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Overview and future needs for ESRF double crystal monochromators dedicated to spectroscopy

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INTRODUCTION





Grenoble 1968, winter Olympic games



Workshop dinner, close to the Olympic springboard



... but also because we propose to adopt for the monochromator workshop ... the Olympic motto, proposed by Pierre de Coubertin on the creation of the International Olympic Committee in 1894...

Citius, Altius, Fortius

... which is Latin for "Faster, Higher, Stronger."

Also because the Olympic symbol, the rings could be a good illustration of the role of the mono ...central ... but inside a complex instrumentation chain





Double Crystal Monochromator for spectroscopy

- Overview of ESRF double crystal monochromators dedicated to spectroscopy

- Present status and future needs

- Monochromator specifications for spectroscopy applications



SPECTROSCOPY AT ESRF

At ESRF, 14 beamlines are performing spectroscopy activity (+ 1 soft ID32 + 1 EDXAS ID24) 6 ESRF beamlines 6 CRG beamlines

ID12	Polarization dependent spectroscopy	Linear and circular dichroism, XANES
ID16B	NINA	Nano-XRF, nano-spectroscopy
ID20	IXS 1	Inelastic X-ray scattering
ID21	X-ray microscopy	Soft X-ray Nano-XRF, nano-spectroscopy
BM23	EXAFS	EXAFS, XANES, micro-XAS, XRF
ID26	XAS-XES	Emission spectroscopy, XANES, EXAFS

BM01B	SNBL	Combined XRD/XAFS/Raman measurements in operando conditions
BM08A	Gilda	EXAFS, XANES, Refl-XAS
BM20B	RoBL	Radiochemistry XAFS
BM25A	SpLine	EXAFS-XANES
BM26A	DUBBLE	EXAFS, XANES, catalysis infrastructure
BM30B	FAME	XAFS on highly diluted materials, XES, XRF and microXAS

All of them use a Double Crystal Monochromator



ESRF BEAMLINES – MONOCHROMATOR TECHNOLOGY

	ID12	ID16	ID20	ID21	BM23	ID26
Туре	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam
Manufact.	Kohzu	Kohzu	Kohzu	Kohzu	Kohzu	Kohzu
Crystals	111	111/311	111	111/220/ Multilayers	111/311/511	220/311
Angular stroke (°)	9 – 78	3 - 26	5 – 30	3 – 75	3 – 30	5-60
resolution	0.1 "	0.2 "	0.2 "	0.2 "	0.1 "	0.1"
Offset (mm)	-12.5	-12.5	Variable	-12.5	+25	-25
Cooling	He gas at -190 ℃, braids	LN2, side cooling	LN2, side cooling	N₂ at -4 ℃	LN2, braids	LN2, side cooling
Upgrade/ modification	Cooling	Cooling, Support	Cooling, geometry, Suppression of horiz. cam	Cooling	Cooling, Support, crystal cage	Cooling, motorization of the horiz. cam
Optimized for	Polarization, S/N	High energy with nano beam	Inelastic scattering	Low energy with micro beam	EXAFS, S/N, $\Delta E \mu XAS$	RXES, High flux



KOHZU DOUBLE CAM FIXED EXIT MONOCHROMATOR PRINCIPLE



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PERFORMANCE : REPEATABILITY

For a XAS beamline, the repeatability of the energy scale is crucial



PERFORMANCE : REPEATABILITY

For a differential measurement (XMCD, linear dichroism), where the difference between two successive spectra is performed, the repeatability of the energy scale is fundamental for the S/N ratio.



A. Ney and V. Ney, Linz University, Austria



PERFORMANCE : PRECISION

$$\frac{\Delta E}{E} = \frac{\Delta \theta}{tg \theta}$$

The energy at the edge can be calibrated But a reasonable precision should be maintained during the EXAFS measurement

 $k = 20 \text{ Å}^{-1}$, 1500 eV after the edge

$$\Delta E_{\rm max} = 0.5 \ eV$$

Error on photoelectron wave vector:

$$\frac{\Delta k}{k} = \frac{1}{2} \frac{\Delta E}{E_{kin}} = \frac{0.5}{1500} < 2 \ 10^{-4}$$



Demeter 0.9.13 © Bruce Ravel 2006-201

Error on distance:

$$\Delta R = R \frac{\Delta k}{k} < 2.5 \times 210^{-4} = 0.0005 \text{ Å}$$



PERFORMANCE : PRECISION

For multi edge XANES measurements, high precision is also necessary.



PERFORMANCE : STABILITY AT FIXED ENERGY

The stability of the beam intensity at fixed energy is very sensitive to any drift (thermal drifts or mechanical vibrations).

Important for XRF, combined diffraction and XAS in general ! Crucial for RXES





PERFORMANCE : STABILITY AT FIXED ENERGY

Mechanical stability, **vibration**: crucial for new applications like hyperspectral mapping where the **continuous scan** acquisition scheme is mandatory.



Upgrade of the monochromator support Kohzu metallic support \rightarrow ESRF granite support Upgrade of the cooling system He gas close circuit \rightarrow ESRF LN2 circuit



Main drawback of BM23 Kohzu monochromator

Crystal parallelism and "fixed" exit of the X-ray beam during scan In average for BM23 :

> 8 μrad/keV (8.5 μrad/deg.) 12 μm /keV (12.6 μm/deg.)



→ feedback on the piezo is mandatory to perform a XAS spectrum







Micro-beam trajectory in sample plane during an energy scan

- Si (111) monochromator -
- KB focused micro-beam _
- Energy range : 3 keV to 7.5 keV and back, 10 eV steps
- Angular range: 41.23 to 15.28 ° -
- Micro-beam position measured on fluorescence screen in KB focal plane with videomicroscope in BPM mode
- Δ =3µm in focal plane corresponds to $\Delta Ry = ~10 \mu rad$



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Micro-beam trajectory in sample plane during an energy scan

- 1- Beam movement is very reproducible
 - \rightarrow Present strategy : Compensation strategy "Spot tracking"
- 2 Better monochromator performance
 - \rightarrow Stronger specifications on $\Delta R_{v}, \Delta R_{x}$ and fixed exit

3 - Choice of the optical configuration to be less sensitive to monochromator imperfection

Vertical:



PERFORMANCE : AGEING BEHAVIOR

	ID12	ID16	ID20	ID21	BM23	ID26
Delivery	1993	1995	1997	1997	1993	1995

Remarkable longevity :

The DCM are operational and daily used for 20 years (ID12/BM23) !

Modifications have been done

- Cooling (all)
- Crystals mounting (all)
- Geometry (ID20)
- Cam system (ID26/ID20)
- Motors

Maintenance performed regularly

- Crystals
- Mechanics (principal gear, reduction)
- Motors
- Setup of the cam/translations

But certain parts have never been modified:

- Main axis
- Ferrofluidics seal



4 generations later !



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PERFORMANCE : AGEING BEHAVIOR

	ID12	ID16	ID20	ID21	BM23	ID26
Delivery	1993	1995	1997	1997	1993	1995

... but start to suffer from ageing





Mechanical wear on the worm gears damaged and replaced



Main rotation motor replaced

Degradation of the energy scale stability around certain very used edges

... And also a conception that is perhaps not adapted to new spectroscopy challenges (thermal, vibration, control, global conception)

Ageing : clear checks and maintenance procedures



FUTURE REQUIREMENT : SCIENTIFIC AND TECHNICAL GOALS

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS	Y	Y	Ν	Y	Y	Y
XANES	Y	Y	Y	Y	Y	Y
XRF	Y	Y	Y	Y	Y	Y
RXES/RIXS/NRIXS	Y	Y	Y	Y	Y	Y
Final state E scan	Ν	Ν	Y	Ν	Ν	Y
Combined diffraction	Y	Y	Y	Y	Y	Y
Micro/nano beam	Y	Y	Ν	Y	Y	Y
XRF Mapping	Y	Y	Ν	Y	Y	Y
Hyperspectral mapping	Ν	Y	Ν	Y	Y	Y
Step by step	Y	Y	Y	Y	Y	Y
Continuous scan	Ν	Y	Ν	Y	Y	Y

No request for asymmetric cuts, detuning mode, sagittal focusing or polarization transfer specifications.

FUTURE REQUIREMENTS : XAS

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS	Y	Y	Ν	Y	Y	Y
XANES	Y	Y	Y	Y	Y	Y

• Cover large number of elements edges \rightarrow Accessible angular range of the mono

•Number and type of crystals mounted inside the monochromator

Scan the angle (energy) → K = 20 Å⁻¹ = 1500 eV after the edge
At high energy = 0.3 deg.
At low energy ... 20 deg.

	ID12	ID16	ID20	ID21	BM23	ID26
Min. Bragg angle (deg.)	7	3	5	10	2	4
Max. Bragg angle (deg.)	80	20	30	81	35	70
Nb. of crystals pairs	2	2	1	2 to 3	2 to 3	>2
Crystal types	111/?	111/311	111	111/311/?	111/311 /511	111/220 311/411
Scan angle range (deg.)	0.1 - 4	0.2 - 4	0.1 - 1	0.5 - 20	0.2 - 8	0.2 - 8



FUTURE REQUIREMENTS : XAS

	ID12	ID16	ID20	ID21	BM23	ID26
XANES	Y	Y	Y	Y	Y	Y
RXES	Y	Y	Y	Y	Y	Y

• Minimum angle step :

 \rightarrow Energy step needed linked to the core hole life time γ

- A minimum step resolution of $\gamma/20$ is needed ($\approx 0.1 \text{ eV}$ step at Fe K edge, for example) $\Delta \theta = 3 \mu \text{rad}$ at Fe K edge ... and $\Delta \theta = 0.5 \mu \text{rad}$ at W K edge
- Advanced spectroscopy : $\gamma/40$ could be needed



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FUTURE REQUIREMENTS : ENERGY RESOLUTION

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS	Y	Y	Ν	Y	Y	Y
XANES	Y	Y	Y	Y	Y	Y
RIXS	Y	Y	Y	Y	Y	Y

•Energy resolution :

$$\Delta E / E = (\omega_D^2 + \Psi^2)^{1/2} \cot \theta_B$$

Darwin width $\omega_{\!\mathsf{D}}$ is linked to the crystal

 Ψ is a complex contribution that includes The divergence of the X-ray beam All deformations of the crystal that can affect the front wave (crystal fixation, thermal deformation, surface polishing ...) $\Psi \ll \omega_D$

	ID12	ID16	ID20	ID21	BM23	ID26
Intrinsic broadening Ψ (μrad)	< 1	< 1	< 1	< 4	< 1	<1

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FUTURE REQUIREMENTS : REPEATABILITY/STABILITY OF THE ENERGY SCALE

	ID12	ID16	ID20	ID21	BM23	ID26
XANES	Y	Y	Y	Y	Y	Y
Hyperspectral mapping	Ν	Y	Ν	Y	Y	Y

In static mode

For XRF, not so crucial \rightarrow < 1 eV at 20 keV (1 arcsec.)

For combined XRD \rightarrow < 1 eV at 25 keV (1 arcsec.)

In scanning mode From one scan to the other : repeatability in the order of 10 meV at Fe k edge $\rightarrow 0.5 \,\mu rad \,(0.1 ")$ on the main Bragg angle

Stability: Maintain the repeatability for 24H00

	ID12	ID16	ID20	ID21	BM23	ID26
θ _B repeatability (arcsec.) over 24H00	<< 0.1	0.1	0.1	0.5	0.1	< 0.1



FUTURE REQUIREMENTS : PRECISION OF THE ENERGY SCALE

	ID12	ID16	ID20	ID21	BM23	ID26
XANES (multi edges)	Y	Y	Y	Y	Y	Y
EXAFS	Y	Y	Ν	Y	Y	Y

EXAFS : the precision of the energy scale determines the precision on the distance of neighbors (over one EXAFS scan) \rightarrow 0.5 eV : 5 arcsec.

XANES/XMCD : multiple edges measurements (over 10 deg.) < 100 meV \rightarrow 1 arcsec.

	ID12	ID16	ID20	ID21	BM23	ID26
Angle precision (arcsec.)	< 0.5	1	1	1	1	< 0.5



FUTURE REQUIREMENTS : BEAM POSITION STABILITY (STATIC MODE)

	ID12	ID16	ID20	ID21	BM23	ID26
XRF	Y	Y	Y	Y	Y	Y
XRF maps	Y	Y	Ν	Y	Y	Y

Notion of stability in **static mode** is linked to the **duration of the experiment** (maps)

Constraints could be different between macro beam operation micro/nano beam operation

The stability requirements are defined **on the sample** and then interpreted in terms of thermal drifts, mechanical drifts and vibrations limits on the monochromator

	ID12	ID16	ID20	ID21	BM23	ID26
∆z, ∆y on sample over 24H00, unfocused (μm)	1 by 1	0.005 by 0.005	0.5 by 0.5	0.03 by 0.03	1 by 1 0.2 by 0.2	1 by 1



FUTURE REQUIREMENTS : BEAM POSITION STABILITY (SCANNING MODE)

	ID12	ID16	ID20	ID21	BM23	ID26
XANES	Y	Y	Y	Y	Y	Y
EXAFS	Y	Y	Ν	Y	Y	Y

Notion of stability in **scanning mode** is linked to the **angular range of the scan**.

Again, constraints could be different between macro beam operation micro/nano beam operation

The monochromator should be intrinsically stable and errors should be reproducible (lookup tables corrections) as much as possible. Residuals errors could be optimized with active feedback : control/communication issue

	ID12	ID16	ID20	ID21	BM23	ID26
∆z, ∆y on sample over 1 deg. (μm)	1 by 1	0.01 by 0.01	0.5 by 0.5	0.05 by 0.05	0.2 by 0.2	1 by 1
∆z, ∆y on sample over 5 deg. (μm)	1 by 1	0.025 by 0.025	NA	0.15 by 0.15	0.5 by 0.5	1 by 1
Δz , Δy on sample over 20 deg. (μ m)	NA	NA	NA	0.15 by 0.15	NA	NA



FUTURE REQUIREMENTS : BEAM POSITION STABILITY (SCANNING MODE)



	ID12	ID16	ID20	ID21	BM23	ID26
∆Ry over 1 deg. (μrad)	0.2	0.15	0.2	0.1	0.1	0.1
∆Ry over 5 deg. (μrad)	0.5	0.5	NA	0.2	0.25	0.25
∆Ry over 20 deg. (μrad)	NA	NA	NA	0.5	NA	NA
ΔRx over 1 deg. (μ rad)	0.2	1.5	3	0.7	0.7	0.7
ΔRx over 5 deg. (μ rad)	0.5	1.5	NA	1.4	1.4	1.4
∆Rx over 20 deg. (μrad)	NA	NA	NA	1	NA	NA
∆Rz (μrad)	10	10	10	10	10	10

FUTURE REQUIREMENTS : CONTINUOUS SCAN

	ID12	ID16	ID20	ID21	BM23	ID26
Continuous scan	Ν	Y	Ν	Y	Y	Y

Stability (vibration) issues are critical for continuous scan mode as the energy (angular) scale becomes also a time scale.

Bi-directional energy scan becomes important for rapid continuous scan.

Control issues:

The monochromator should communicate with the detection and with the source.

Complex trajectory of the Bragg angle could be envisaged Accurate measurement of the Bragg angle at full speed

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS (s/scan)	NA	3	NA	10	1	1
XANES (s/scan)	NA	1	NA	3	0.2	0.2



CONCLUSIONS

Vertical and Horizontal homogeneity of the requests

Angle range: Low energy / high energy monochromator compatible ?

We have presented a list of requirements for a future double crystal monochromator dedicated to spectroscopy....

.... That are in general a factor 10 more stringent than the present performance announced by the main industrial suppliers

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CONCLUSIONS



And for the double crystal monochromator dedicated to spectroscopy ... the winners are....



Thank you for your attention

