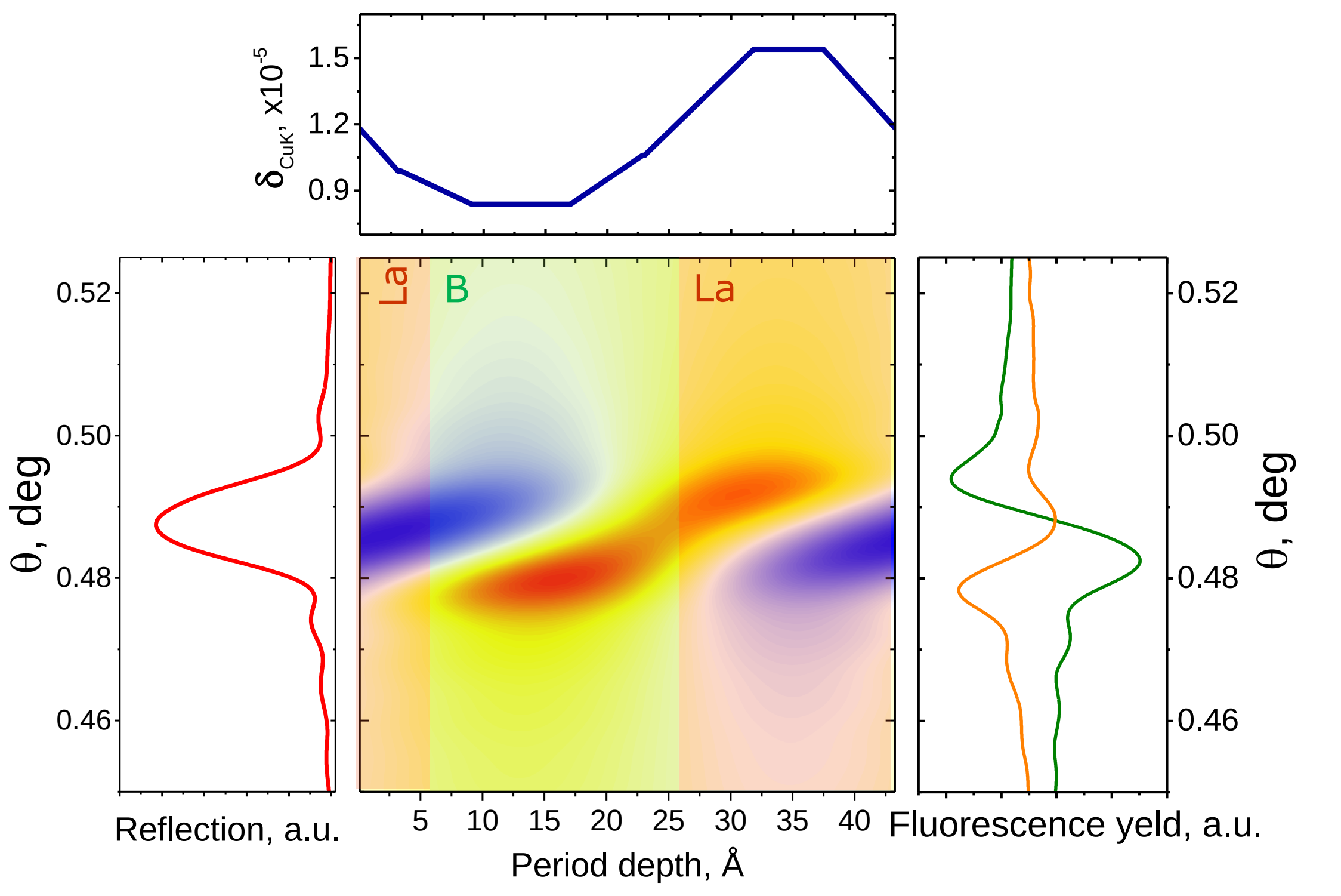
$$(\hat{A} + \lambda\cdot D)\cdot\mathbf{x} = \mathbf{b}$$

$$D = \begin{vmatrix} 2 & -1 & 0 & 0 & 0 & -1 \\ -1 & 2 & -1 & 0 & 0 & 0 \\ \ldots & & & & & \\ -1 & 0 & 0 & 0 & -1 & 2 \end{vmatrix}$$

λ – regularization coefficient



Experimental scheme: classical scheme for GIXR measurements + energy dispersive X-ray fluorescence detector.



Effective EM field from the periodic part of the La/B MLM in the first Bragg reflection condition (center graph), calculated GIXR (left graph) and fluorescence yield from La and B (right graph), and the δ profile used for calculation of EM field (top graph).

Conclusion

→ Model independent GIXR analysis allows a fast optical constant profile determination for following XSW analysis;

→The XSW technique is atomic sensitive which allows it also to differentiate between layers and materials that do not show any optical contrast;

→ It is capable of discerning small differences in intermixing between 2 layers.

→ Allows to find very small contaminations in thin films and also locate them.

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