The ODE beamline at SOLEIL: first results on XMCD and EXAFS under extreme conditions and kinetics experiments

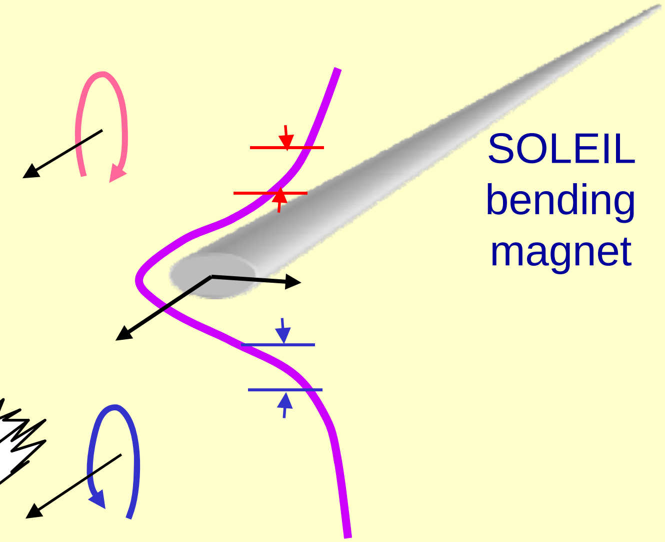
Alberta Congeduti, Qingyu Kong, Sébastien Chagnot, Alexandre Monza,
Gwenaëlle Abeille, Aurélien Delmotte, Olga Roudenko, and François Baudelet

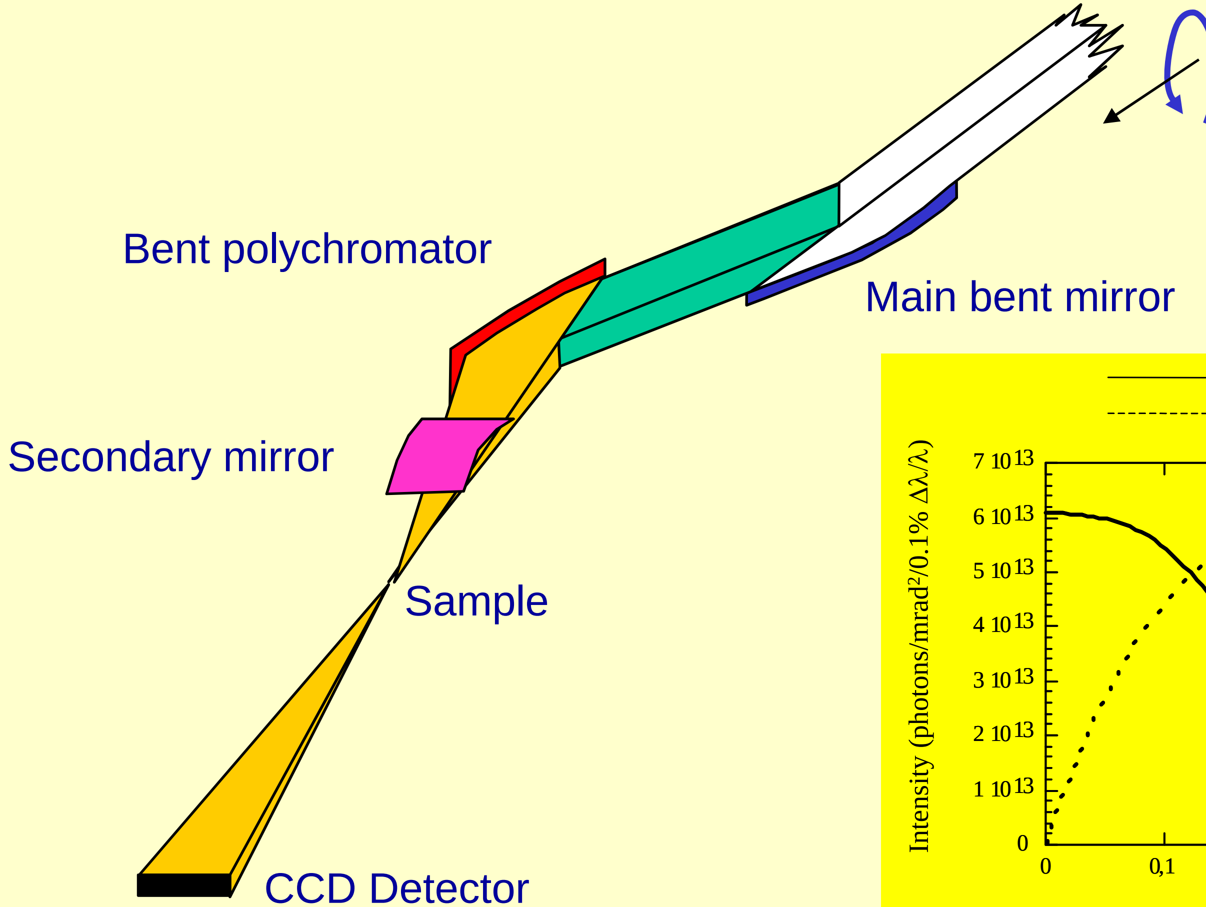
Source: Bending Magnet

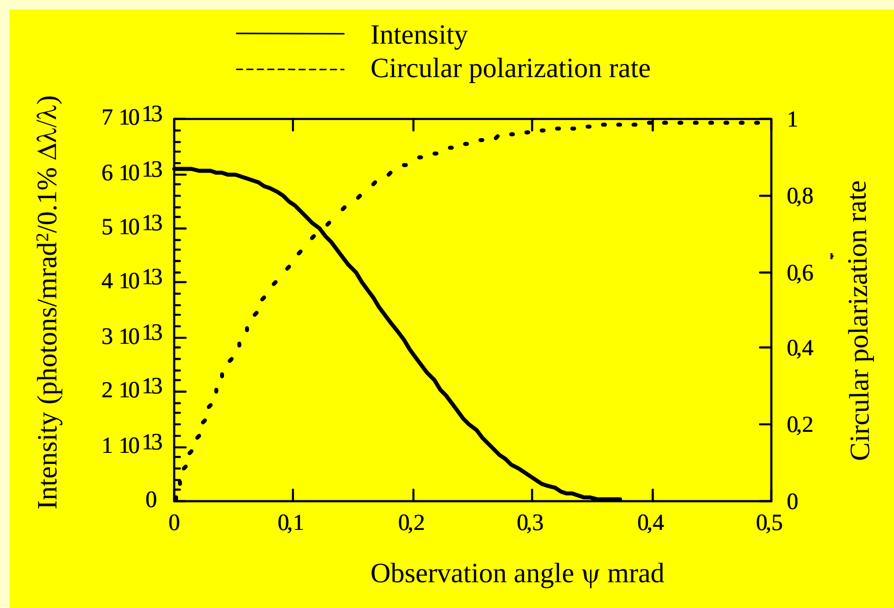
⇒ intrinsic angular aperture → few optical elements, short optical paths → huge stability

⇒ absence of spatial coherency effects

⇒ spatial distribution of X-ray polarization





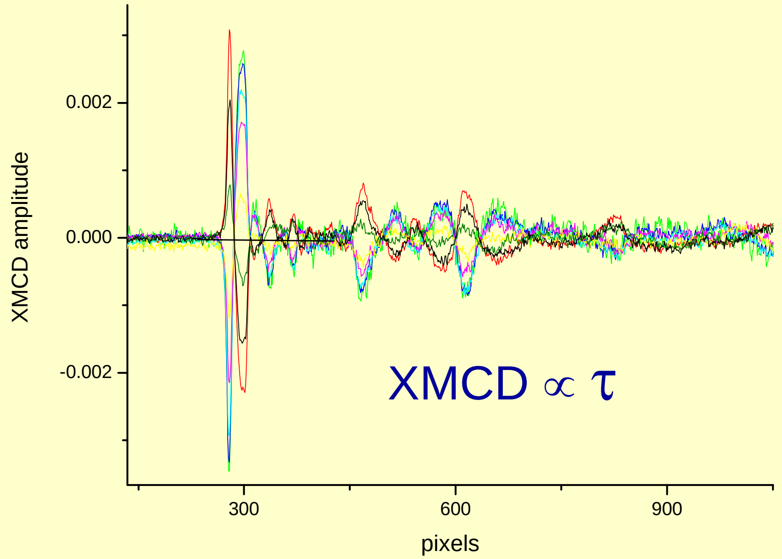


ODE Bending Magnet Circular Polarization

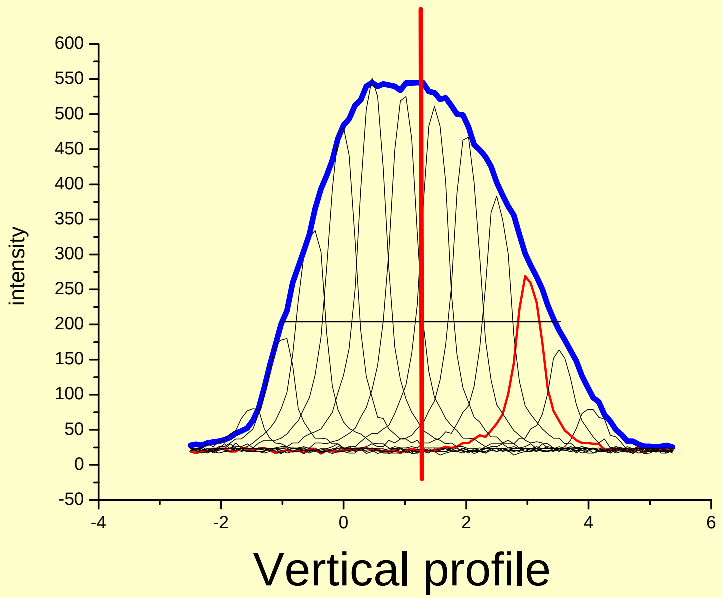
Fe K edge 7112 eV

τ circular polarization rate

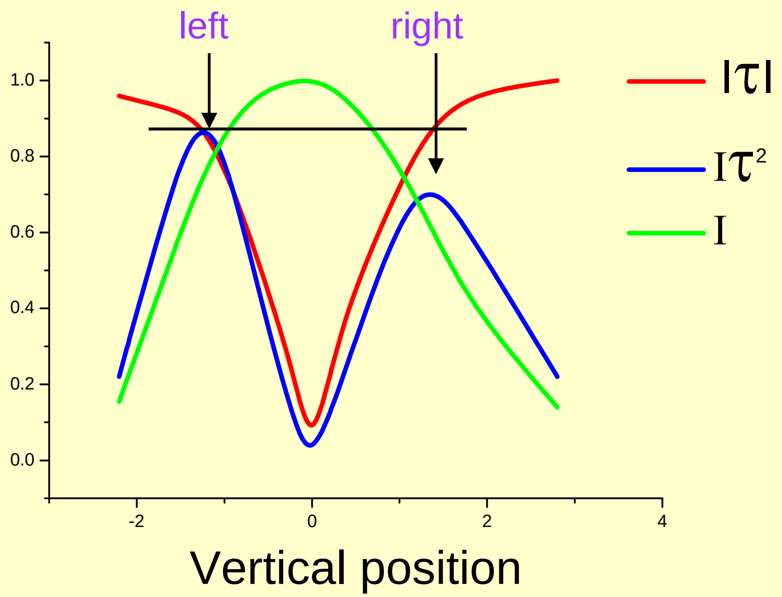
$I\tau^2$ merit factor



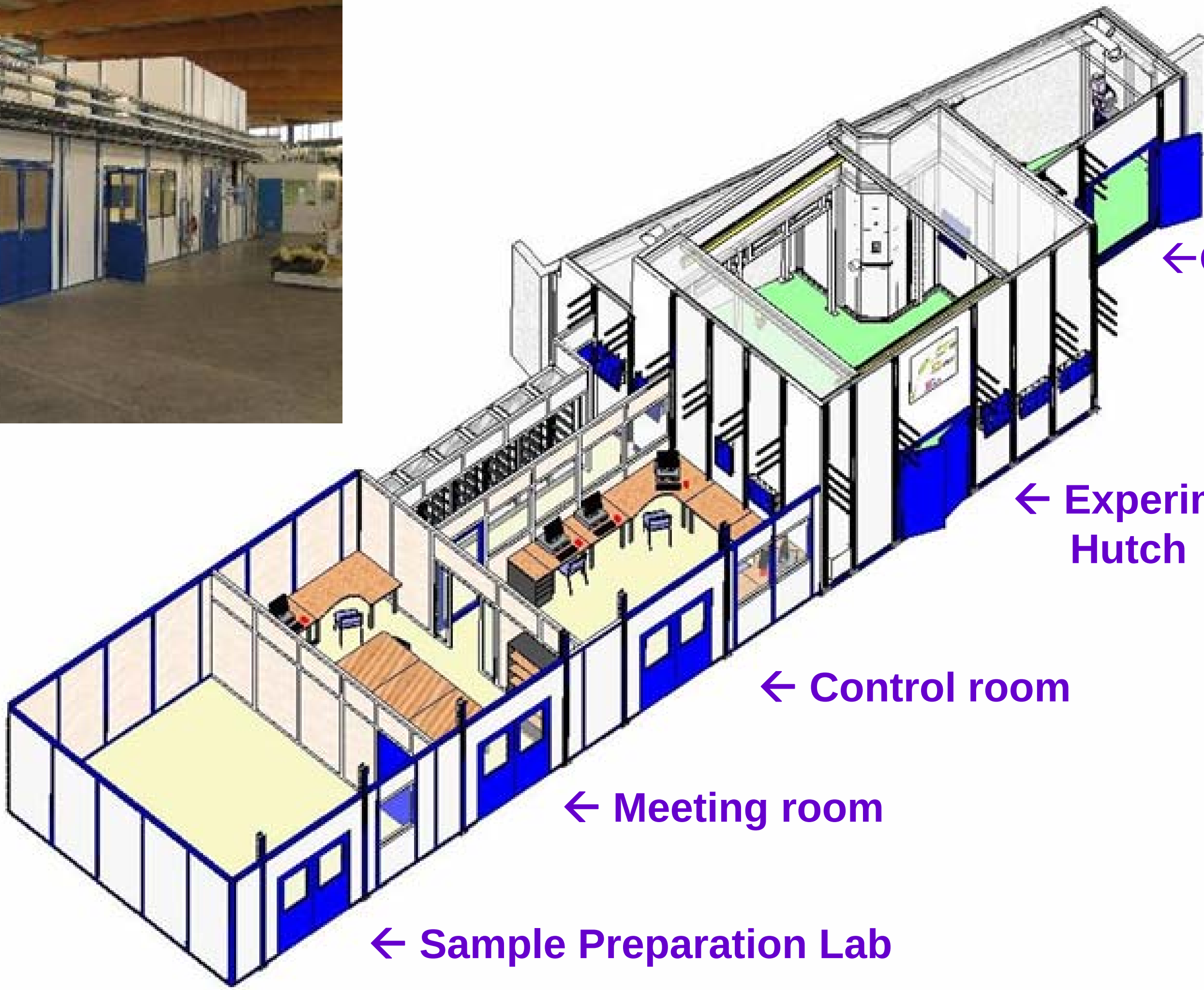
Intensity I



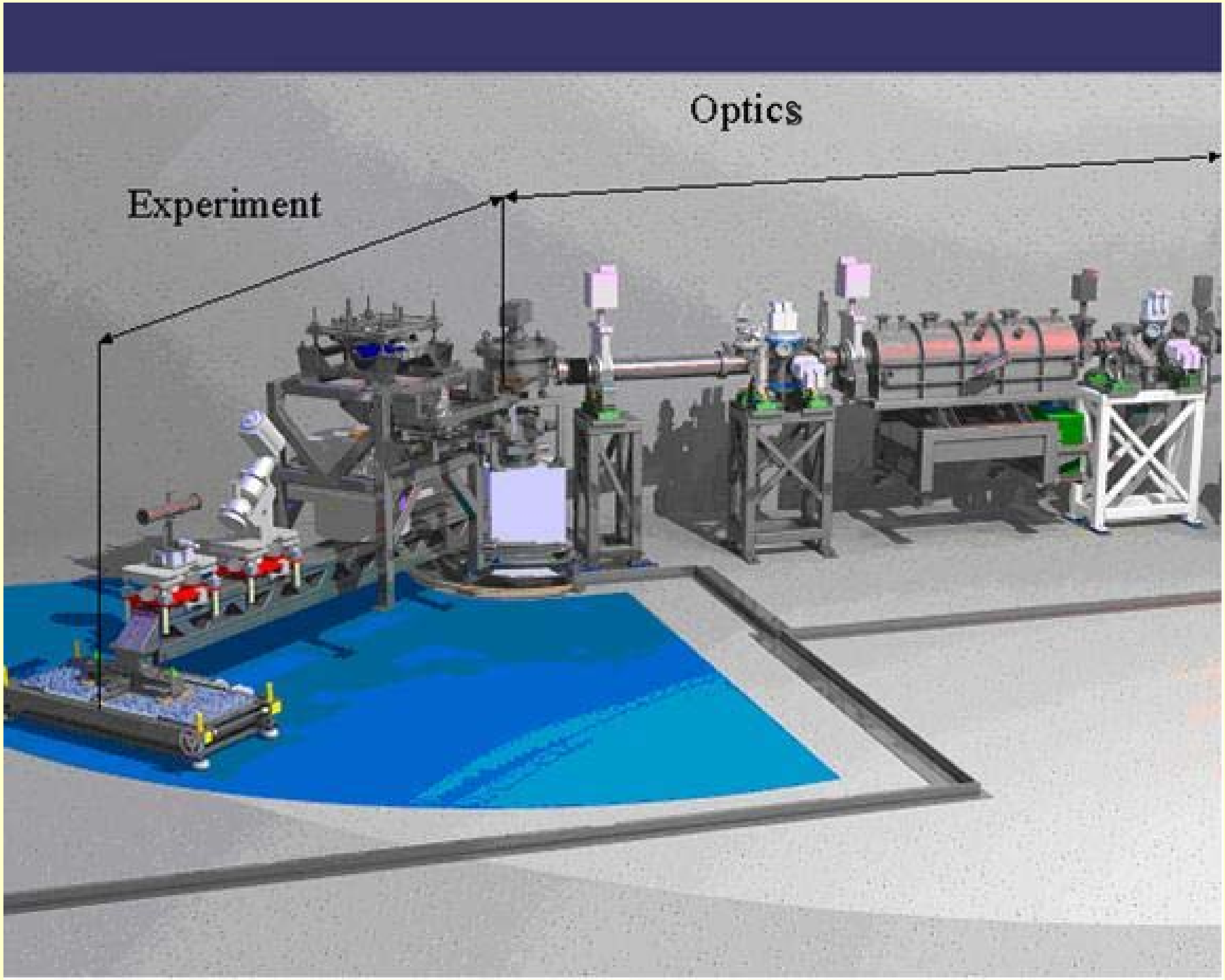
Best positions



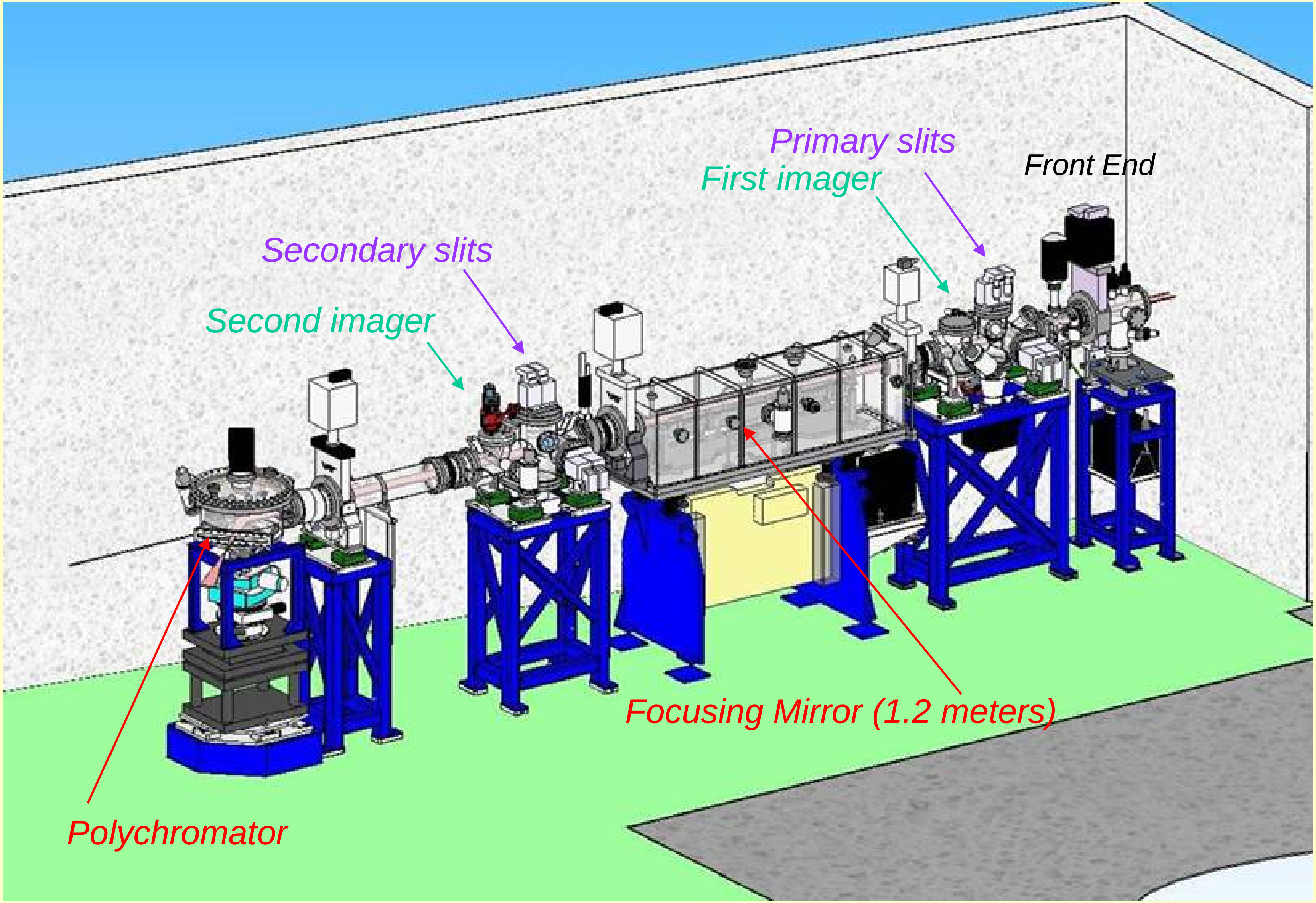
ODE layout: hutches and control rooms



ODE layout: optics and experiment



Optics

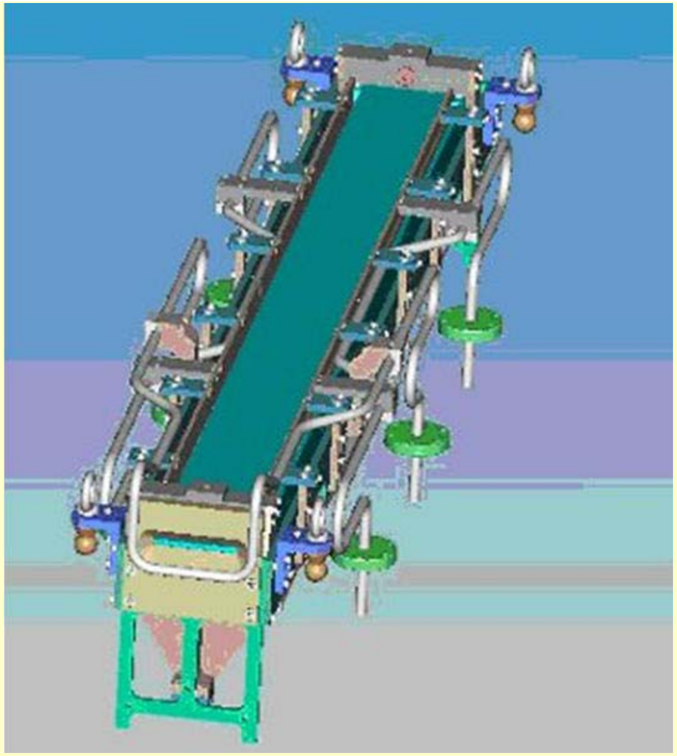


Primary slits
First imager
Front End
Secondary slits
Second imager
Focusing Mirror (1.2 meters)
Polychromator

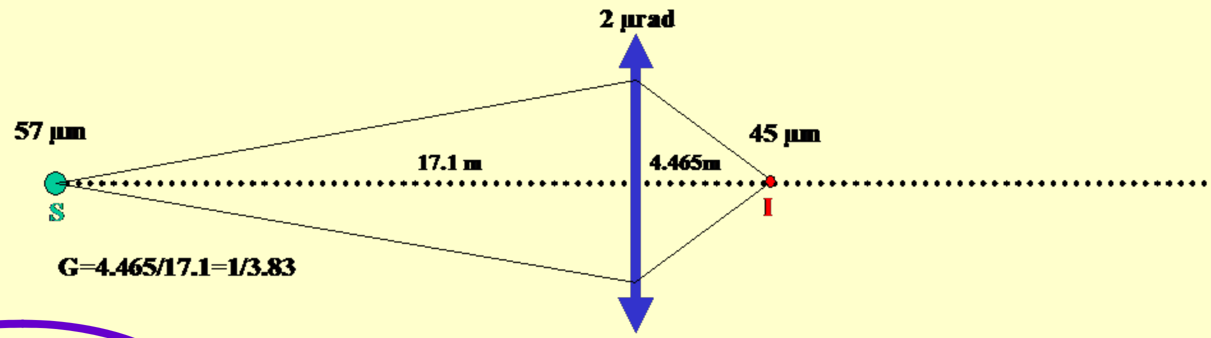
Optics: Focusing mirror

Winlight System

Useful area : 88 x 1200 mm
Coating Pd / Si
Slope error : 1.0 µrad RMS on 1m
Roughness : 1 à 2 Å RMS
Double bender → Curvature radius 0.8 km to ∞
Water cooled (InGaSn bath)
Focusing 8 mm → 35 µm



Optics: Vertical focusing

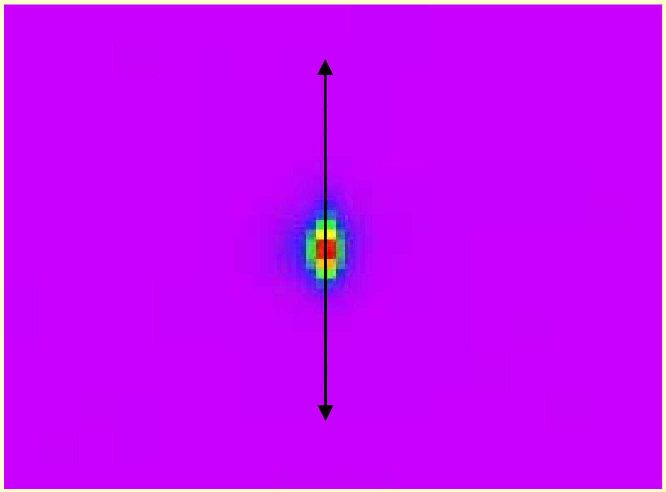


$$I = \sqrt{(S \times G)^2 + (2 \times 2.35 \times 2\mu rad \times 4.465m)^2}$$

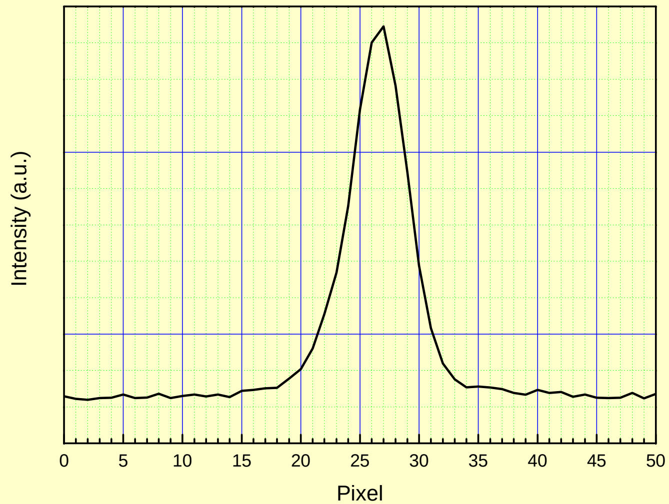
$$I \approx 45\ \mu m$$

Optical simulations by
Mourad Idir Thierry Moreno

Real Focus Image

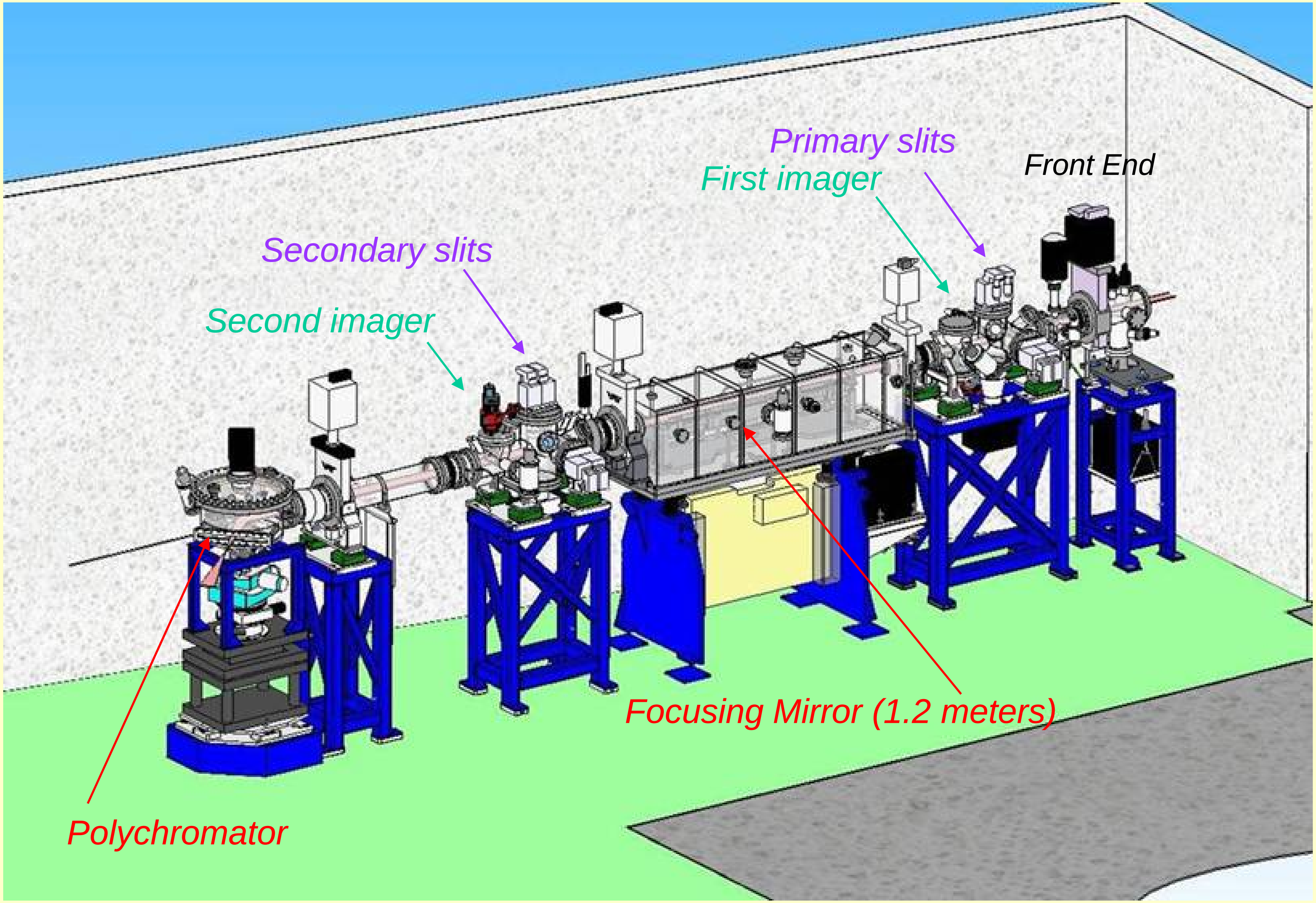


Real Vertical Profile



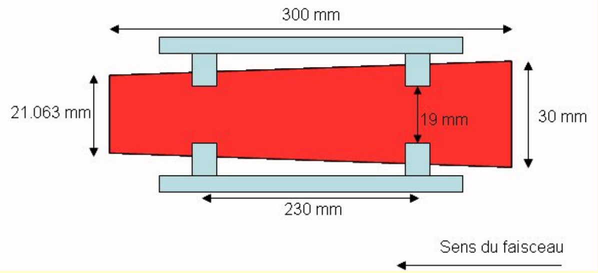
1 pixel = 7 µm → measured FWHM = 35 µm

Optics

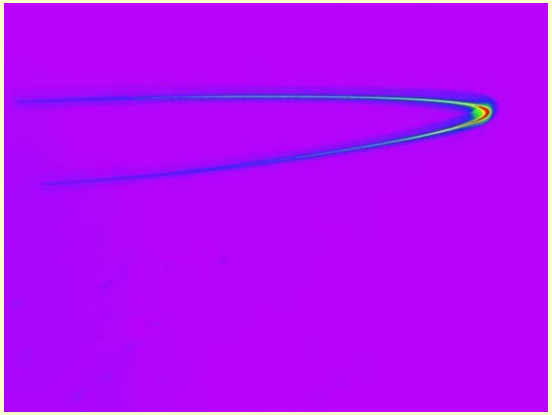

Primary slits
First imager
Front End
Secondary slits
Second imager
Focusing Mirror (1.2 meters)
Polychromator

Optics: polychromator focusing

Si 111 and 311

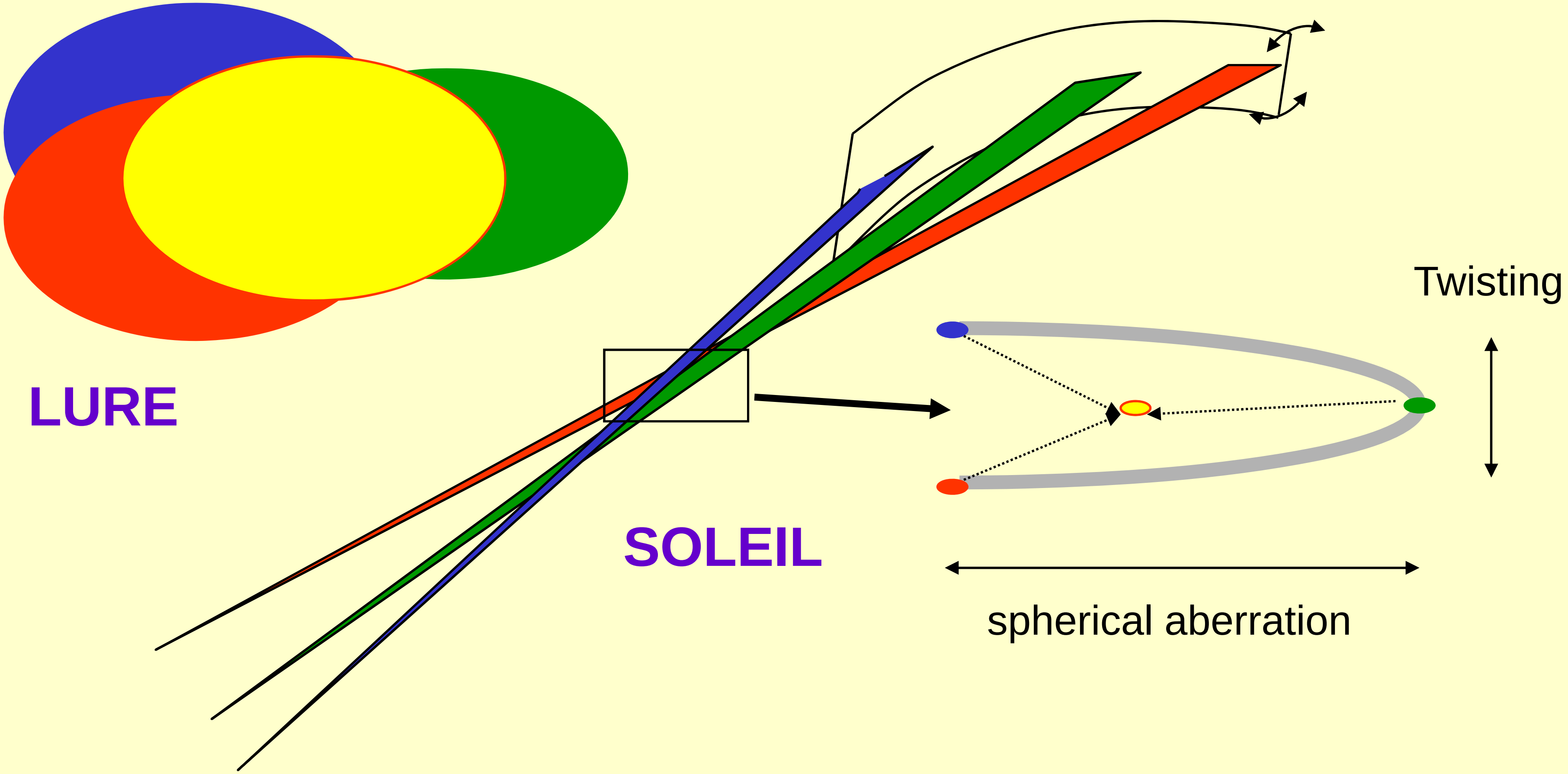


Thierry Moreno and Mourad Idir



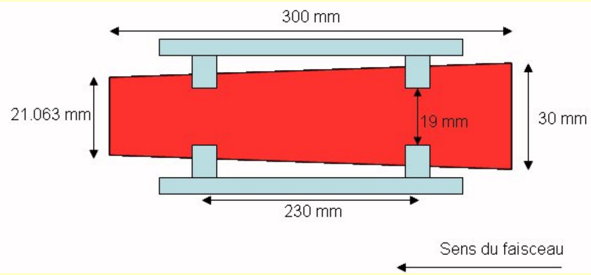
Optics: polychromator focusing

	σ_x (µm)	σ_z (µm)	σ'_x (µrad)	σ'_z (µrad)
Soleil	60.1	24.9	134.8	2.1
Lure	2500	1580	1070	170

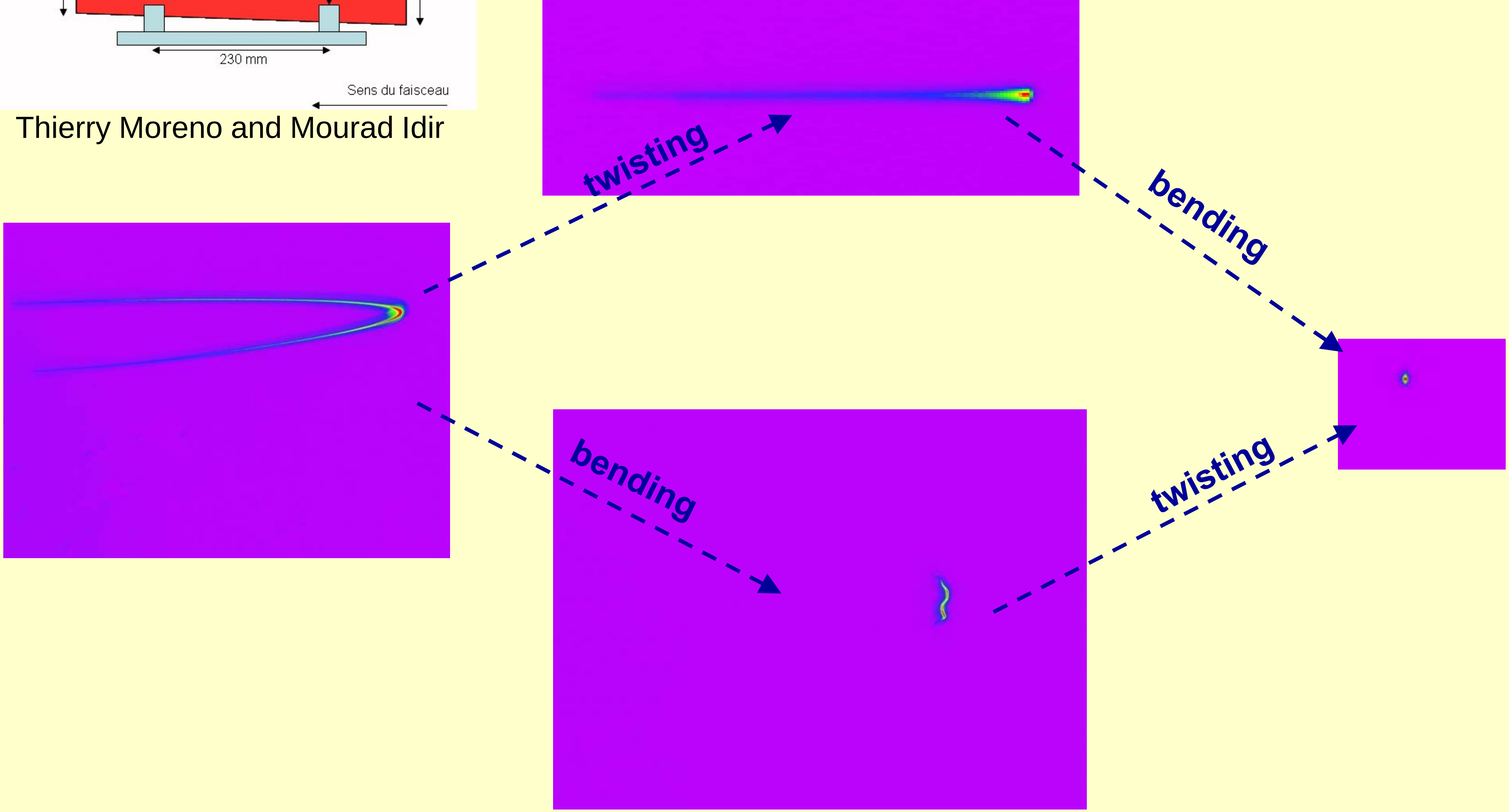


Optics: polychromator focusing

Si 111 and 311

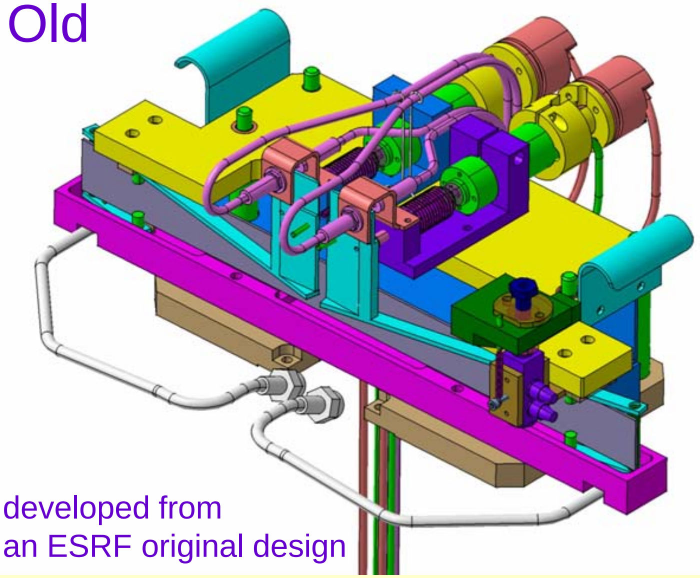


Thierry Moreno and Mourad Idir



Optics: bender mechanical improvements

Old

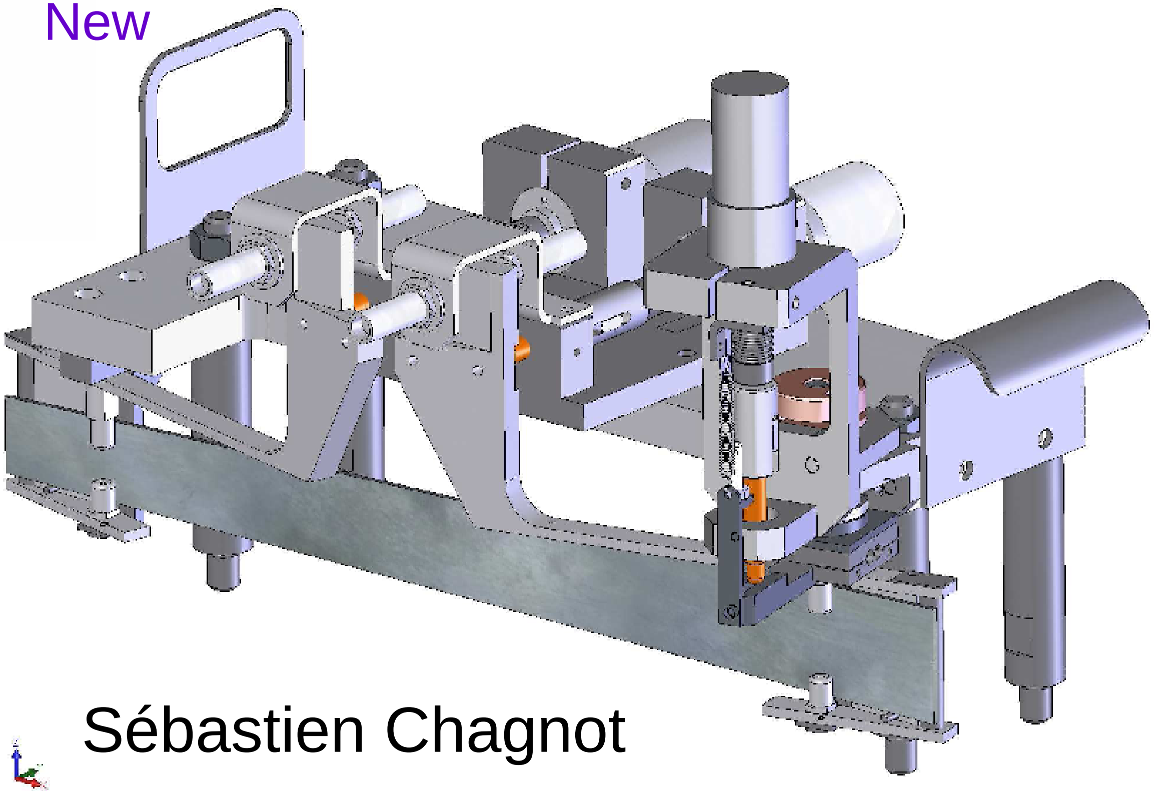


developed from
an ESRF original design

- Better contact between blade and bender
- Decoupling twisting and bending movements

$\Rightarrow$ Very small spot size

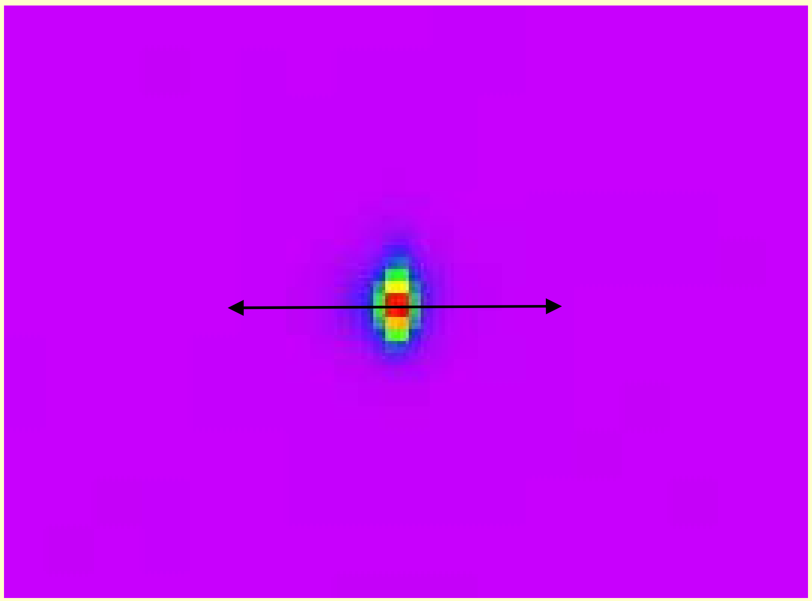
New



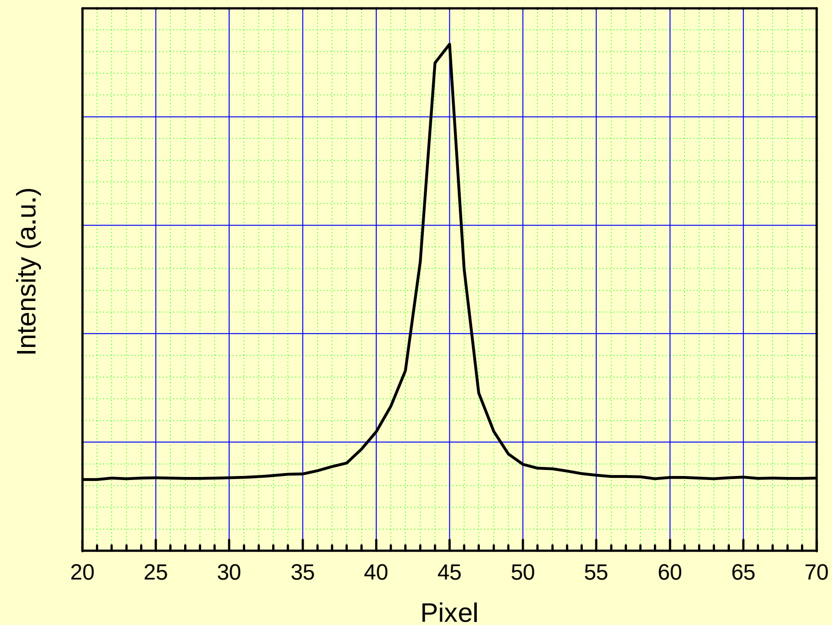
Sébastien Chagnot

Optics: 311 focus @ Fe K-edge

Focus Image

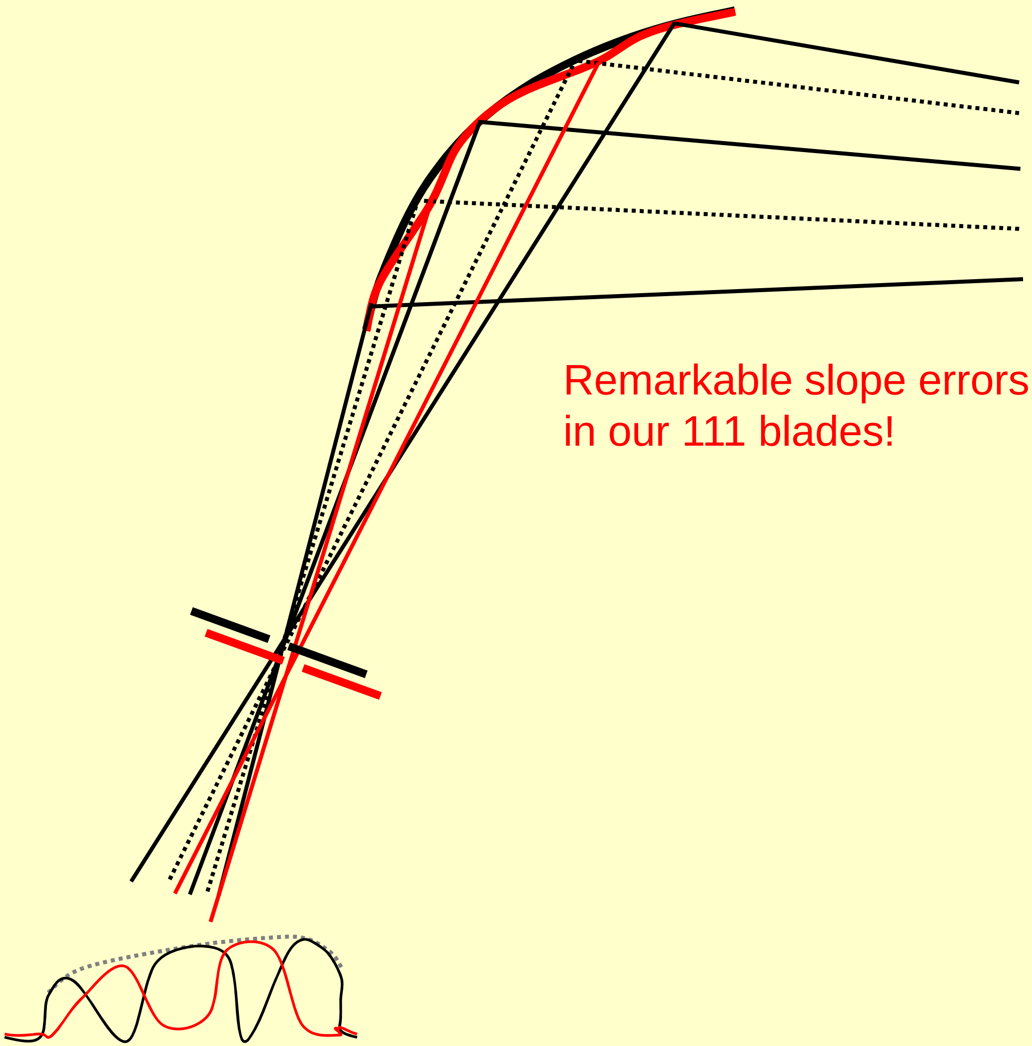


Horizontal Profile

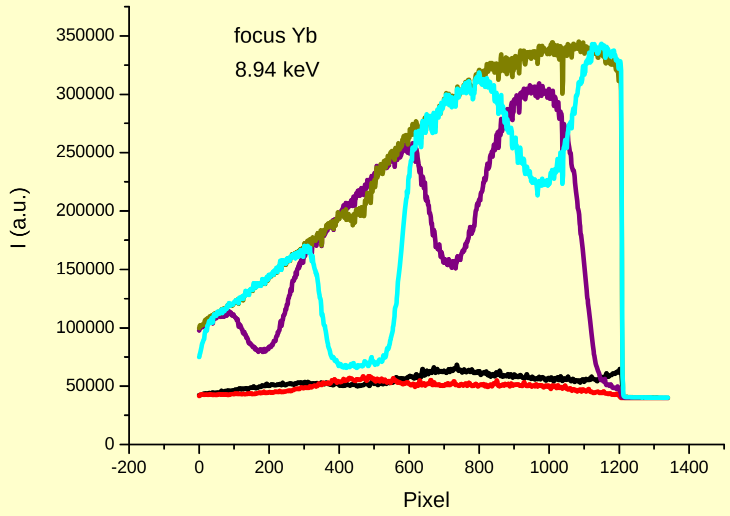


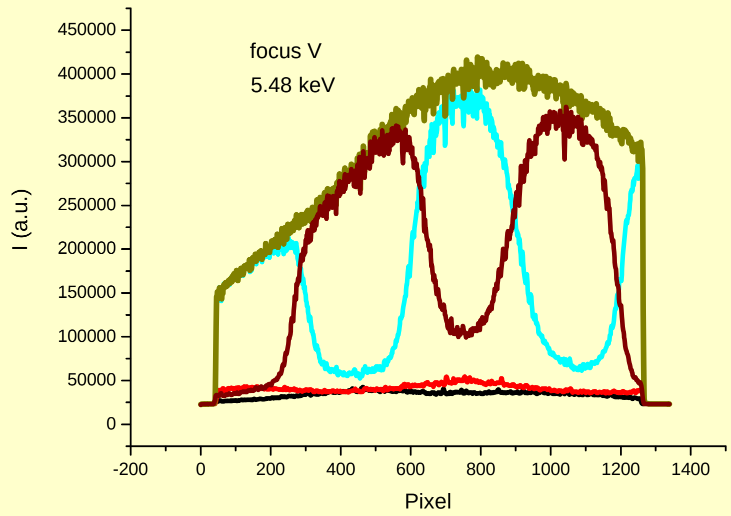
1 pixel = 7 µm → FWHM 20 µm

Optics: high sensitivity to slope errors



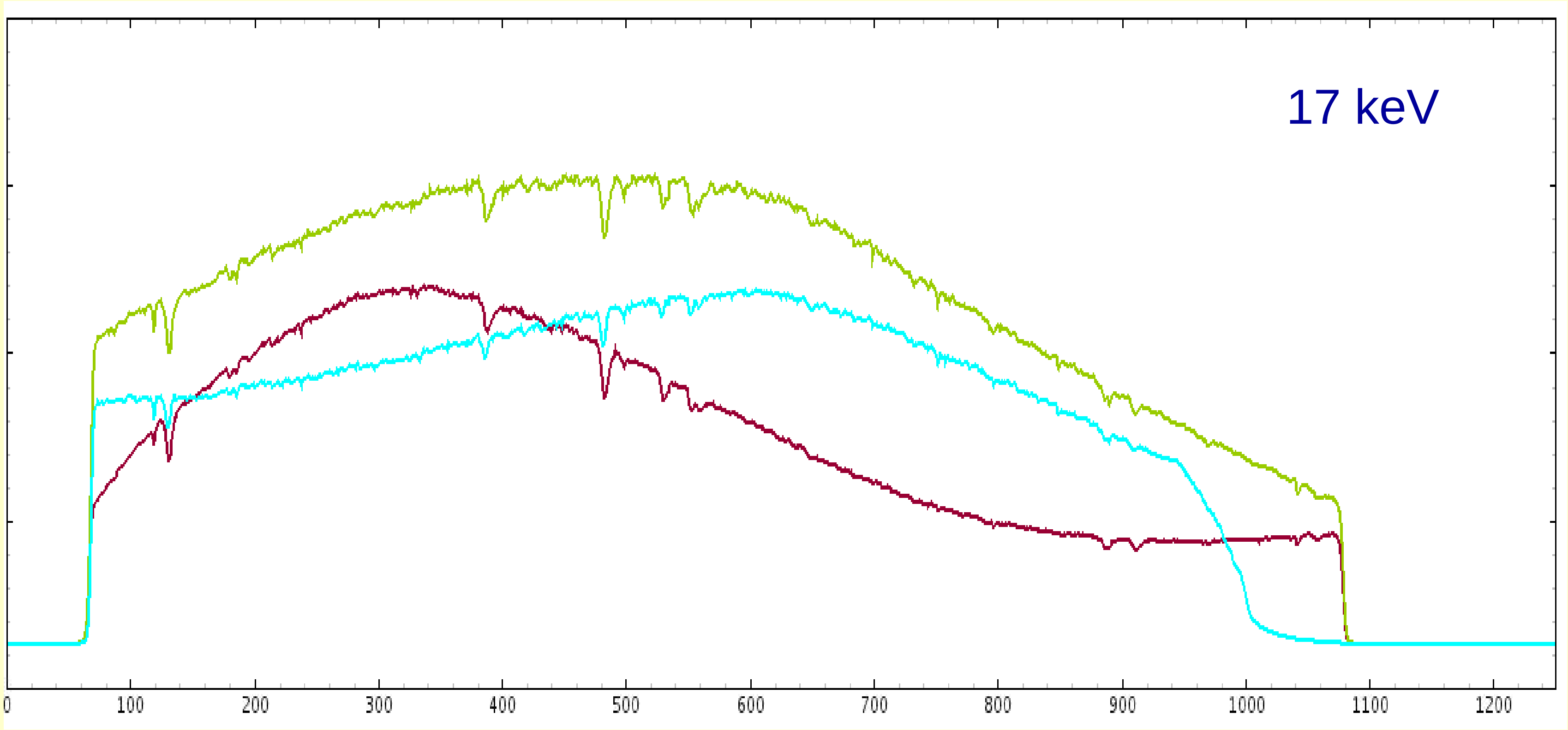
Remarkable slope errors
in our 111 blades!



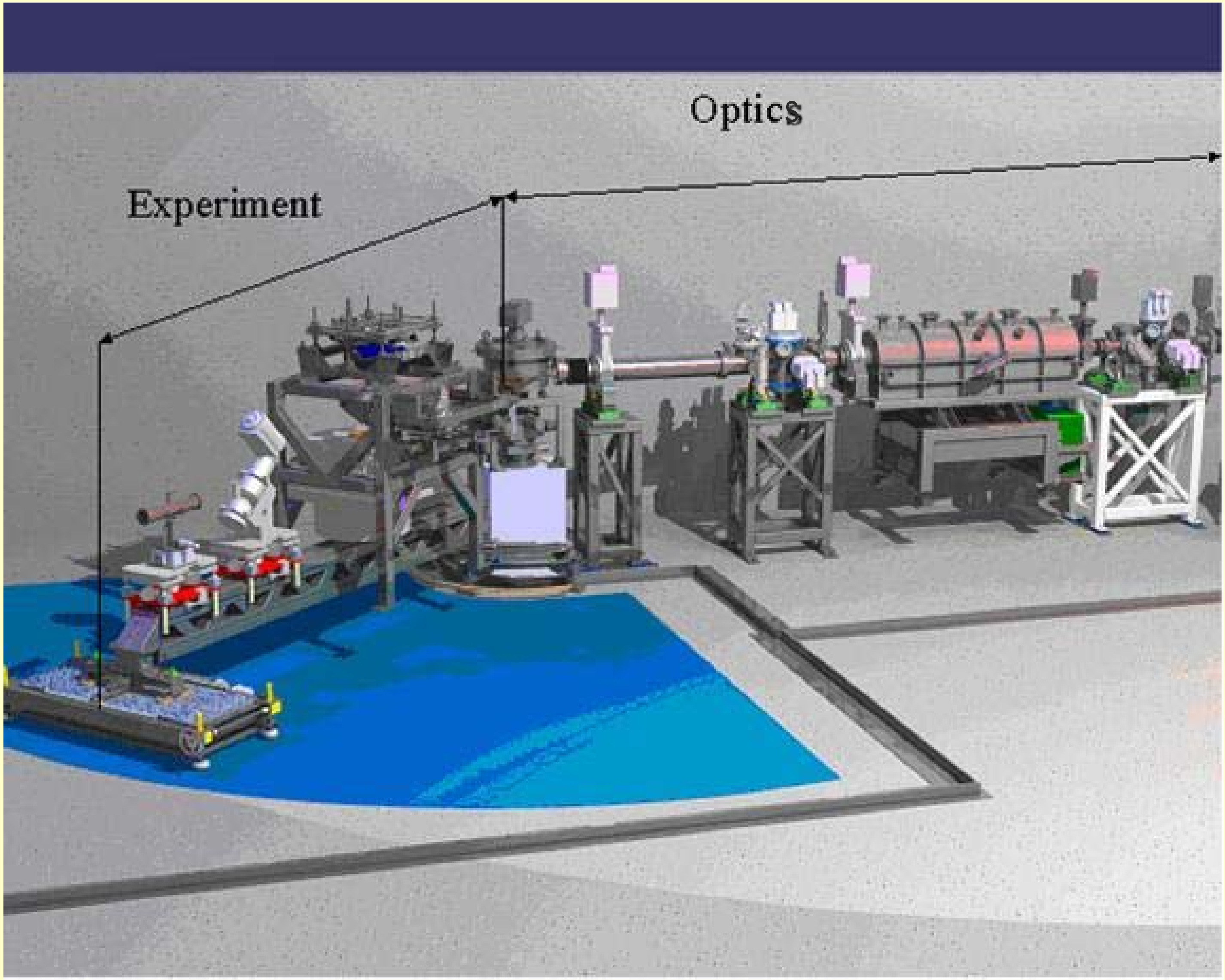


More details in Thierry Moreno's talk

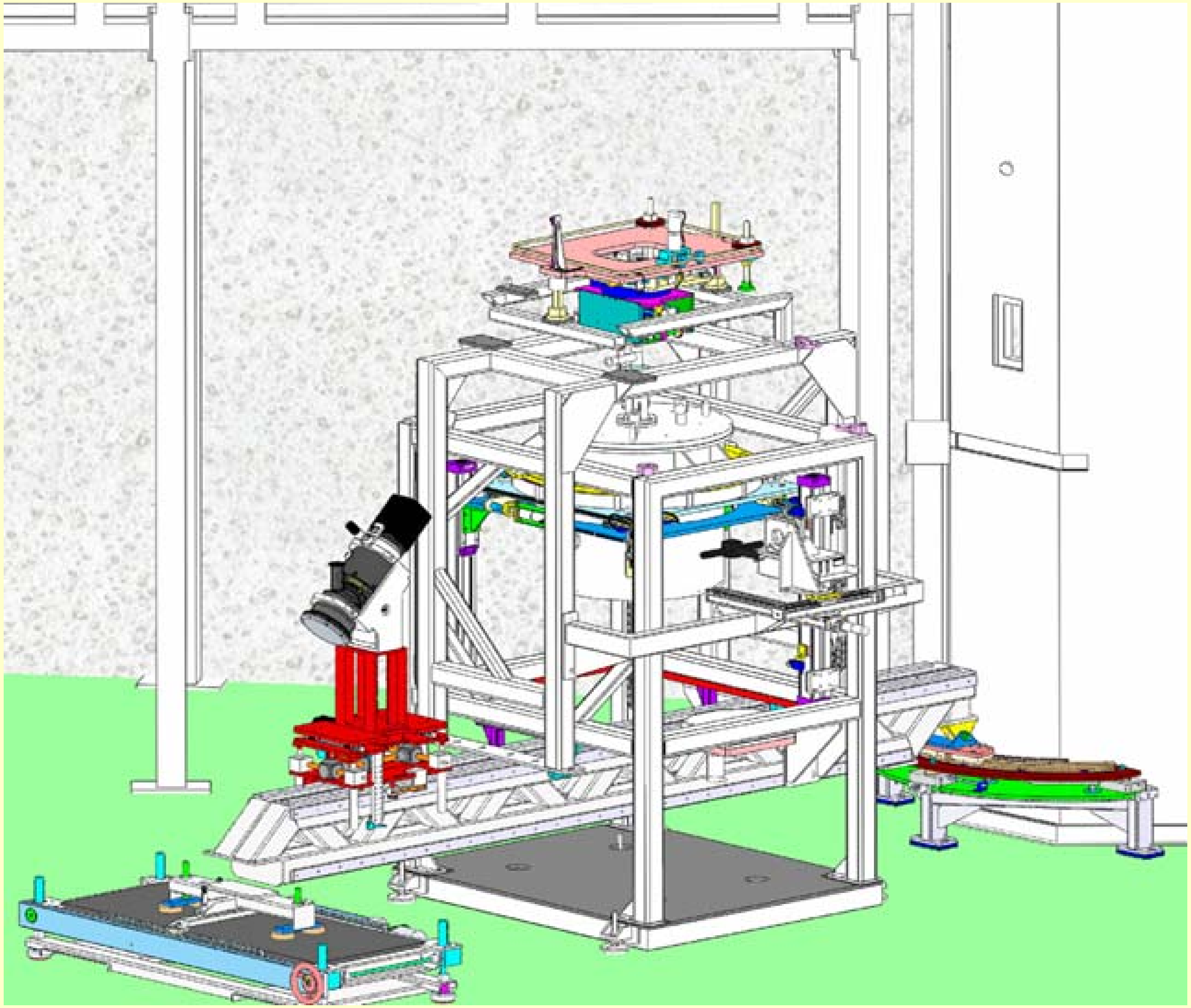
Optics: more homogeneous focus with the new Si 311 blade



ODE layout: optics and experiment



Sample environment



Multipurpose sample environment adapted to various kinds of studies: pressure, temperature, magnetic field, gas or liquid environment

Sample Environment:

in situ Pressure Measurement

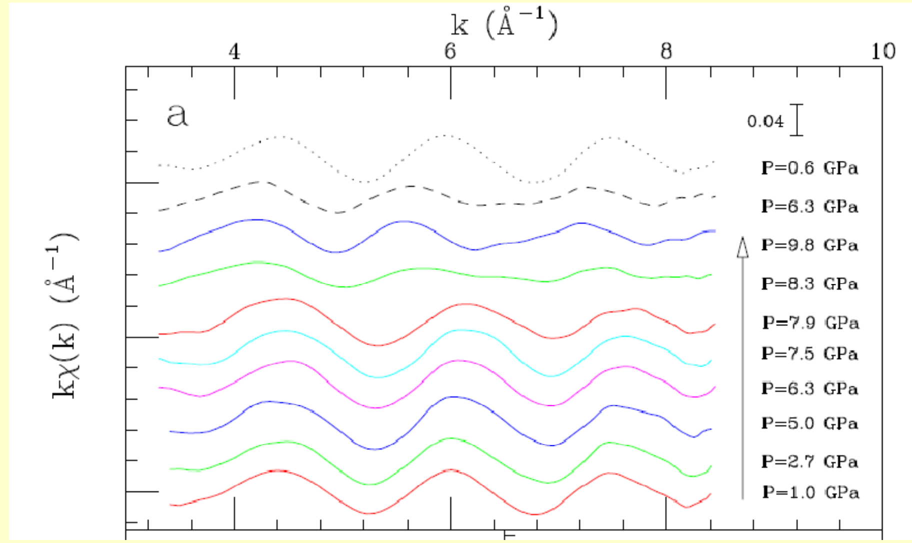
Results: EXAFS STUDY OF a-Ge AT HIGH PRESSURE

ODE's first measurement in Diamond Anvil Cell (0 – 10 GPa) at the Ge K-edge (11100 eV)

First high-pressure measurements in a diamond anvil cell (DAC) in dispersive mode using ODE beam-line at Soleil. Ge K-edge XAS a-Ge films of about 3 x 3 μm thickness (obtained by evaporation).

The films were amorphous as confirmed by XRD.

Spectra taken with less than 1 s of integration.

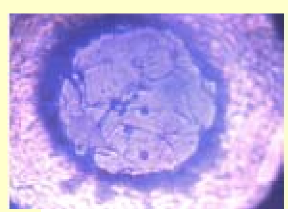


STRUCTURAL MODIFICATIONS:

- A clear transition is evidenced above 7.9 GPa.
- The spectra at 8.3 GPa and 9.8 GPa are different.
- The weak XAS structural signal obtained at 8.3 GPa is compatible with the presence of strong structural disorder (different amorphous phase).
- The strong signal at 9.8 GPa is compatible with a crystalline structure with elongated first-neighbour distances (like Ge II)

- At ~ 8 GPa: the surface shows a metal-like reflectivity, loss of the Raman signal and strong diffuse scattering.

P=7.5 GPa



P=7.9 GPa

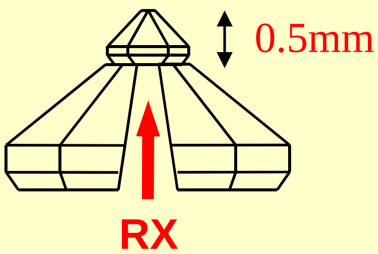
P=8.3 GPa

Di Cicco et al.Phys. Rev. B **78**, 033309 (2008)

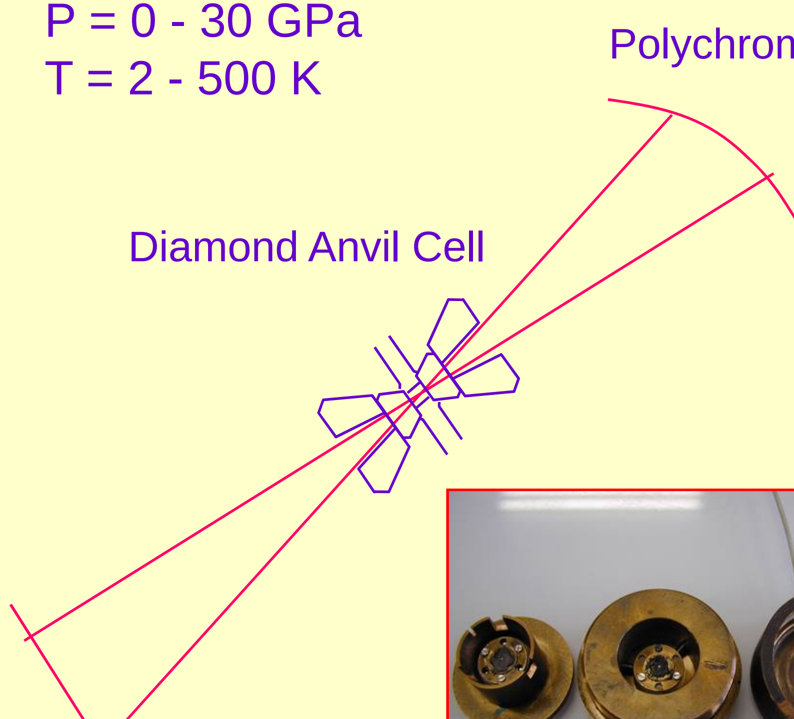
Andrea Di Cicco, Alberta Congeduti, Federica Coppari, Jean Claude Chervin, Francois Baudelet, Alain Polian

Sample Environment: Drilled Diamond Anvil Cell for Low Energy HP measurement

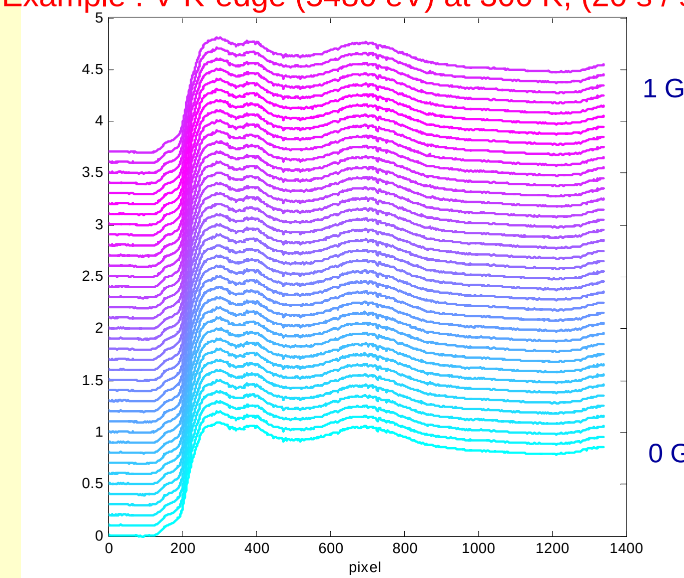
Drilled diamond cell ⇒ thickness reduced of a factor 2.5
⇒ e.g. a factor ~150 on the transmitted intensity at the Mn k-edge



P = 0 - 30 GPa
T = 2 - 500 K



• Example : V K-edge (5480 eV) at 300 K, (20 s / scan)



F. Rodolakis (LPS / SOLEIL), J.-P. Rueff (SOLEIL / LCPMR), M. Marsi (LPS)

Collaboration with Bernard Couzinet IMPMC

Sample Environment:

in situ Pressure Measurement

2T Magnetic Coil for XMCD

+ Fast Feadback

Results: Magnetic transitions under pressure in magnetite

0 – 30 GPa, 2T

Verwey transition disappears at 8 GPa

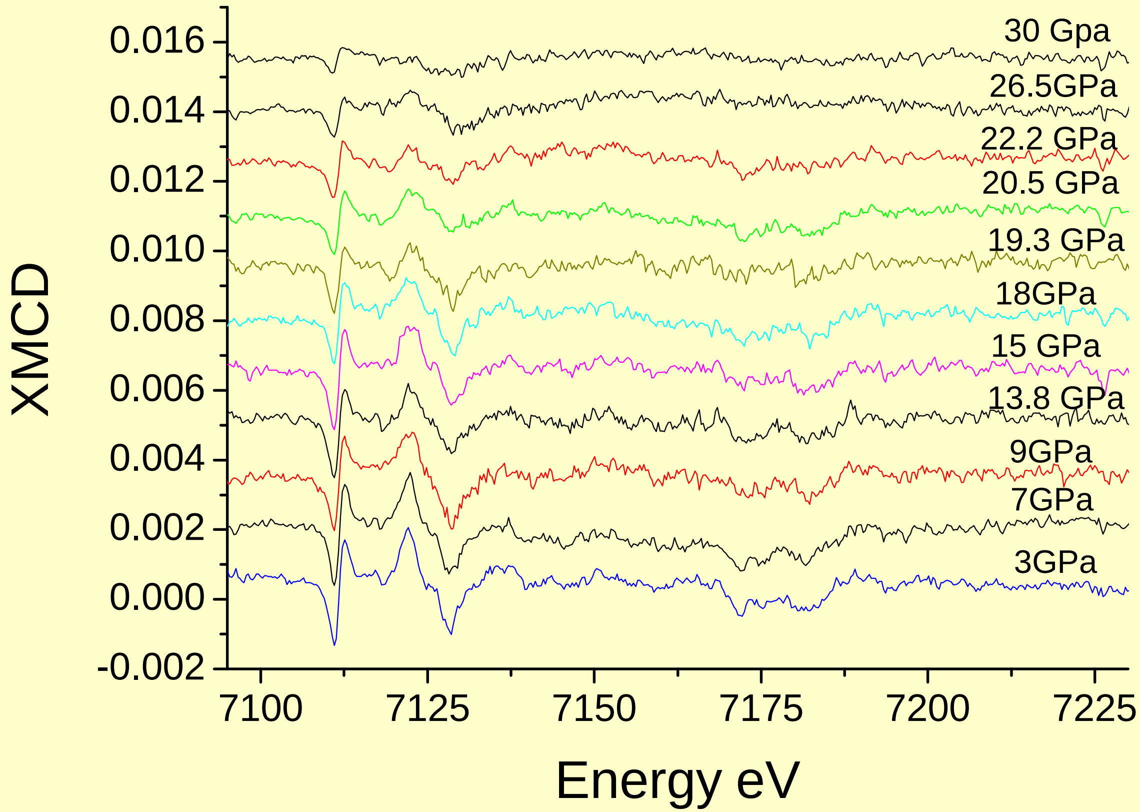
S. Todo et al. J. Appl. Phys. **89**, 11 7347 (2001)

Transition from indirect to direct spinel at 8 GPa

G. Kh. Rozenberg, et al. PRL **96**, 045705 (2006)

Only an abrupt magnetic transition between 12 and 16 GPa

Yang Ding et al. PRL **100** 045508 (2000)



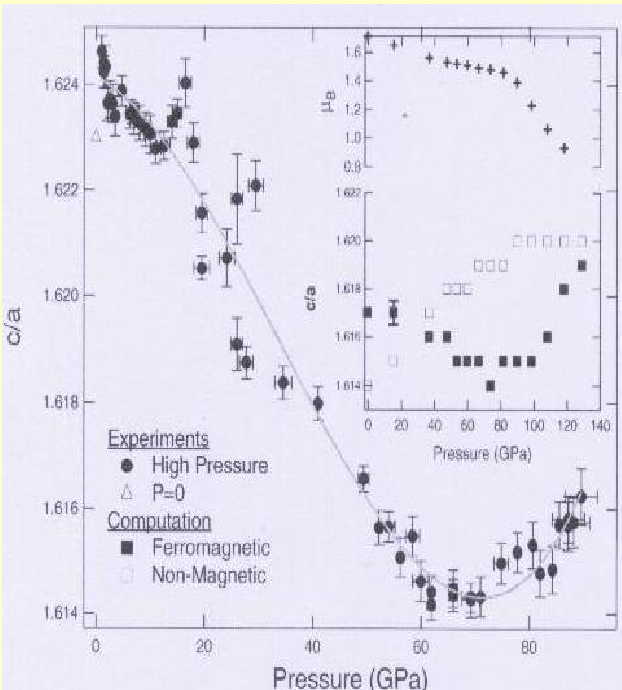
No transition from indirect to direct spinel but a continuous decrease of the magnetic moment between 8 and 30 GPa

F. Baudelet, O. Mathon, J.P.Itié, S.Pascarelli, A.Polian, M. d'Astuto and J.C. Chervin

Results: XMCD on Co at HIGH PRESSURE

0 – 94 GPa, 2T

Anomalous c/a ratio behaviour at HP

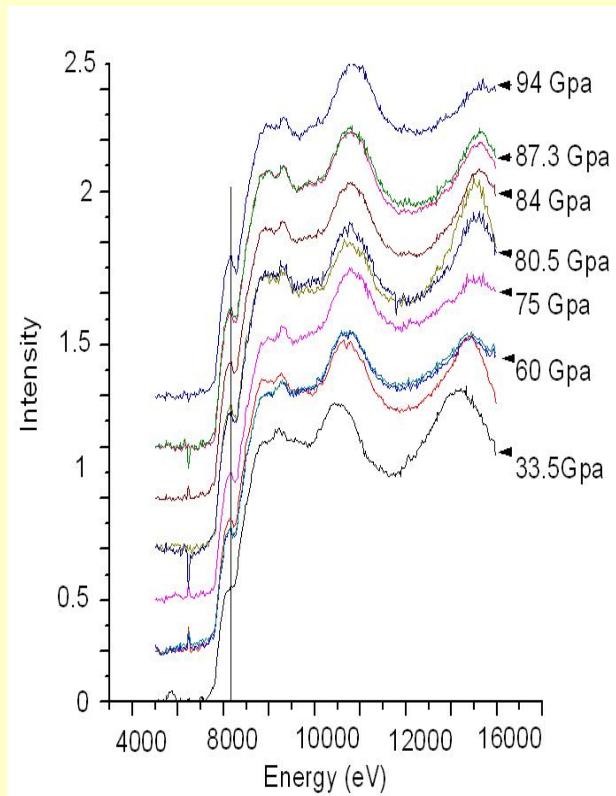


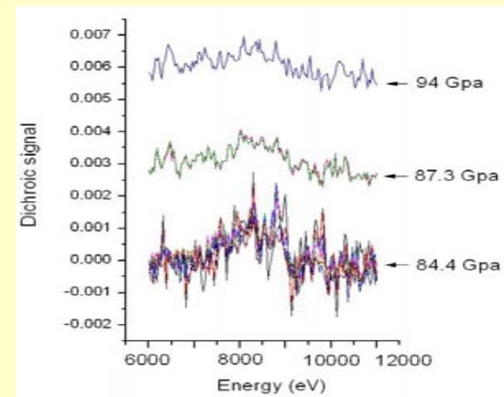
Antonangeli *et Al*, App. phys. Lett., 92, 111911 (2008)

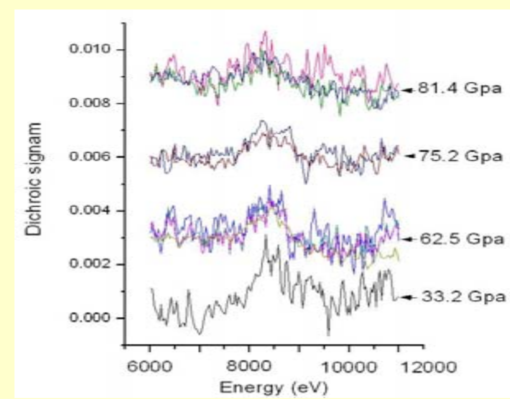
Magnetic moment vanishes at HP?

XAS

XMCD







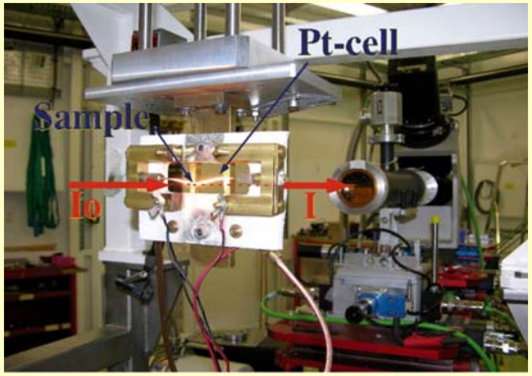
Olivier Mathon, Sakura Pascarelli, François Baudelet, Alexandre Monza, Matteo D'Astuto, Daniele Antonangeli, Jean-Paul Itié, Emma Pugh, Jean-Claude Chervin, Alain Polian,

Beamline limit → ID24 to get over

In good agreement with Iota *et al.*

Results: Kinetics of iron redox in aluminosilicate glasses and melts

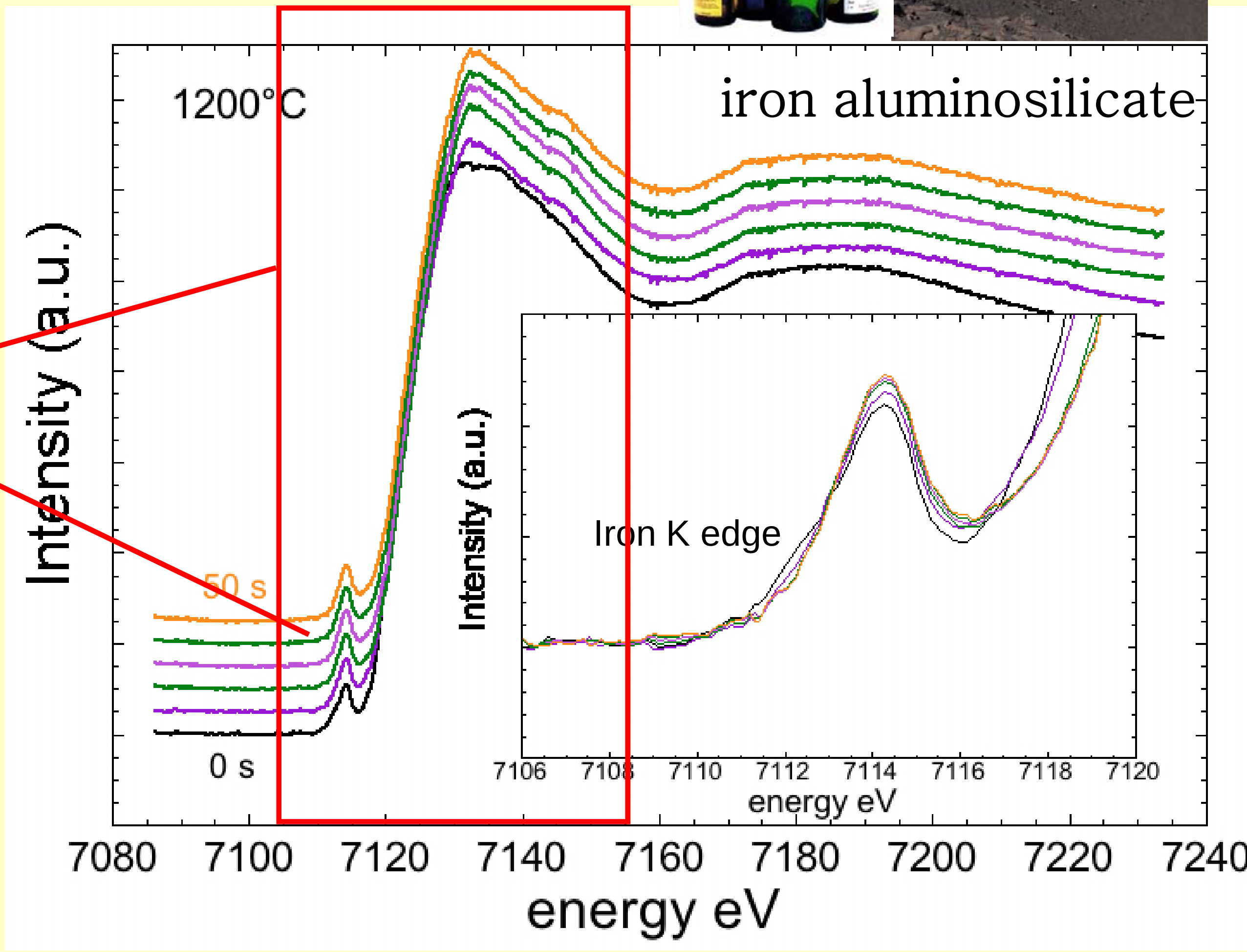
25° – 1700°C

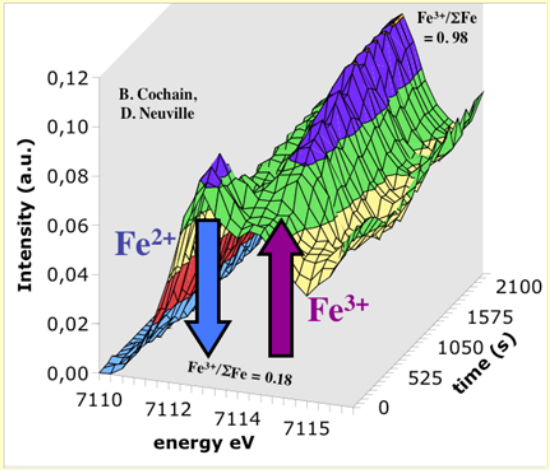


Temperature variation induces a change in the redox state. It can be followed by:

Changes in the White Line

Changes in the Pre-Peak



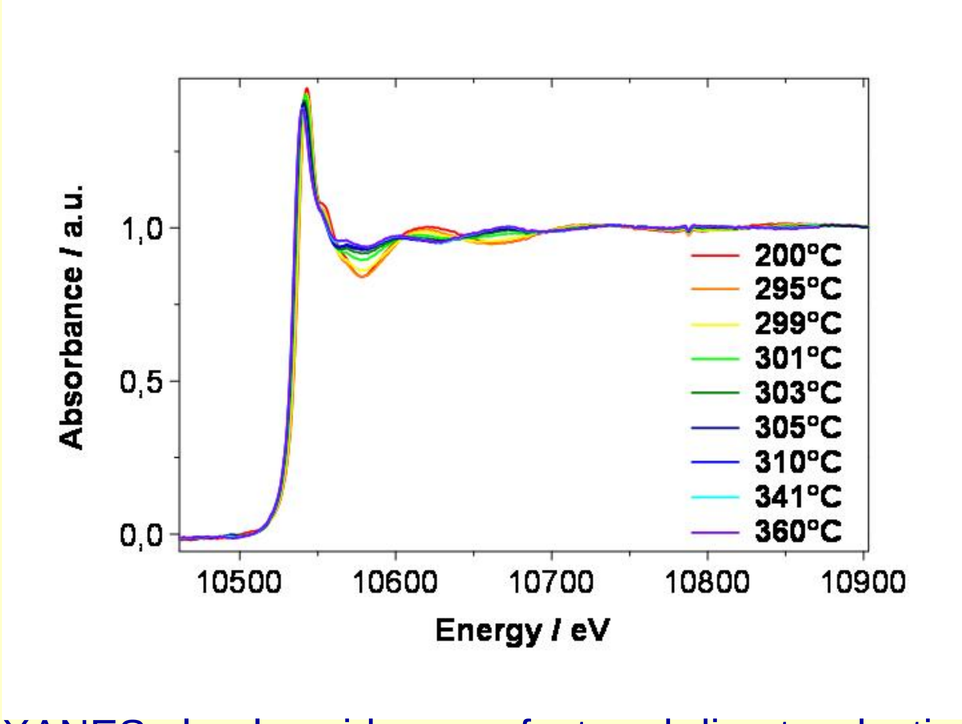


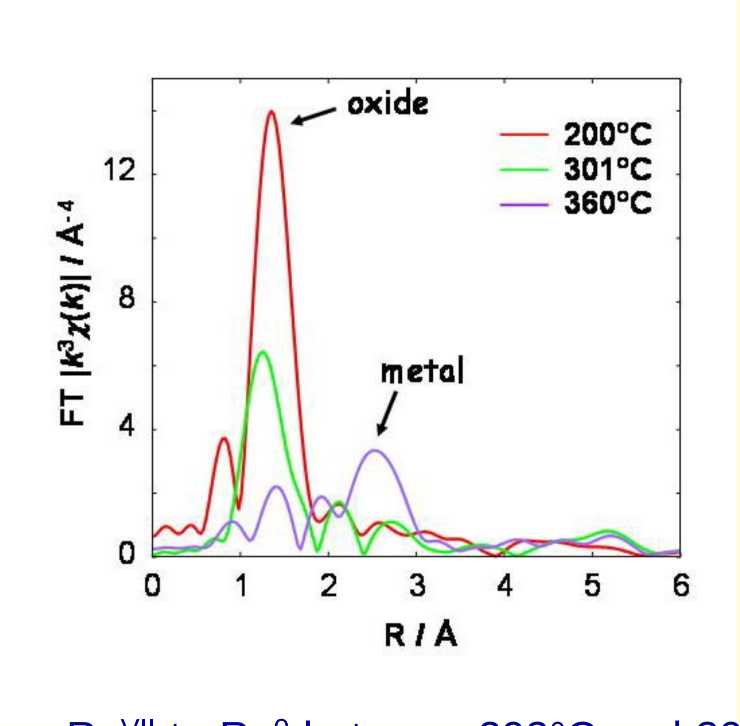
Benjamin Cochain, Daniel R. Neuville, Dominique de Ligny, Denis Testemalem, Eozen Strukelj, Pascal Richet

Results: Time-resolved reduction of ReO_x/Al_2O_3 catalysts

Supported rhenium oxide is very selective towards dimethoxymethane during methanol partial oxidation. It has been proposed that an original redox couple (Re^{VI}-Re^{IV}) could be at the origin of this behavior.

Experimental setup: Powdered catalyst in a Lytle-type cell, with a mica window for Raman. EXAFS (Re L3-edge) spectra recorded on ODE beamline (ca. 2 spectra/minute).





XANES clearly evidence a fast and direct reduction from Re^{VII} to Re^{0} between 293°C and 303°C, well confirmed by the EXAFS analysis

Elise Berrier, Sylvain Cristol, Camille La Fontaine, Valérie Briois, Francoise Villain

Developments in progress

- XMCD @ 7T, 2K, HP
- XRD/XAS combination
- Raman/XAS combination
- Stopped Flow
- New benders and blades for the polychromator (220 and 111, 311 with lower slope error)
- Fluorescence measurements
- Turbo EXAFS for diffusing samples' kinetics
- Acquisition Graphic Interface and Live Energy Calibration

Gwenaëlle Abeillé

Alberta Congeduti

Qingyu Kong

François Baudelet

Sébastien Chagnot

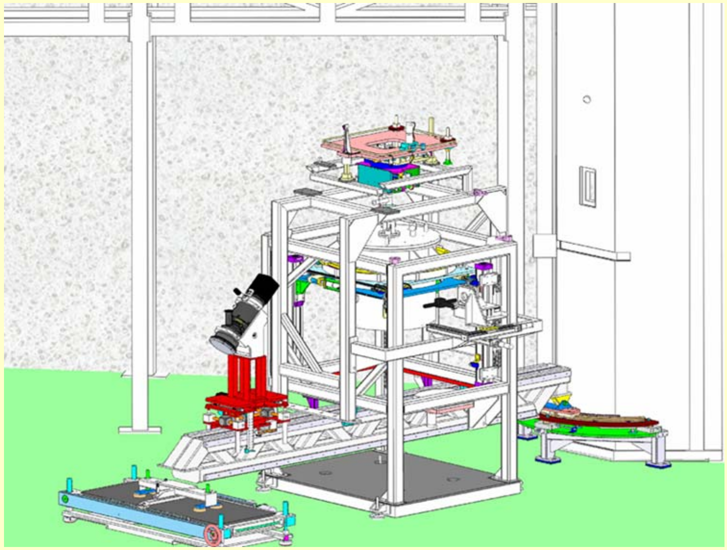
Olga Roudenko

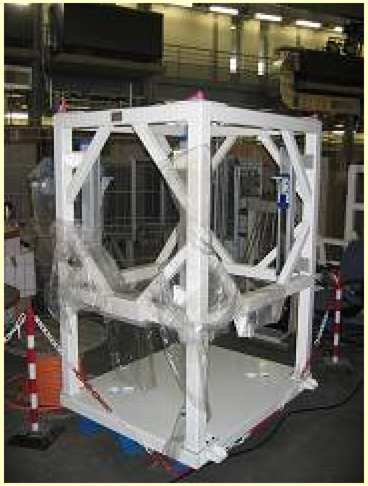
Aurélien Delmotte

Alexandre Monza

THANKS FOR YOUR ATTENTION!

Developments in progress: XMCD @ 7T, 2K, HP

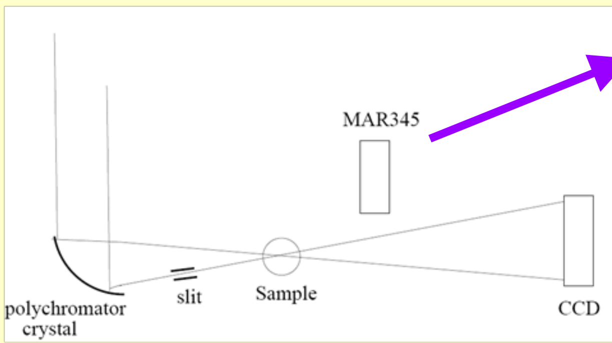


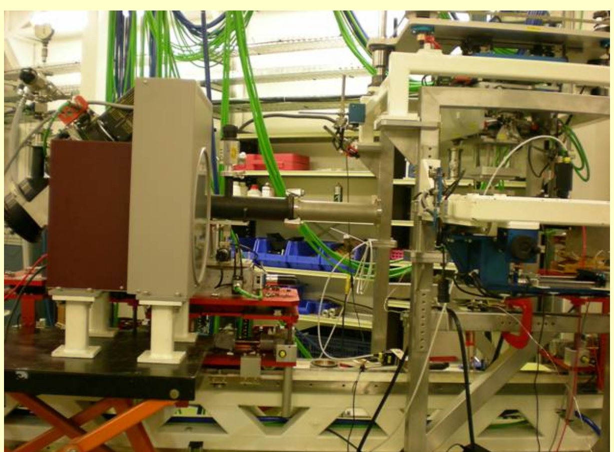


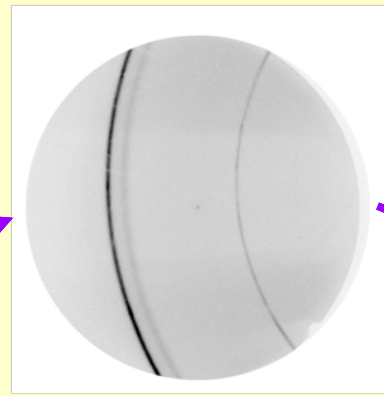
Developments in progress: Combination of XAS/XRD

• First attempt: crystallization of a-Ge upon decreasing pressure at 6 GPa

Set-up

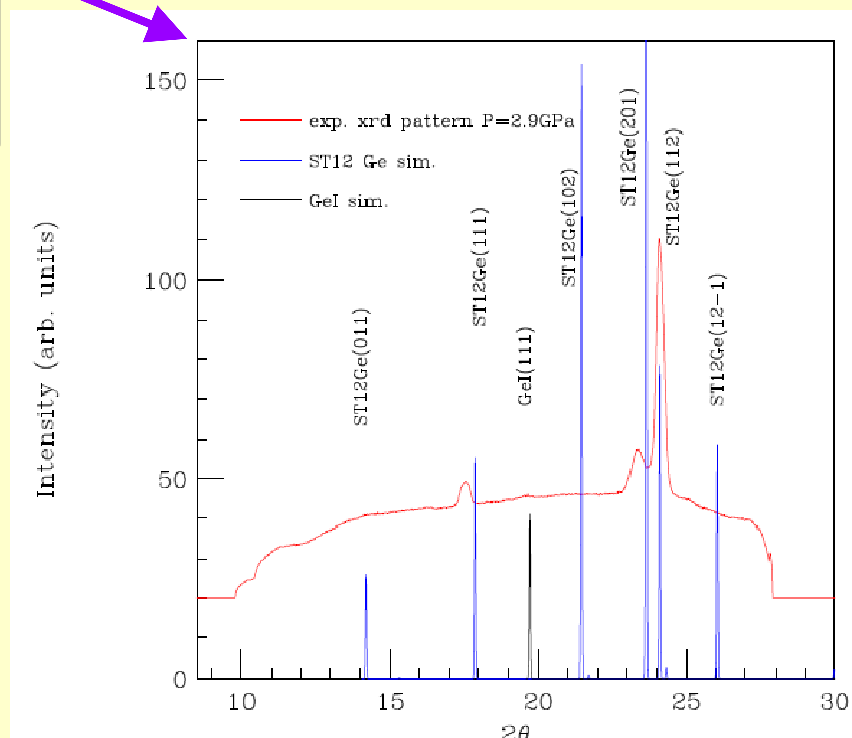






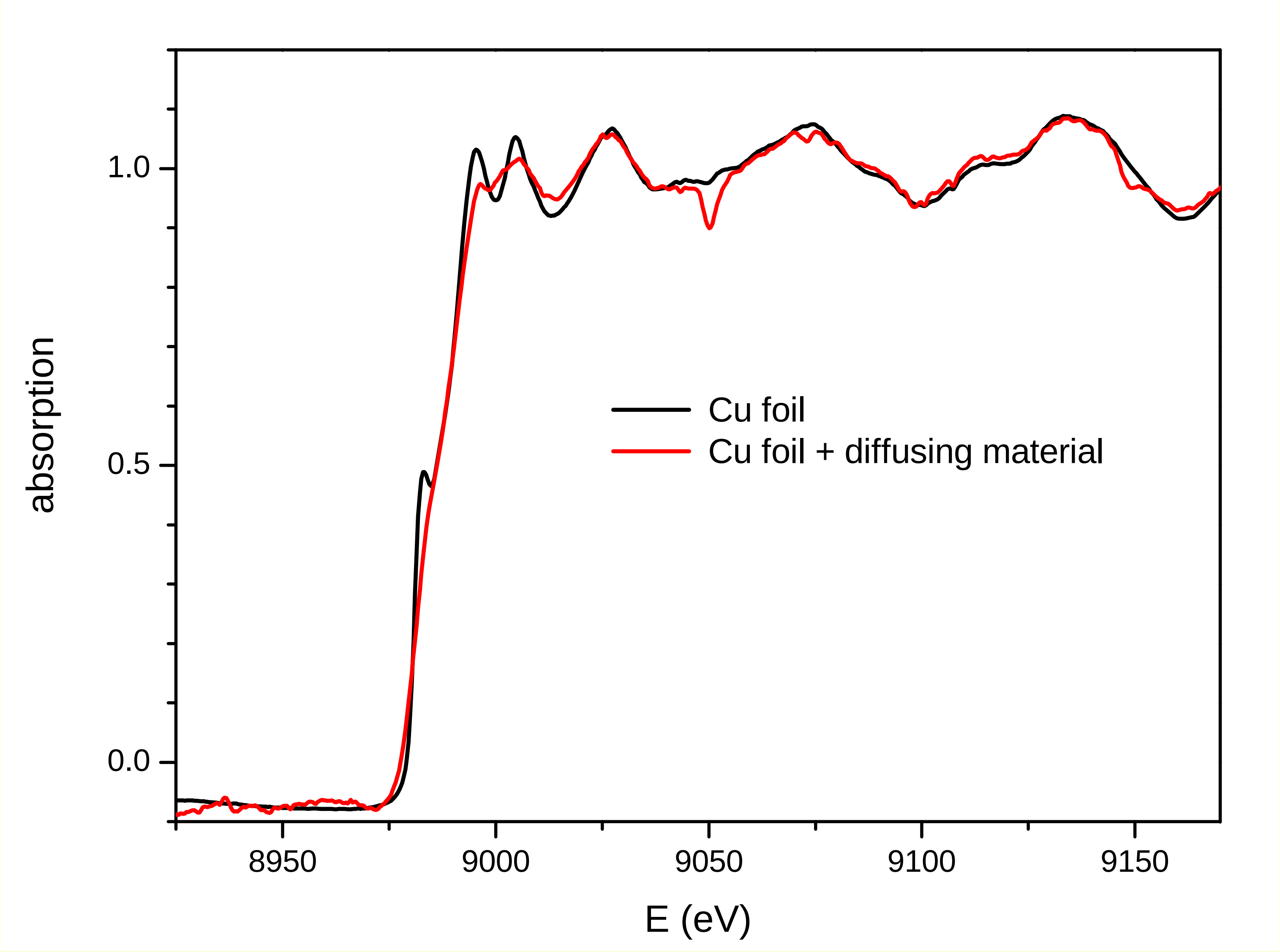
MAR345 image

2.9 GPa diffraction pattern upon depressurization



Federica Coppari, Emiliano Principi, Alberta Congeduti, Sebastien Chagnot and Andrea Di Cicco

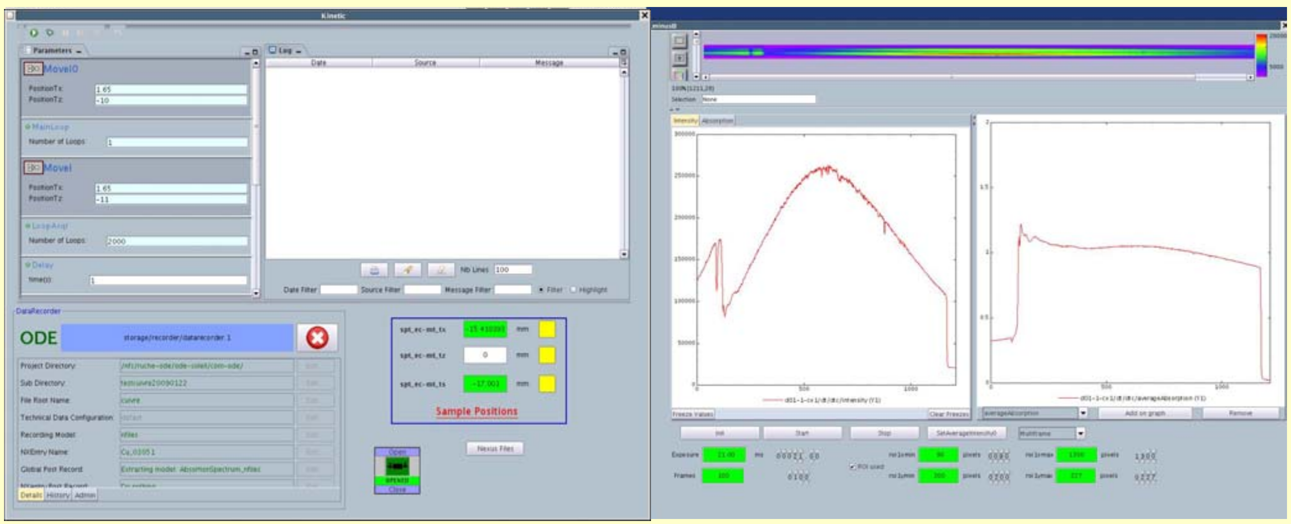
Effect of diffusing materials on resolution



Informatics interfaces

- XMCD

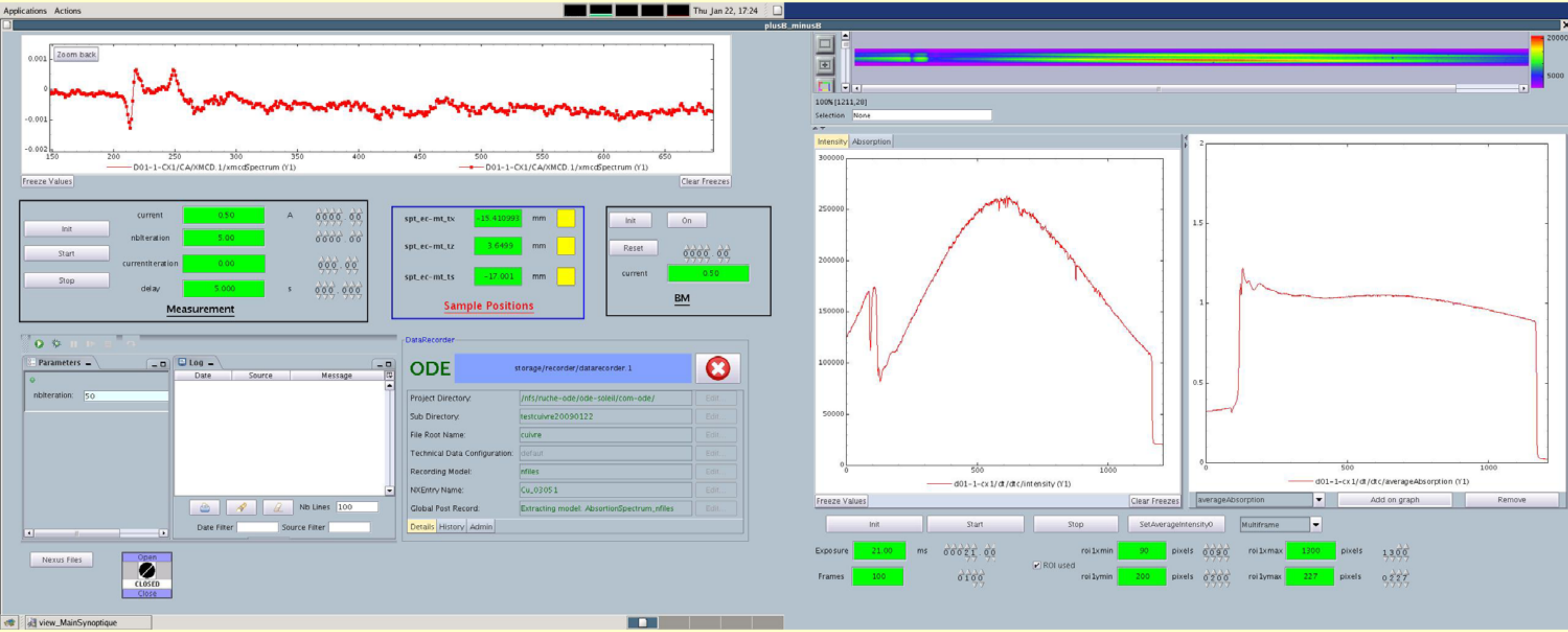
- Kinetics



Qingyu Kong, Gwenaelle Abeillé, …

Informatics interfaces

- XMCD

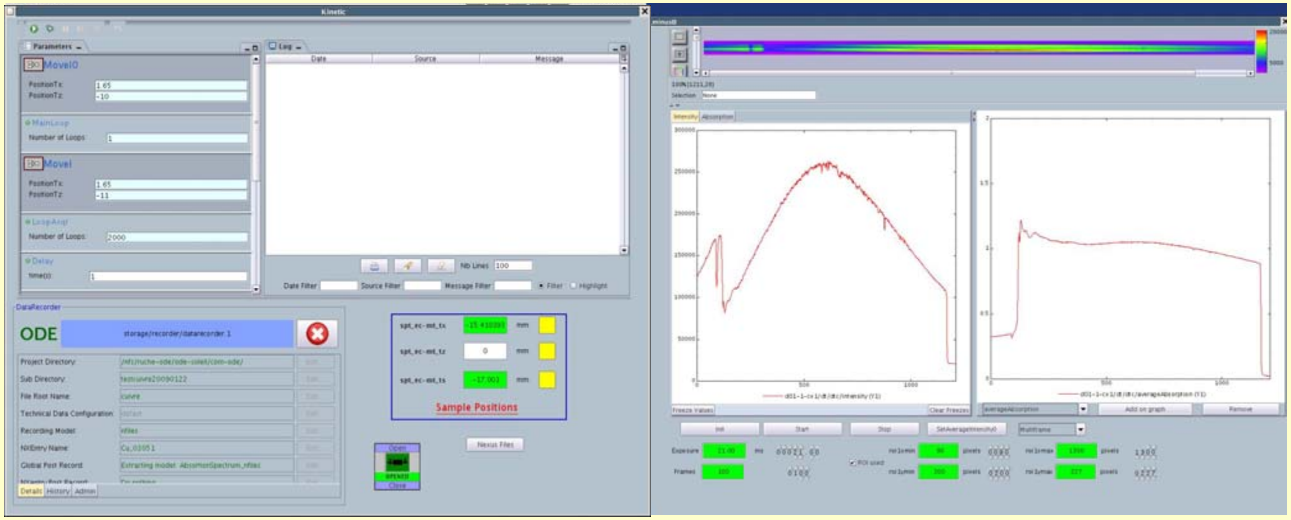


Qingyu Kong, Gwenaelle Abeillé, …

Informatics interfaces

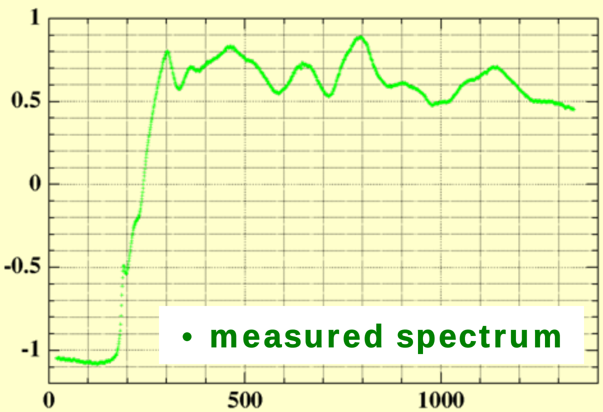
- XMCD

- Kinetics

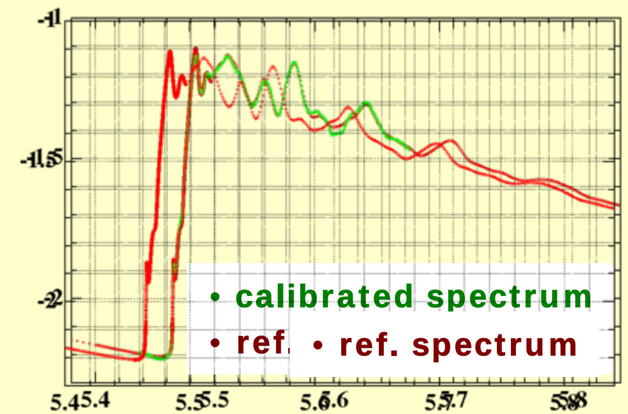


Qingyu Kong, Gwenaelle Abeillé, …

Energy calibration



$$\Phi(\bar{c}, \bar{a}, \bar{b})$$



$$E_i = \sum_{0}^{K} c_k x^k$$

$$A_i = a_0 + a_1 y_i + \sum_{1}^{M} b_m T_m(E_i)$$

Calibration of spectra from DXAS beamlines
M.P. Ruffoni and R.F. Pettifer
J.Synchrotron Rad.(2006). 13, 489-493

Background correction using Chebyshev polynomials T_m

Highly multi-modal optimization problem

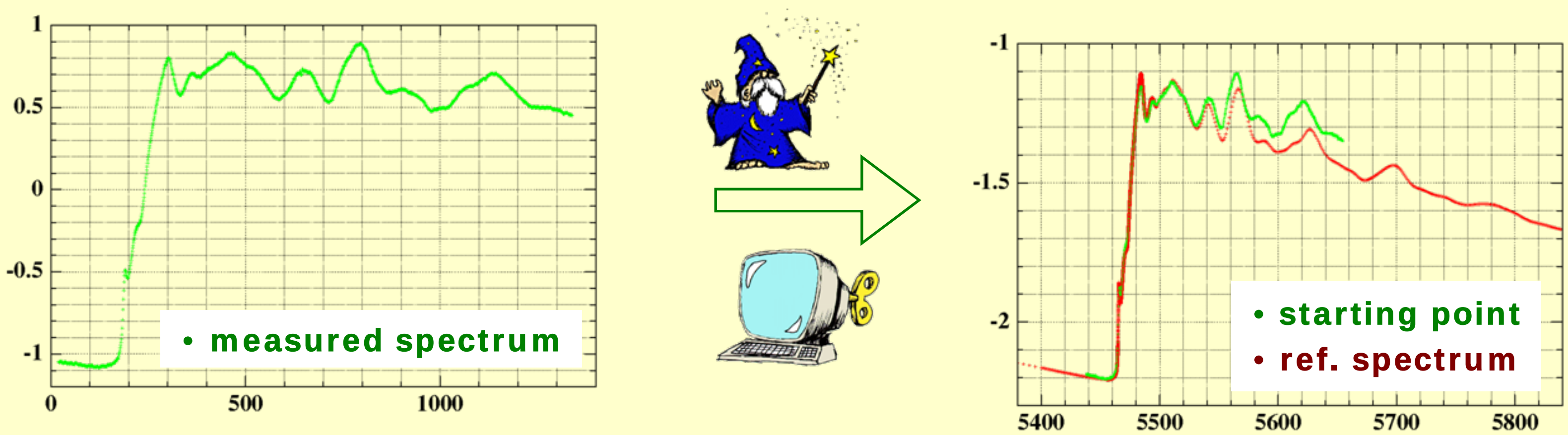
Find $\{c_k, a_l, b_m\}$, $k = 1, \ldots, K$, $l = 1,2$, $m = 1, \ldots, M$

so as to $\min_{c,a,b} \sum_i \left\langle \Phi(\bar{c}, \bar{a}, \bar{b})(x_i, y_i) - (E_i^{ref}, A_i^{ref}) \right\rangle^2$

The solution found by a local algorithm (such as Levenberg-Marquardt) is <u>very</u> sensitive to the starting point

Improved calibration tool

1. **Finding a good starting point given measured and reference spectra.**

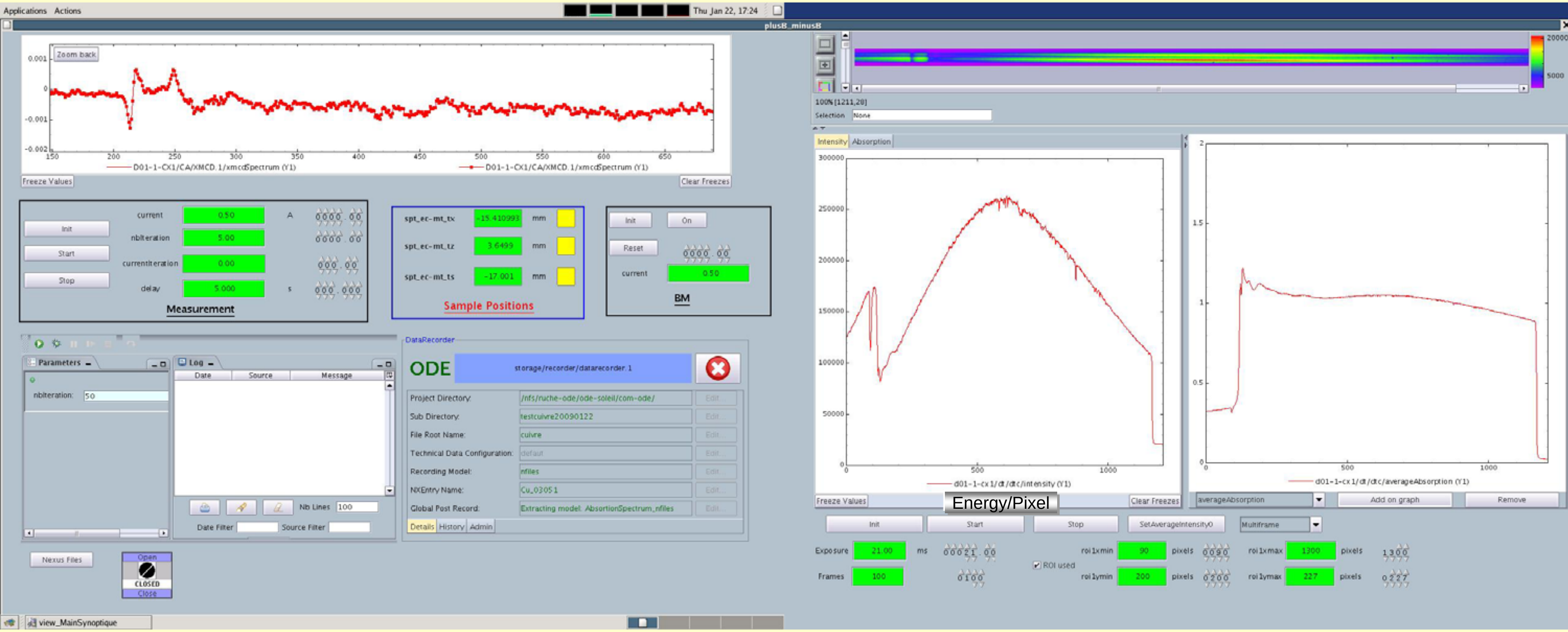


2. **A slower <u>global</u> optimization algorithm replaces quick local optimization (L.-M.) when the latter one fails.**

The optimization method, based on ***Covariance Matrix Adaptation,*** avoids local optima traps and achieves satisfactory calibration in more cases. (Many thanks to **Nikolaus Hansen**, INRIA Saclay!)

Contact: olga.roudenko@synchrotron-soleil.fr

Live Energy Calibration



Olga Roudenko, Julien Malik …

Developments in progress

- XMCD @ 7T, 2K, HP
- XRD/XAS combination
- Raman/XAS combination
- Stopped Flow
- New benders and blades for the polychromator (220 and 111, 311 with lower slope error)
- Fluorescence measurements
- Turbo EXAFS for diffusing samples' kinetics
- Acquisition Graphic Interface and Live Energy Calibration

Alberta Congeduti

Qingyu Kong

François Baudelet

Sébastien Chagnot

Gwenaëlle Abeillé

Olga Roudenko

Aurélien Delmotte

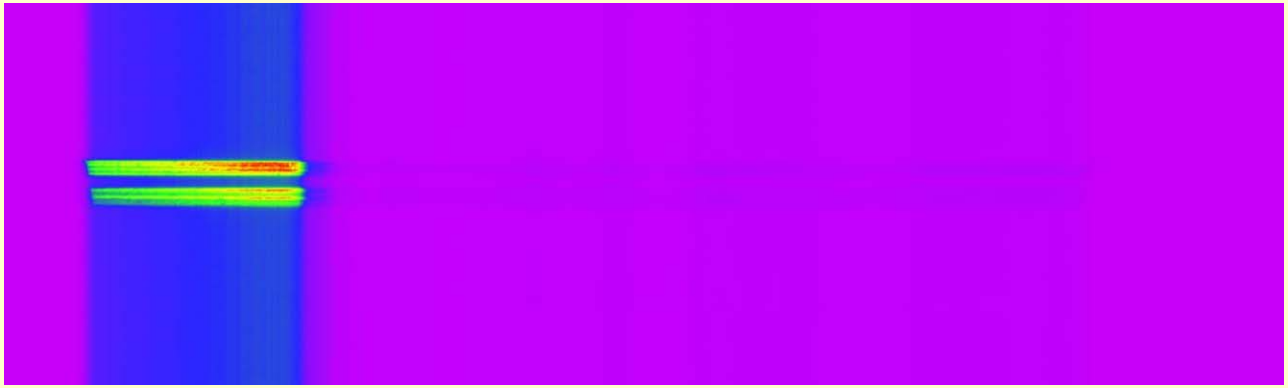
Alexandre Monza

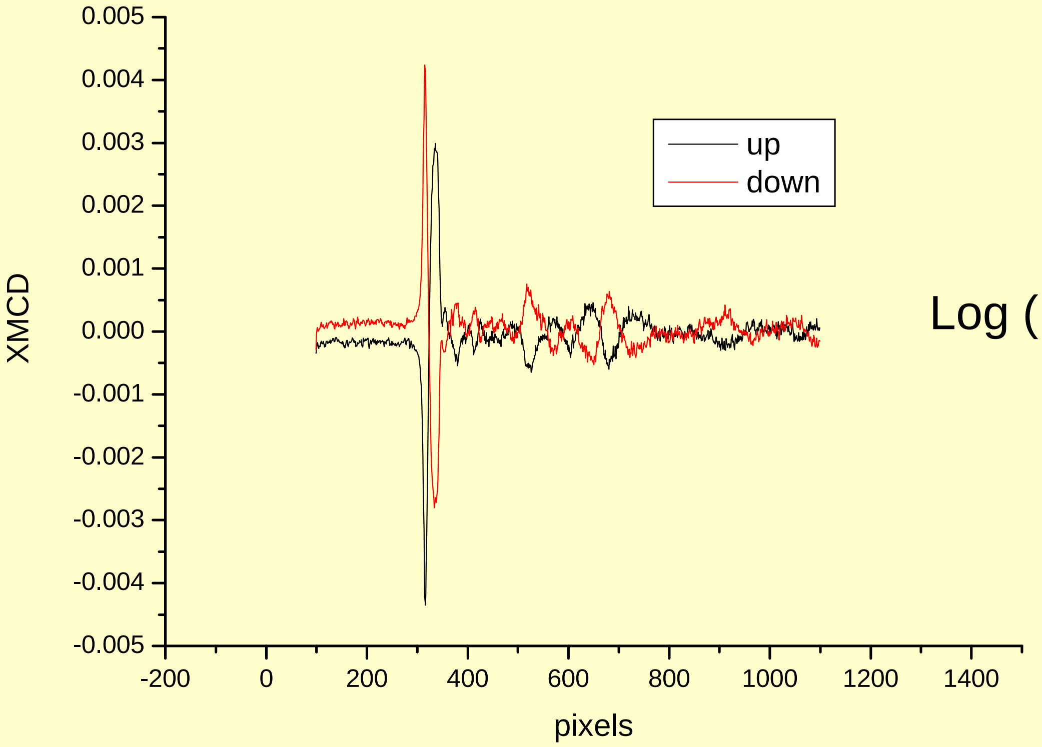
THANKS FOR YOUR ATTENTION!

Beamline specification

EXAFS	Measurements from 5 keV to ~~25~~ keV
XANES and XMCD	From 3.5 keV to 25 keV
Resolving power	E/ΔE: 3 10^4 for Si_{311} 0.7 10^4 for Si_{111}
Focus size:	~~40~~ µm x ~~40~~ µm FWHM
Detection mode:	Transmission mode with a photodiode array or a CCD camera Fluorescence mode
Source:	Bending magnet
First mirror:	1.2 meter long Ir and Rh bent mirror
polychromator:	Bragg geometry, Si_{111}, Si_{311}
High temperature limit:	1100 K under controlled atmosphere for heterogeneous catalysis 800 K for high-pressure measurements
Cryogenic temp. limit:	Down to 2 K for ambient and high pressure conditions
Pressure:	Up to 100 GPa in quasi hydrostatic conditions More than 100 GPa in non-hydrostatic conditions
Magnetic field:	Up to ~~6T~~

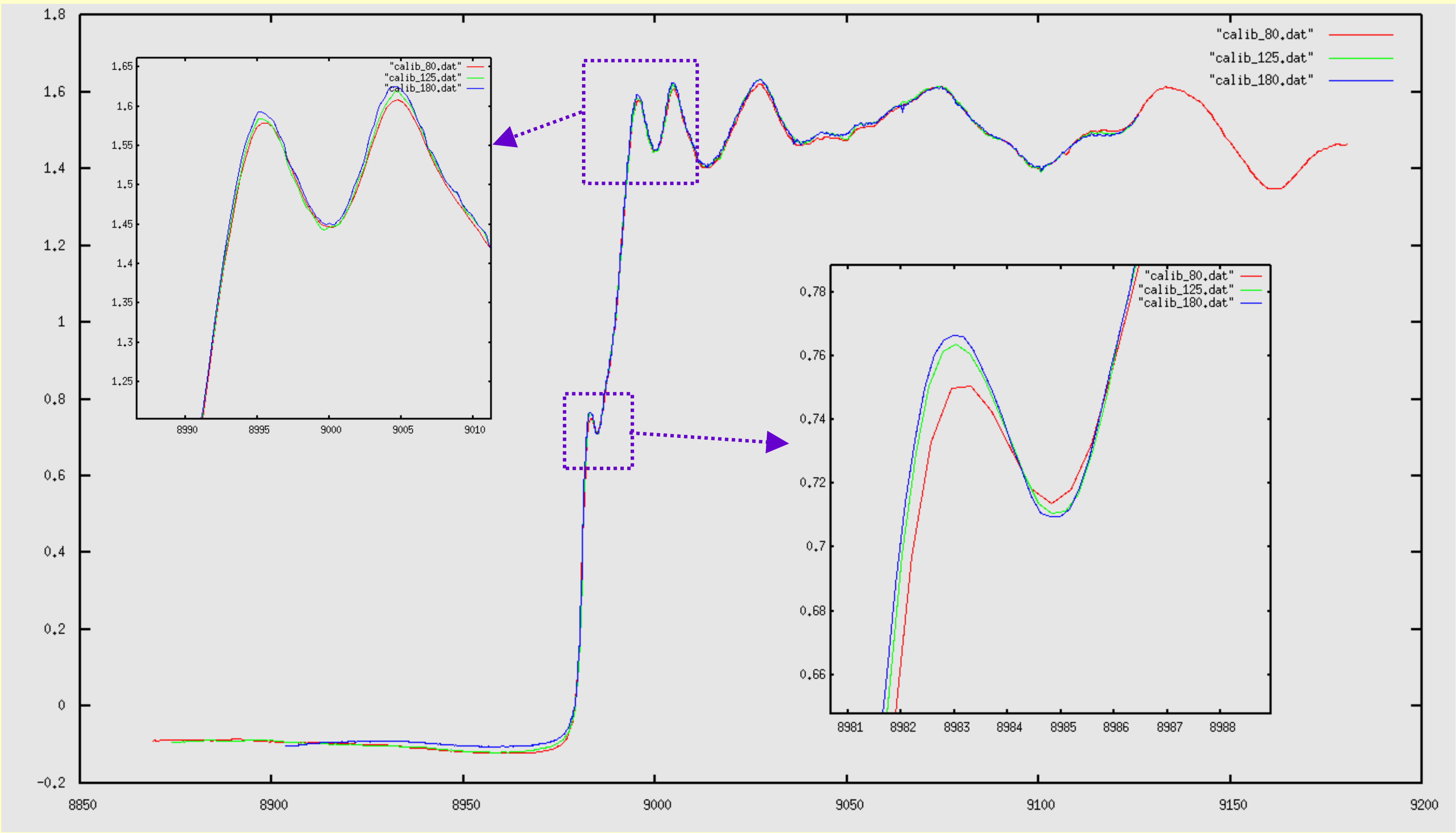
Future development: double beam XMCD ?





Log (I0up/Iup)-log (I0down/Idown)

Quarter wave plate?



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"calib_125.dat"
"calib_180.dat"
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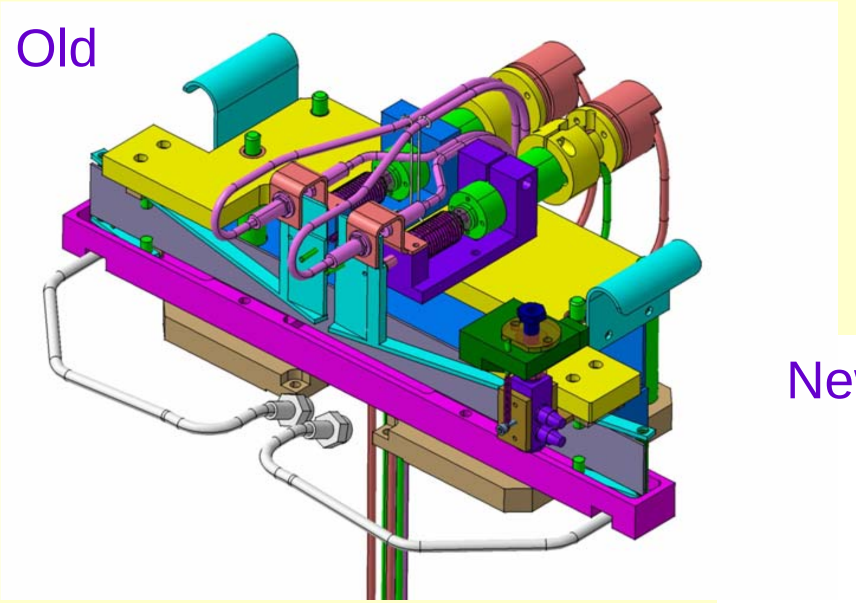
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
-0.2
8850
8900
8950
9000
9050
9100
9150
9200

Optics: bender mechanical improvements

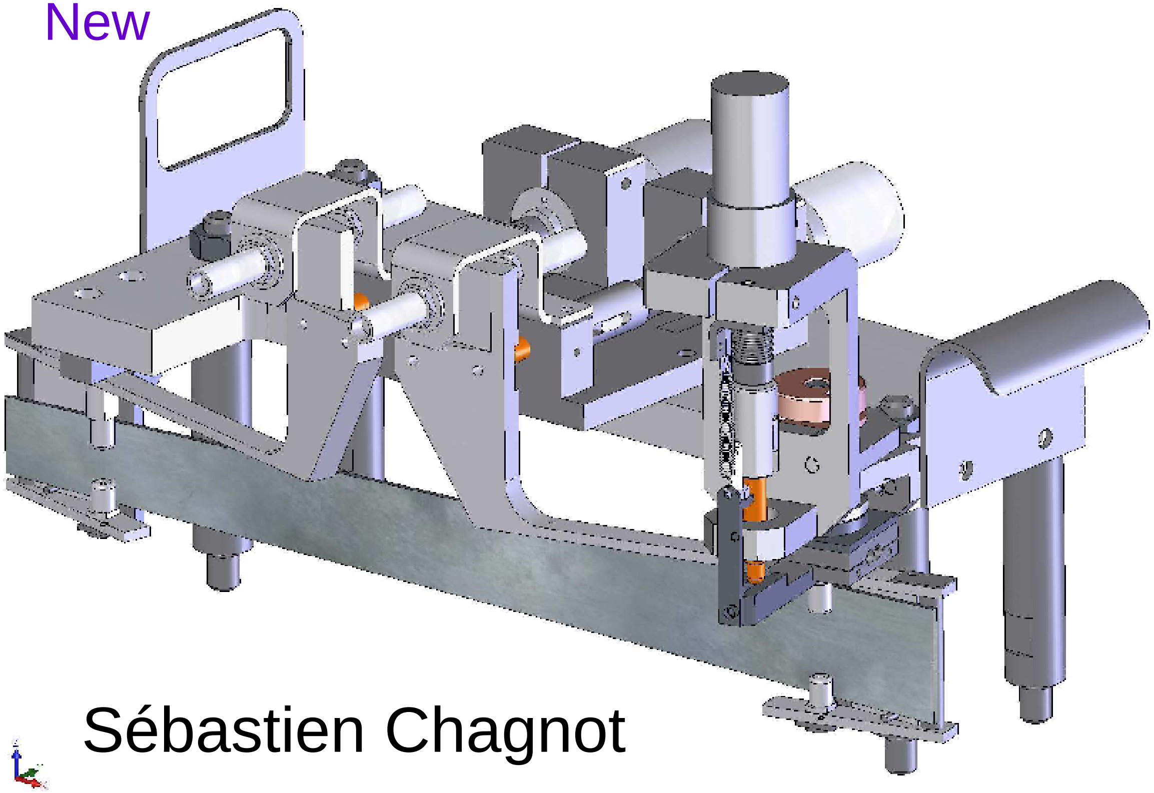
Old



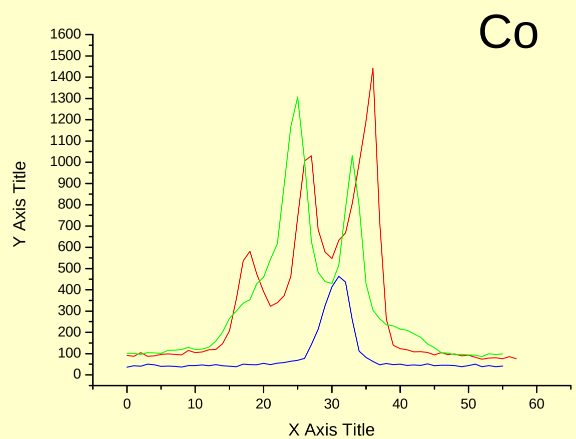
- Better contact between blade and bender
- Decoupling twisting and bending movements

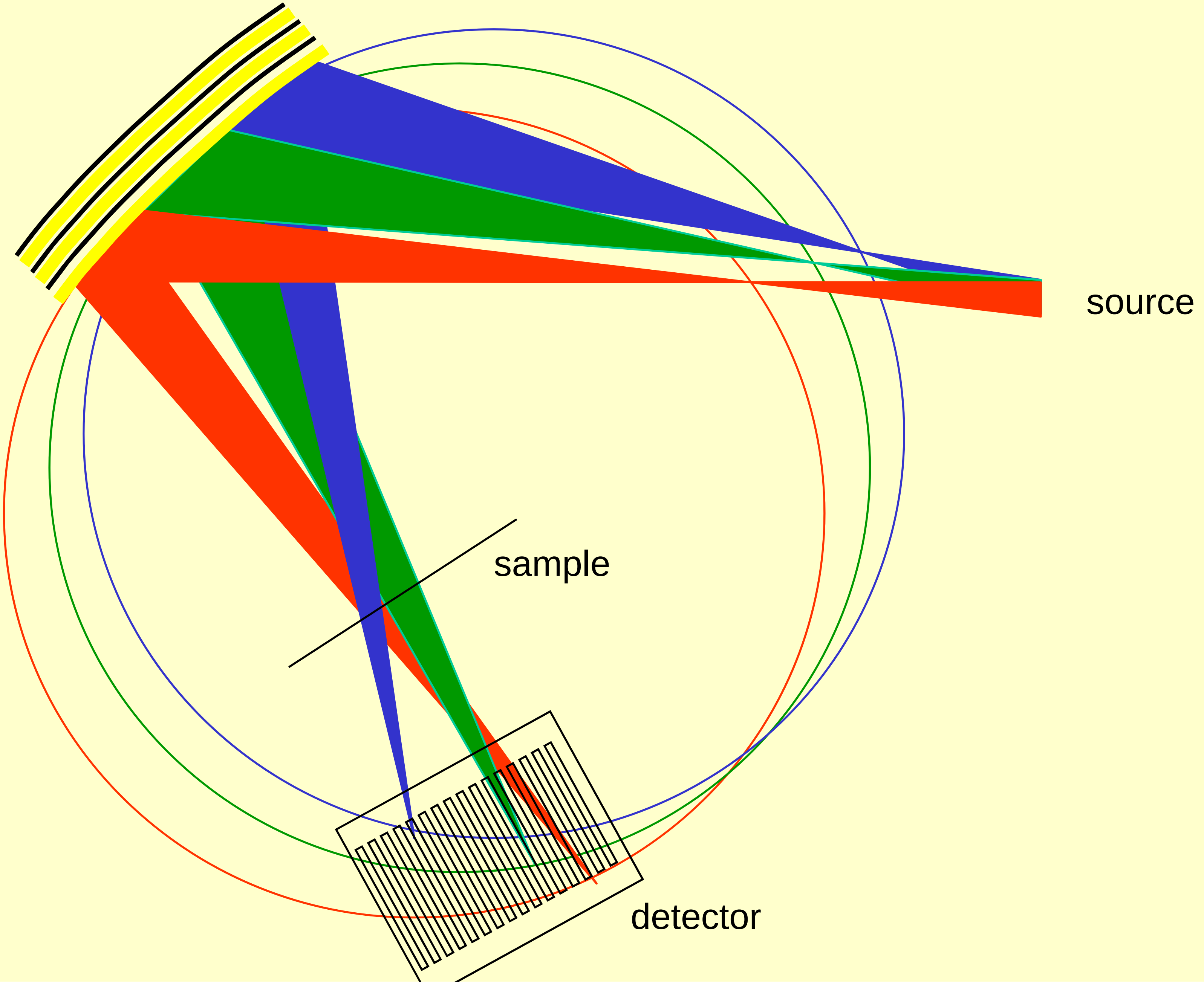
$\Rightarrow$ Very small spot size

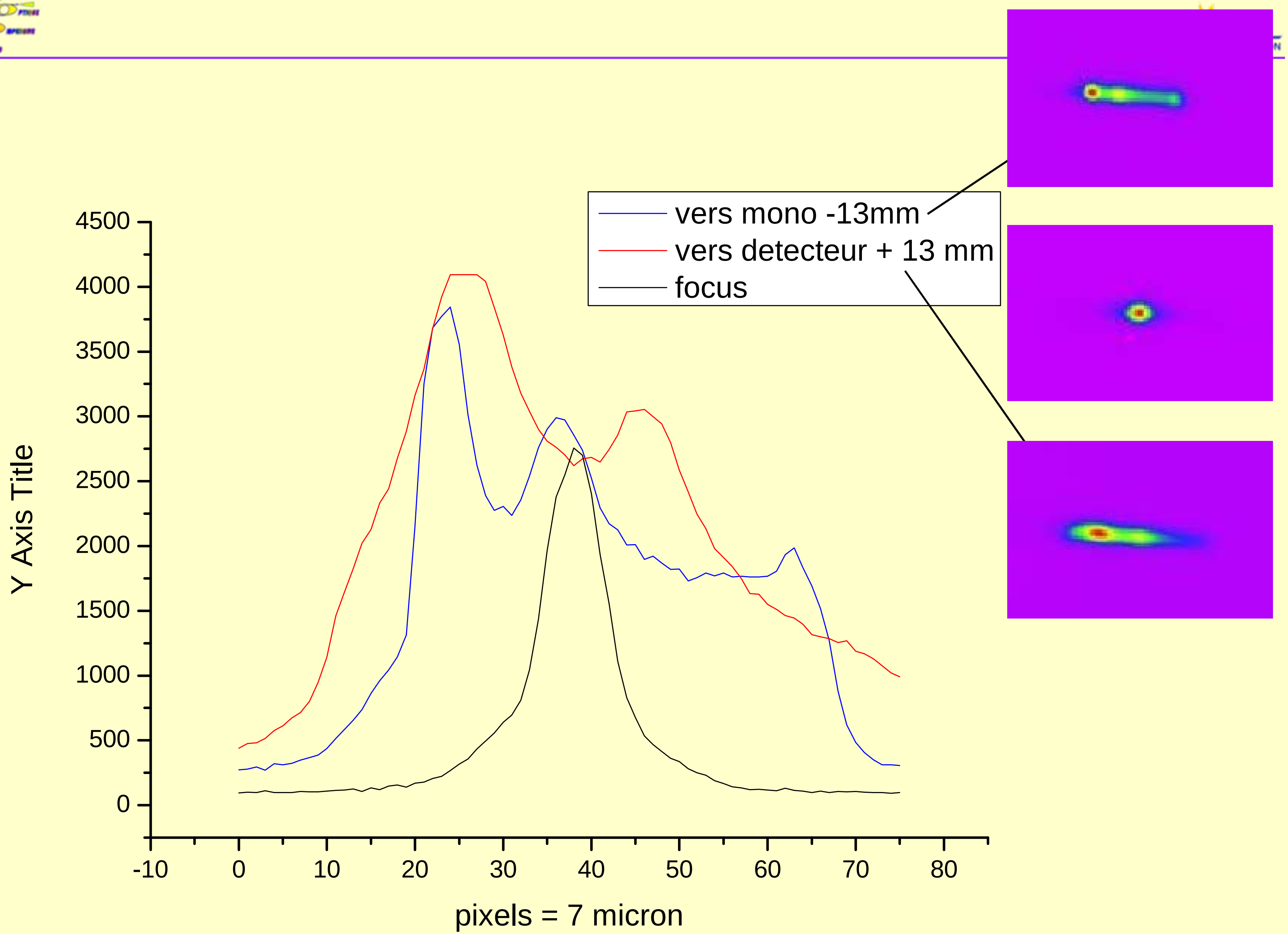
New



Sébastien Chagnot




source
sample
detector


vers mono -13mm
vers detecteur + 13 mm
focus
Y Axis Title
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3500
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2500
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1500
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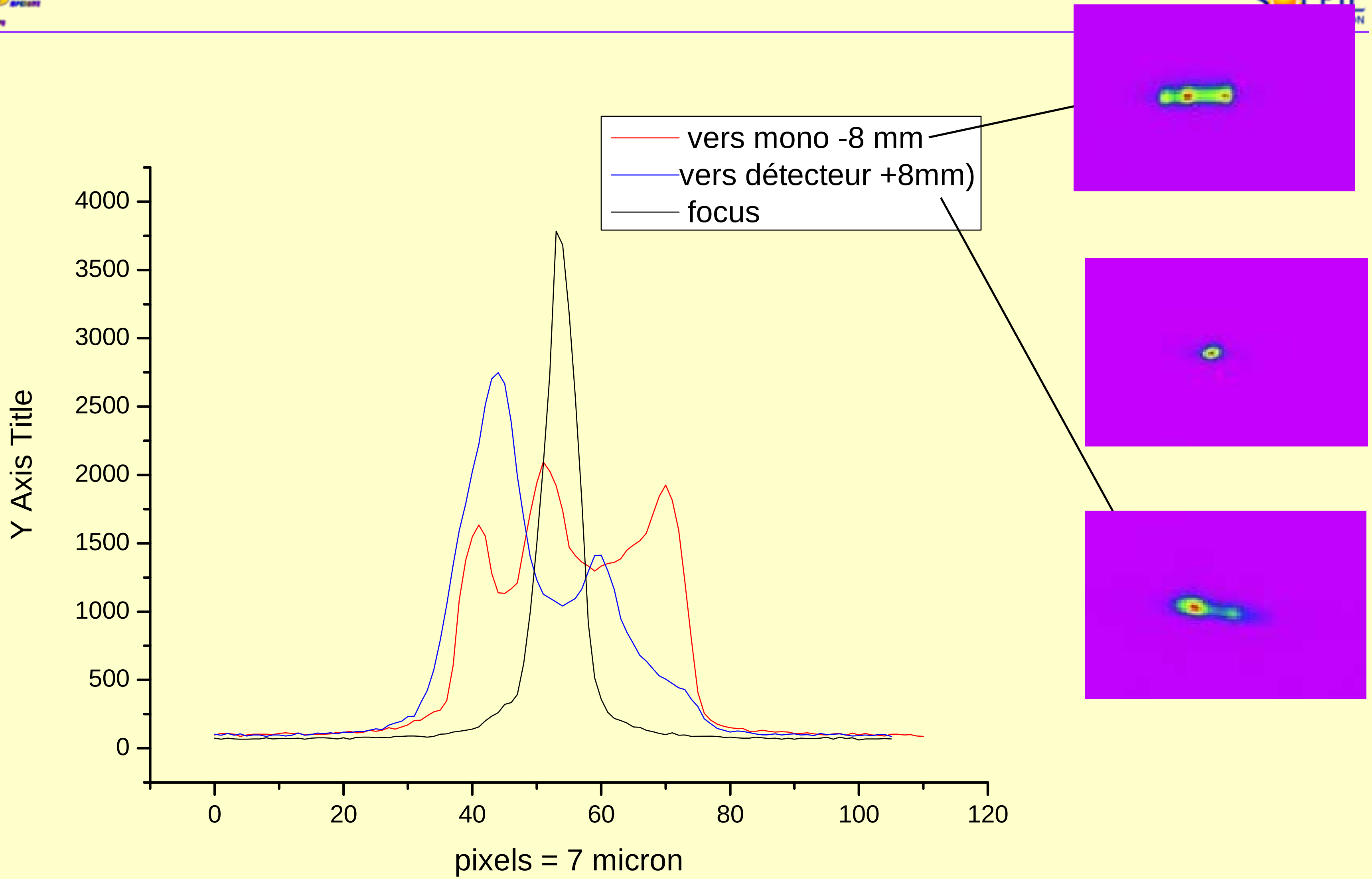
-10
0
10
20
30
40
50
60
70
80

Se


vers mono -8 mm
vers détecteur +8mm)
focus
Y Axis Title
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1000
500
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20
40
60
80
100
120
pixels = 7 micron

Zn