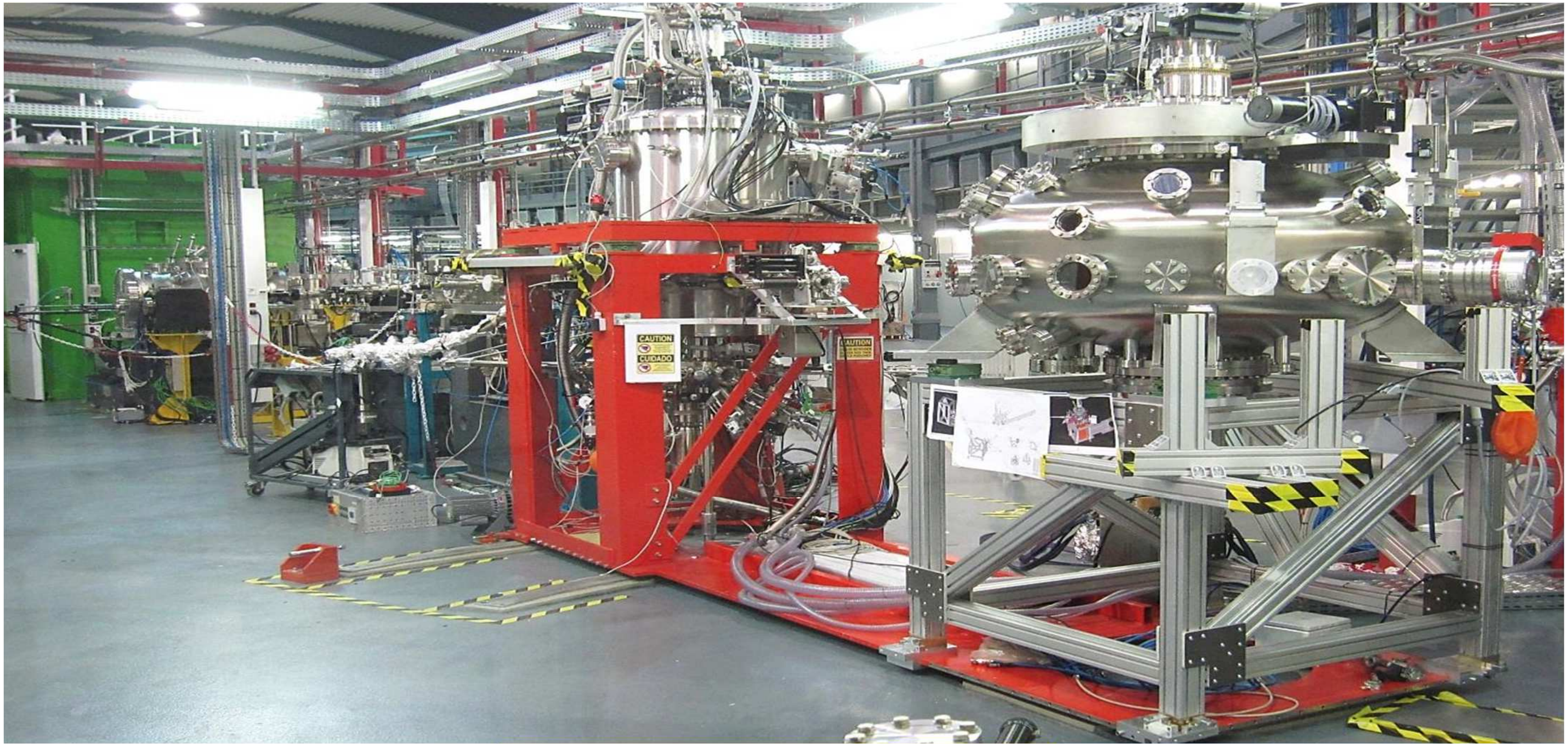
# BL29 BOREAS

## Resonant X-ray Absorption and Scattering



# Soft XAS/XMCD & RMXS beamline



*Scientific case*

> Apple II undulator + VLS gratings mono → 80-4000 eV energy range

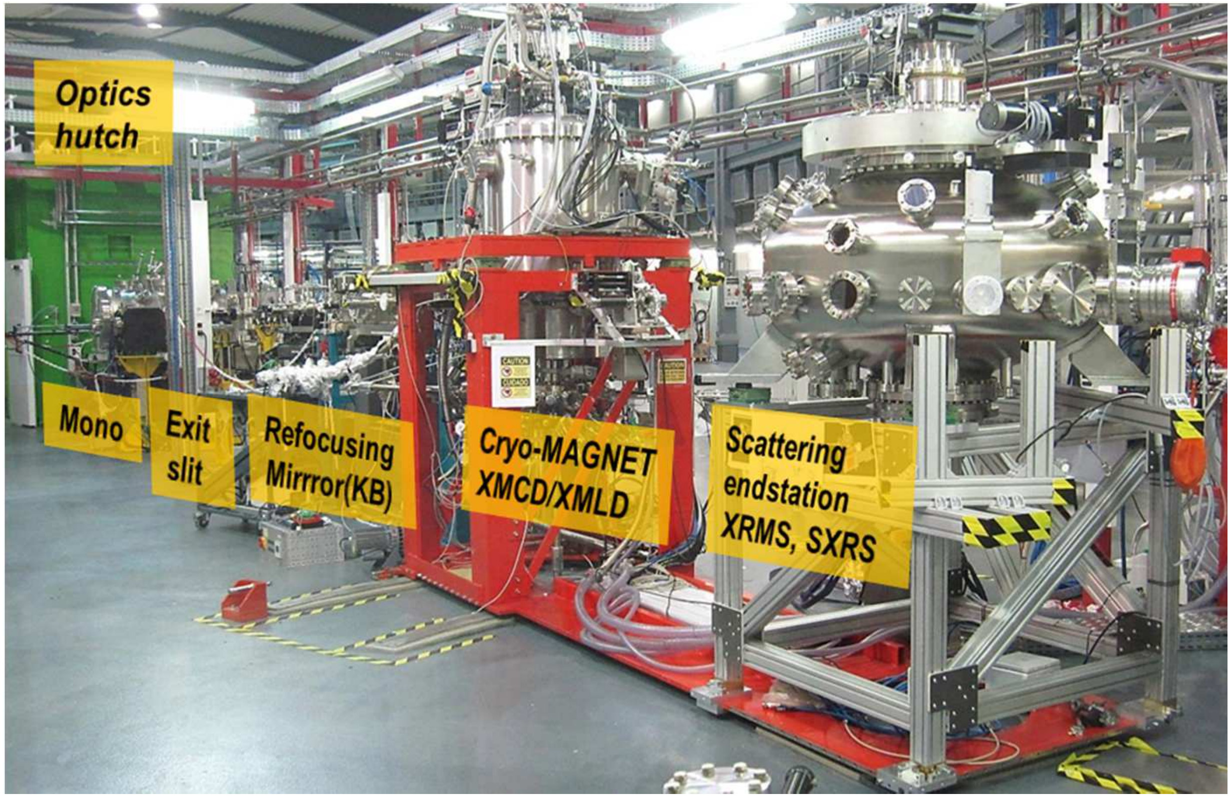
> 2 endstations - *HECTOR* 6 T cryomagnet & *MARES* chamber+ 2 T magnet

*Outline*

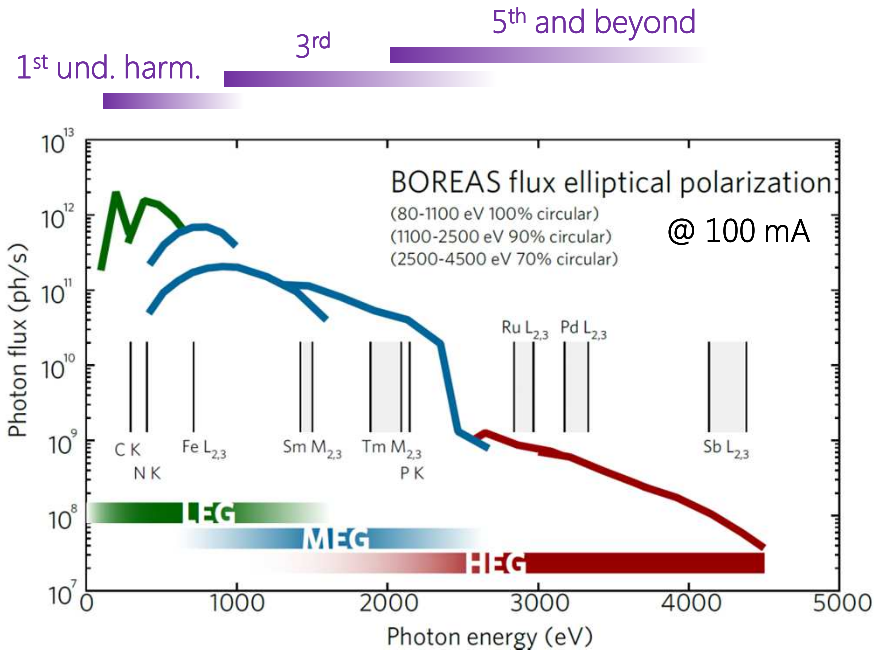
1) BL performance

2) Endstations status

3) Users' output



# BL performance - monochromator




## *Issues & Interventions (by Toyama)*

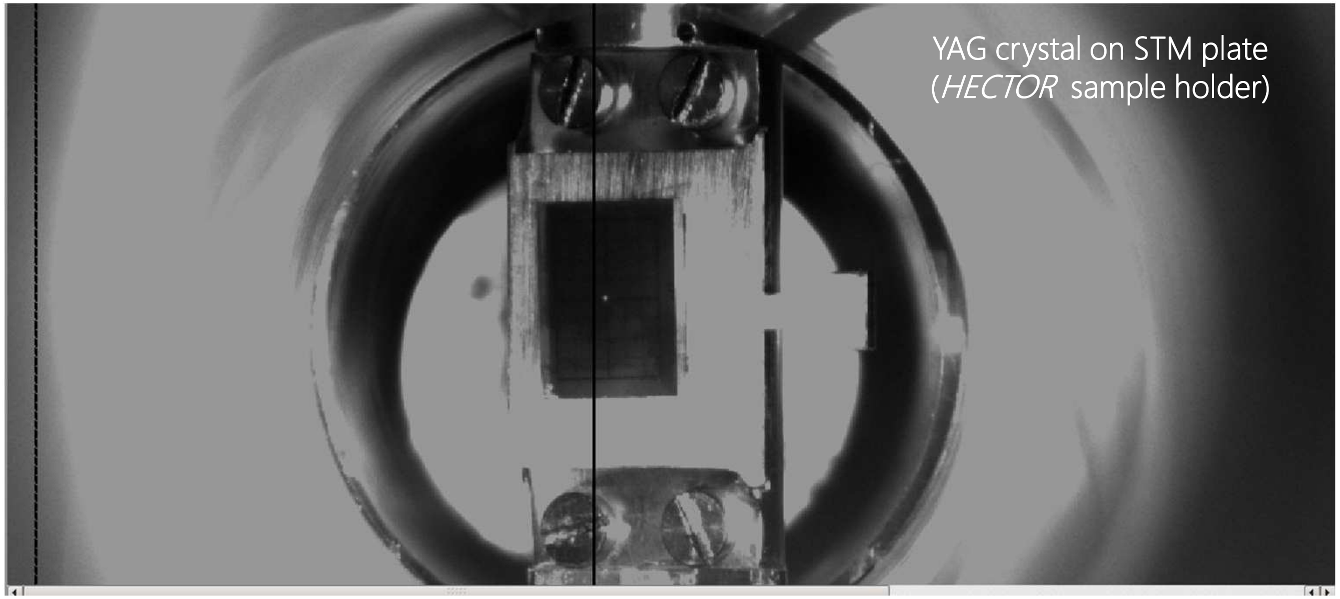
> March'13 : grating exchange motion friction (risk of leak opening)

> April'14 : SM1 pitch blockage and SM double piping (DP) leak, friction (E reproducibility)

> end 2014 (foreseen) : grating DP leak (Temp. stability)

# BL performance – KB mirrors

*Intervention on april '14*



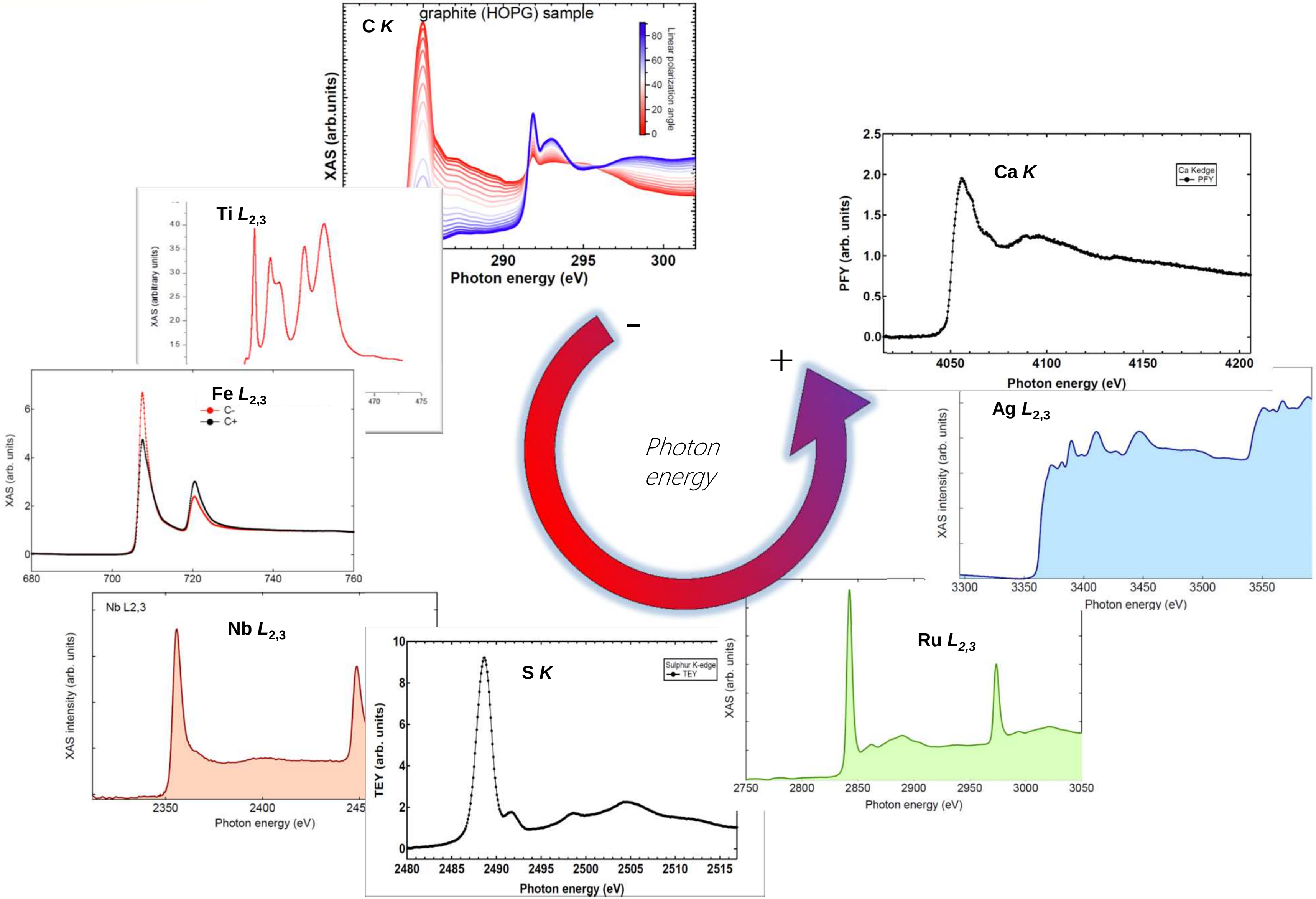

> Blocked translations fixed (replacement of damaged IRELEC motors)

> Beam spot size not tunable before april'14 ~ 300 (H) x 800 (V) um (fixed)

Variable beam spot size from april'14. Best focusing ~ 80 (H) x 30 (V) um

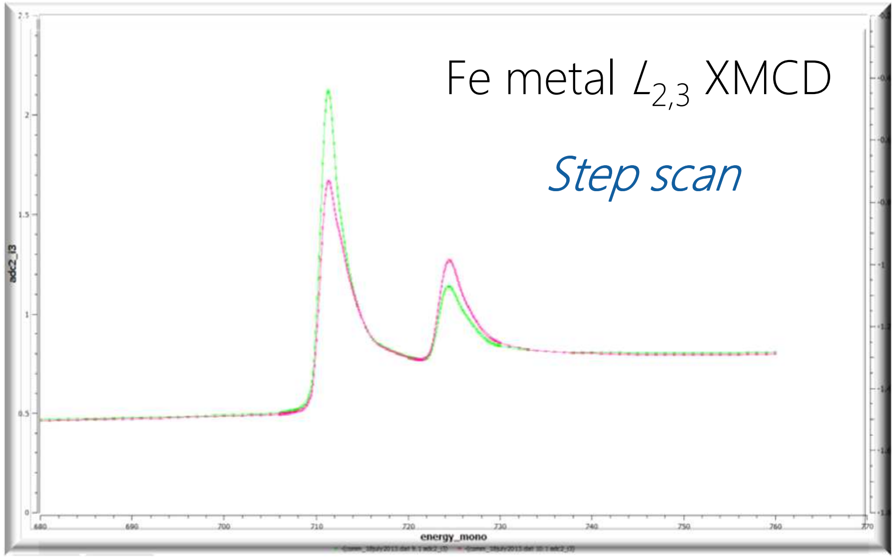
# BL performance - E calibration & refs.



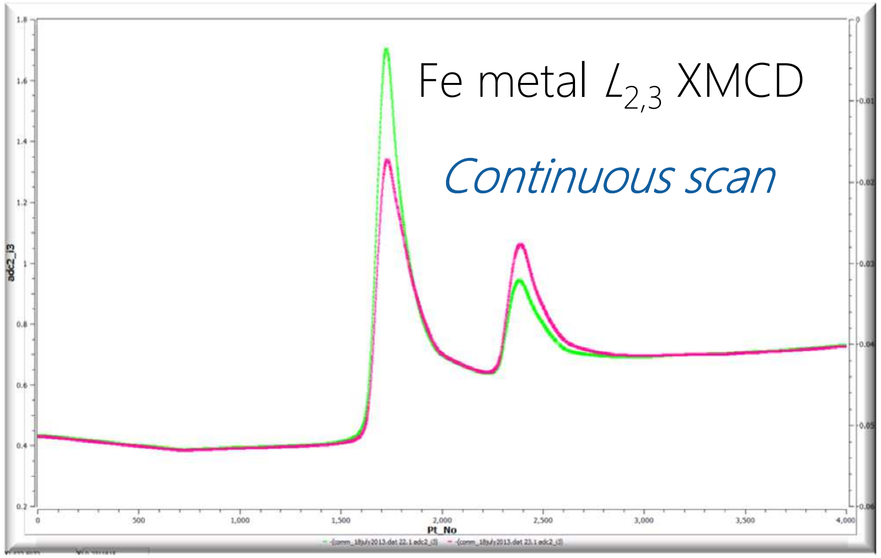


# BL performance - continuous scans






Variable step (open loop) und.+mono movement
Total scan time ~ 18 min.

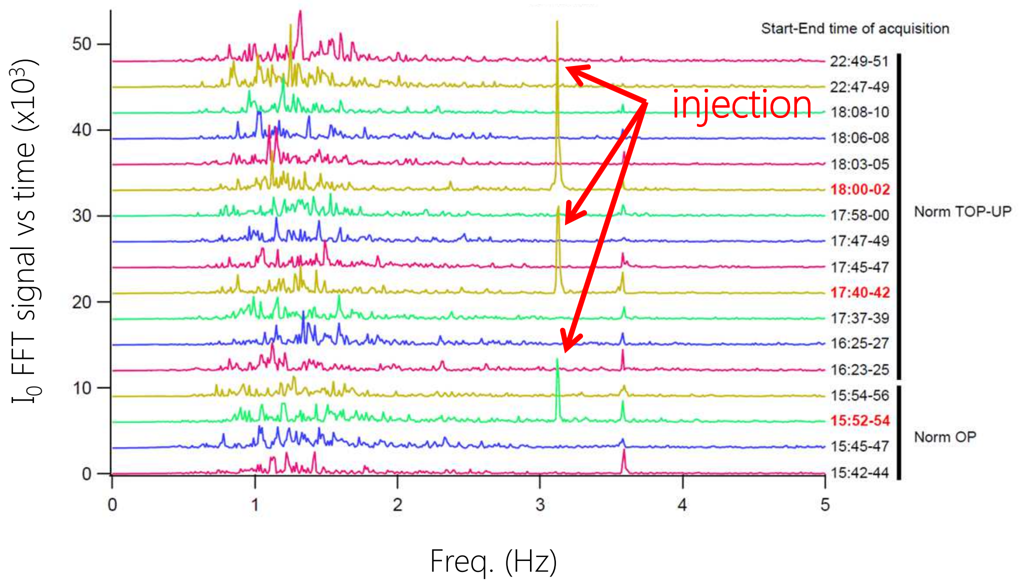


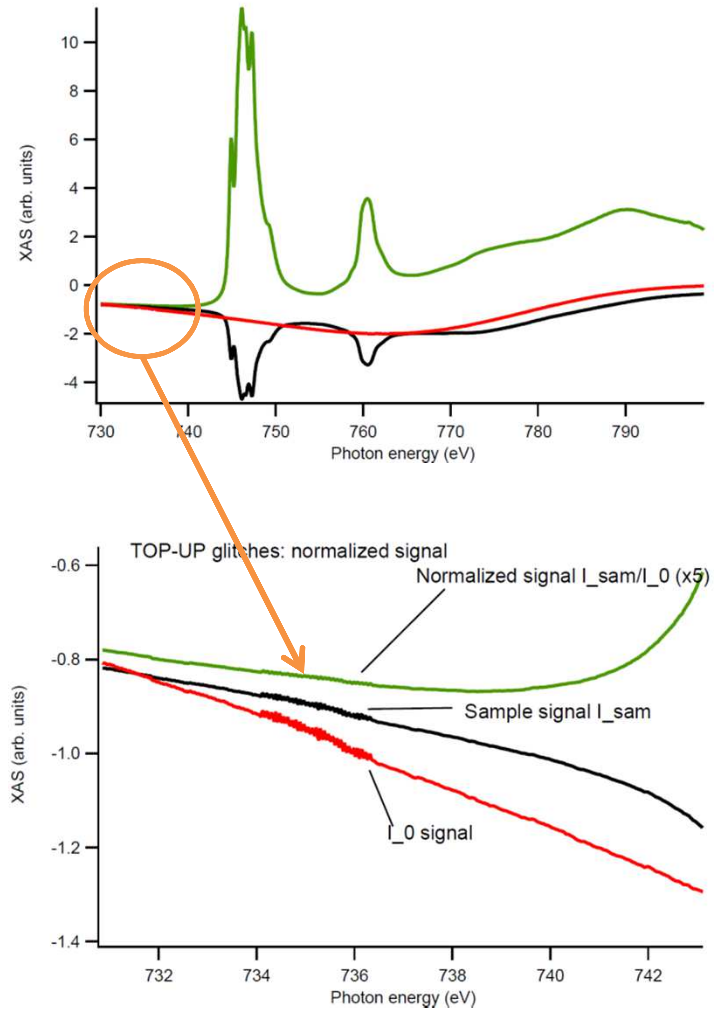

Continuous und.+ mono movement.
Total scan time ~ 2 min. (same resolution as step mode)

- High sampling frequency → larger density of pts/eV
- Less time between scans → better reproducibility
- Faster scans → more efficient use of beamtime
- *Pending:* implementation for "antiparallel mode" (i.e. linear intermediate pol.)

# BL performance - TOP UP tests





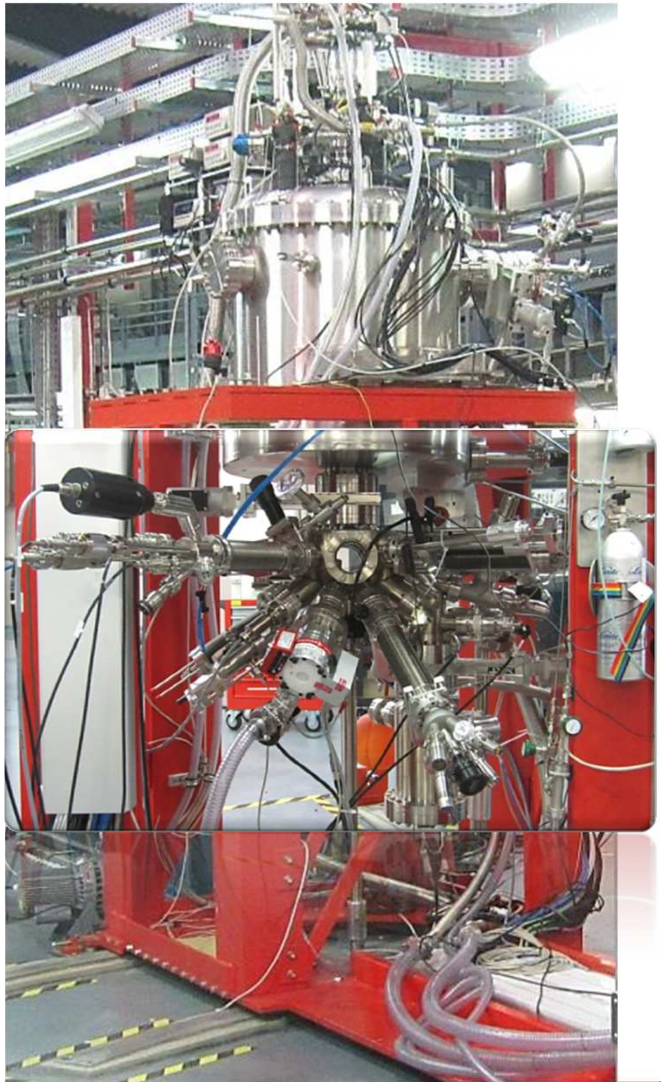

> During injection we see **noise increase** @ 3.125 Hz due to kickers operation

> **Limited impact** on BL op. since number of affected scans is small

> The injection process ~10/20 s usually affects **small portion of the spectra** (typical scans take ~2 min)

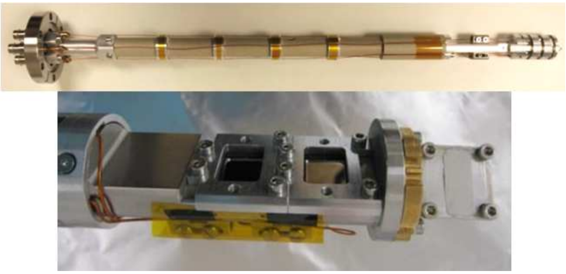
*Conclusion* : No gating needed so far at BOREAS for TOP-UP operation

# BL endstation 1 - XMCD cryomagnet



1st endstation, "HECTOR" SC 6T/2T/2T magnet with VTI

> Running user experiments since May 2012 (29 till today, >5 days long in average)
> Performs according to specs ($B_y$ up to 6 Tesla, 1.5 K< *T* <370 K)
> Detection modes: TEY + TFY, transmission



Recent upgrades

> Improved vacuum scheme (safe if blackout)
> Broadened remote *macro*-controlled operation
> Motorization of transmission diode arm
> Auto *magnetic field-on* flashing lights

In progress upgrades

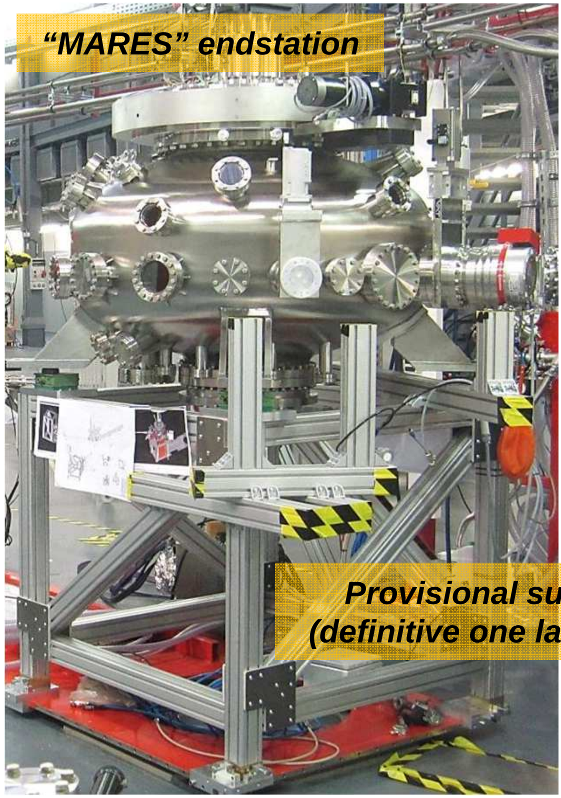
> Fast loadlock (up to 4 sample holders simultaneously)
> Sample bias (to enhance TEY with X, Z- applied field)
> Anti-ice / condensation barriers
> Definitive Scaffold (for LN, LHe refill…)
> PFY detection (collaboration agreement with MPI Dresden under study)
> Dock for travelling suitcase, STM (coll. agr. with ICN2 under study)

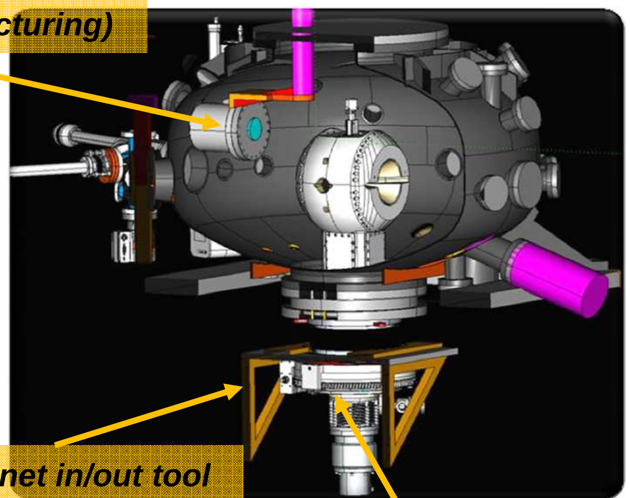
Growing equipment @ preparation chamber

> Heating stage, evaporators, wobble stick, gas lines, ion gun, quartz balance, mini LEED
> New large turbo pump + ion pump → base pressure ~ $10^{-10}$ mbar

# BL endstation 2 - Magnetic Scattering

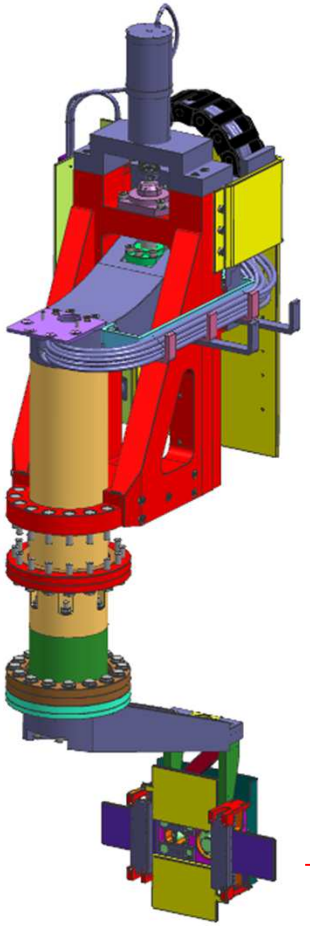
> Final assembly and commissioning mid-late 2014
> Open to users from feb. 2015 (i.e. available in currently open call)
> *Availability*: reflectometry exps. without applied magnetic field

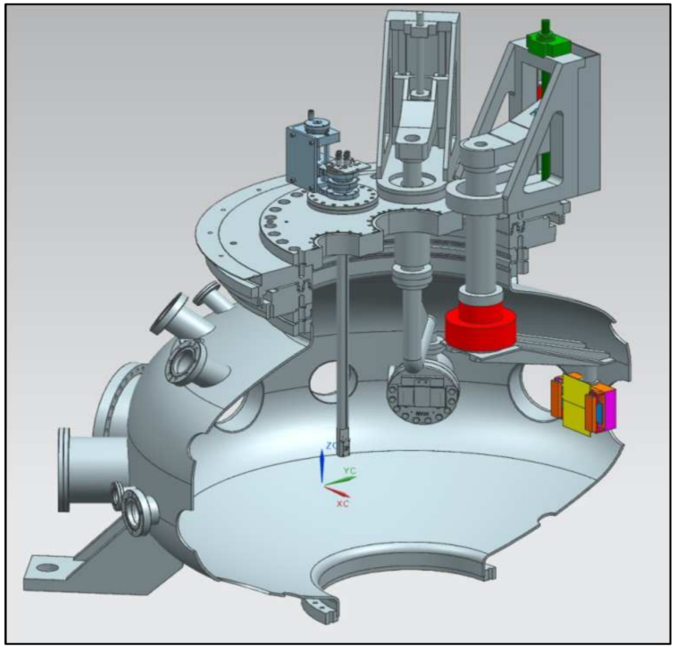


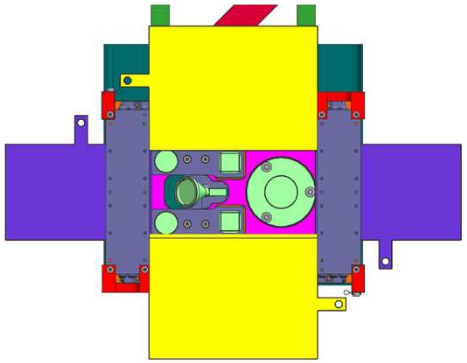



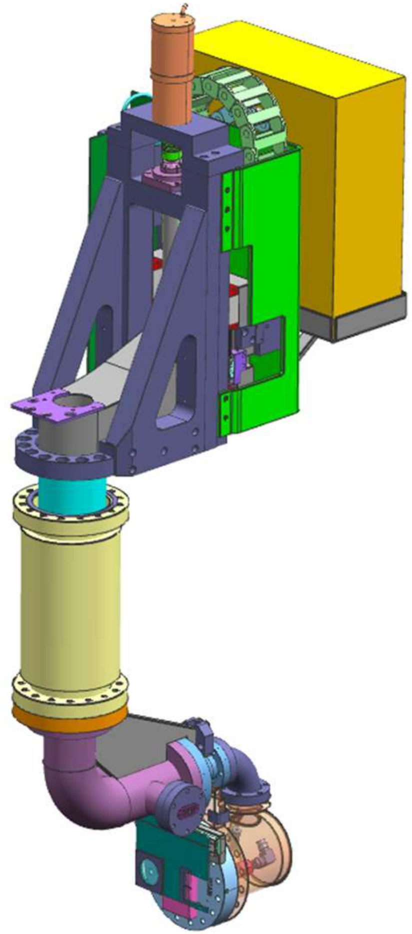

> Assembly, first tests with point detector
> Cabling, control (pool) in progress
> CCD commissioning in progress

# BL endstation 2 - detector arms





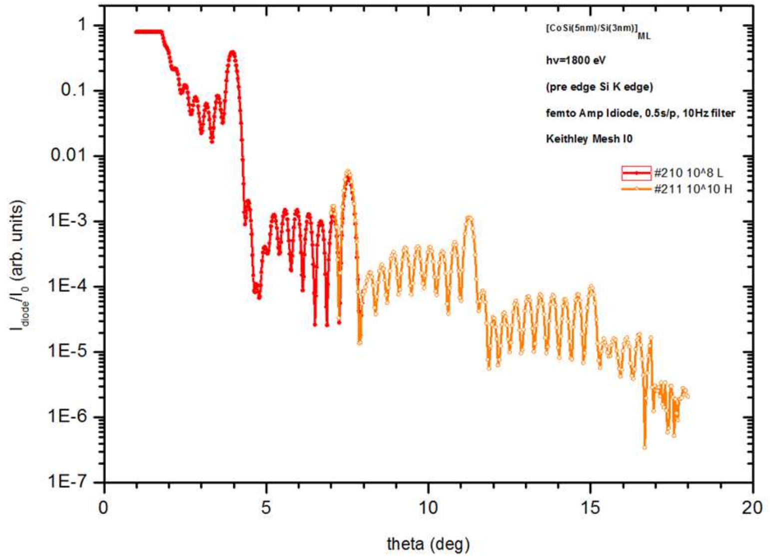


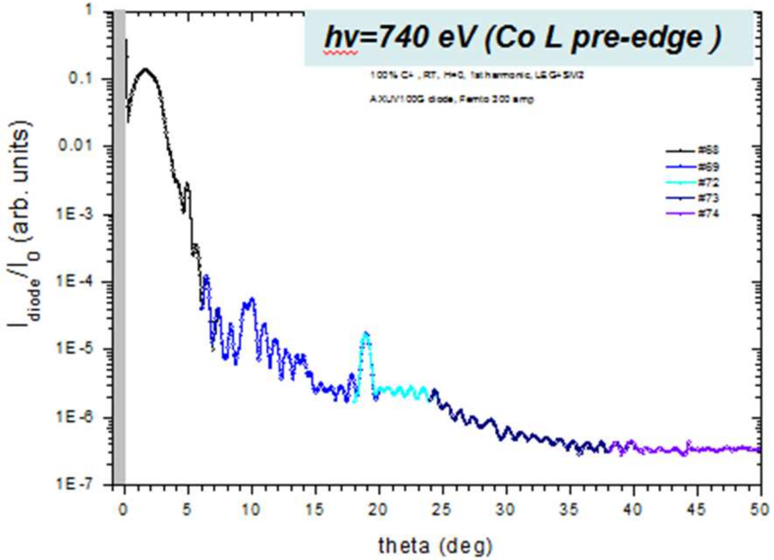


> Arm 1 – channeltron, microchannel plate, diodes, slits

> Arm 2 – XCAM CCD (SAT OK, june'14)

# BL endstation 2 – first spectra

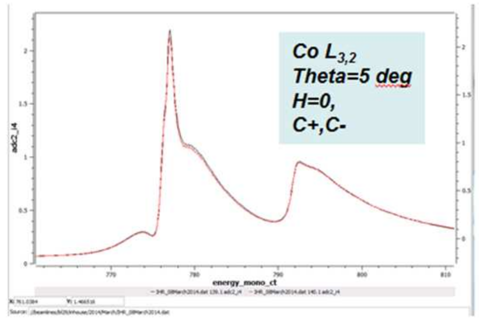


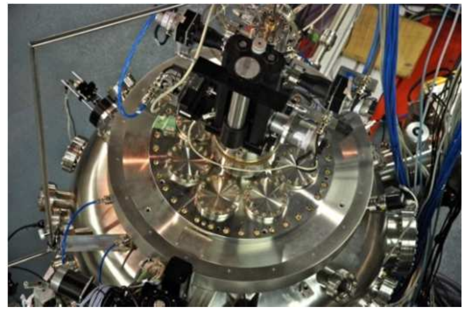



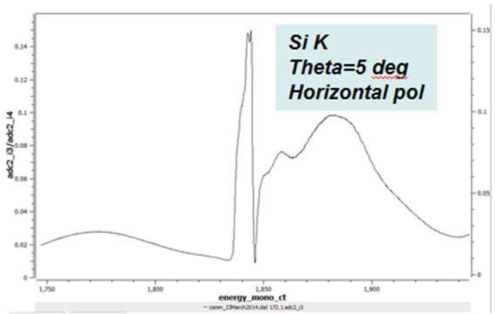

> March 2014 IHR/Commissioning beamtime

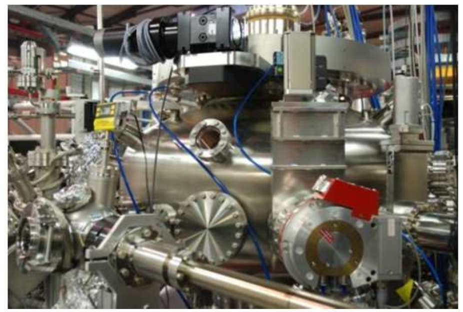
[CoSi(5nm)/Si(3nm)] ML test sample reflectivity

Diode detection on temporary arm support



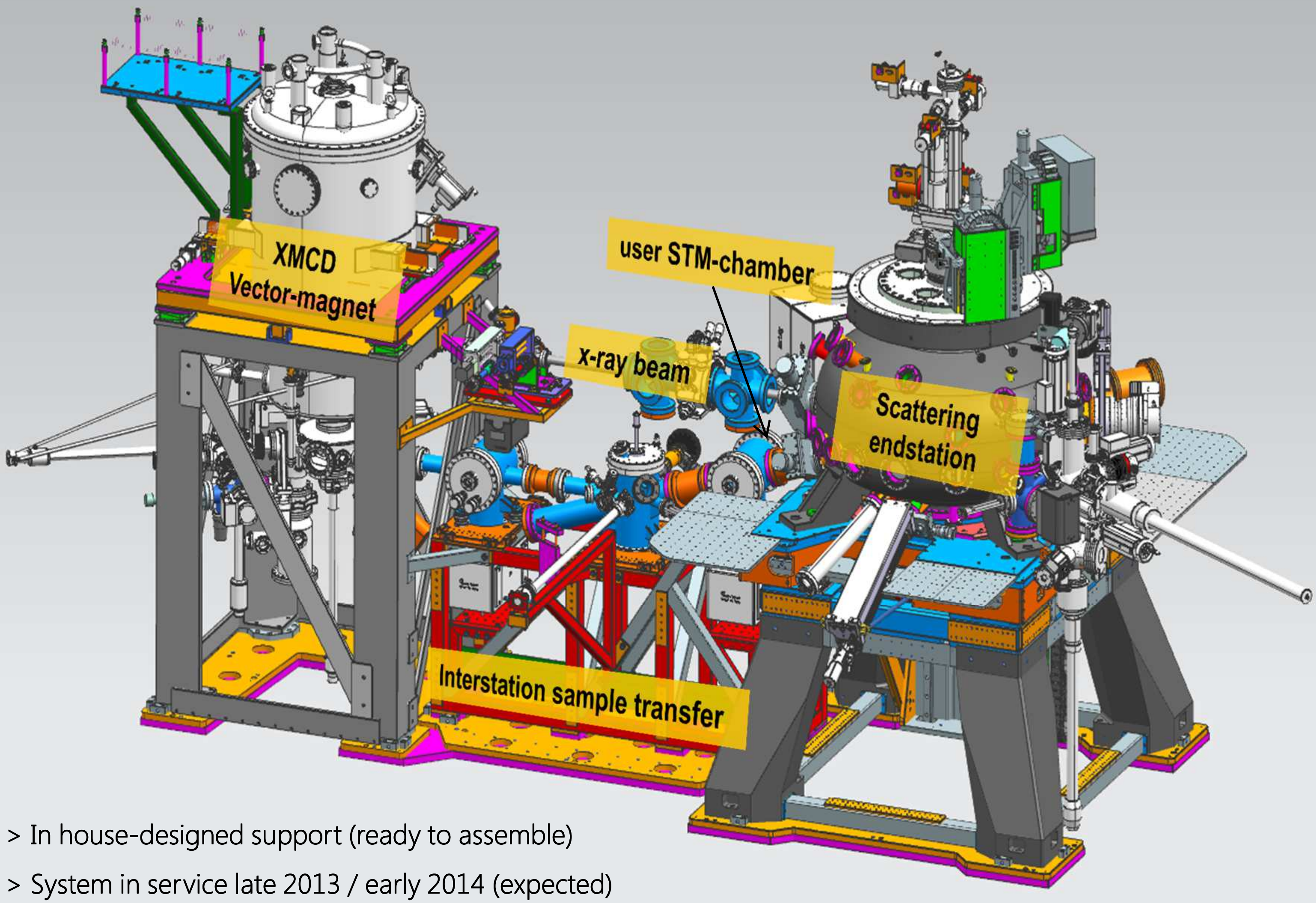





# Interstations transfer system – STM coupling





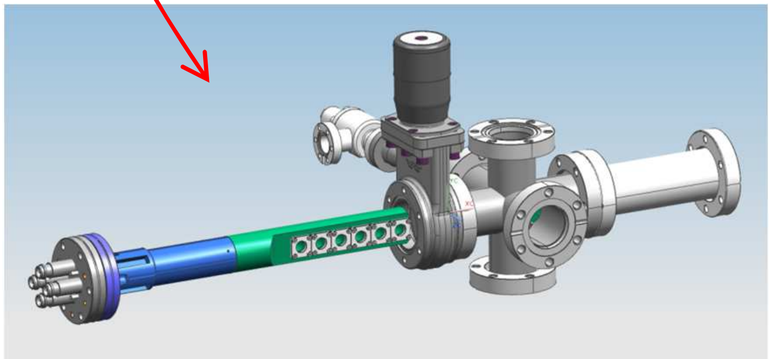

> In house-designed support (ready to assemble)

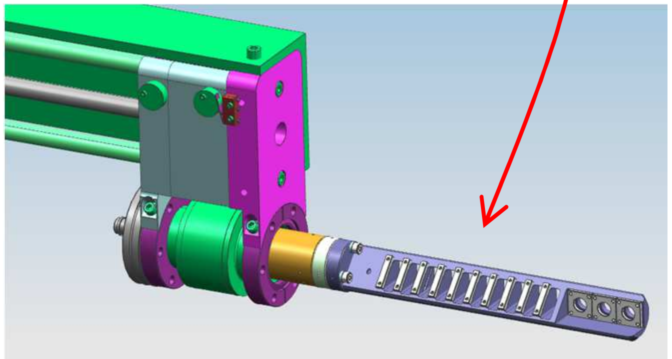
> System in service late 2013 / early 2014 (expected)

# Other developments - upgrades

## equipment

> Small UHV prep. chamber, coupling to interstations transfer system
> "OESS" (off-endstation samples stage) for quick exchange of *dirty samples*
> Motorized system for (more) E reference samples
> Surface science instrumentation for MARES (mini-LEED head, linear translator...)





## controls

> Deeper macro / GUI control of vectorial magnetic field & sample temperature
> New macros for cooling down/warming up VTI, valves & EMs control...
> Long macros (overnight...) stability
> *MARES* macros

# BL users operation 2013-2014

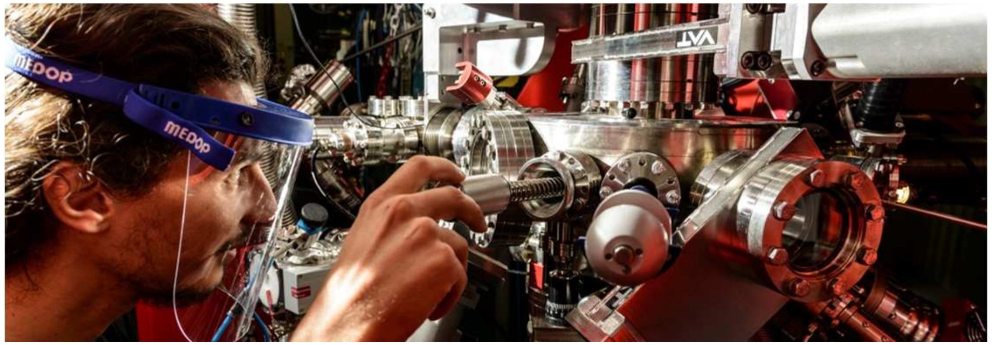


*User experiments in HECTOR*

> Jan. 2013– Mar. 2014 : 14 user experiments performed (14 beamtime granted user proposals from 2012 call)

> May 2014 – Dec. 2014 : 13* accepted proposals (5 so far carried out) from 2013 call

* 7/13 require in-situ surface preparation (single crystal cleaning, evaporation etc.)

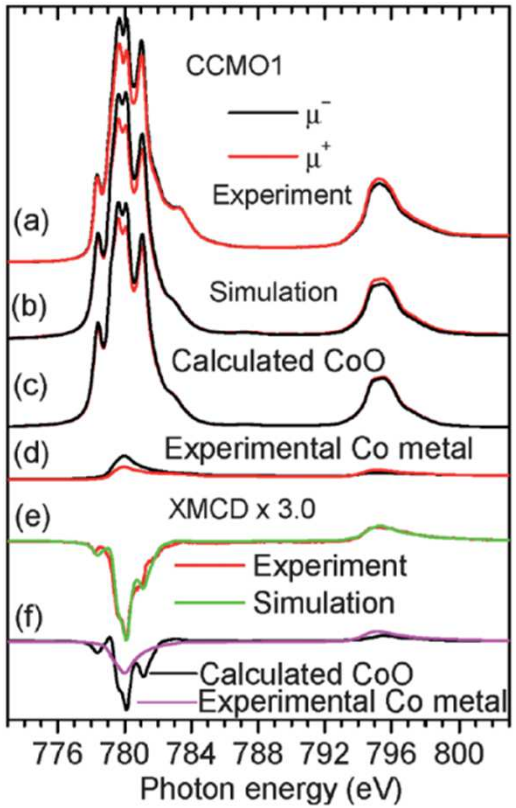



*Major issues / technical interventions in HECTOR*

> Cold He-leak in VTI tail, drain current and 4-point contact wires, sample heater)

> Oct. – Nov. 2013 : liquid He supply shortage (global issue) → some experiments cancelled, beam profited to: 1) perform friendly users RT/zero B-field measurements, and 2) improve continuous scan mode)

# IHR experiments – first publications




*2012 beamtime*

Nanoscale

RSC Publishing

PAPER

View Article Online
View Journal | View Issue

## Direct observation of rotatable uncompensated spins in the exchange bias system Co/CoO–MgO

**Cite this:** *Nanoscale*, 2013, **5**, 10236

Chuannan Ge,[ab] Xiangang Wan,*[a] Eric Pellegrin,*[c] Zhiwei Hu,[d] S. Manuel Valvidares,[c] Alessandro Barla,[ce] Wen-I. Liang,[f] Ying-Hao Chu,[f] Wenqin Zou[a] and Youwei Du[a]

Received 23rd April 2013
Accepted 27th July 2013

DOI: 10.1039/c3nr02013d

**www.rsc.org/nanoscale**

We have observed a large exchange bias field $H_E \approx 2460$ Oe and a large coercive field $H_C \approx 6200$ Oe at $T = 2$ K for Co/CoO core–shell nanoparticles (~4 nm diameter Co metal core and CoO shell with ~1 nm thickness) embedded in a non-magnetic MgO matrix. Our results are in sharp contrast to the small exchange bias and coercive field in the case of a non-magnetic $Al_2O_3$ or C matrix materials reported in previous studies. Using soft X-ray magnetic circular dichroism at the Co-$L_{2,3}$ edge, we have observed a ferromagnetic signal originating from the antiferromagnetic CoO shell. This gives direct evidence for the existence of rotatable interfacial uncompensated Co spins in the nominally antiferromagnetic CoO shell, thus supporting the uncompensated spin model as a microscopic description of the exchange bias mechanism.

# User experiments – first publications

*2012 beamtime*

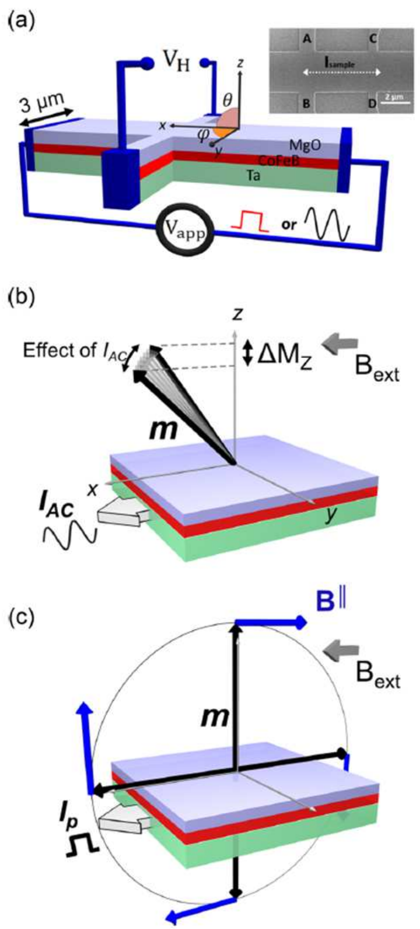



FIG. 2. (Color online) (a) Schematic of the sample and measurement geometry. Inset: Scanning electron micrograph image of a sample. (b) Small oscillations of the magnetization induced by an ac current. (c) Switching of the magnetization induced by the combined action of $B^{\parallel}$ and the external field.

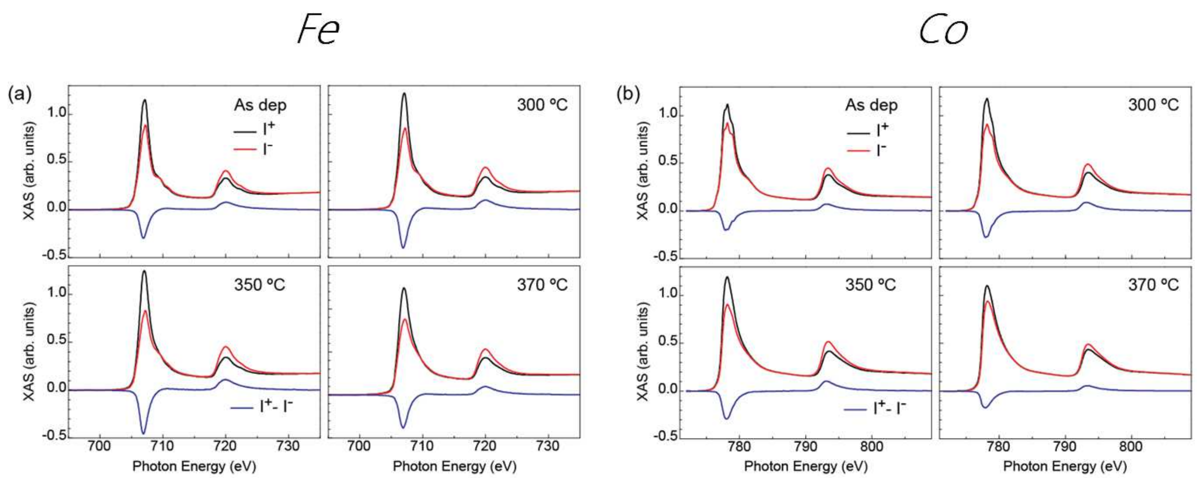
PHYSICAL REVIEW B **89**, 214419 (2014)

## Fieldlike and antidamping spin-orbit torques in as-grown and annealed Ta/CoFeB/MgO layers

Can Onur Avci,[1,2] Kevin Garello,[1,2] Corneliu Nistor,[1,2] Sylvie Godey,[2] Belén Ballesteros,[2] Aitor Mugarza,[2] Alessandro Barla,[3] Manuel Valvidares,[4] Eric Pellegrin,[4] Abhijit Ghosh,[1] Ioan Mihai Miron,[5] Olivier Boulle,[5] Stephane Auffret,[5] Gilles Gaudin,[5] and Pietro Gambardella[1,2]

[1]*Department of Materials, ETH Zurich, Hönggerbergring 64, CH-8093 Zürich, Switzerland*
[2]*Catalan Institute of Nanoscience and Nanotechnology (ICN2), UAB Campus, E-08193 Barcelona, Spain*
[3]*Istituto di Struttura della Materia (ISM), Consiglio Nazionale delle Ricerche (CNR), S.S. 14 Km 163.5, I-34149 Trieste, Italy*
[4]*ALBA Synchrotron Light Source, E-08290 Cerdanyola del Vallès, Barcelona, Spain*
[5]*SPINTEC, UMR-8191, CEA/CNRS/UJF/GINP, INAC, F-38054 Grenoble, France*

(Received 31 January 2014; revised manuscript received 12 May 2014; published 23 June 2014)

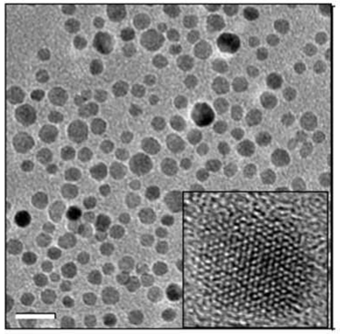


# User experiments – PhD thesis contribs.



*2012 beamtime*

> High T SC with embedded nanoparticles

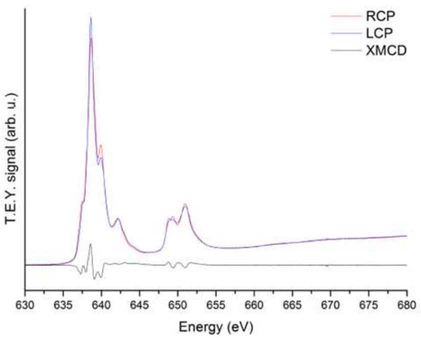


Chapter 9

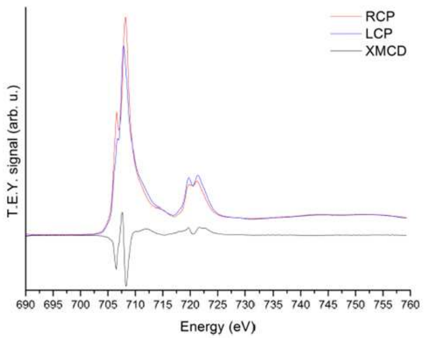
Synchrotron XAS & XMCD

Contents






(a) $MnFe_2O_4$-T Mn edge




(b) $MnFe_2O_4$-T Fe edge

E. Solano et al, UAB-ICMAB-ALBA (PhD thesis, publication in prep.)

*2013 beamtime*


Inorganic Chemistry

Article

pubs.acs.org/IC


## Stability of the Cationic Oxidation States in $Pr_{0.50}Sr_{0.50}CoO_3$ across the Magnetostructural Transition by X-ray Absorption Spectroscopy


Jessica Padilla-Pantoja,*[,†] Javier Herrero-Martín,[‡] Pierluigi Gargiani,[‡] Manuel Valvidares,[‡] Vera Cuartero,[‡,§] Kurt Kummer,[§] Oliver Watson,[§] Nicholas B. Brookes,[§] and José Luis García-Muñoz[†]

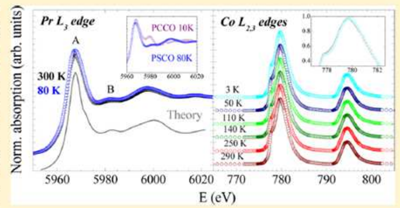
[†]Institut de Ciència de Materials de Barcelona (ICMAB)-CSIC, Campus Universitari de Bellaterra, E-08193 Bellaterra, Barcelona, Spain

[‡]ALBA Synchrotron Light Facility, 08290 Cerdanyola del Vallès, Barcelona, Spain

[§]European Synchrotron Radiation Facility, F-38043 Grenoble Cedex 9, France



**ABSTRACT:** The possible hybridization between Pr 4f and O 2p states in $Pr_{0.50}Sr_{0.50}CoO_3$ at low temperatures was investigated by different techniques. First, using neutron diffraction we observed a strong contraction of some Pr−O bonds across the magnetostructural transition at $T_S \sim 120$ K. In contrast to the Pr−O bond contraction in $Pr_{0.50}Ca_{0.50}CoO_3$, this transition is not accompanied by the appearance of $Pr^{4+}$ at low temperatures, as revealed by X-ray absorption spectroscopy at Pr edges. Despite the fact that a Pr valence change is not the mechanism that drives this transition, we point out an active participation of Pr ions across $T_S$. Moreover, Co $L_{2,3}$-edge and O K-edge X-ray absorption spectra did not reveal any spin-state variation and showed the stability of the average formal valence of cobalt ions. The large density of empty $t_{2g}$ symmetry states in the studied thermal range does not suggest the occurrence of $Co^{3+}$ in a pure low-spin state. The overall metallic behavior agrees with our findings. We propose a mixture of $Co^{3+}$ ions in the intermediate-spin or high-spin configuration together with $Co^{4+}$ ions in a low- or intermediate-spin state.





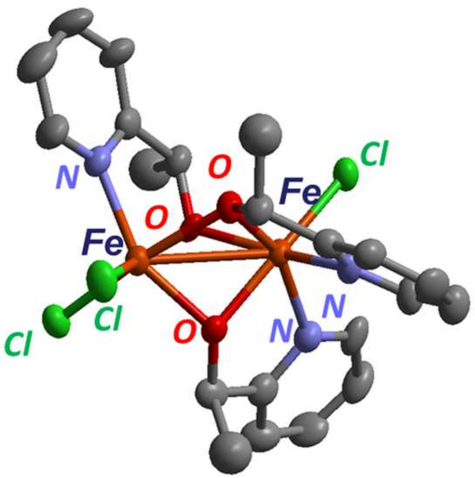
J. Padilla-Pantoja et al (in press, PhD thesis in prep.)

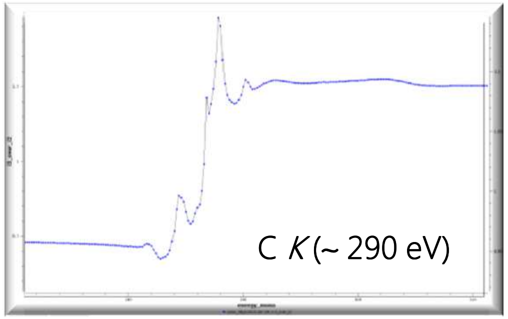
# IHR projects - collaborations

## > Di-Fe (III) chiral complex molecular magnets (preliminary measurements)

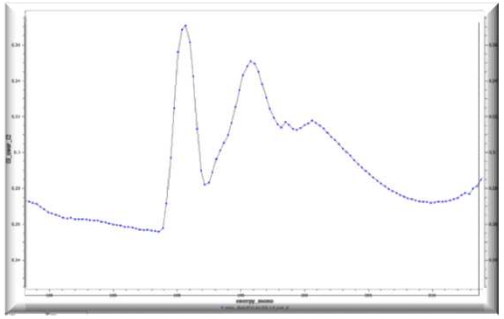
J. Giner-Planas, F. Di Salvo, M. Y. Tsang, S. M. Valvidares, P. Gargiani, J. Herrero-Martín, F. Teixidor, C. Viñas, N. Aliaga-Alcalde, E. Ruiz, D. Choquesillo-Lazarte,

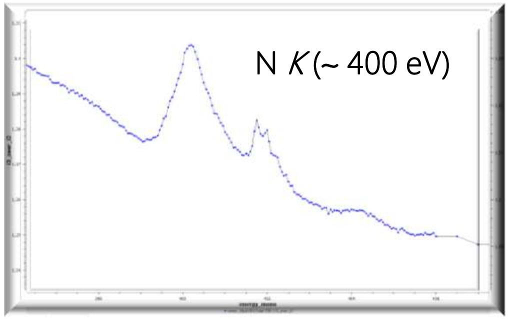
*ICMAB-CSIC, Univ. Barcelona, IACT-CSIC (Granada), ALBA*

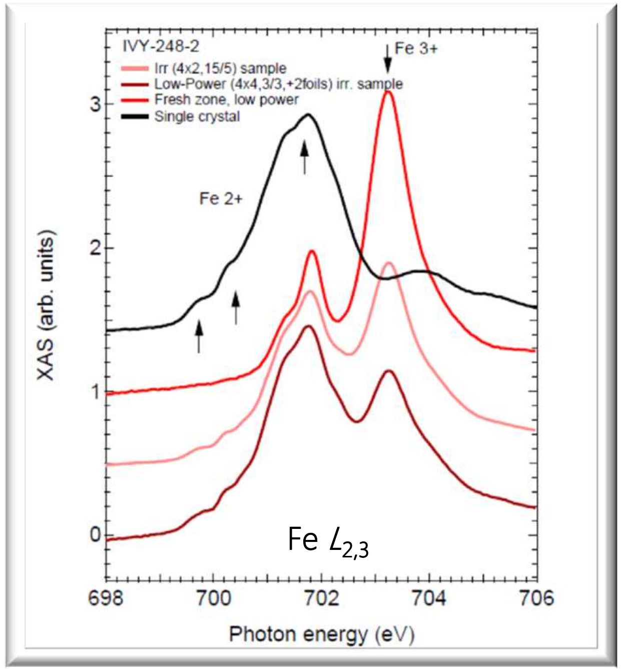


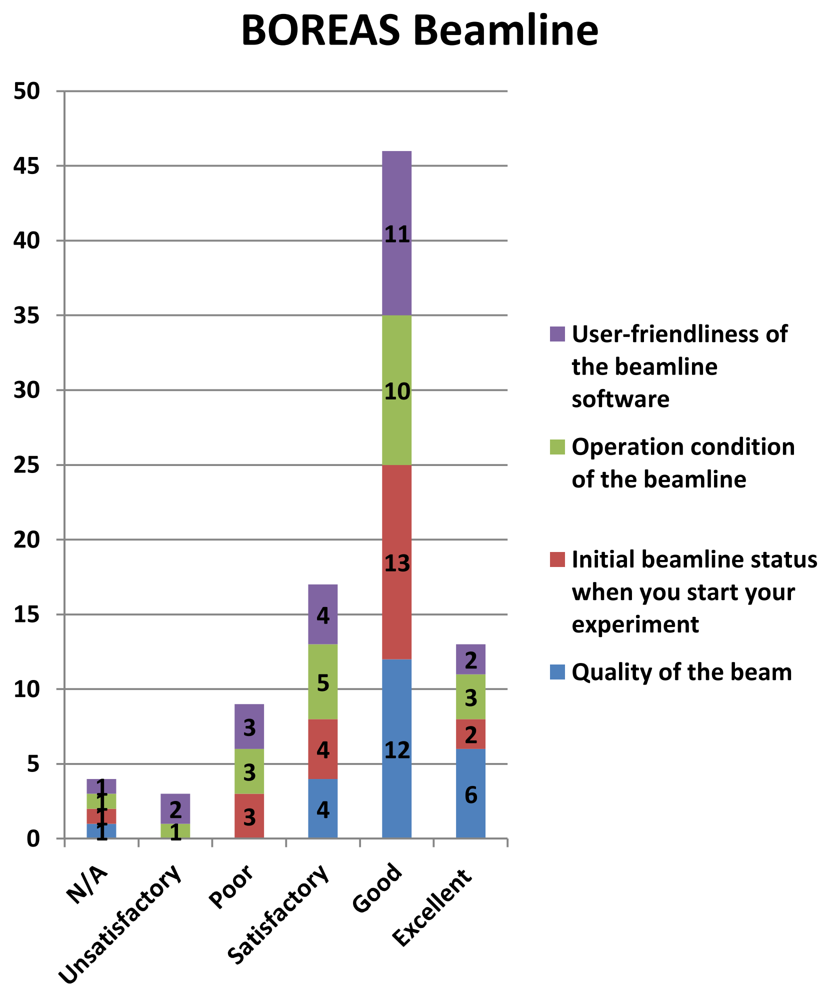

Cl $L_1$ edge (~ 200 eV)
(20 times flux attenuation)

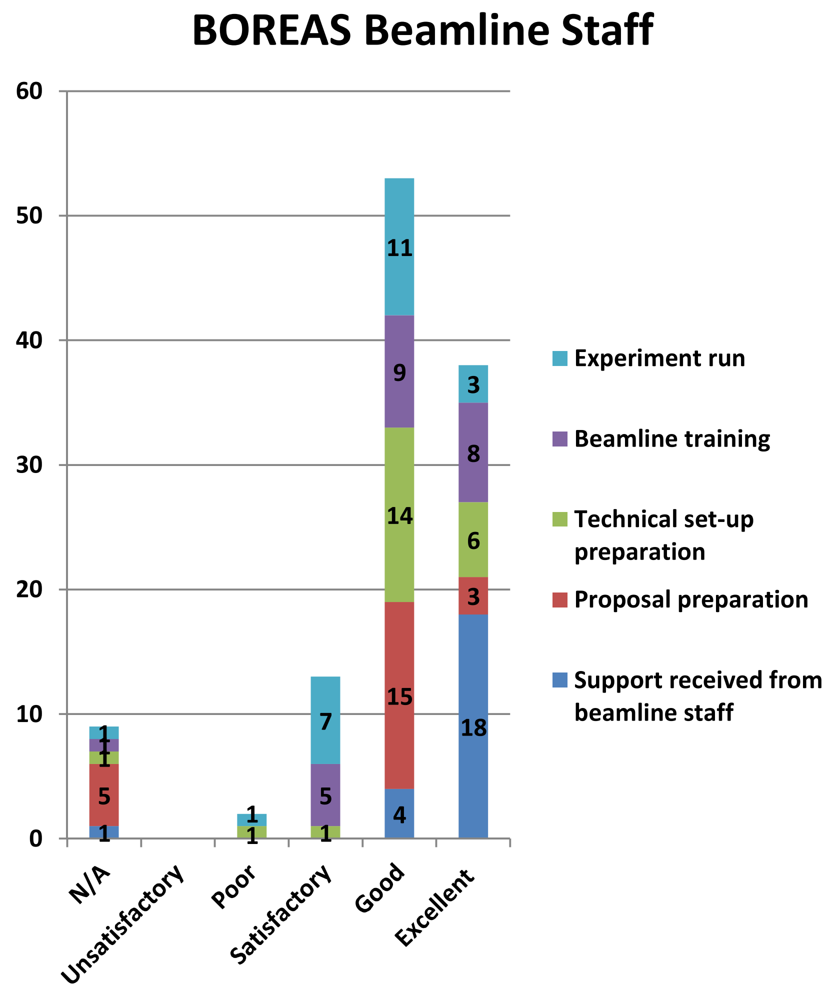




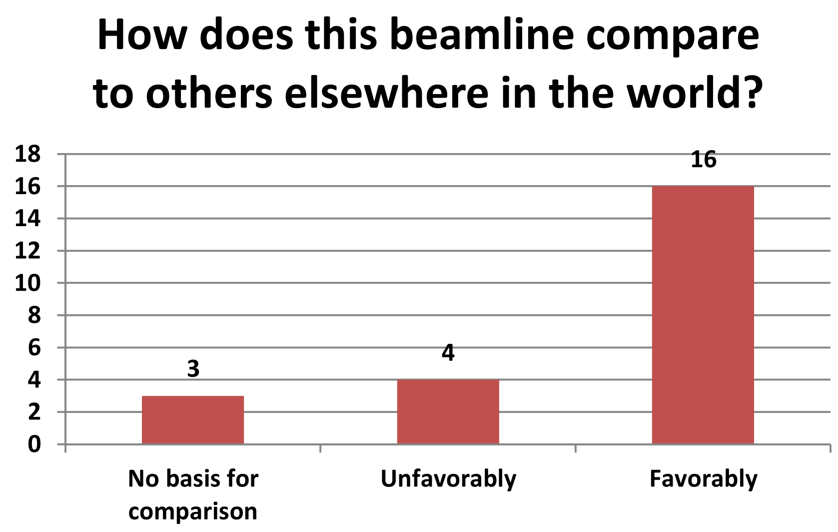
# Users evaluation (1/2)



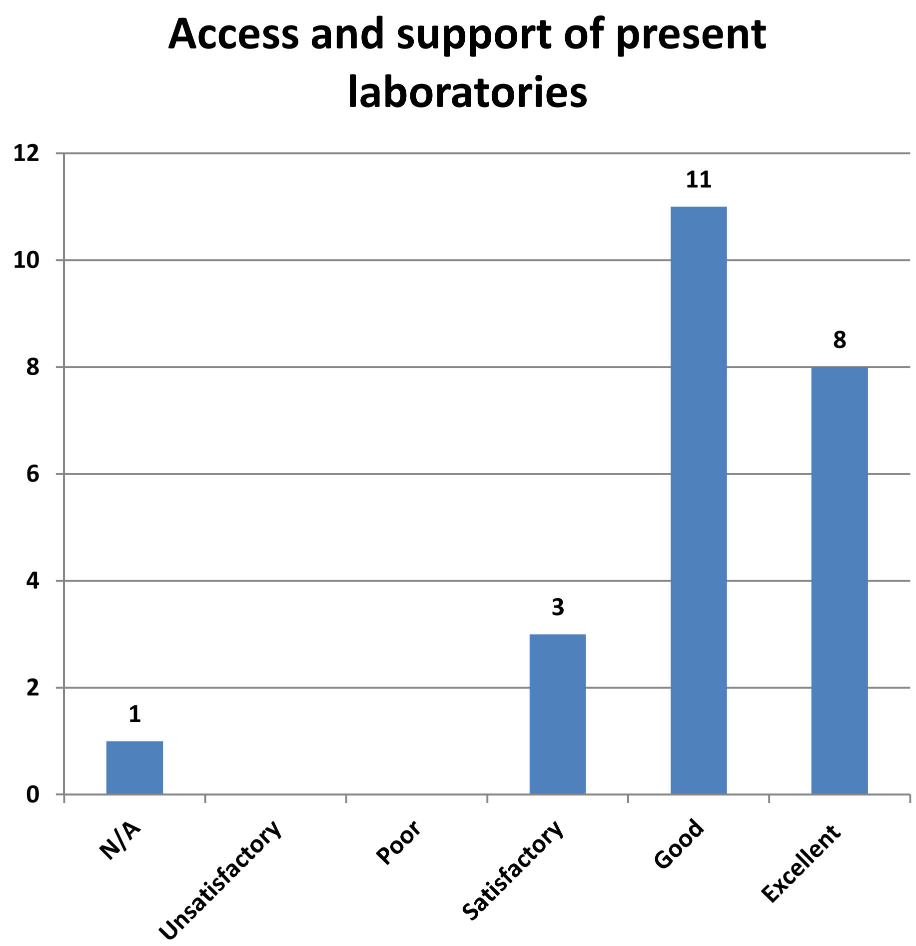
# Users evaluation (2/2)



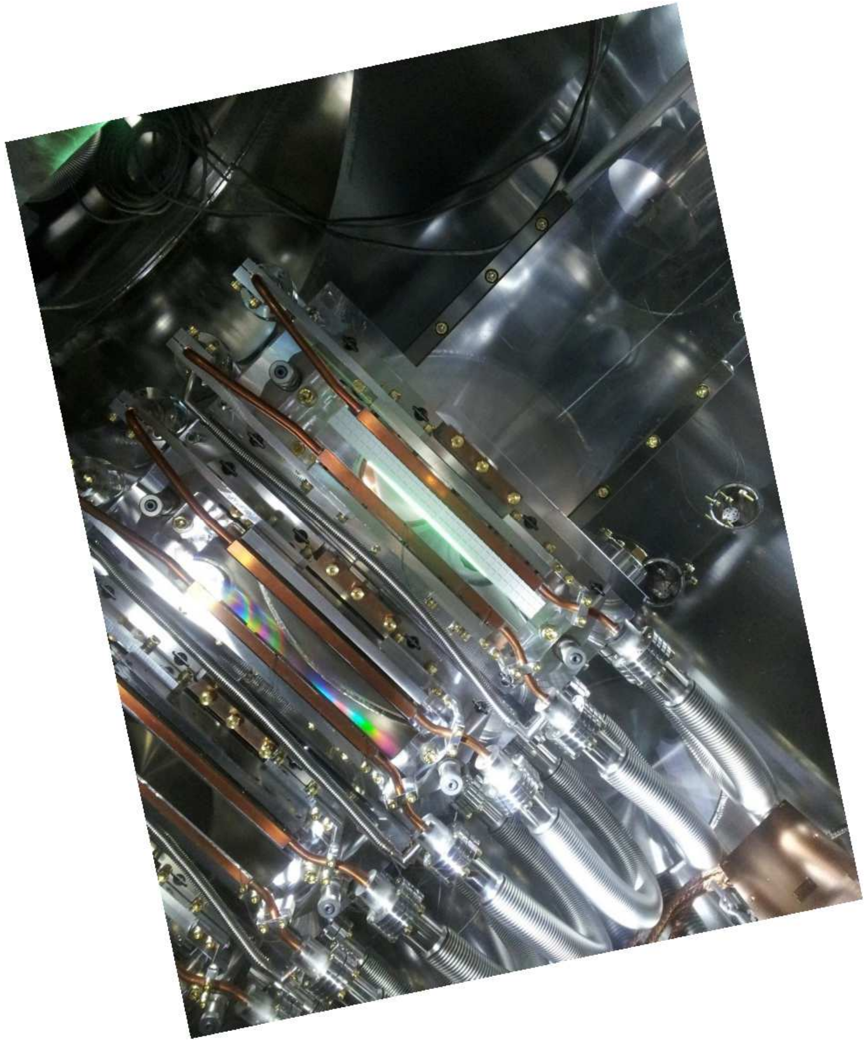



*Users' comments*

- Bad pressure conditions in analysis chamber
- Some of the equipment is new and needs to be debugged
- J. Herrero, P. Gargiani and M. Valvidares were supporting us the whole time



# Acknowledgements



*BL29 Staff*

M. Valvidares, P. Gargiani (BL scientists),
J. Moldes (controls), A. Crisol (eng.), F. Farre (tech.),
X. Fariña and X. Serra (electronics)

*Special ALBA contributors*

E. Pellegrin, S. Ferrer, C. Colldelram, J. Nicolas, D. Fernandez,
G. García, M. A. García-Aranda, L. Campos, User Office

*Former staff & technical help*

A. Barla, R. Martin, J.F. Moreno, D. Bacescu, J. C. Martinez,
J. Lidon, Y. Nikitin, O. Matilla, S. Rubio, F. Becheri, J. Navarro,
Ll. Ginés, A. Pascual, C. Ortiz, M. Llonch, C. Ruget, Alex, Paco,
Eshraq, Xavi P. , C. Orozco, D. Iriarte, Thiago, A. Ruz, S. Astorga,
Bern, Oscar, David, J. Ferrer, legal section (CFTs)

*Externals:* J. B. Kortright, N. Brookes, J. Criginski, C. Quiros, J. M. Alameda, C. Piamonteze, T. Schroder

*External companies*

TOYAMA, SCI-MAG, CVT, VG, HTS-110, PINK, SPECS,
DODECON, FERROVAC, GAMMVAC, TECNOVAC, VAT,
IBERLASER, HOSITRAD, VCS, AVS, AVACTEC,VAQTEC,
MCALLISTER, HAMAMATSU, XCAM, AMBAR, INSERTY,
SPARK,NANOTEC, SJUTS, KONIK, MDC, TRINOS, VAT