

# Study of nano-scale supperlattices by proton induced Kossel diffraction

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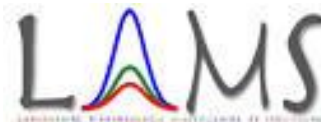
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# ● Motivation

# ● Superlattices

# ● Methodology

- Particle Induced X-ray Emission (PIXE)
- Kossel diffraction
- Combination of Kossel diffraction and PIXE
- X-ray color camera

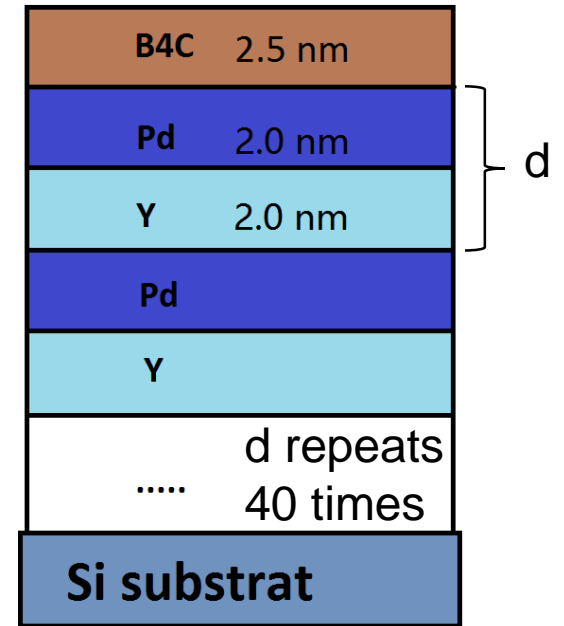
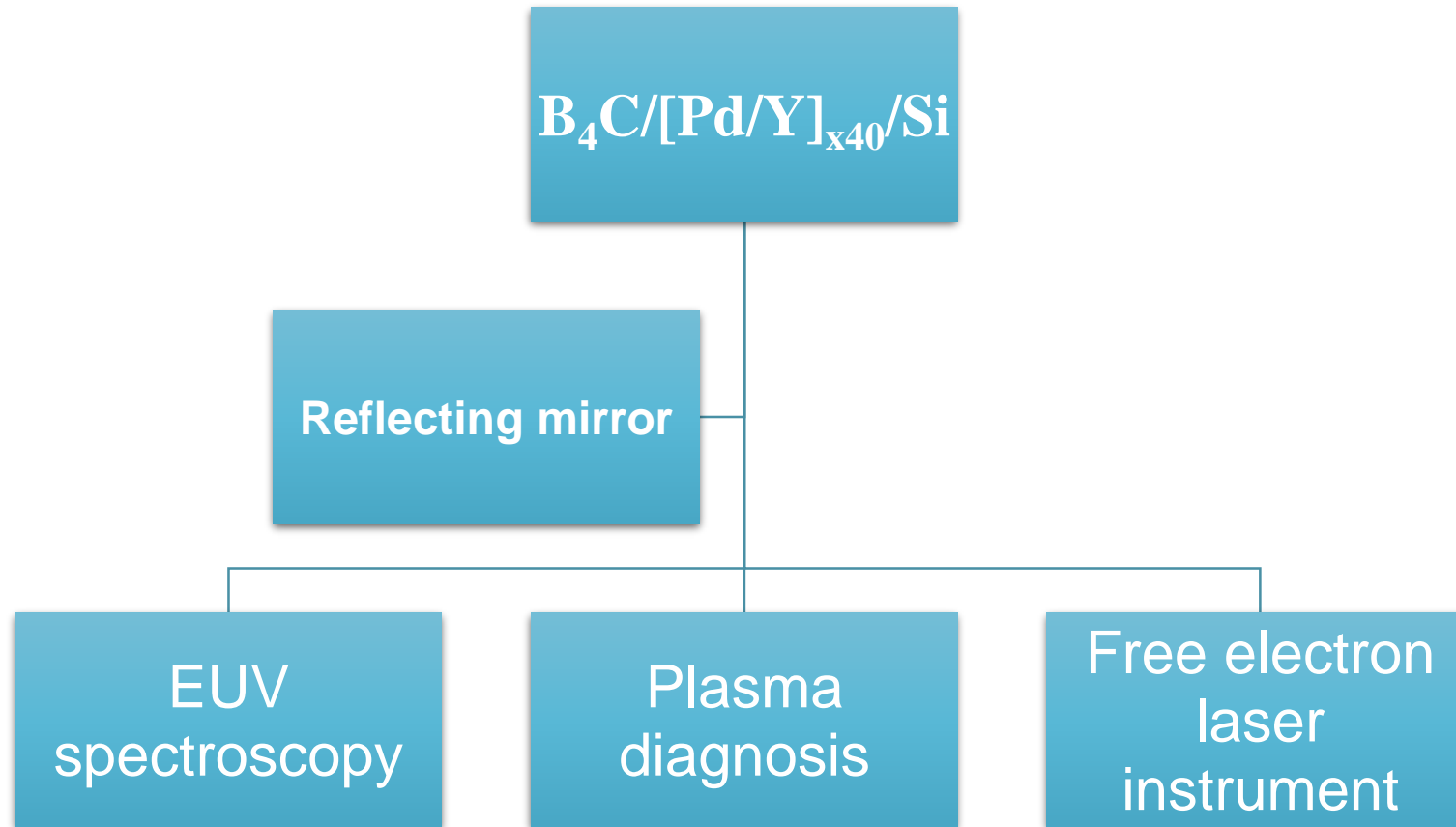
# ● Results and Discussions

# ● Conclusions

# Motivation

- Widely used superlattices, or periodic multilayers.
- Characterization of the structure of periodic multilayers is dominated by X-ray reflectometry.
- Do we have more options for the characterization?

# Pd/Y superlattices



Designed structure of  $B_4C/[Pd/Y]_{x40}/Si$

Pd/Y multilayers designed to work in the 7.5-11 nm wavelength range. Theoretical calculation indicates that Pd/Y has a high reflectance in this range.

# Pd/Y based superlattices

- **Problem: the original design has bad optical performance**

- Grazing incident X-ray reflectometry (Cu K radiation 8048 eV):

**Low reflection, bad periodicity**

- Severe interdiffusion between Pd and Y layers.

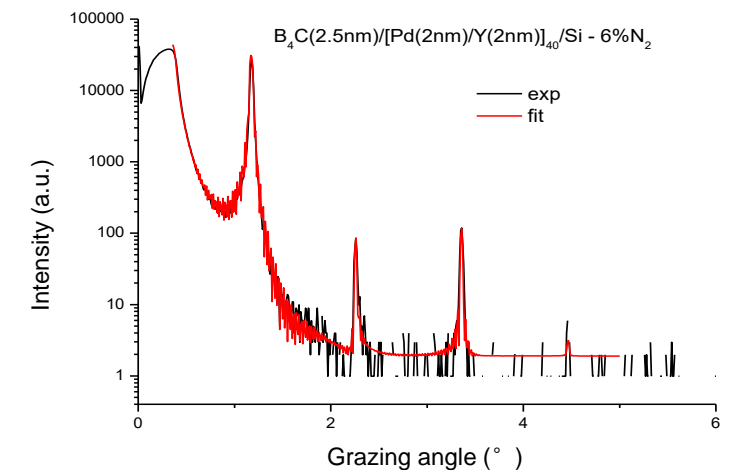
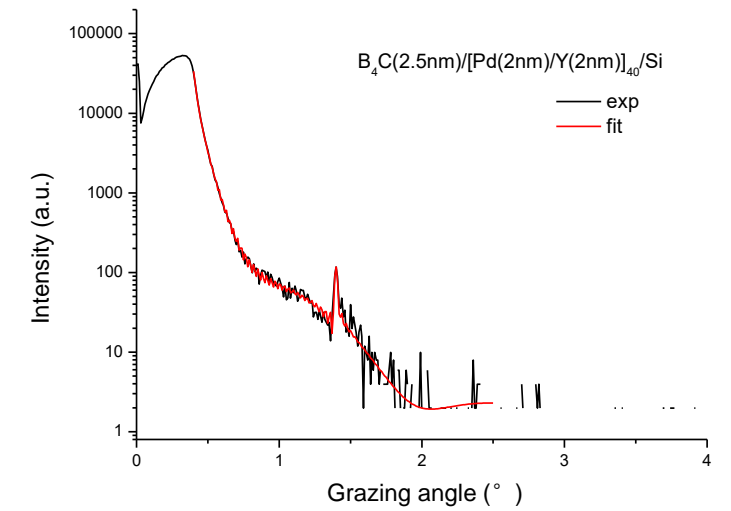
- **Solution: Derivative systems**

- N<sub>2</sub> in the sputtering gas [1].

**0%      2%      6%**

- B<sub>4</sub>C barrier layers [2].

- **Pd-on-Y interfaces**
- **Y-on Pd interfaces**
- **all interfaces**



[1] Xu *et al.*, "Enhancement of soft X-ray reflectivity and interface stability in nitrated Pd/Y multilayer mirrors," *Opt. Express*. 23, 33018 (2015).

[2] Prasciolu *et al.*, "Thermal stability studies of short period Sc/Cr and Sc/B<sub>4</sub>C/Cr multilayers," *Appl. Opt.* 53(10), 2126 (2014).

# Particle Induced X-ray Emission

## ● Particle Induced X-ray Emission (PIXE)

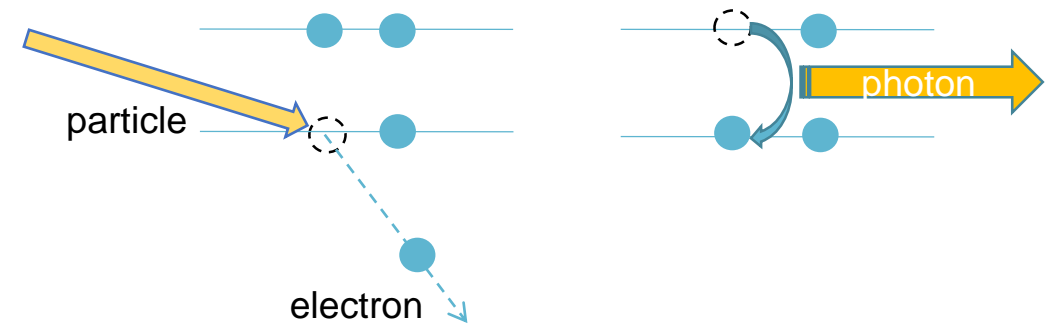
- Ionization: particle generated core-hole.
- X-ray emission (fluorescence).

## ● SAFIR platform in UPMC

- Système d'Analyse par Faisceaux d'Ions Rapides  
(Analysis System using fast ions).
- Van de Graaff accelerator of positive ions.
- 2 MeV proton beam, ionization of Pd L shells.

## ● Advantages of protons

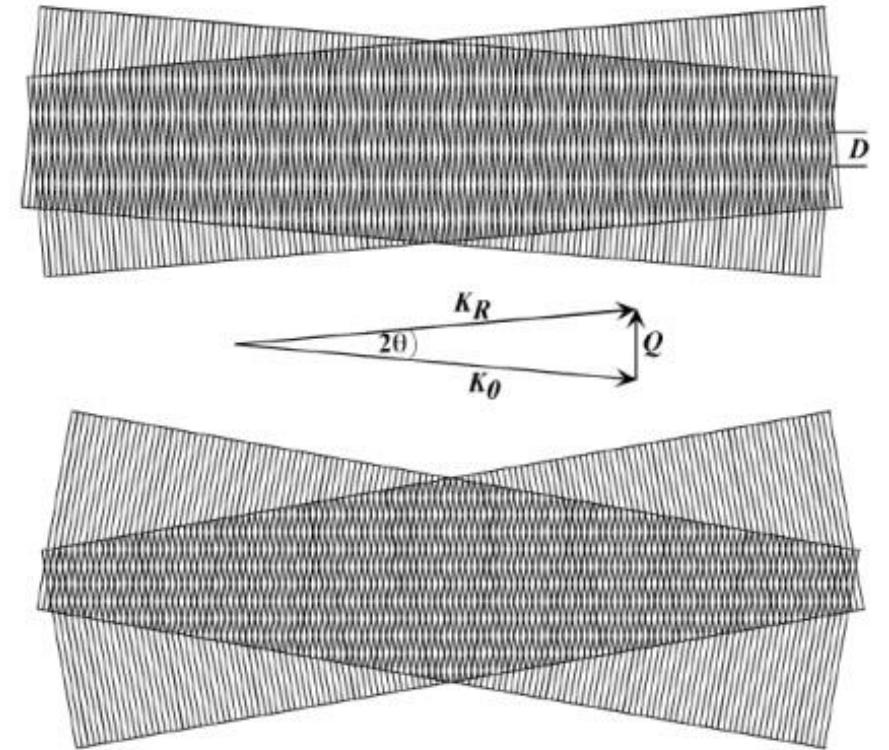
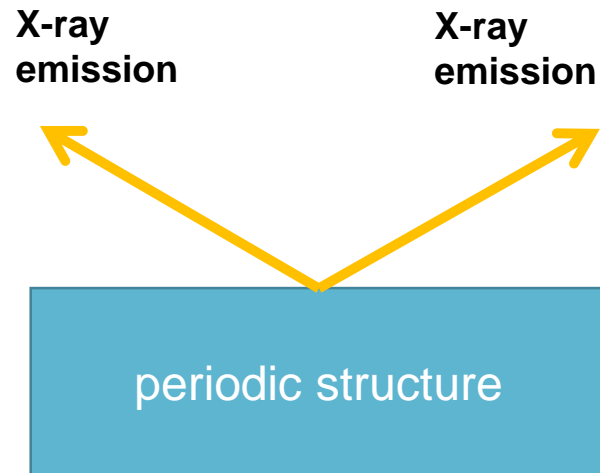
- Low scattering.
- Low bremsstrahlung.
- Low energy loss (0.5% after penetrating the multilayer).
- Uniform ionization cross section.



Accelerator in SAFIR

# Kossel diffraction

- Kossel diffraction [3]
- Interference of fluorescence diffracted by a periodic micro-structure.
- X-ray standing waves within the superlattice: periodic electric field with nodal plan and anti-nodal plan.

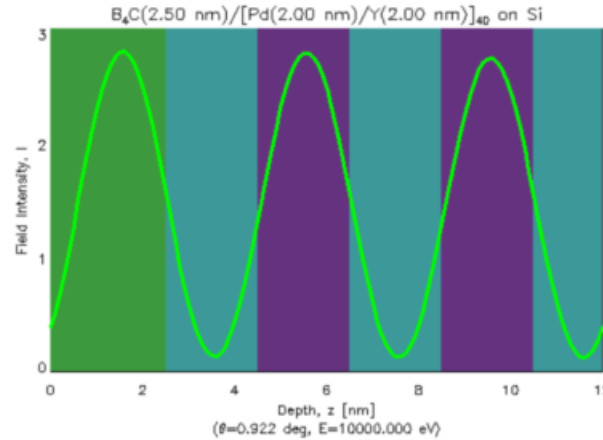


$$D = \frac{\lambda}{2 \sin \theta} = \frac{2\pi}{Q}$$

[3] W. Kossel, V. Loeck, and H. Voges, Z. Für Phys. **94**, 139 (1935).

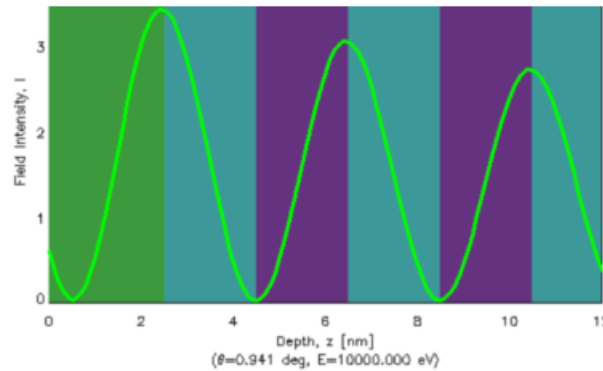
# Combine PIXE with Kossel diffraction

$\theta < \theta_{\text{Bragg}}$

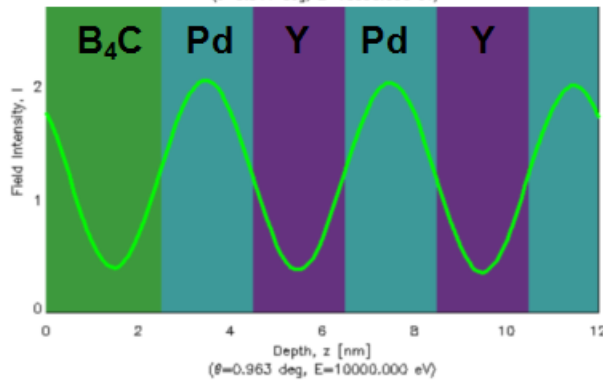


$$D = \frac{\lambda}{2 \sin \theta} = \frac{2\pi}{Q}$$

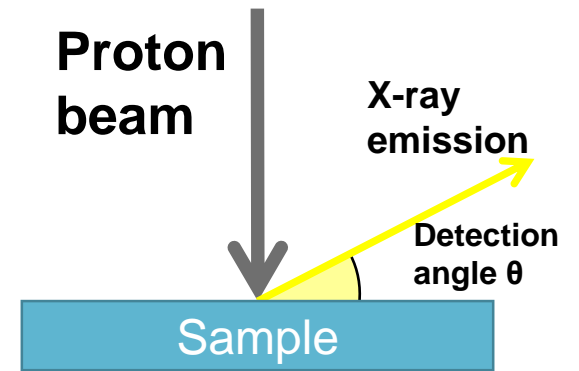
$\theta = \theta_{\text{Bragg}}$



$\theta > \theta_{\text{Bragg}}$



Measurement of X-ray emission intensity as a function of the detection angle.

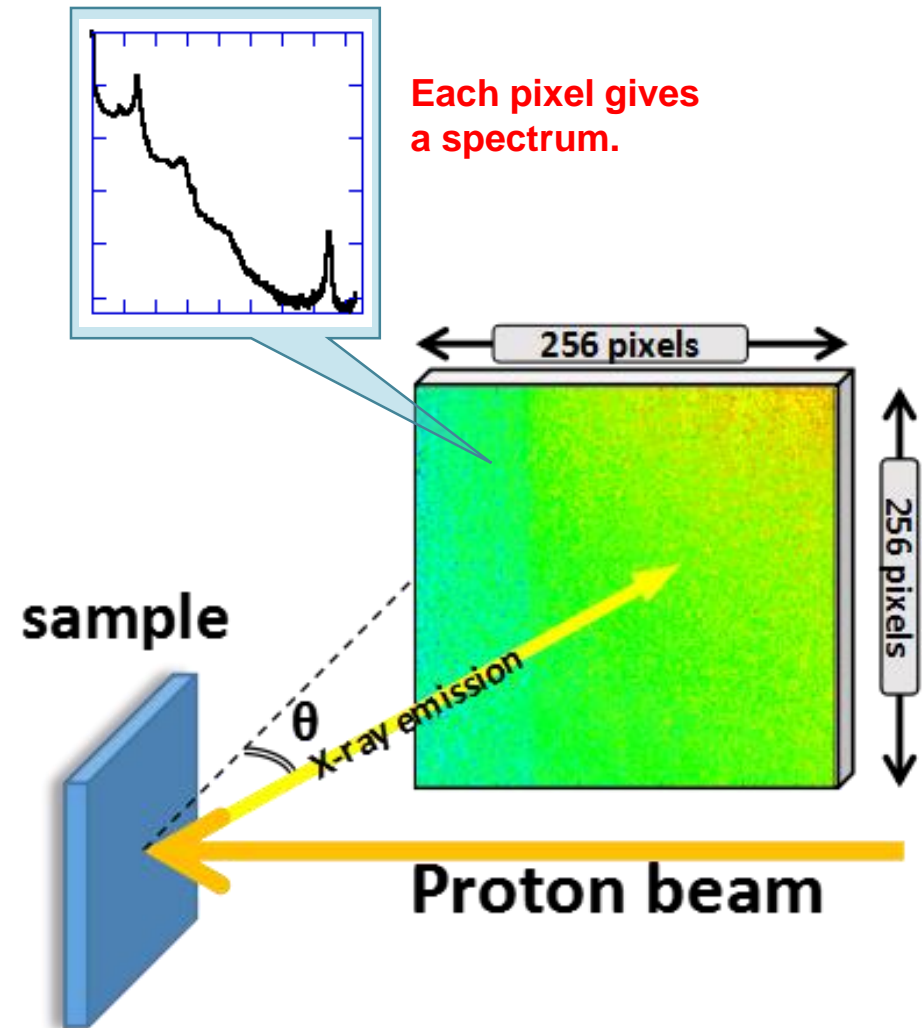




# X-ray color camera

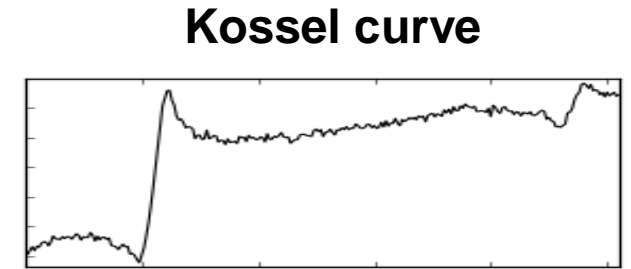
- Advantage: spatially resolved.
- Measurement of X-ray emission intensity in a squared area of about 13 x 13 mm.
- Acquisition time significantly reduced compared to SDD.

Days to 2 hours >>> potential of in-situ measurements (annealing test, oxidation test)

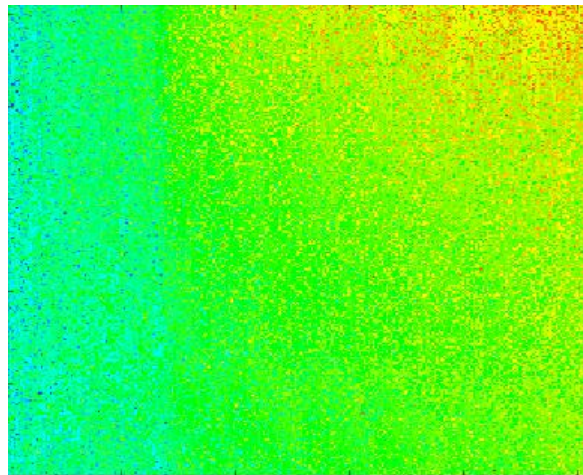


# Kossel curves

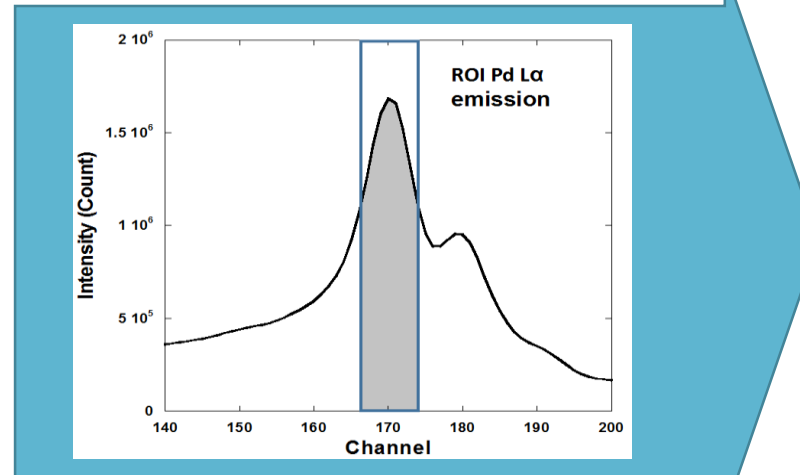
- Kossel curve: the angular distribution of the characteristic X-ray emission.
- The original image obtained by X-ray color camera contains the intensity of emitted photons of all energies.
- To get the Kossel curve of Pd L $\alpha$  emission, we need 3 steps:
  1. Select region of interest (ROI) on the spectrum.
  2. “Filter” the original image by applying ROI.
  3. Integrate the “filtered” image along the vertical pixels.



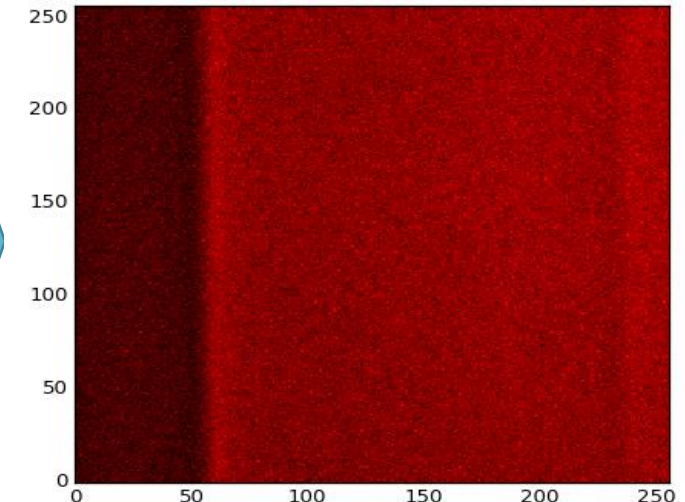
Integrate along the vertical pixels



Distribution of X-ray



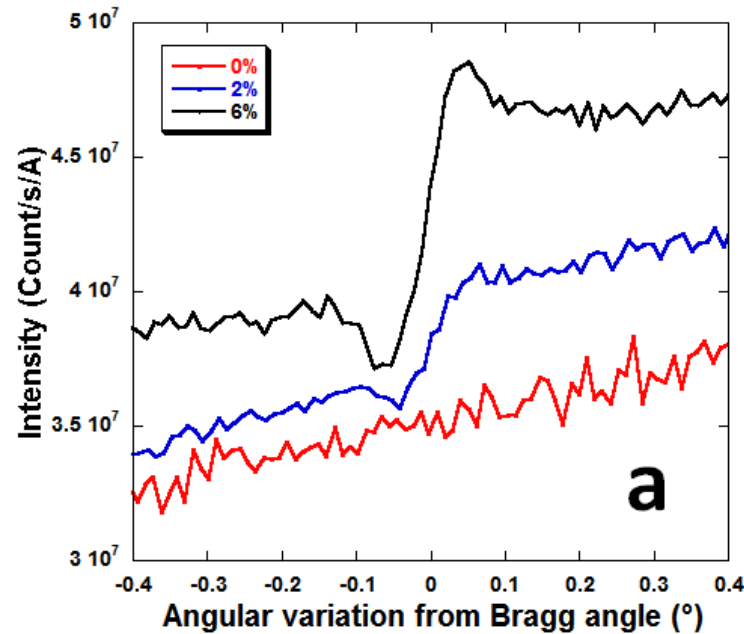
ROI selection



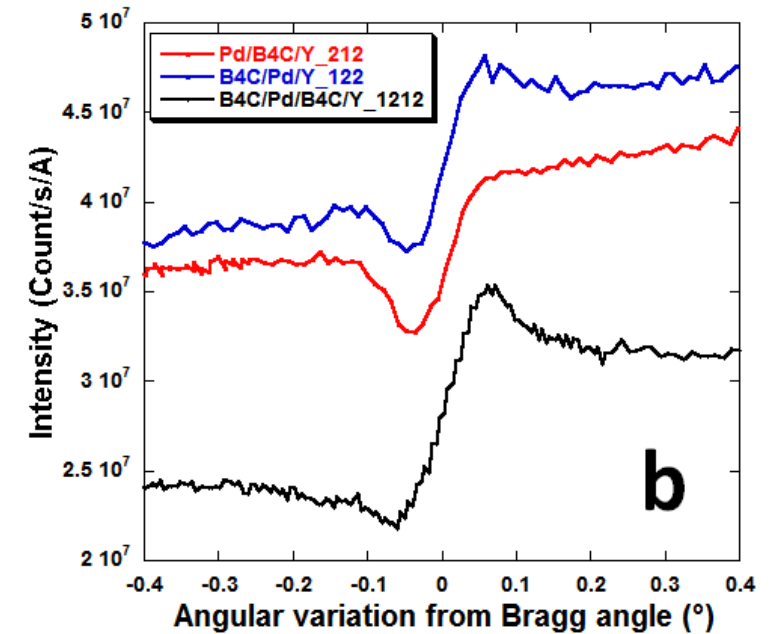
Distribution of Pd L $\alpha$  emission

# Kossel curves of Pd La emission

- 2-hour acquisition time. Curve statistics can be further improved by increasing the acquisition time.
- Angles are adjusted to the Bragg angle, which is in most cases located in the center of the Kossel oscillation.
- The periodicity of the originally designed Pd/Y multilayer is totally compromised.
- Nitrogen reduces the interdiffusion.
- Kossel features can be distinguished for samples with different B<sub>4</sub>C barrier layers. **Structural sensitivity.**



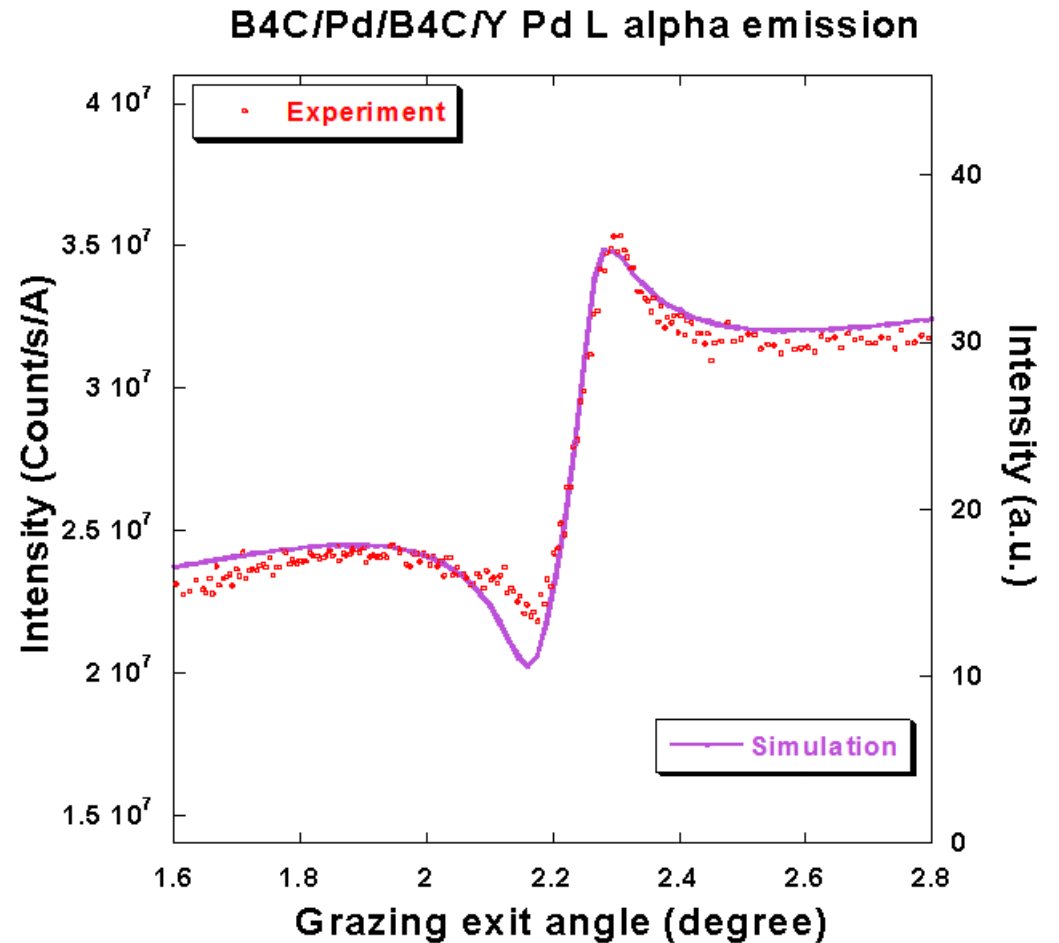
Kossel curves of the series of samples:  
a) Deposited with nitrogen in the sputtering gas.



Kossel curves of the series of samples:  
b) With B<sub>4</sub>C barrier layers. The notation gives multilayer structure and layer thickness respectively.

# Simulation

- Simulation of the PIXE-Kossel curve.
- Code for XRF-Kossel curve.
- Approach.
- Uniform ionization.
- Still not perfect due to background.
- Improvement needed.



# Conclusion

- We have observed the Kossel diffraction of proton induced Pd L $\alpha$  emission in Pd/Y based superlattices.
- Nitridation has positive effect in preventing or reducing the interdiffusion between Pd and Y layers.
- The Pd/Y based superlattices with B<sub>4</sub>C barrier layers located at different interfaces can be distinguished by their Kossel curves.
  
- We have developed a new method to characterize the superlattices: PIXE-Kossel.
- Simple experimental setup.
- Element specific diffraction patterns.



**Thank you for your attention!**