



# Energy dispersive x-ray CCD camera for PIXE-Kossel and imaging purposes

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# Energy dispersive CCD camera

PIXE – Kossel

Spectral imaging

# Energy dispersive CCD camera

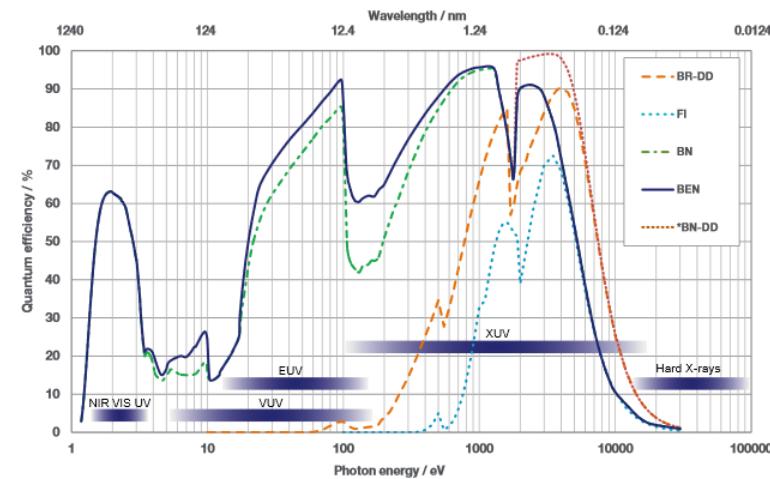
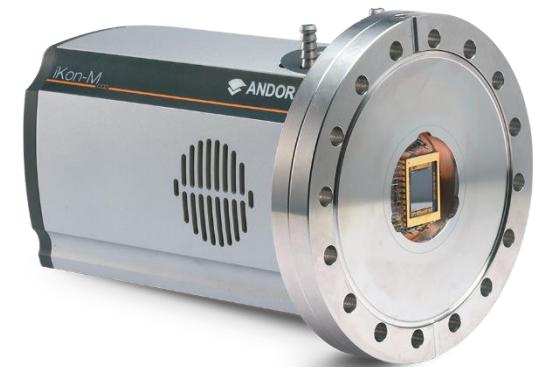
- **Each pixel of the CCD camera is an EDS detector  
(Energy Dispersive Spectrometer)**
- Functioning equivalent to a SDD (silicon drift detector)
- Work in a large spectral range (5 eV - 20 keV, depending on the CCD)
- Spectral resolution around 150 eV @ 6 keV
- Quantum efficiency > 10% over the whole spectral range
- Single-photon counting mode

# Energy dispersive CCD camera

- The pnCCD Color X-Ray Camera® (CXC) de PNDetector

- **ANDOR iKon-M**

- Back-illuminated deep-depletion
- Size of pixels  
     $13 \mu\text{m} \times 13 \mu\text{m}$  ( $1024 \times 1024$ )
- Pelletier cooling  
     $-80^\circ\text{C}$  (low electronic noise)
- Spectral range  
    from 1 keV up to 20 keV
- Efficiency  
     $>10\%$
- Software  
    P. Walter (LAMS), P. Sarrazin (SETI), I. Ismail (LCPMR)



Energy dispersive CCD camera

PIXE – Kossel

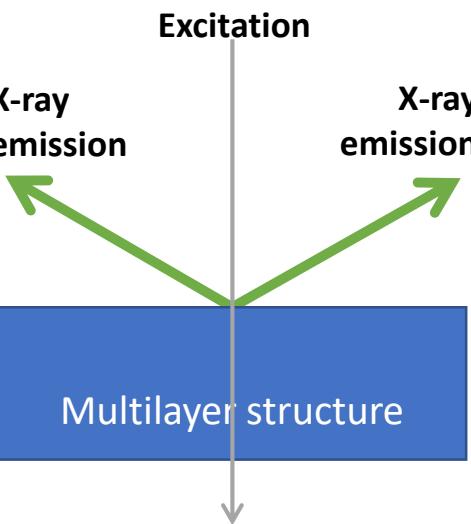
Spectral imaging

# PIXE – Kossel

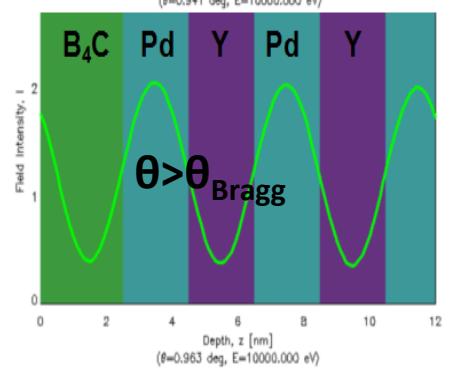
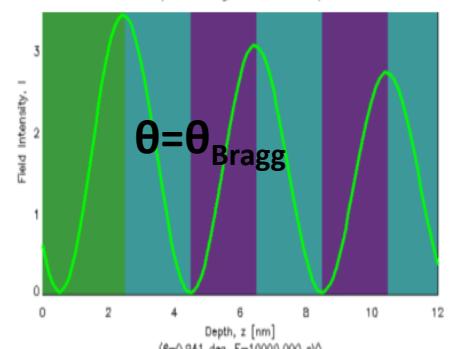
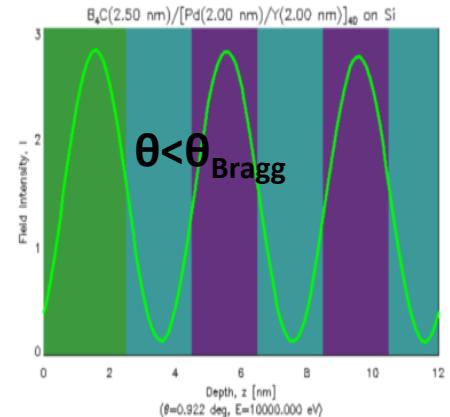
## Principle of Kossel diffraction

(GEXRF = grazing exit x-ray fluorescence)

- Generation characteristics x-rays
- Interference between emitted radiations
- Diffraction of the radiation emitted by the atoms of the multilayer by the multilayer structure itself under a given detection angle (Bragg for a periodic structure)
- Study of thin films
- Excitation
  - Protons (PIXE)
  - X-rays (XRF)
  - Electrons (XES)



Pd / Y  
periodic  
multilayer



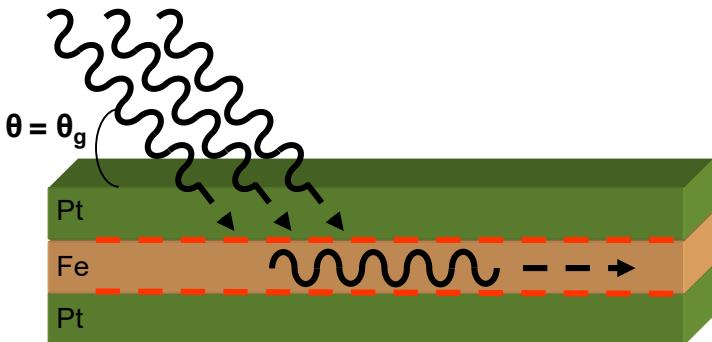
Depth distribution of the  
electric field

# PIXE – Kossel

## Study of a Pt/Fe/Pt waveguide

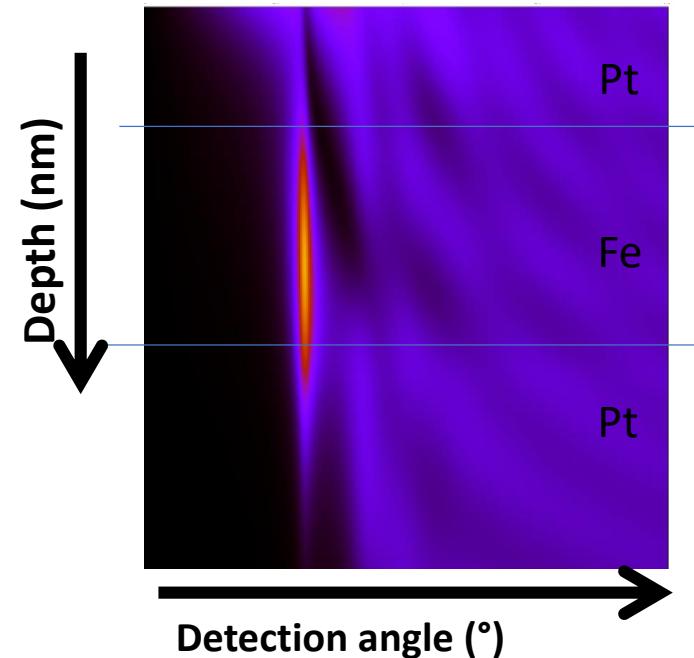
### Planar x-ray waveguide

- A “light” guiding material between two “heavy” cladding materials
- Total reflection

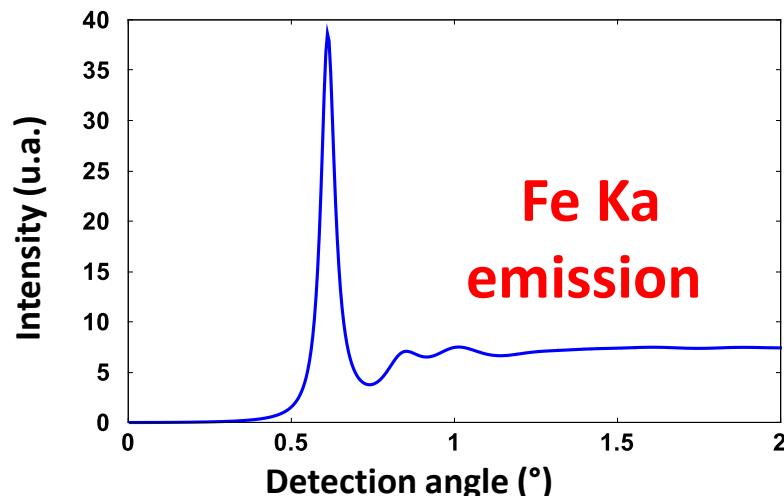


**Kossel curve: observation of the angular distribution of the emitted characteristic radiation**

**Modeling Kossel curves  
depth distribution of the emitting atoms**



Simulations of the depth and angular distribution of the electric field and of the Kossel curve

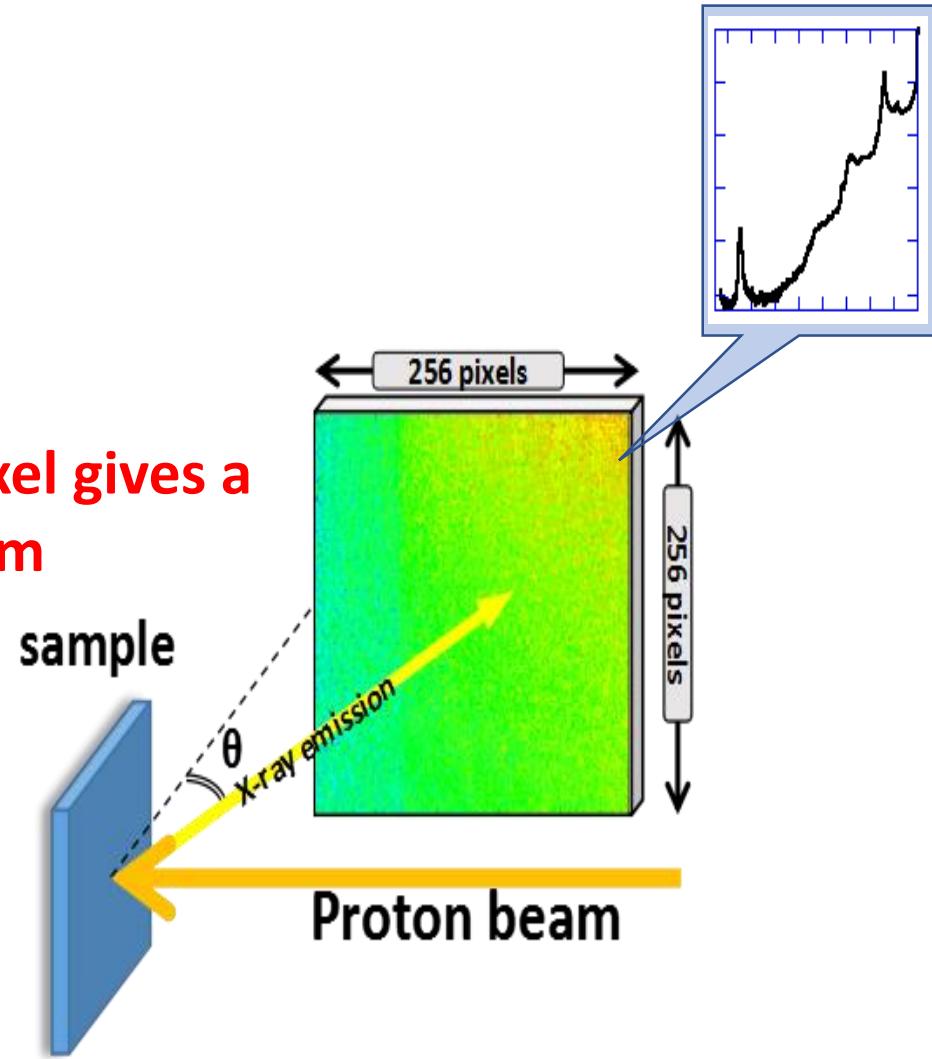
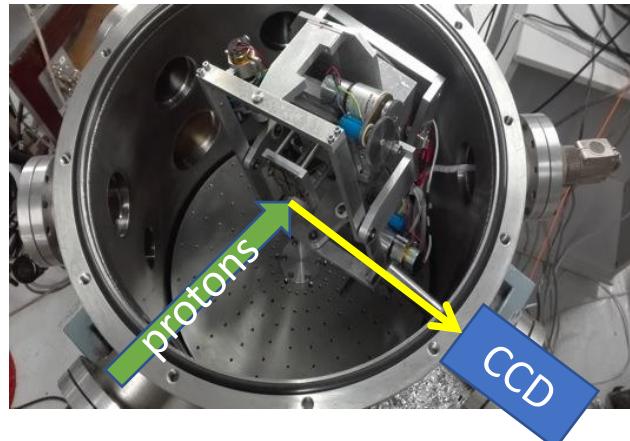


# PIXE – Kossel

- **Experimental process**
- Be filter (protons dangerous for pixels)
- Bining 256 x 256 pixels ( $52 \times 52 \mu\text{m}$ )
- No angular scan
- “Short” acquisition time, 2h

**Each pixel gives a spectrum**

Top view of the goniometric chamber



# PIXE – Kossel

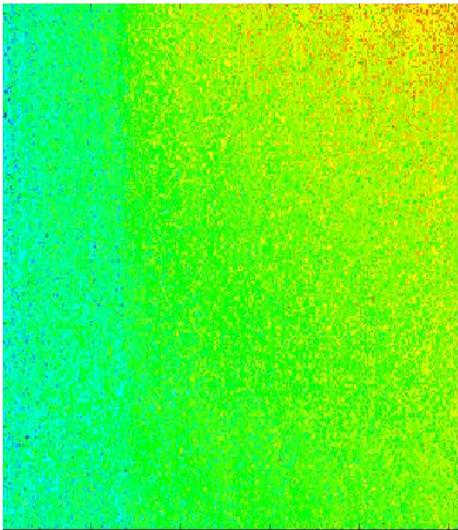
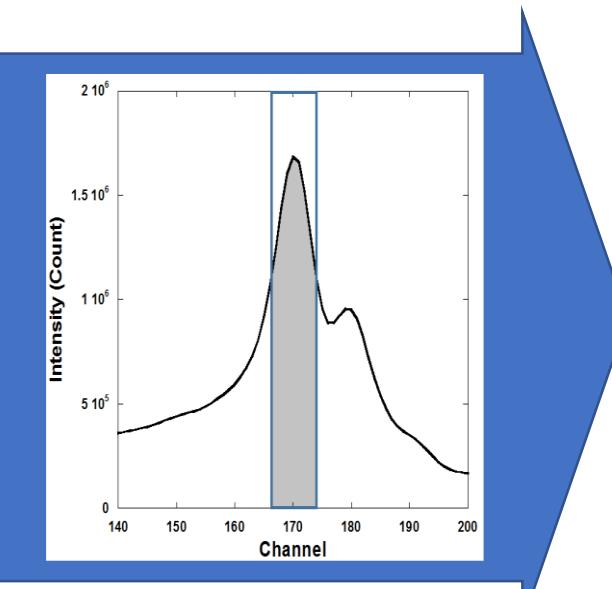
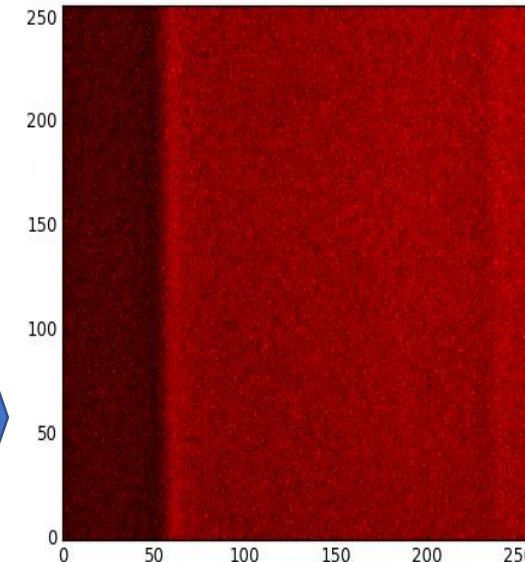


Image obtained  
with the intensity  
collected over the  
full spectral range



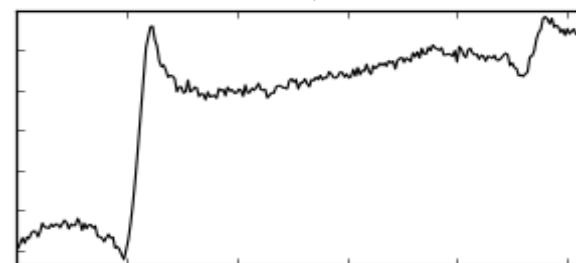
Selection of a spectral range  
(characteristic emission)

Kossel curve  
Angular distribution of the intensity of  
a characteristic x-ray line



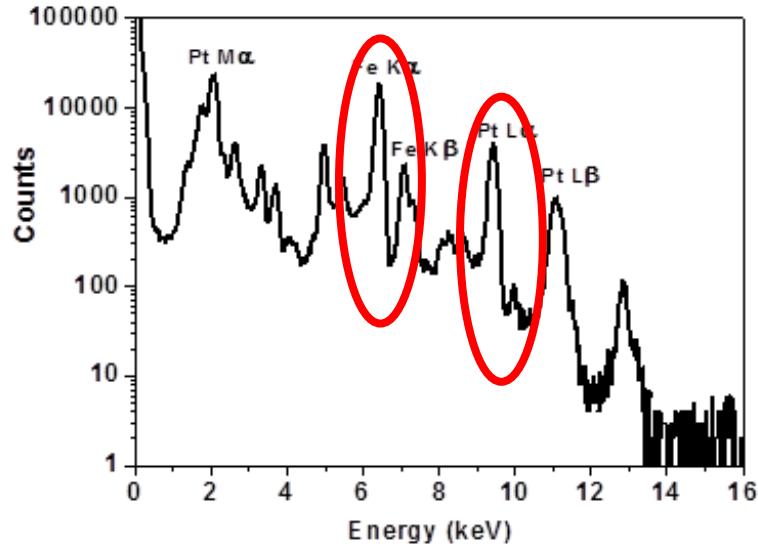
Spectral image

Column  
integration



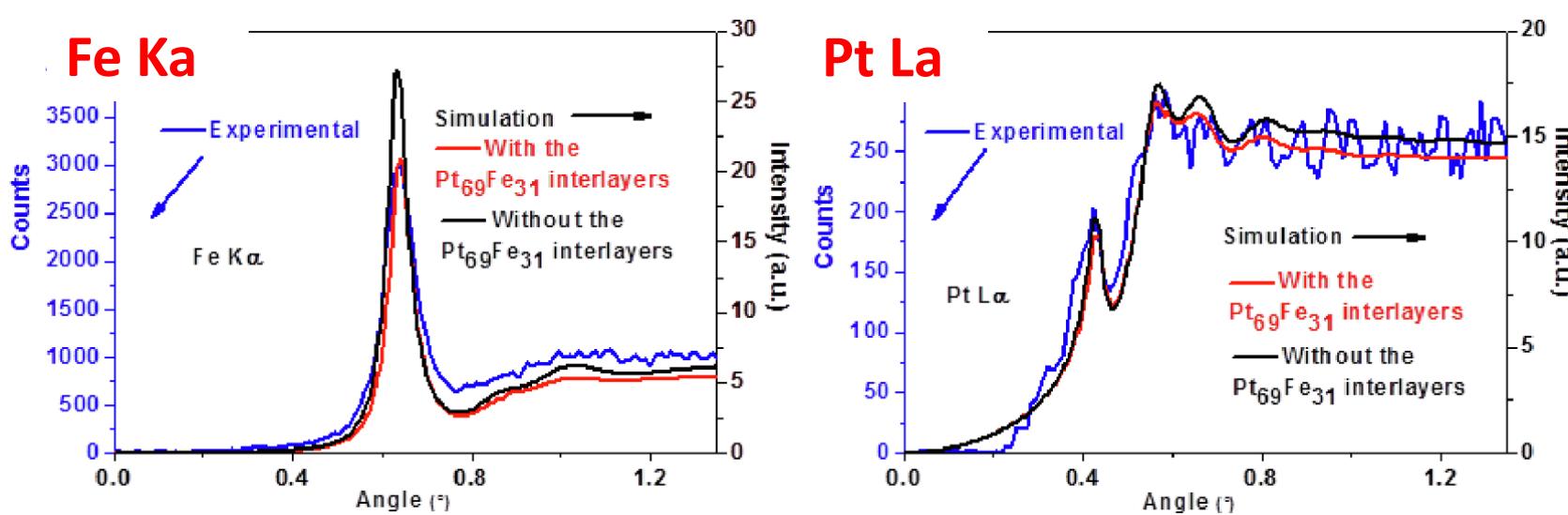
Detection angle

# PIXE – Kossel

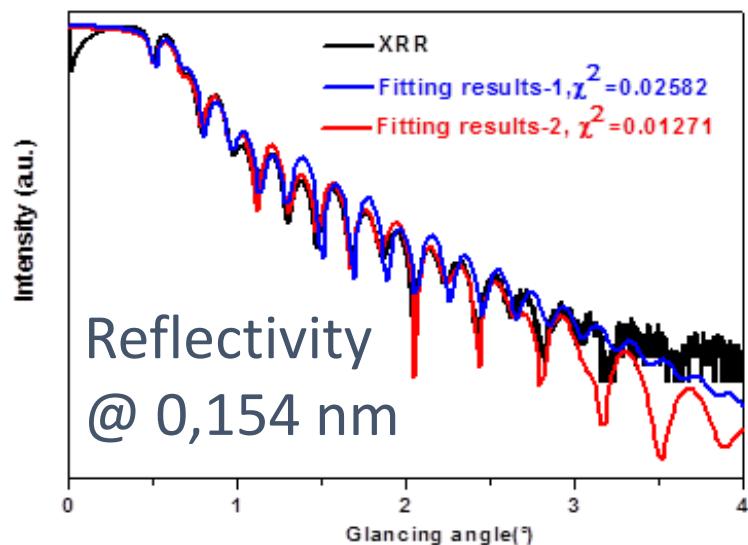
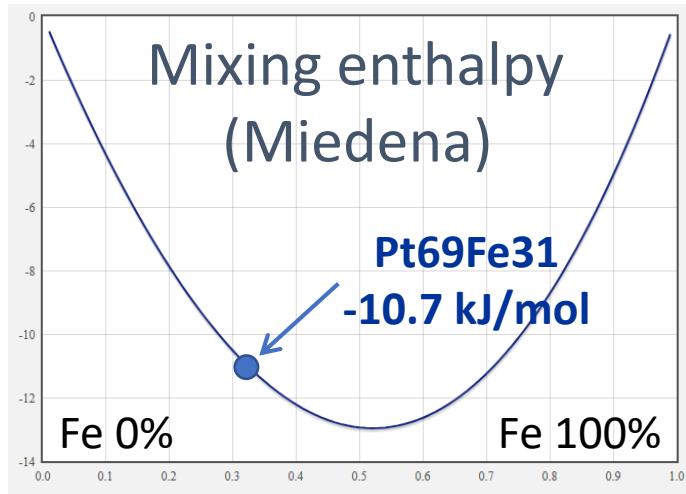


X-ray spectrum of the  
**Pt/Fe/Pt waveguide**  
Fe Ka and Pt La emissions

## Kossel curves



# PIXE – Kossel



Structure with 5 layers  
**Pt / PtFe / Fe / PtFe / Pt**

	Thickness / Roughness (nm) ( $\pm 0.05$ nm)	Density (g/cm <sup>3</sup> )
Pt	3.83 / 0.57	bulk
Pt <sub>69</sub> Fe <sub>31</sub>	1.62 / 0.90	16 ± 2
Fe	5.19 / 1.20	bulk
Pt <sub>69</sub> Fe <sub>31</sub>	1.41 / 0.79	16 ± 2
Pt	10.25 / 0.39	bulk
Si substrate	- / 0.50	bulk

Interfaces PtFe  
≈1.5 nm

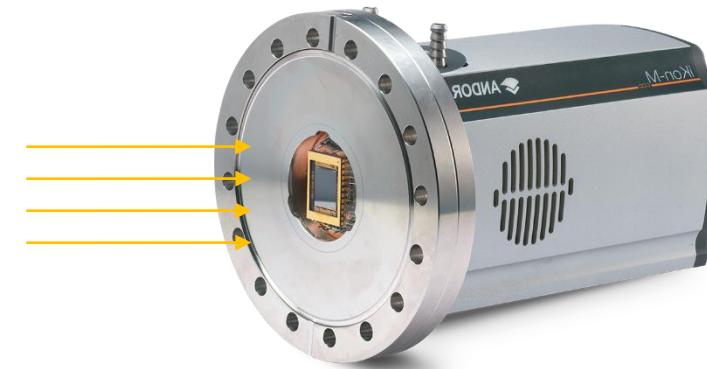
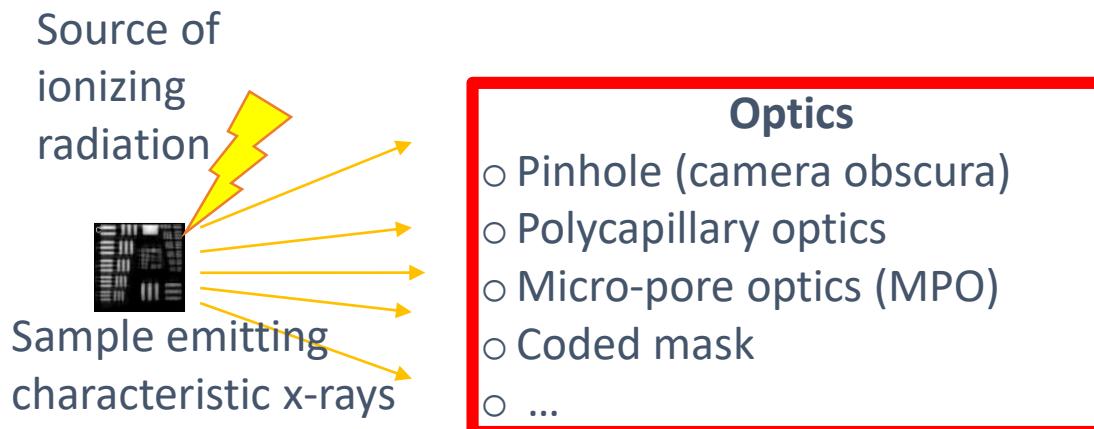
Energy dispersive CCD camera

PIXE – Kossel

Spectral imaging

# Spectral imaging

- **Full field x-ray fluorescence X (FF-XRF) in fact FF-PIXE!**
- Spectral imaging without scanning of the beam or sample
- Isotropic radiation of the characteristic emission
- Need to put an optics between the emitting sample and the detector (CCD camera)
- So that one pixel of the CCD corresponds to one point of the sample

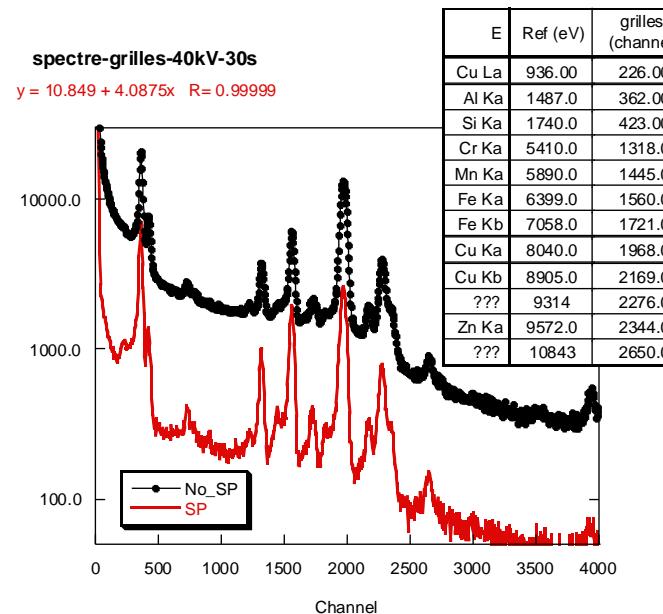


# Spectral imaging

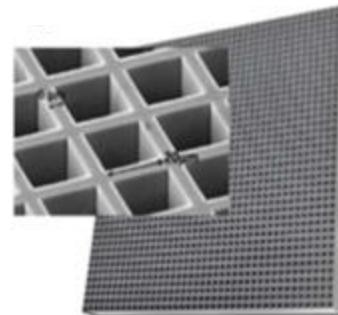
- First try with MPO at LCPMR with an x-ray tube (30 kV – 400 µA – 2h) FF-XRF



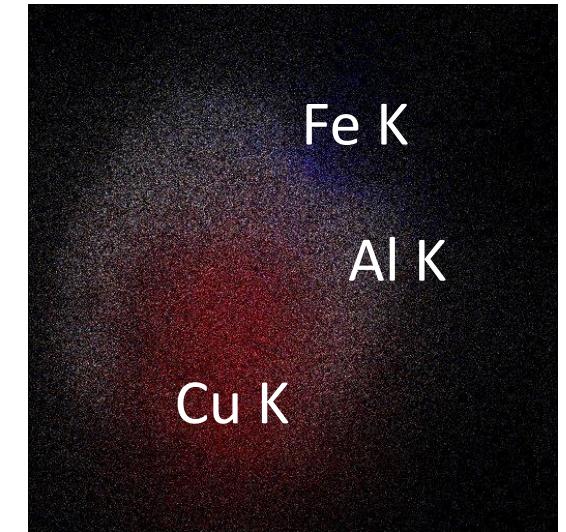
Visible image  
- Copper grid  
- Al sample holder  
- Iron screws



Emitted x-ray characteristic emissions



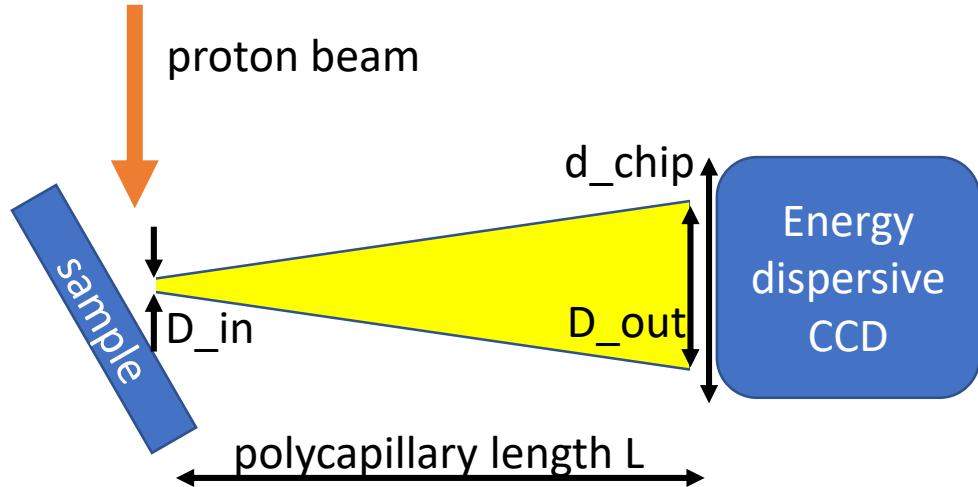
MPO



X-ray image  
Lot of scattered radiation  
Poor spatial resolution

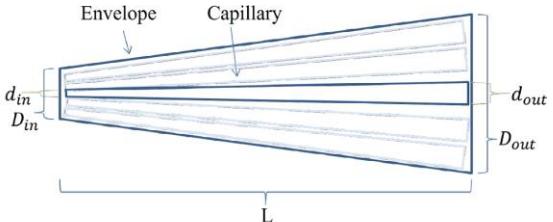
# Spectral imaging

- Project with a **conical polycapillary**



Spatial resolution depends on

- Size of pixels
- Diameter of capillaries
- Magnification
- Aberrations, ...



Magnification	20–40
$D_{in}$ [mm]	$0.42 \pm 0.02$
$D_{out}$ [mm]	6.56–0.2
$d_{in}$ [ $\mu\text{m}$ ]	$0.5 \pm 0.5$
$d_{out}$ [ $\mu\text{m}$ ]	$7 \pm 0.5$
$L$ [mm]	$53.34 - 0.5 \pm 0.2$

## We expect

- Spatial resolution  $1 \mu\text{m}$
- Large field of view  $1 \text{ mm} \times 1 \text{ mm}$
- Short acquisition time min to h
- Qualitative and quantitative mapping
- No scanning
- Study of geochemical markers of the environment obtained from biogenic carbonates (mollusc shells)

# Conclusion

- X-ray energy dispersive CCD is a versatile instrument spectroscopy – imaging
- PIXE-KOSSEL not only limited to periodic multilayers analysis of stacks of nanometer thin films
- Imaging possible without scanning
  - short acquisition time
  - large number of count – quantitative maps
  - possibility to deploy this tools on electron microscopes



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MERCI  
THANK YOU