First test experiments with FMB-Oxford direct drive DCM at the Sirius beamline of Synchrotron SOLEIL

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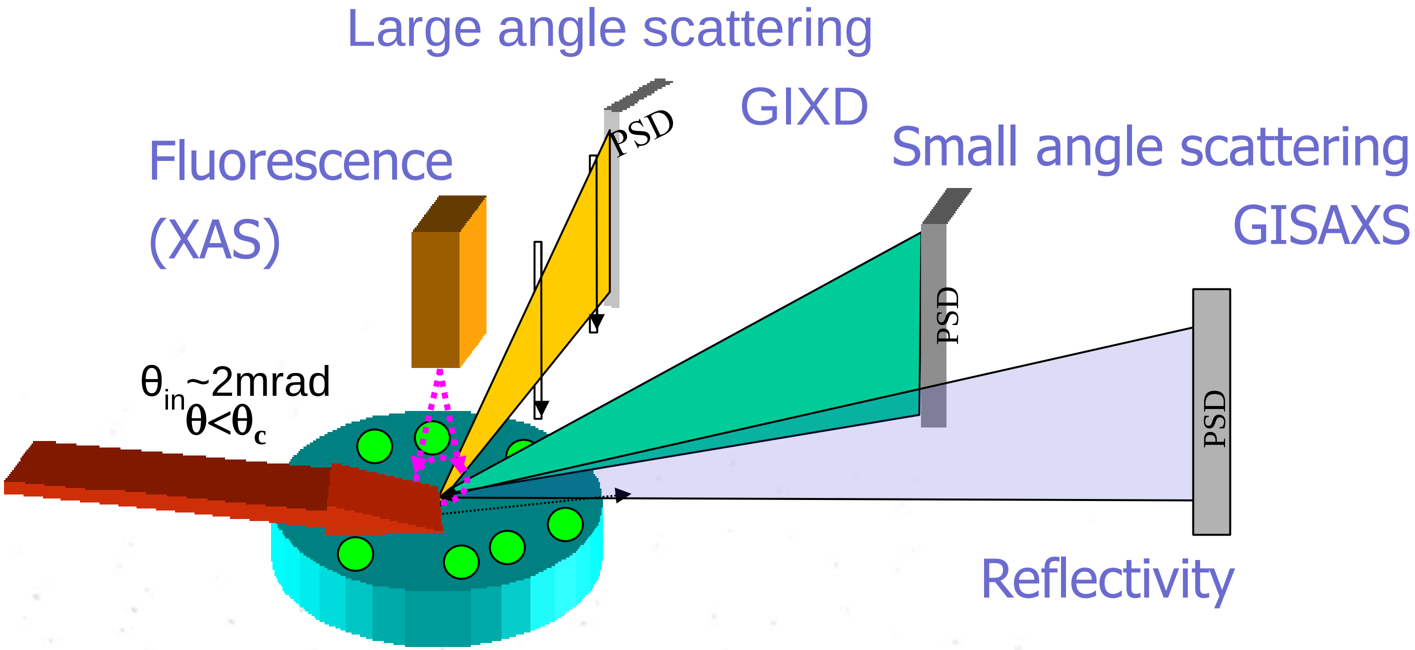
Outline

- The Sirius beamline at SOLEIL: experimental requirements and optical path chosen
- The FMB-Oxford DCM: direct drive vs. worm wheel
- DCM geometry and specifications, offline tests
- Performances at fixed energy (XRD test experiments)
- Performances in energy scan (XAS test experiments)
- Issues and correction strategies

The Sirius beamline at SOLEIL: experimental requirements

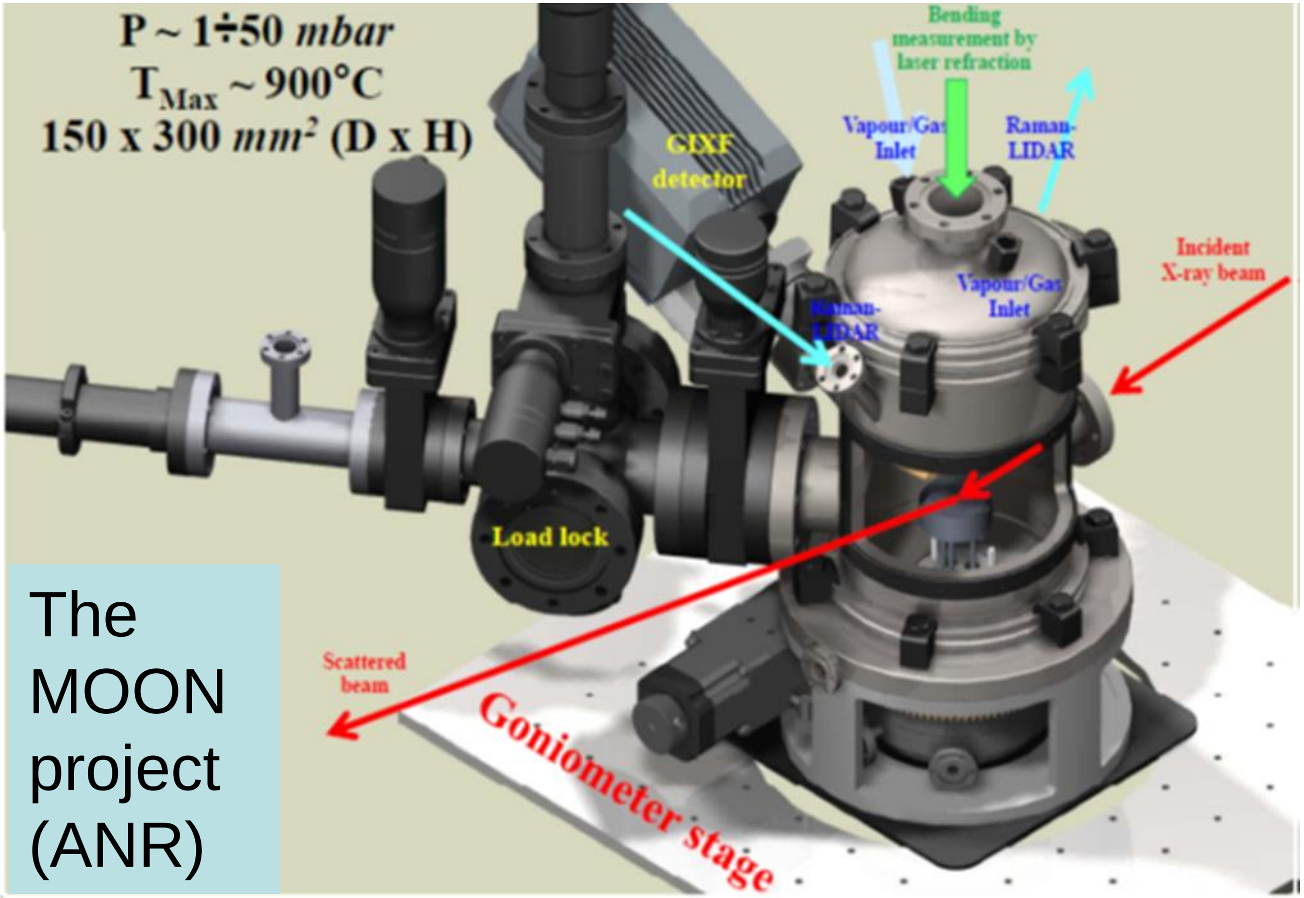


Energy range: 1.4–12 keV
tender x-ray
variable polarisation

Goal: to combine different scattering and spectroscopy techniques at grazing incidence for surface/interface studies

GIXD, GISAXS, FLY-XAS, DAFS, Reflectivity, XRMS

The Sirius beamline at SOLEIL: experimental requirements



The MOON project (ANR)

In situ / in operando experiments

Monitoring the nucleation process and the early stages of MOCVD/ALD growth of ZnO via X-ray techniques

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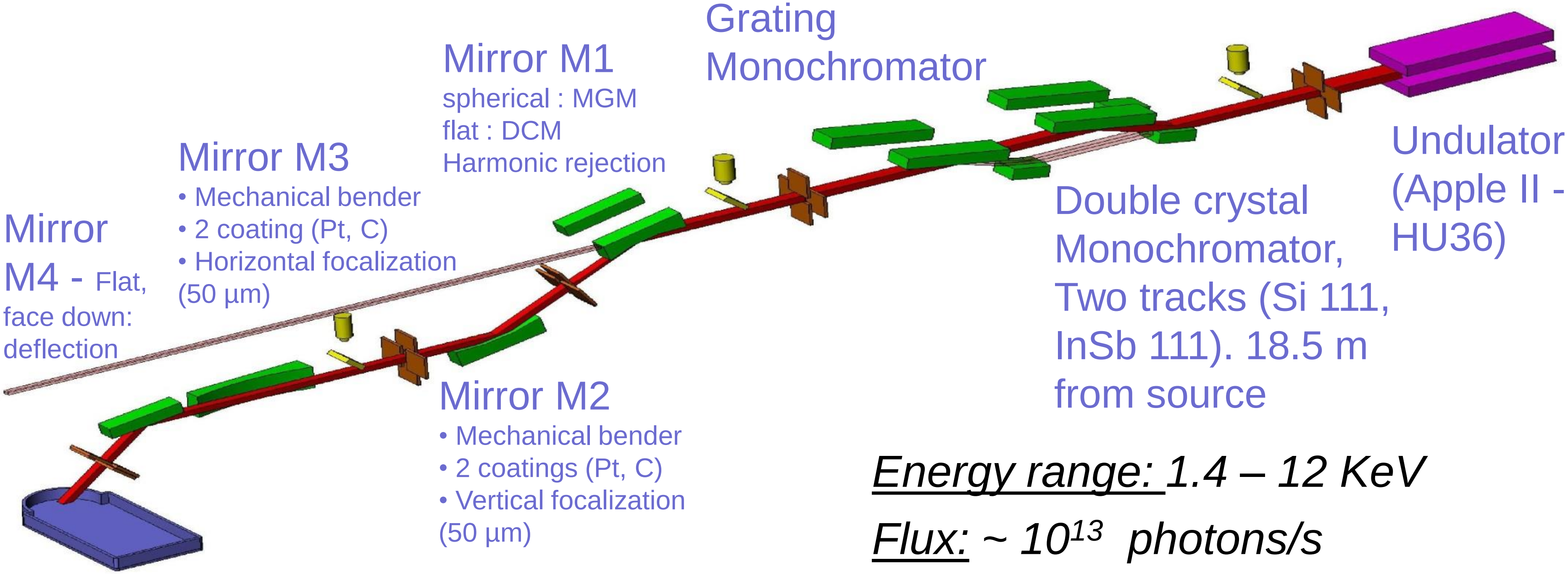
We need fast scanning to follow kinetics !

The Sirius beamline at SOLEIL: technical solutions/selected optics



Multilayer Grating Monochromator

Mirror M1
spherical : MGM
flat : DCM
Harmonic rejection

Mirror M3
- Mechanical bender
- 2 coating (Pt, C)
- Horizontal focalization (50 µm)

Mirror M4 - Flat, face down: deflection

Undulator (Apple II - HU36)

Double crystal Monochromator, Two tracks (Si 111, InSb 111). 18.5 m from source

Mirror M2
- Mechanical bender
- 2 coatings (Pt, C)
- Vertical focalization (50 µm)

Energy range: 1.4 – 12 KeV

Flux: ~ 10^{13} photons/s

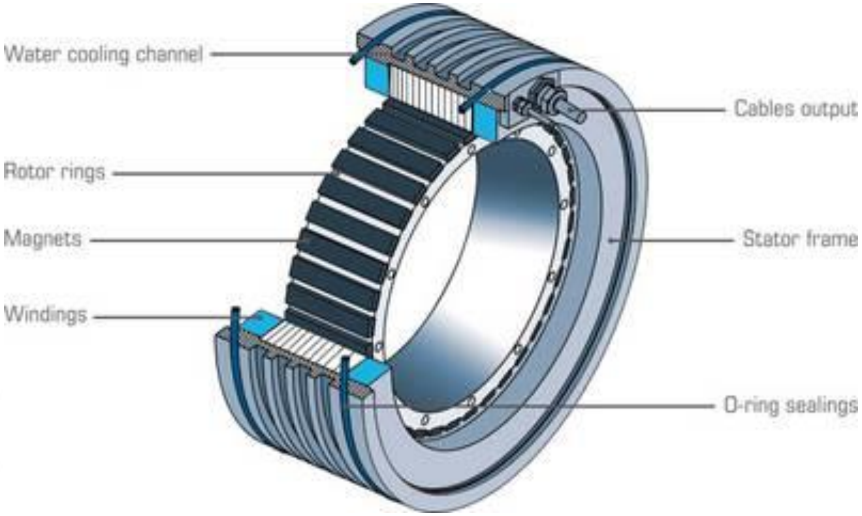
Beam size: 0.05 x 0.05 mm^2

2 (3) end-stations

The FMB-Oxford DCM: worm-wheel vs. direct drive

worm - wheel

direct drive (torque)



Sirius DCM uses a synchronous brushless motor (ETEL) with 44 poles (22 pairs) → backlash-free and dynamic response

Renishaw encoder + interpolator = 144x10^6 counts/revolution

The FMB-Oxford DCM: worm-wheel vs. direct drive

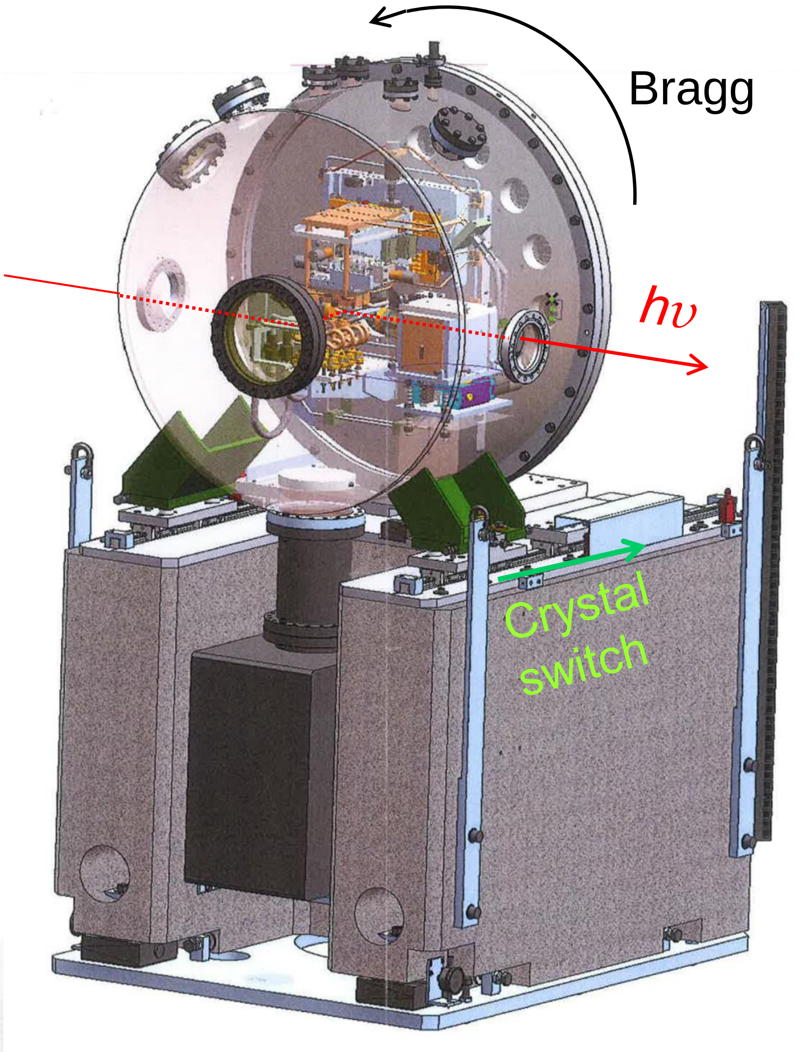
Parameter	Worm Wheel	Direct Drive
Resolution	<0.04 arc seconds (0.18 µrad)	<0.02 arc seconds (0.09 µrad)
Repeatability	<0.15” (0.7 µrad) over 35° <0.2” (1 µrad) over 90°	<0.1” (0.45 µrad) over 35° <0.15” (0.7 µrad) over 90°
Velocity Range	0.000 1°/sec - 1°/sec	0.000 1°/sec - 4°/sec
Velocity Accuracy*	1.5% (20” at 0.5°/s)	0.1% (2.5” at 1°/s)
Settling Time*	150ms from 0.5°/s	50ms from 1°/s

Direct drive enables →

- Slightly better repeatability
- Superior dynamic performances
- Very small increments without any backlash

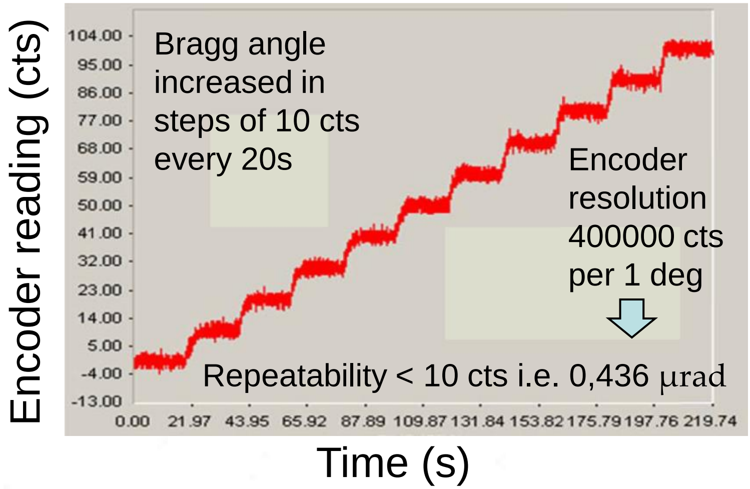
With 4°/s at 10 keV we can cover ~ 700 eV in 0.2 s → kinetics monitoring

DCM geometry and specifications



- Two crystal pairs: Si (111) and InSb (111) selected by translation of the whole vessel
- Perpendicular translation of 2nd crystal for fixed exit (xtal2perp)
- Beam walks on the second crystals (longer)
- Very large Bragg angle range (3° to 85°) and xtal2perp (9-73mm crystal separation) to allow access to 2 KeV with Si (111)

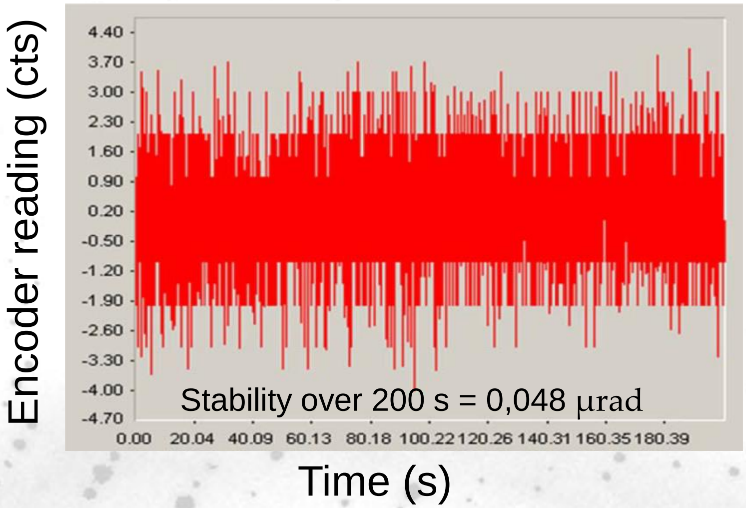
DCM typical offline tests at the factory



Bragg angle increased in steps of 10 cts every 20s

Encoder resolution 400000 cts per 1 deg

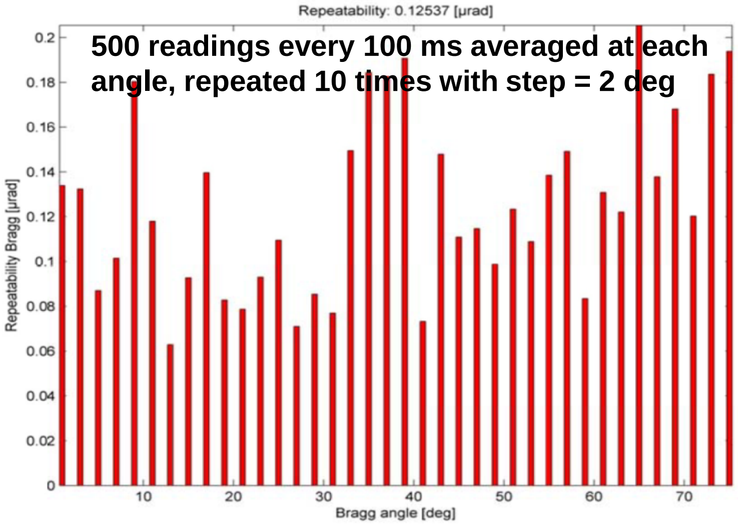
Repeatability < 10 cts i.e. 0,436 µrad



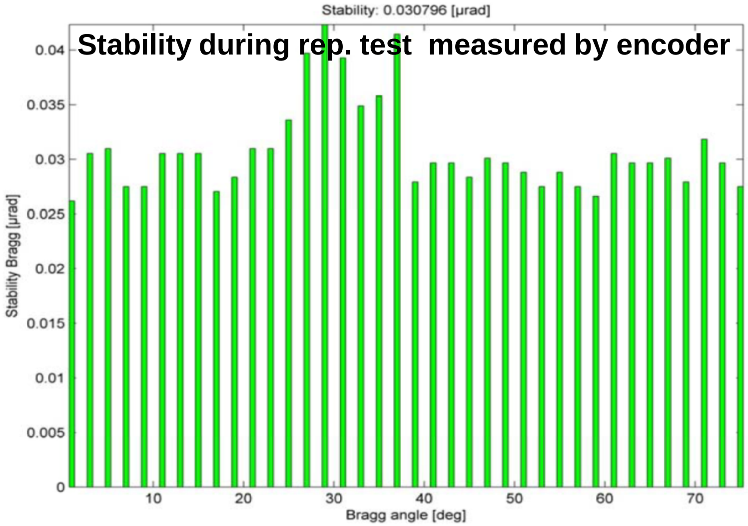
Stability over 200 s = 0,048 µrad

Measurements with DCM internal encoder

Measurements with Zygo ZMI-2000 laser interferometer

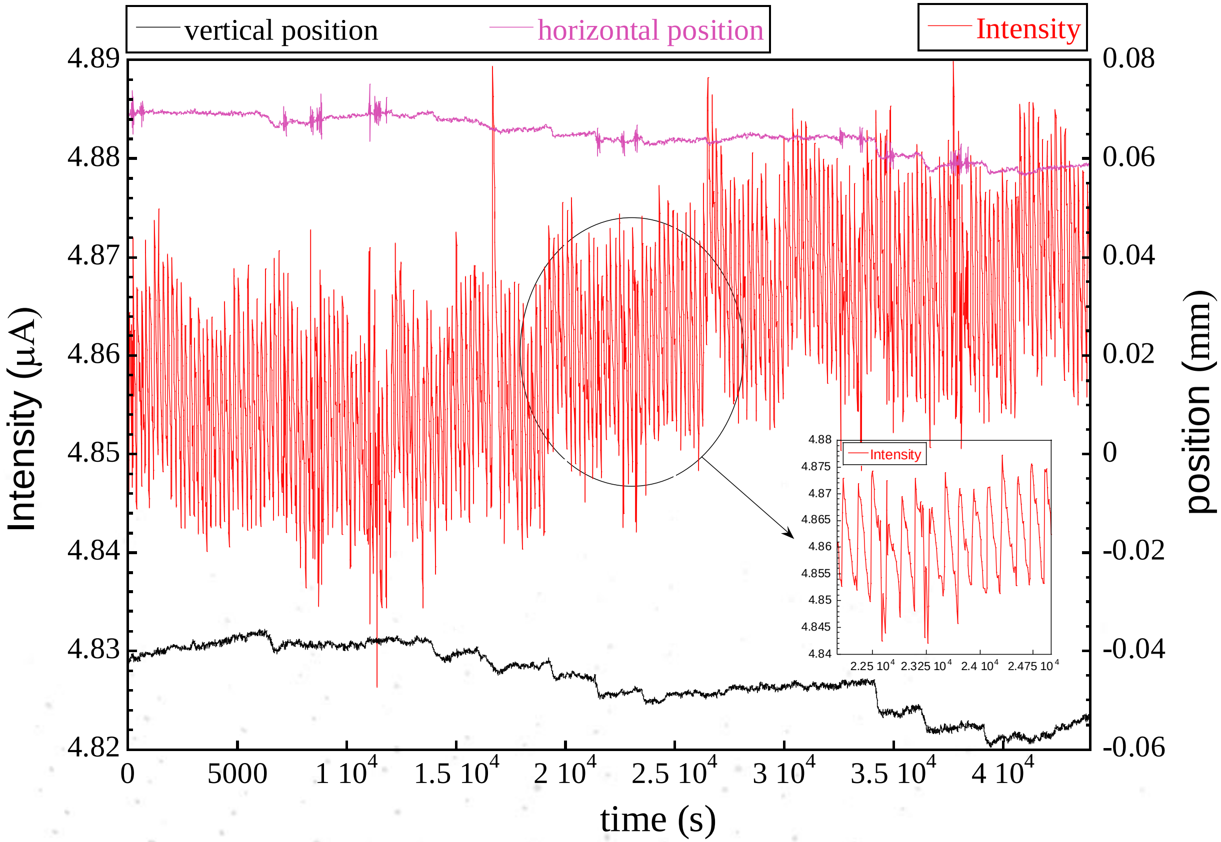


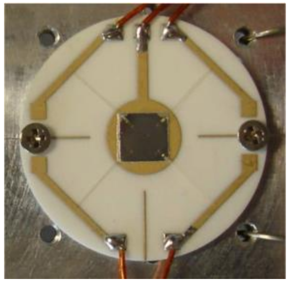
500 readings every 100 ms averaged at each angle, repeated 10 times with step = 2 deg

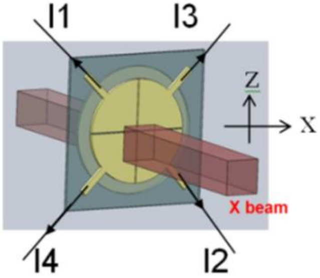


Stability during rep. test measured by encoder

Performances at fixed energy (on line)







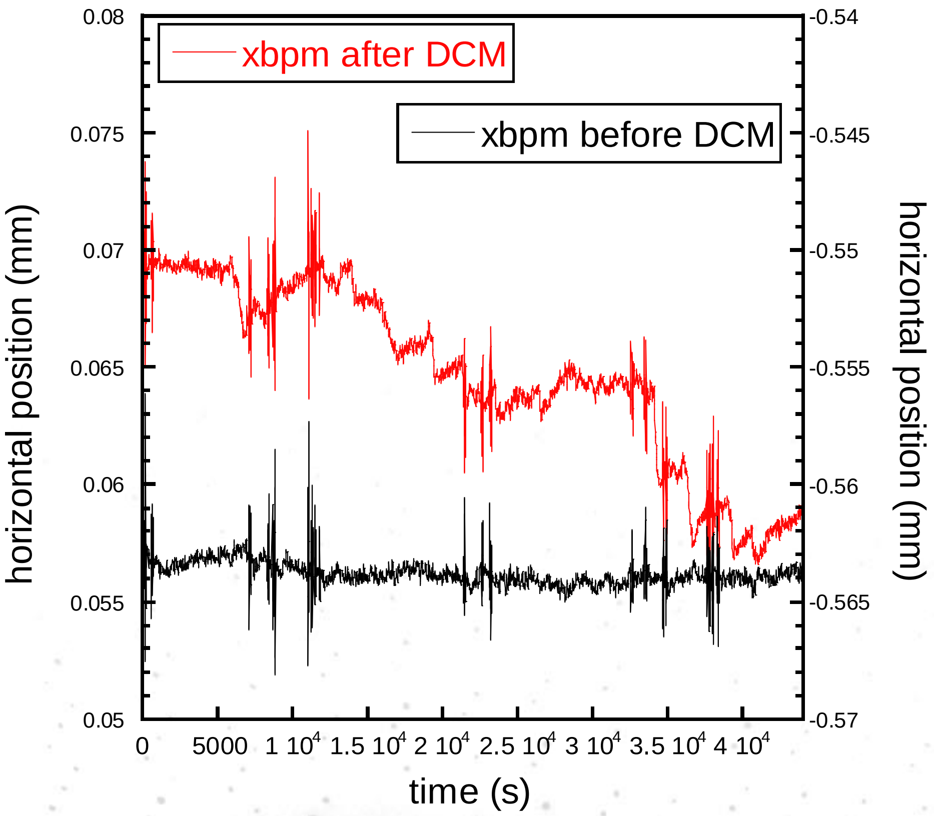
Beam intensity and position monitored with 4Q scCVD diamond [thickness =50µm, ϕ= 4mm, gap=50µm]. 27 m from source.

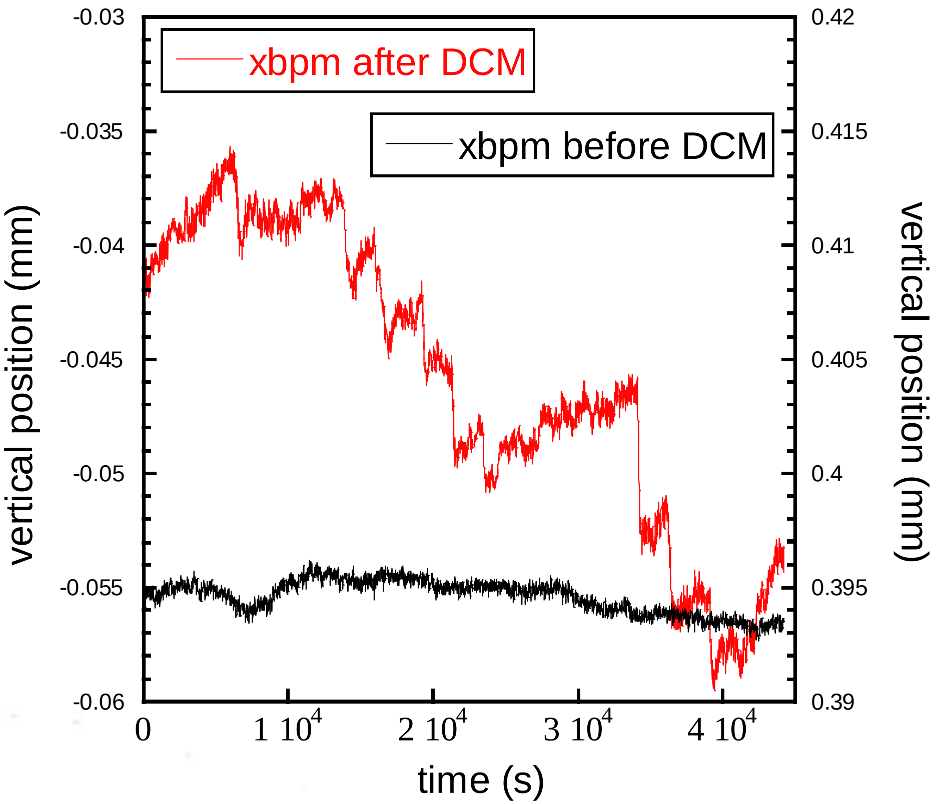
K. Desjardins et al., Journal of Physics Conferences Series 425, 212004, (2013)

- Vertical beam movement of 24 µm in ~12 hours
- Horizontal beam movement of 18 µm in ~12 hours
- Intensity variation < 0.4 % in ~12 hours

No stabilisation feedback

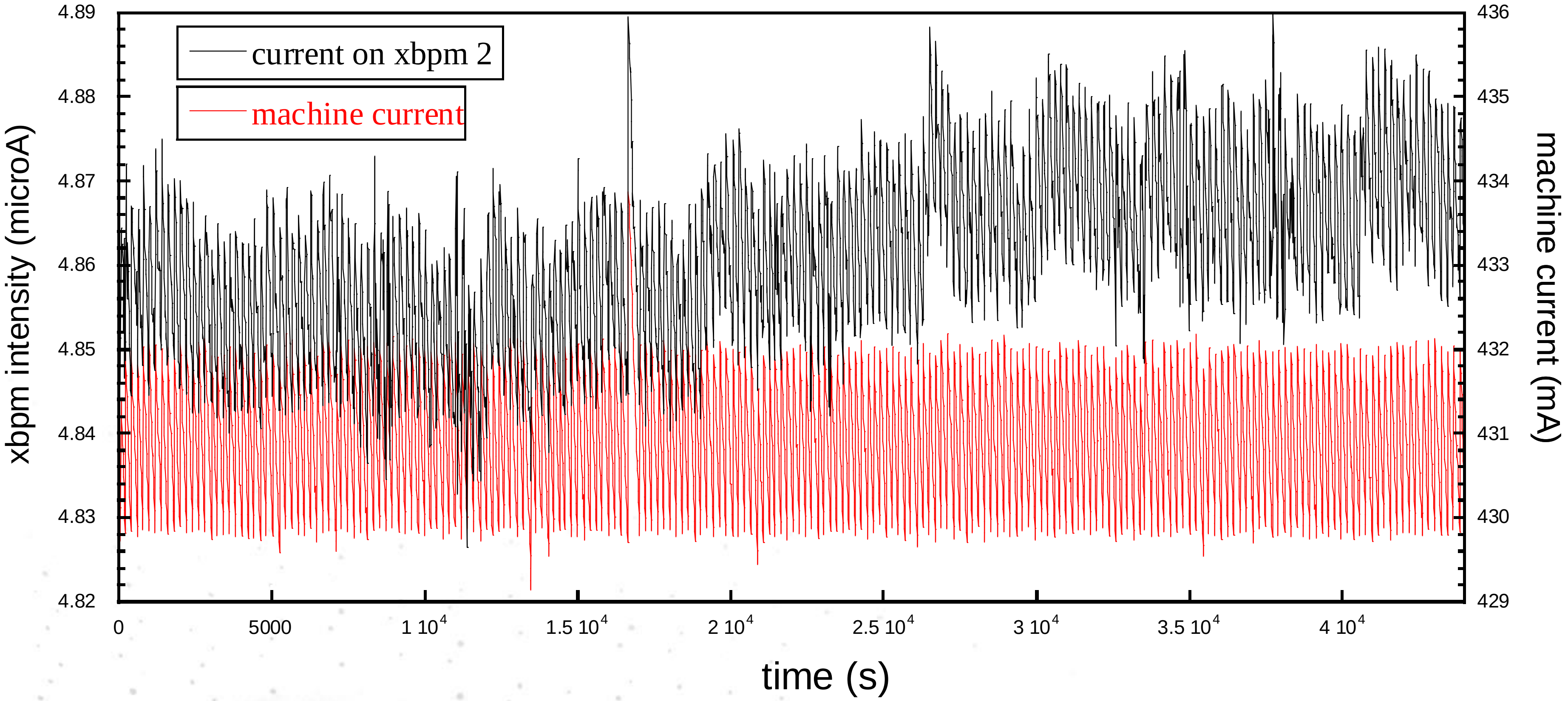
Performances at fixed energy





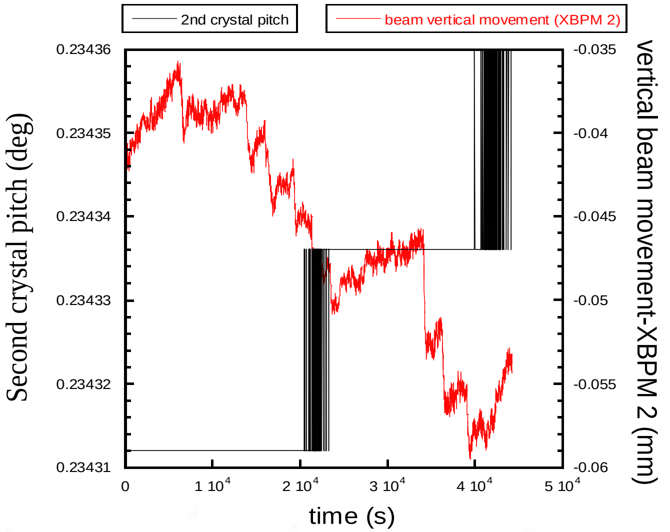
Horizontal and vertical beam movements not due to the source, they origin from the DCM

Performances at fixed energy

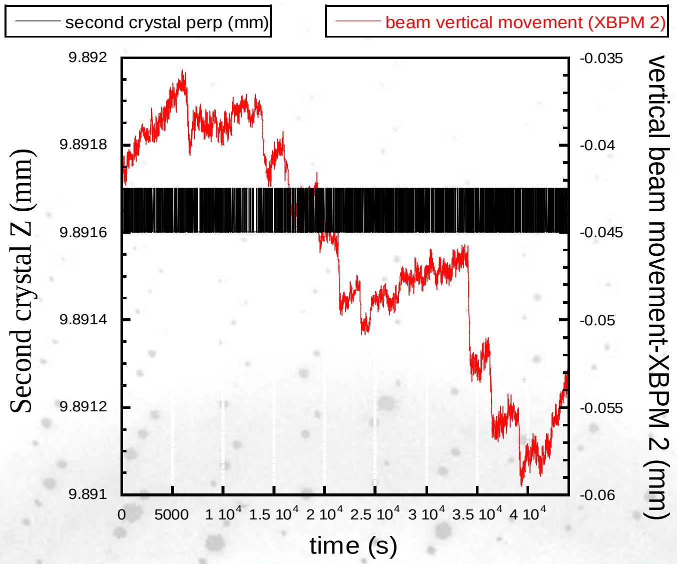


Intensity variations read by XBPM2 not related to oscillation in machine current

Performances at fixed energy



V

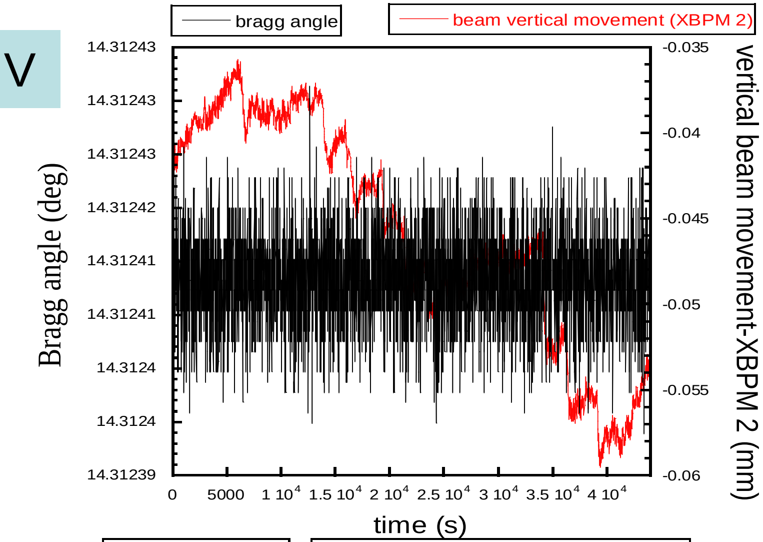


V

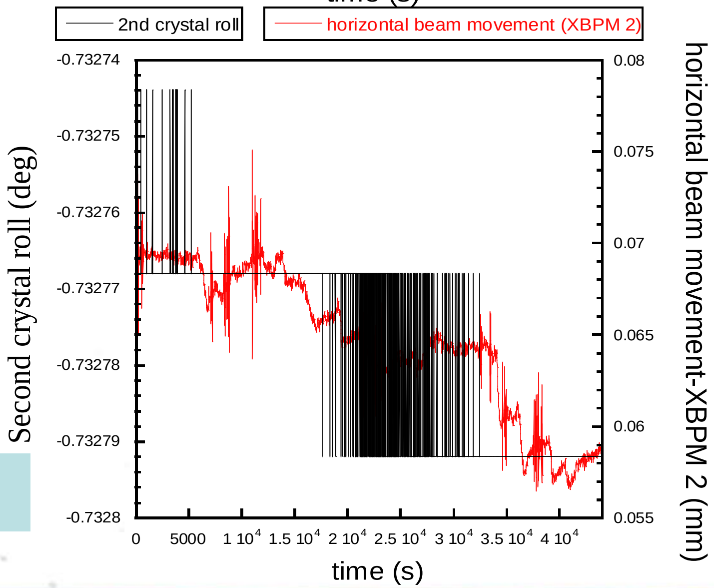
2nd crystal pitch, roll and perp (along with Bragg) do not change within repeatability specifications

But this can still give a movements of 13 microns for pitch and roll !!

V

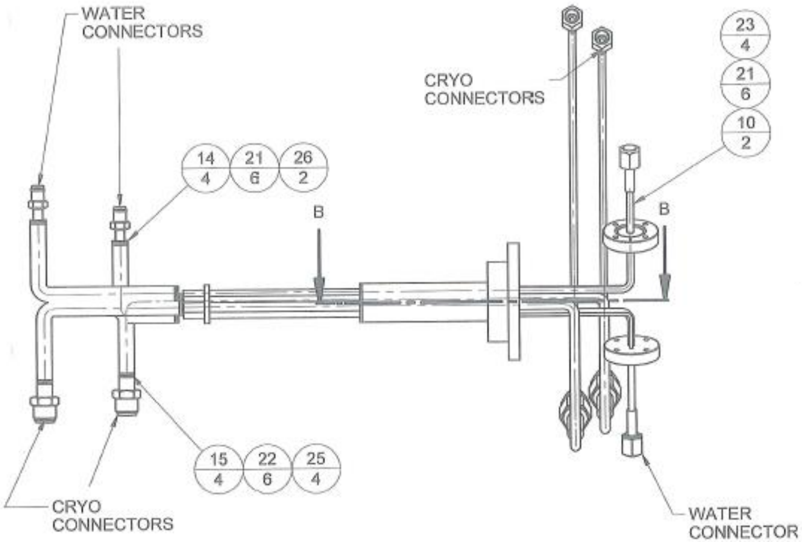


H

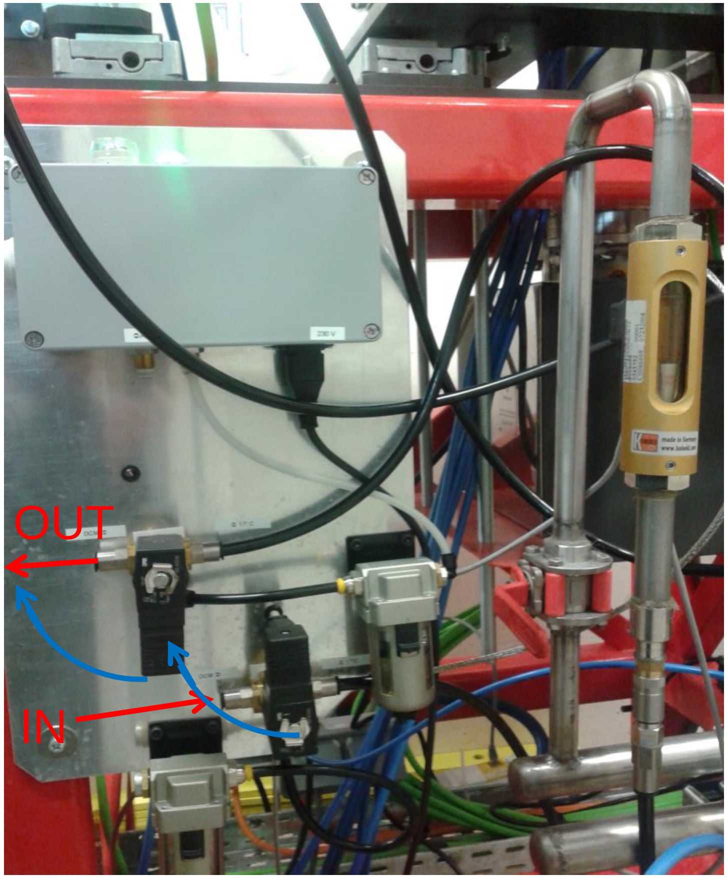


Performances at fixed energy/issues

Stability issues may be due to the fact that we do not use water cooling



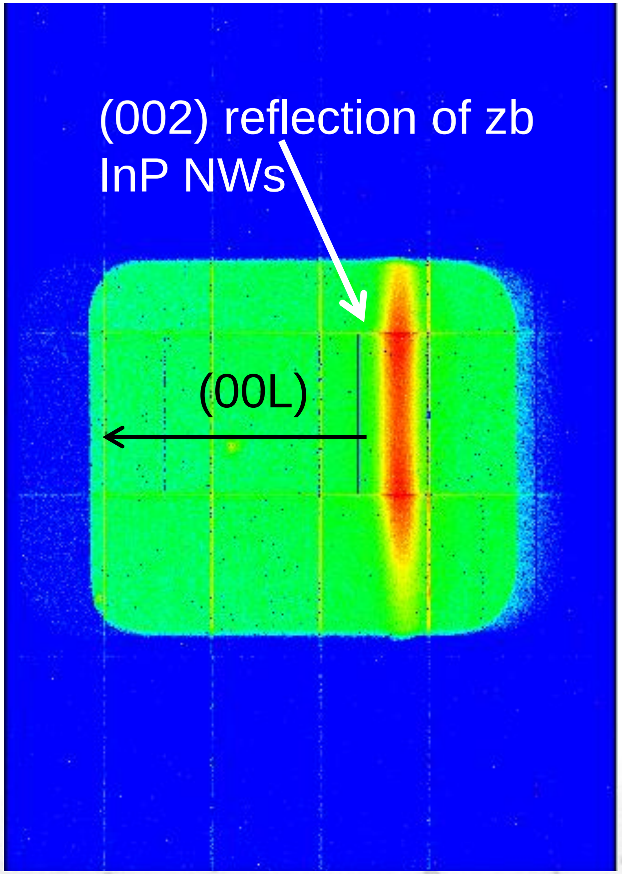
Water connectors too close to LN one: freezing when water supply fails !

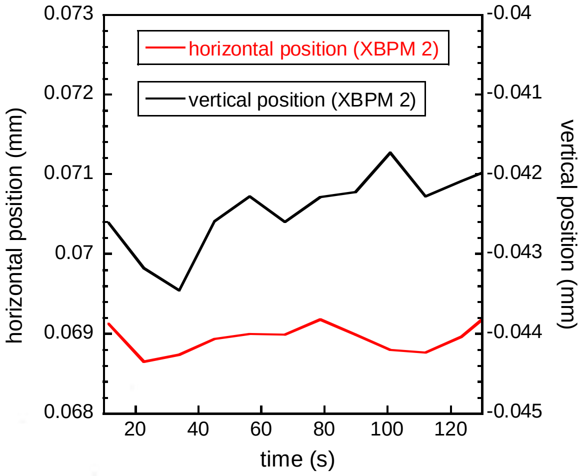


Home-made solution: it does not work yet. Improving it or chiller solution.

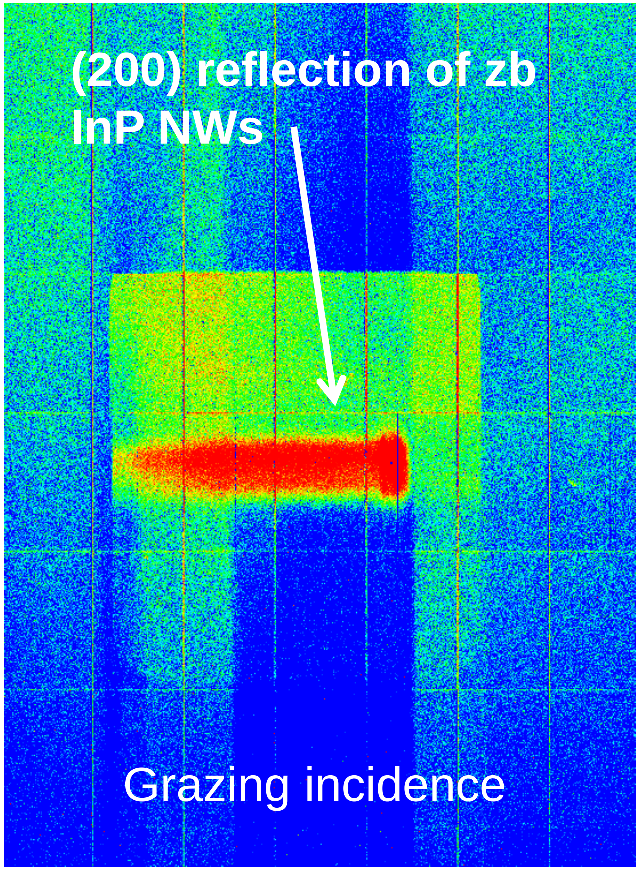
Performances at fixed energy - XRD

XRD test on low-density zincblende InP nanowires on Si substrate





We can obtain satisfying XRD data (even at GI) since the flux is high and beam movements are small over the integration time (<1 µm in 100 s)



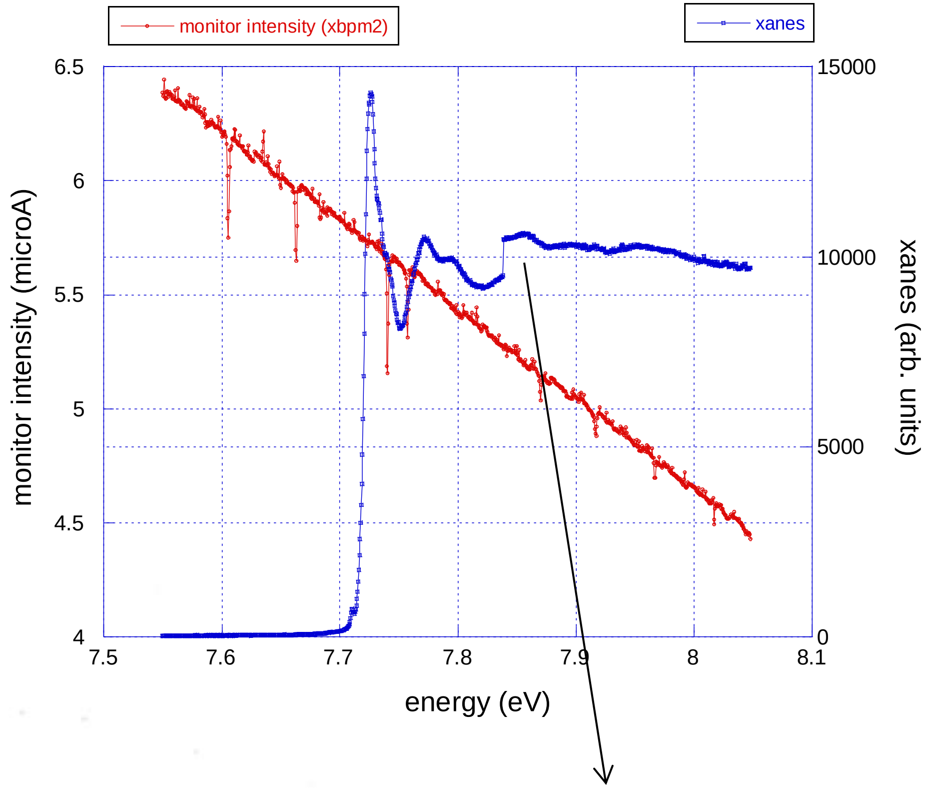
Performances in energy scan

Scans are performed by moving simultaneously monochromator Bragg angle and undulator gap

2 7 E	Rs2
5.0	-0.7134
6.0	-0.7121
7.0	-0.7333
8.0	-0.7335
9.0	-0.7337
10.0	-0.7030
11.0	-0.7030

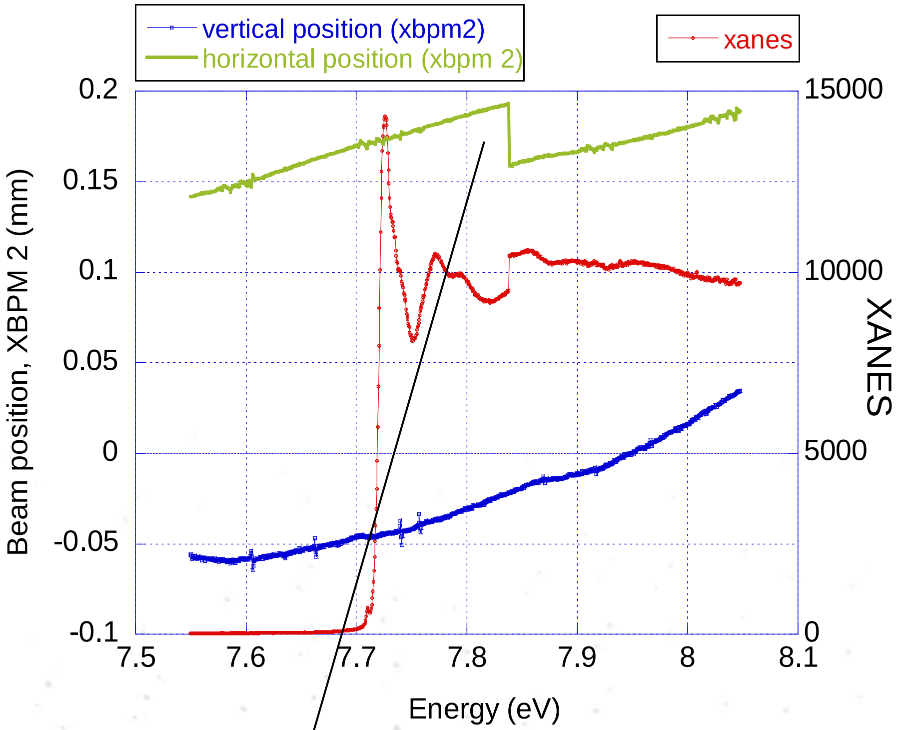
Tables used for 2^{nd} crystal pitch, roll and perp calibration during scan

No piezo used, no feedback to stabilise beam position/intensity

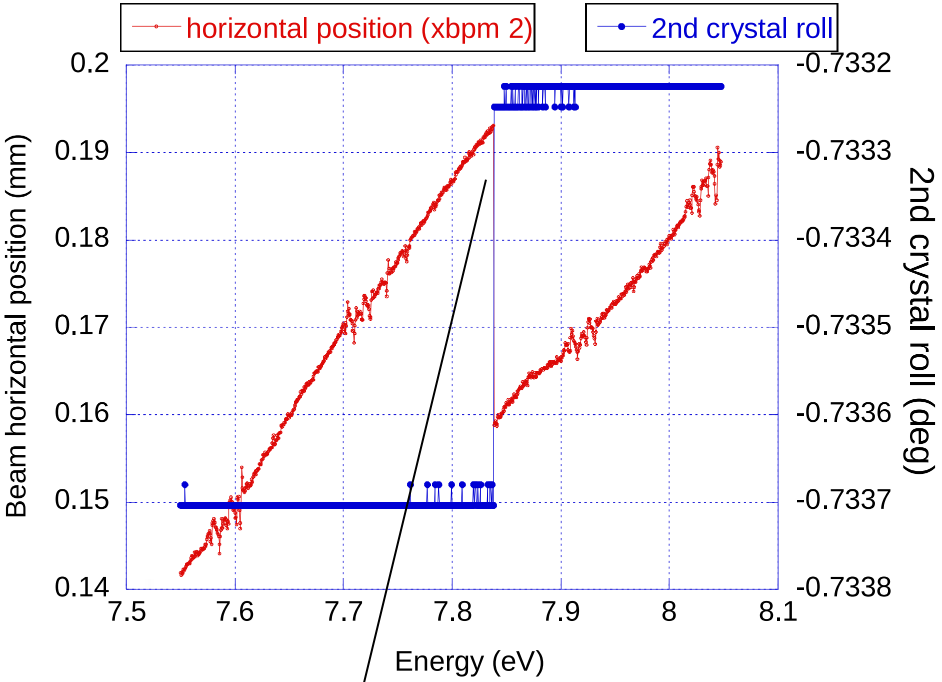


Step observed in XANES, not in monitor, spoils the spectrum

Performances in energy scan



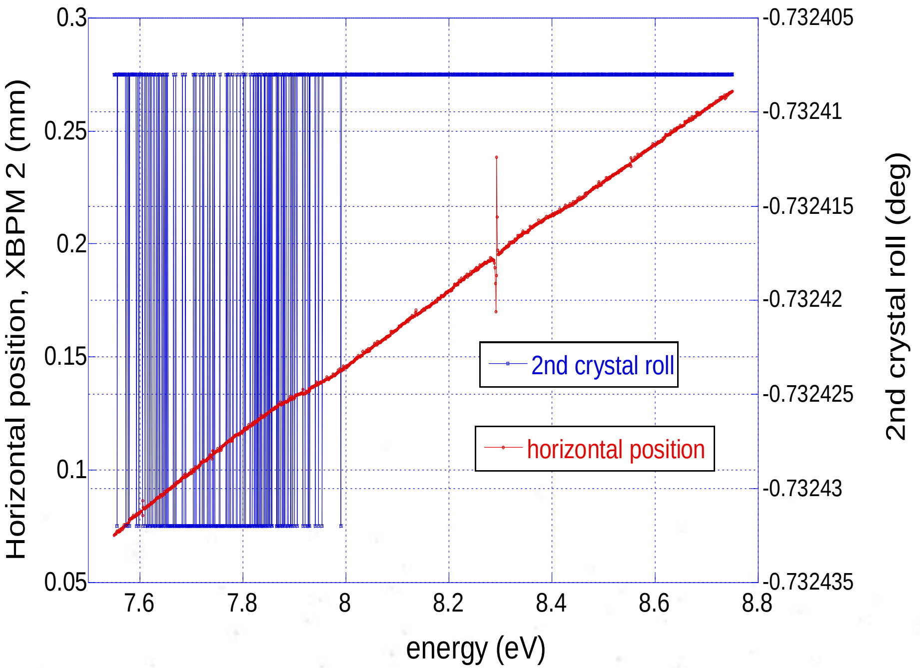
Step corresponds to beam horizontal movement of 40 microns

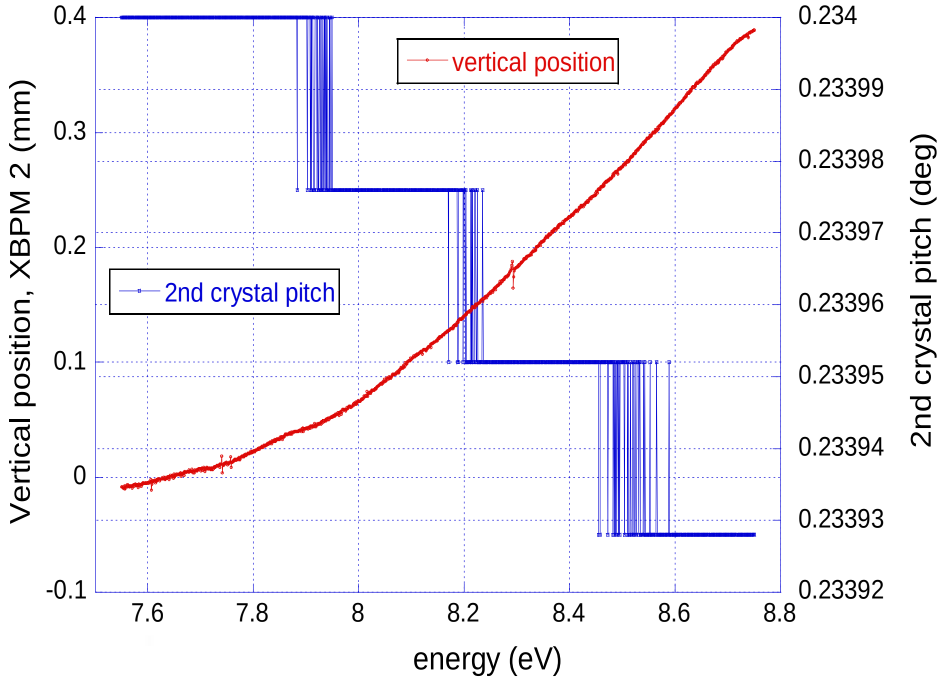


Beam horizontal movement is related to change in 2nd crystal roll ($5*10^{-4}$ deg)

Problem with roll resolution !! (due to Tango, not to Mono)

Performances in energy scan





Second roll fixed to average of optimized values in energy range of interest.

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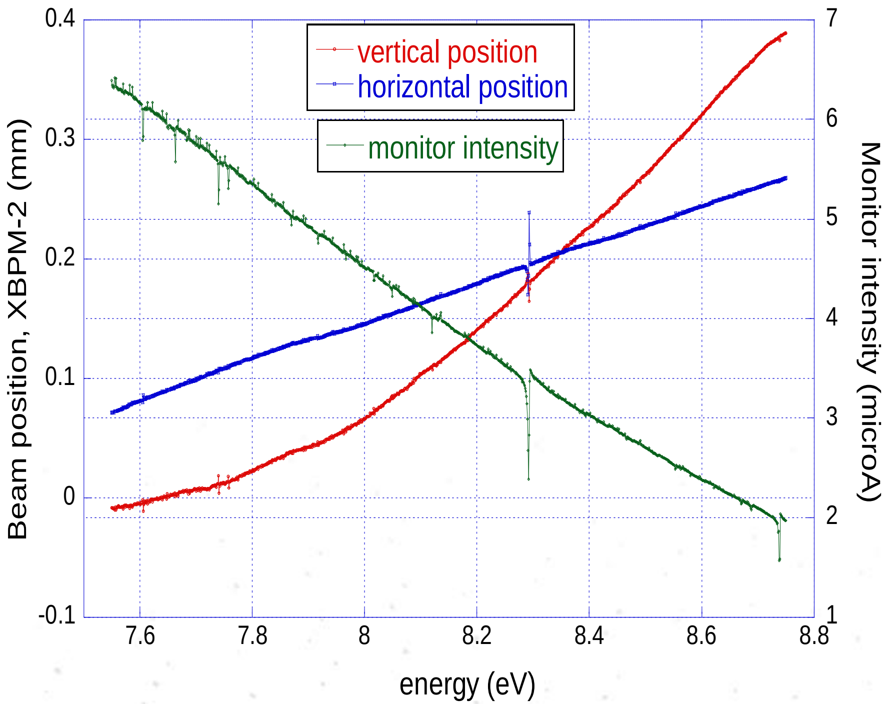
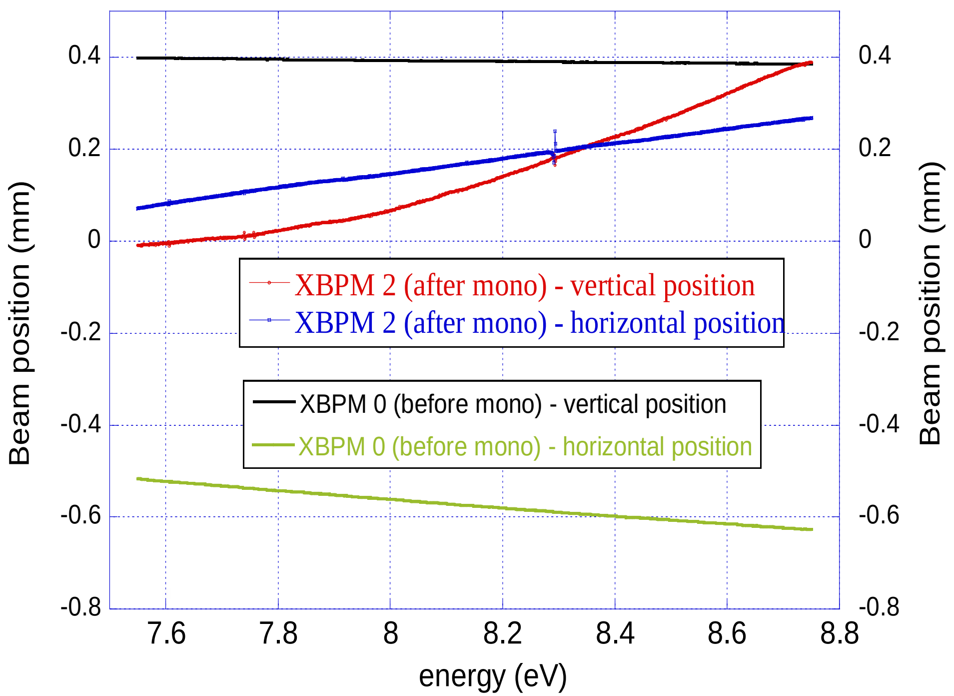
Pitch and roll do not vary more than repeatability now, but there are important beam movements during the scan.

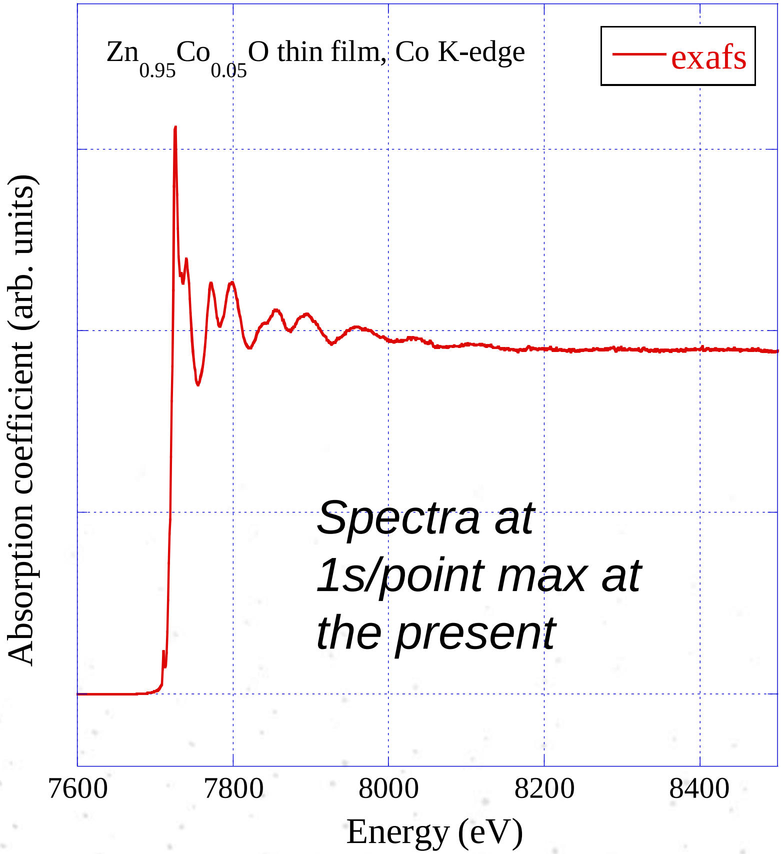
Performances in energy scan



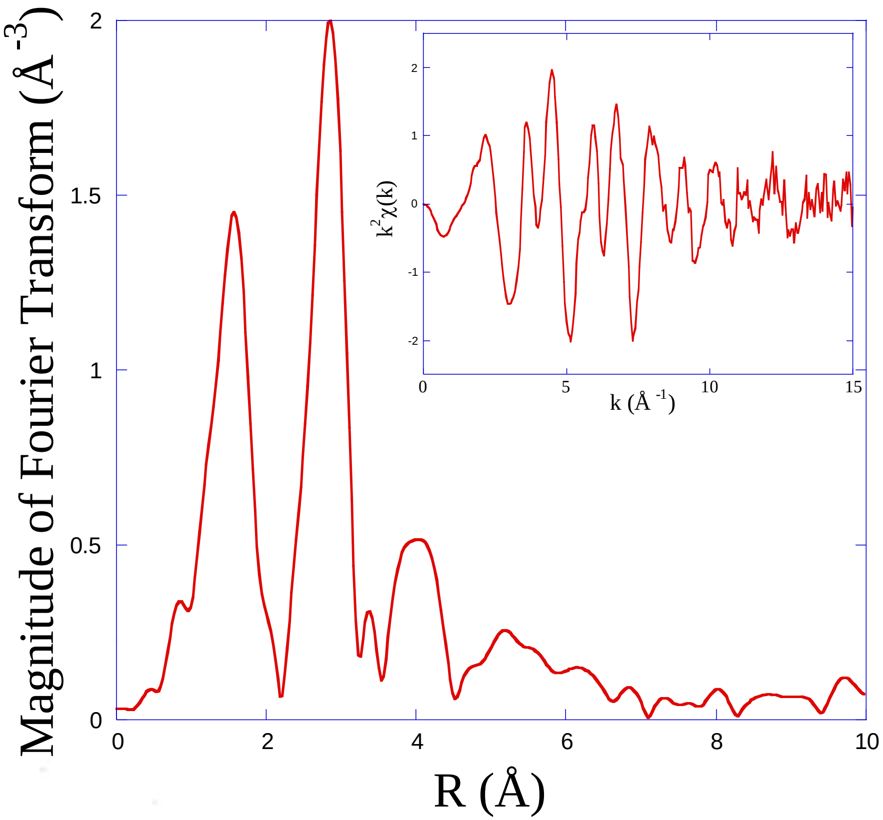


Beam movements after DCM larger than source movements, even if a contribution from the source is possible, especially in horizontal. Main contribution comes from the DCM.

Performances in energy scan

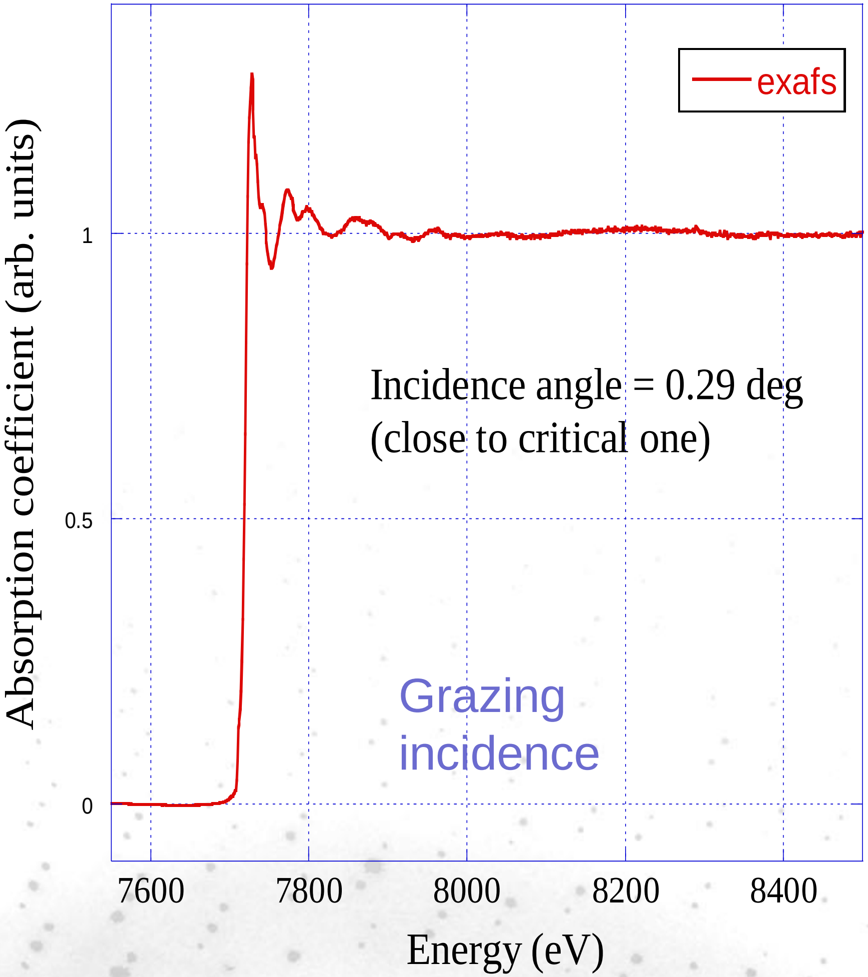


Spectra at 1s/point max at the present

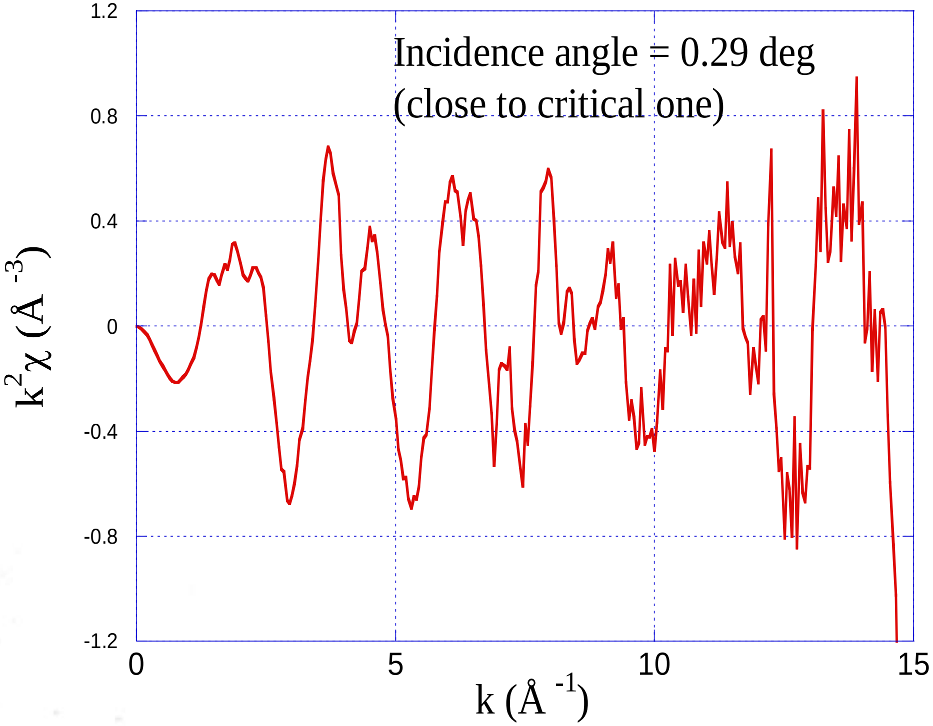


No more steps and good exafs spectra of dilute elements in nanostructures possible at Sirius fixing the crystal 2 roll, even without feedback.

Performances in energy scan

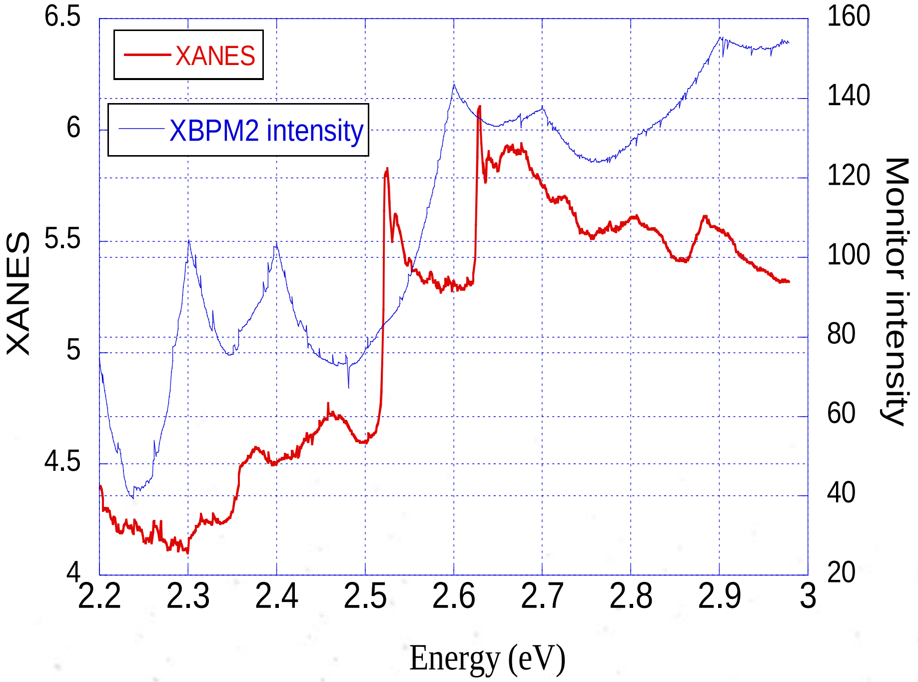


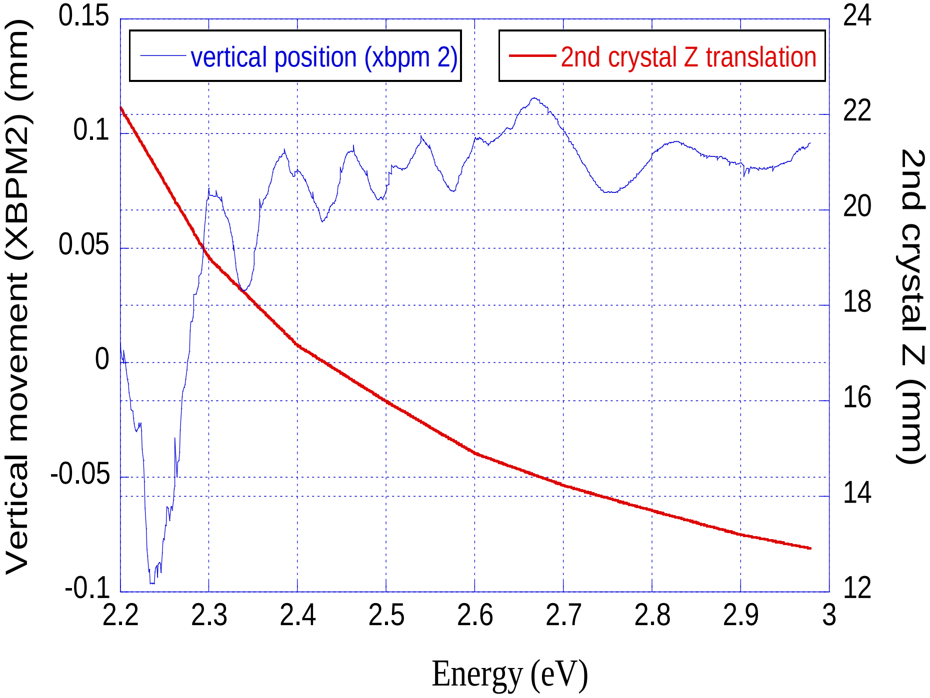
Grazing incidence



Also grazing incidence EXAFS feasible by placing a 50-100 micron slit before the sample to define beam spot (samples 5x5 mm)

Performances in energy scan/issues





Problems in beam intensity stabilisation during scans at low energy (< 3 keV) due to large variation of 2nd crystal perp. translation ⇨ many points needed in table.

Solutions: use of piezo on 2nd crystal perp/feedback ? InSb instead of Si at E < 3 ?

Issues and correction strategies

- Solving problem with 2nd crystal roll minimum step (Tango configuration): done !
- New test to re-introduce water cooling for long time stabilization
- Improving stability in beam position using one or two of the piezo on pitch/roll/perp (depending on experiment) along with the I-200 two-channel digital electrometer with analog output for servo control (Successfully tested at other beamlines in SOLEIL !)
- Increase of velocity in scans to fully exploit the DCM dynamics thanks to continuous energy scan/fly scan