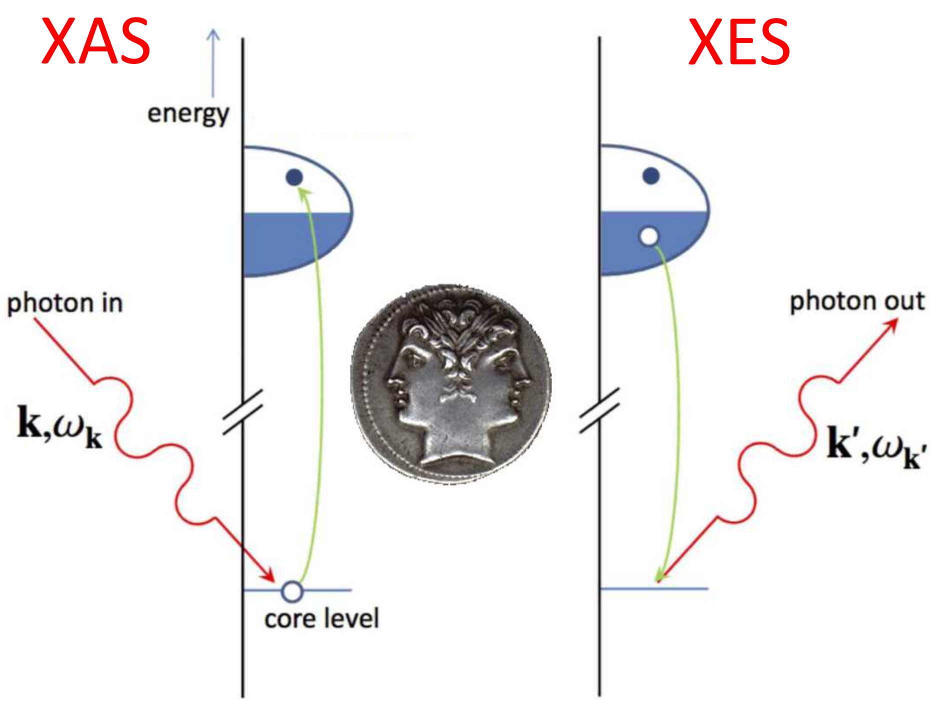
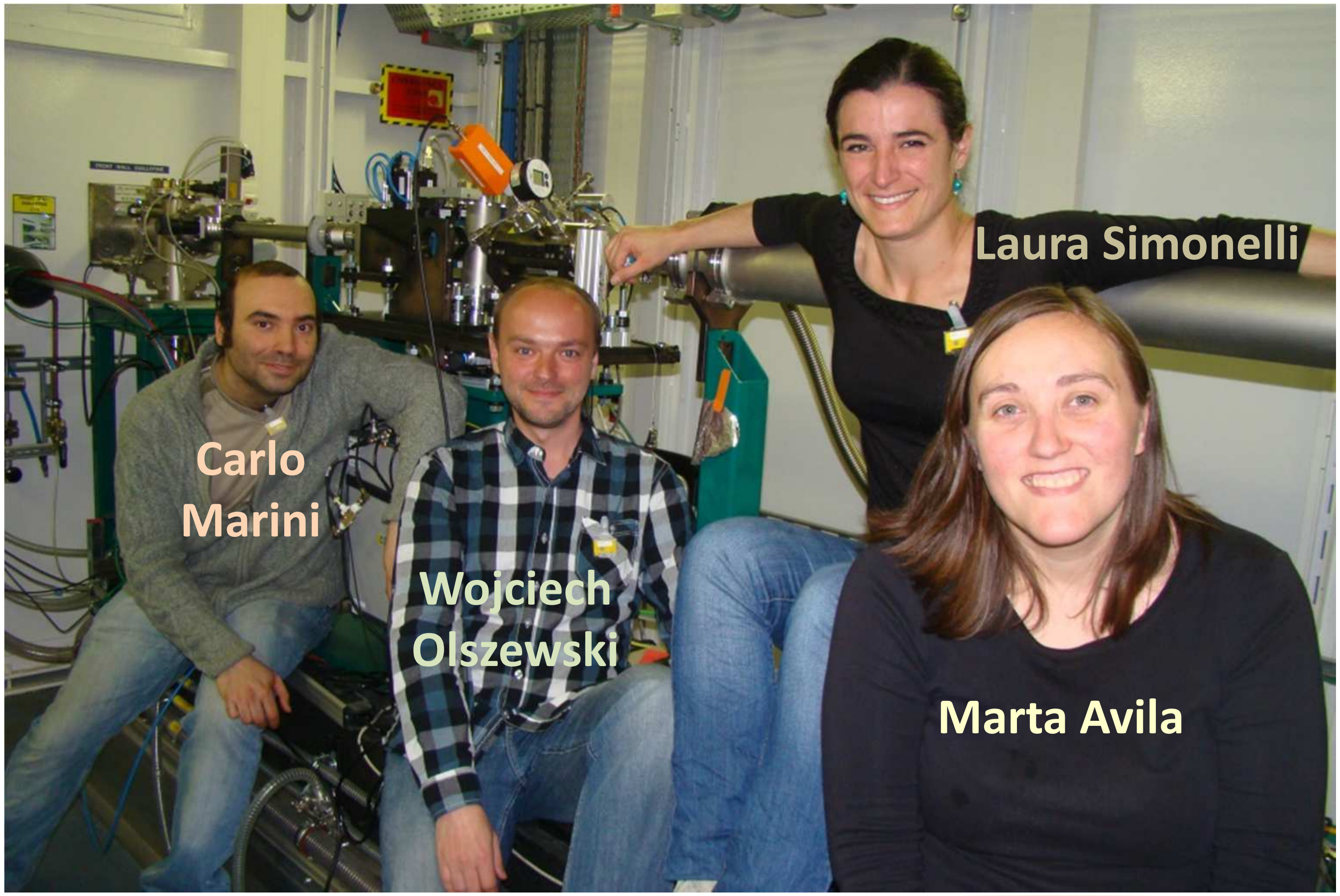
Core Level Absorption & Emission Spectroscopies

Laura Simonelli



Core **L**evel **A**bsorption & **E**mission **S**pectroscopie**s**

The completed CLÆSS team



OUTLINE

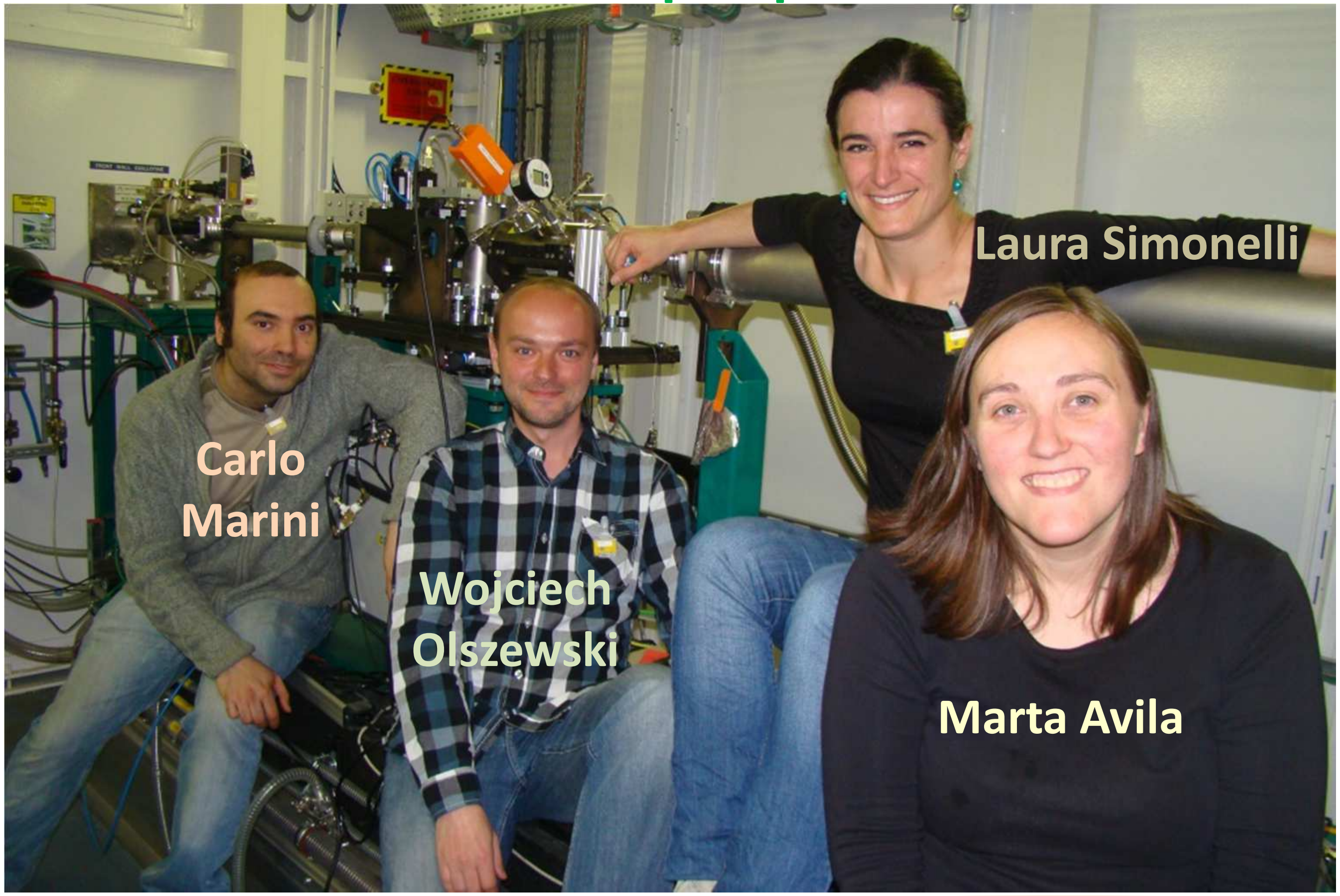
Beam time allocation

Users feedback

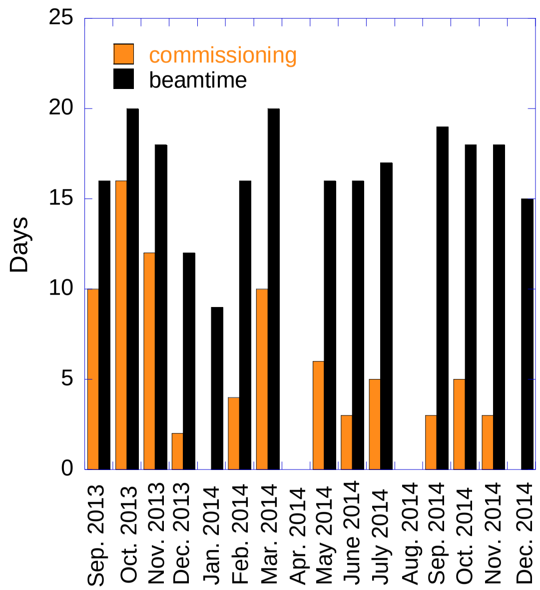
Users/IH research

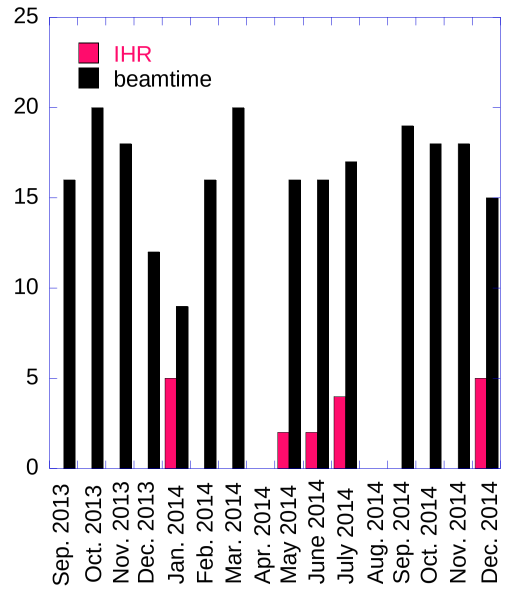
BL22 status

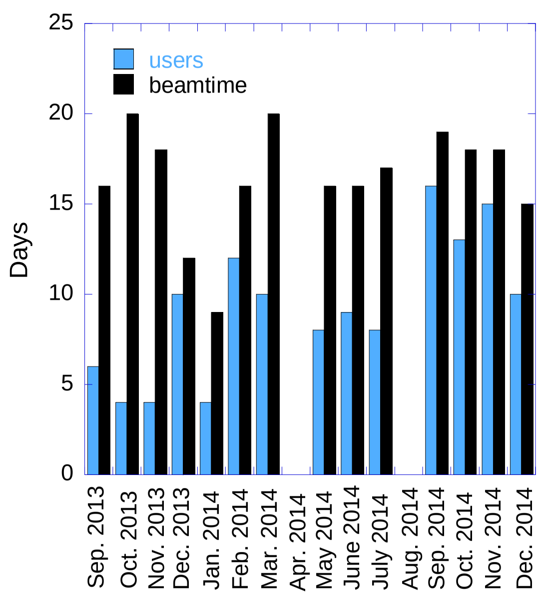
Future and perspective

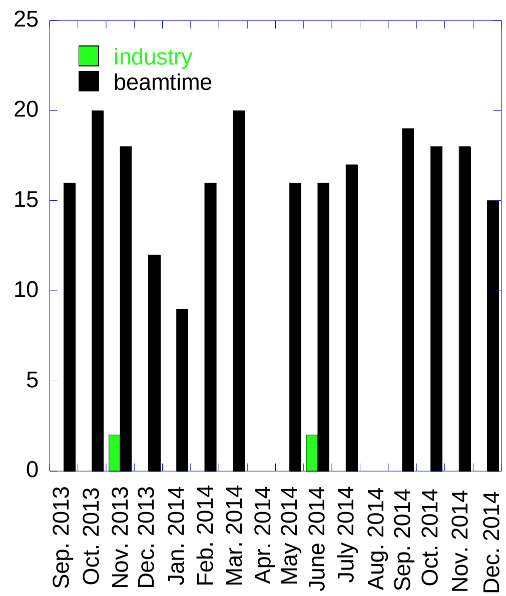


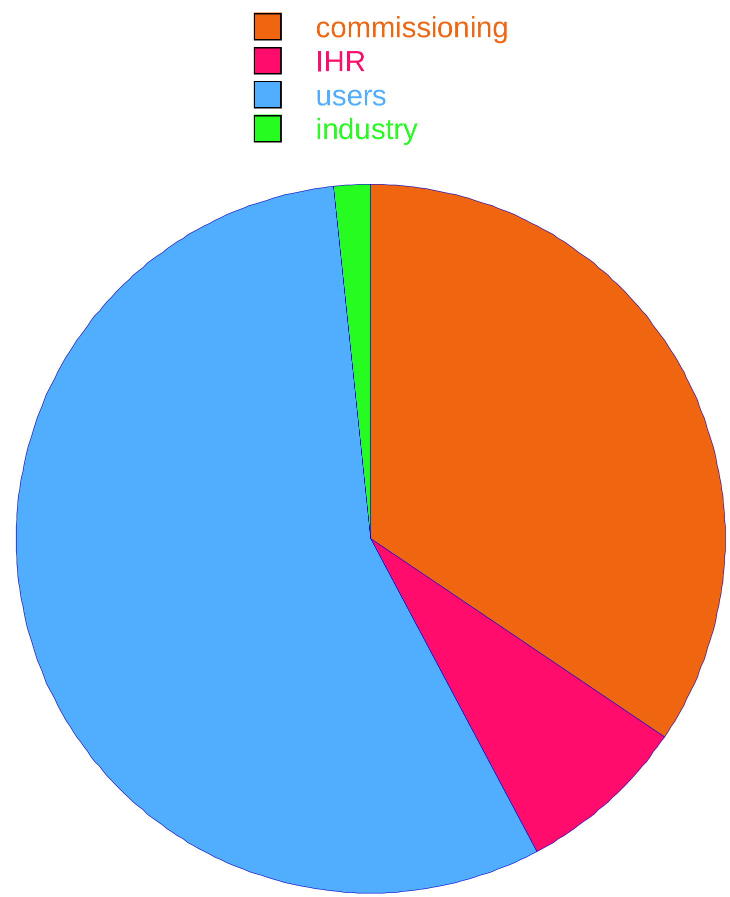
Beam time allocation



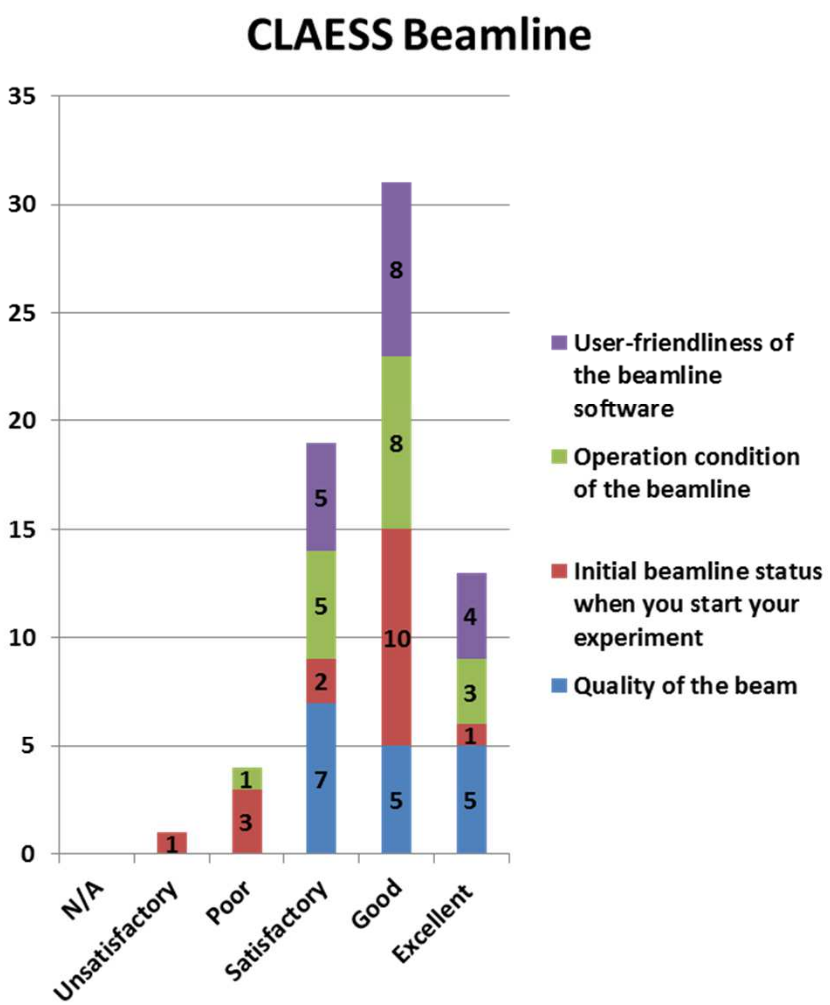


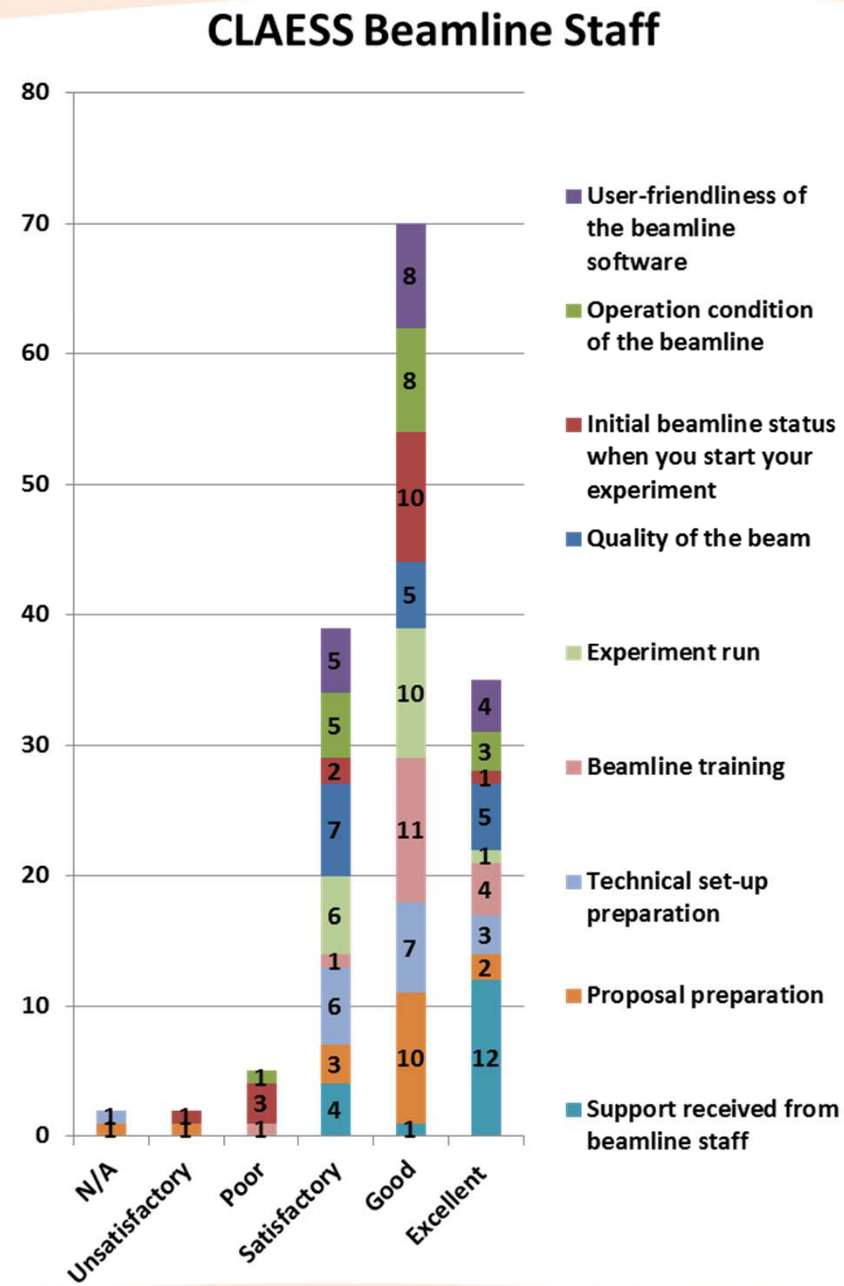




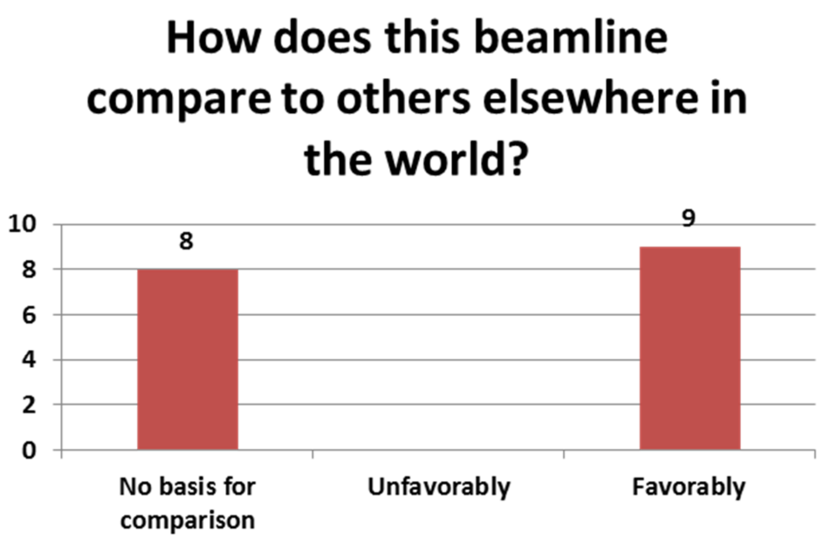


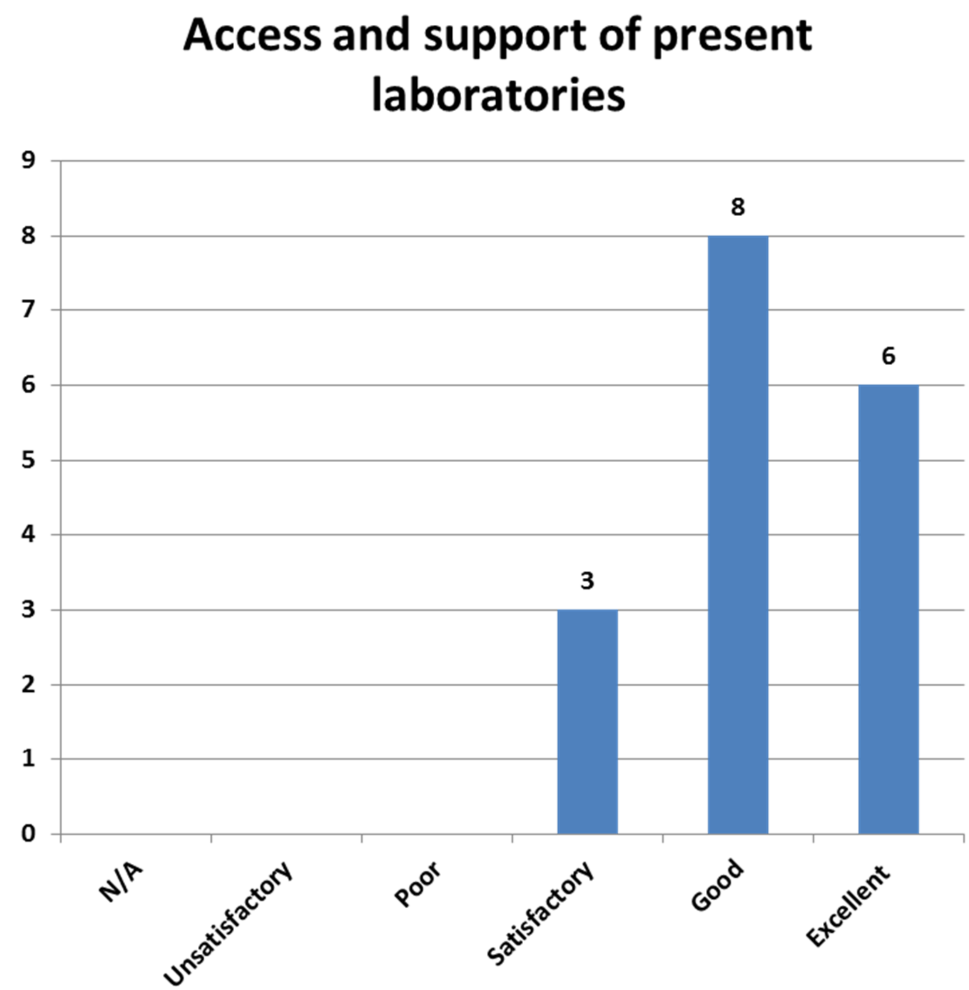
Users feedback received during 2013



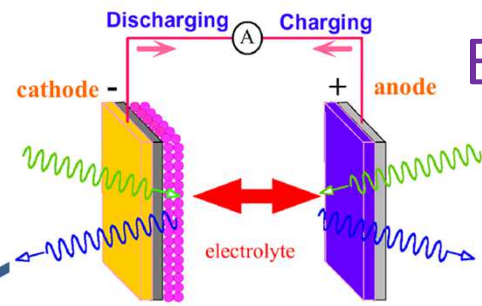


Users feedback received during 2013

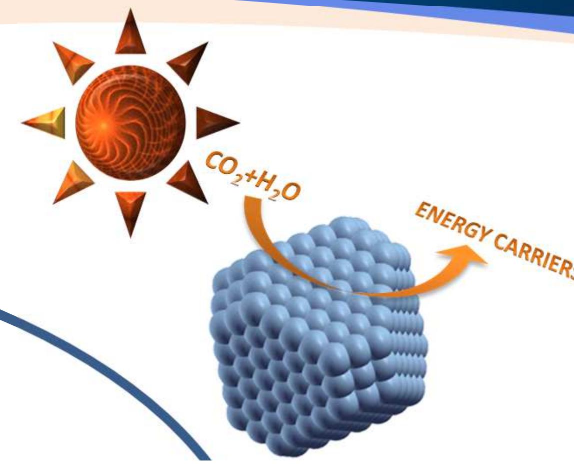




Users/IHR production



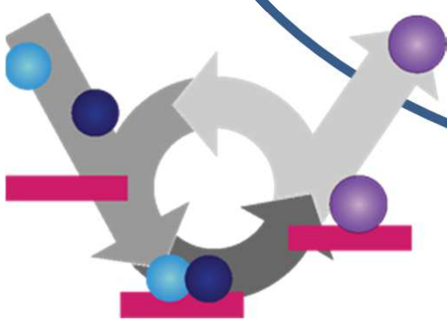
Batteries



Cultural Heritages

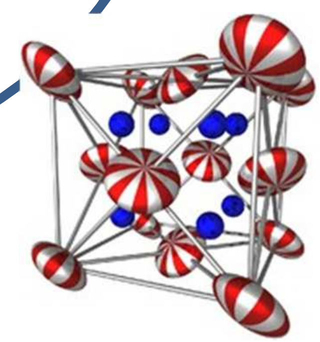
CLÆSS
beamline

Artificial Photosynthesis



Catalysis

Environmental Science



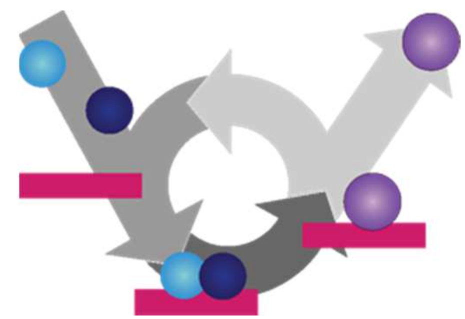
Solid State Physics

publications

Exceptional oxidation activity with size-controlled supported gold clusters of low atomicity
Avelino Corma et al., NATURE CHEMISTRY 5,775 (**2013**)

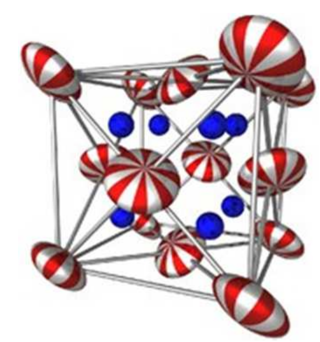
Low-Temperature Oxidation of Carbon Monoxide with Gold(III) Ions Supported on Titanium Oxide
Wolfgang Grünert et al., Angew. Chem. Int. Ed. 2014, 53, 3245 (**2014**)

Isomerization of -pinene oxide using Fe-supported catalysts: Selective synthesis of campholenic aldehyde
Martina Stekrova et al., Applied Catalysis A: General 470, 162 (**2014**)



Catalysis

Strong local lattice instability in hexagonal ferrites RFe_2O_4 (R = Lu,Y,Yb) revealed by x-ray absorption spectroscopy
Sara Lafuerza et al., PHYSICAL REVIEW B 89, 045129 (**2014**).

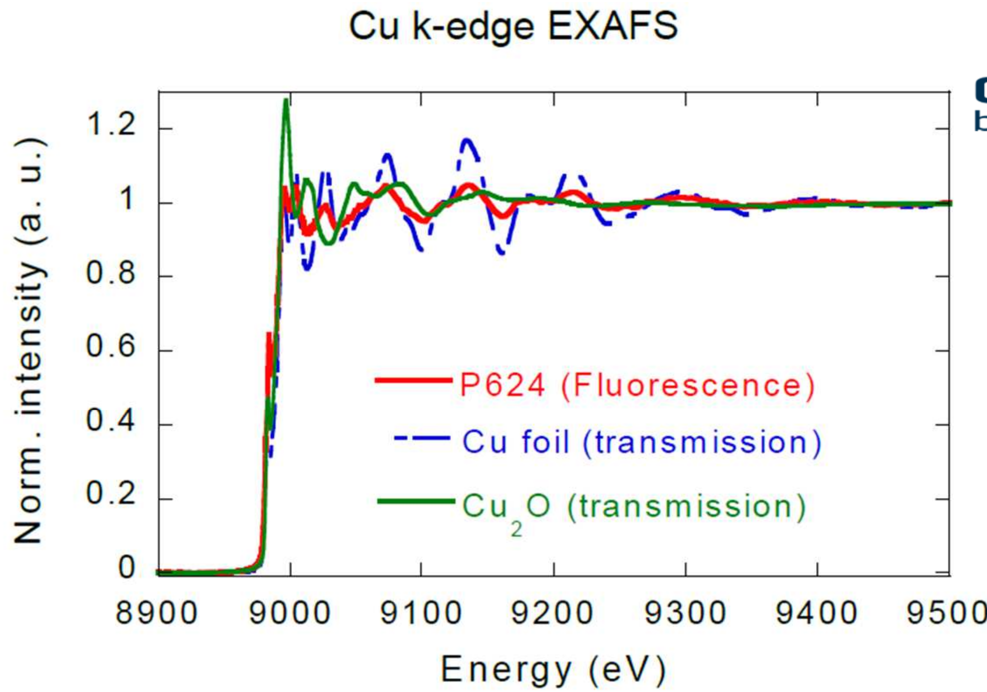


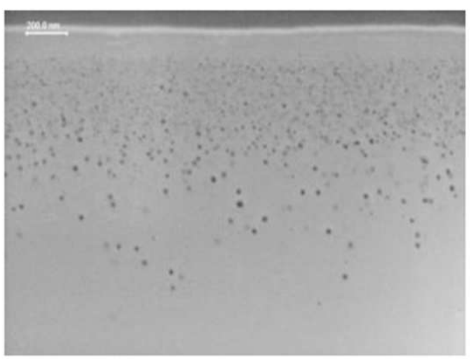
Solid State Physics

Cultural Heritages
(first fluorescence scans Dec. 2013)

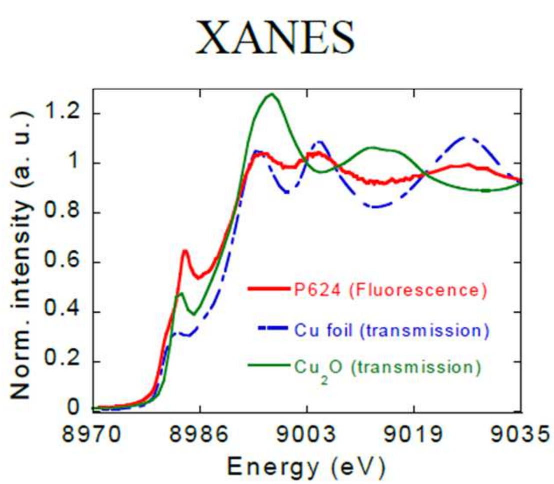
P624

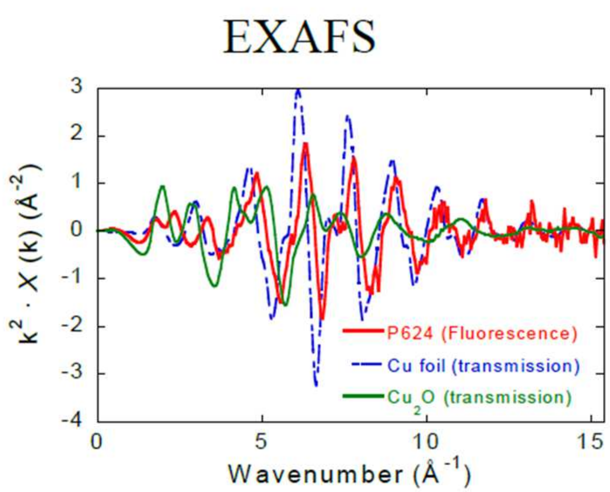
Courtesy of T. Pradell





P624 dark decoration



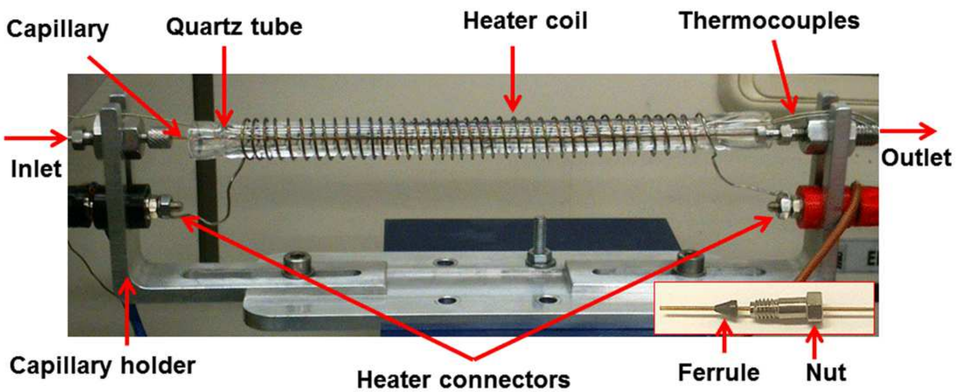


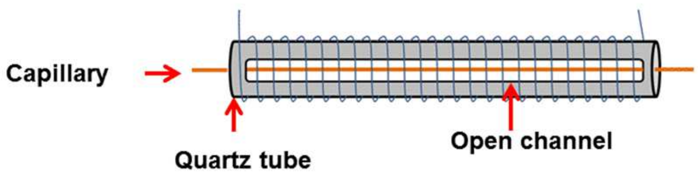
Users research

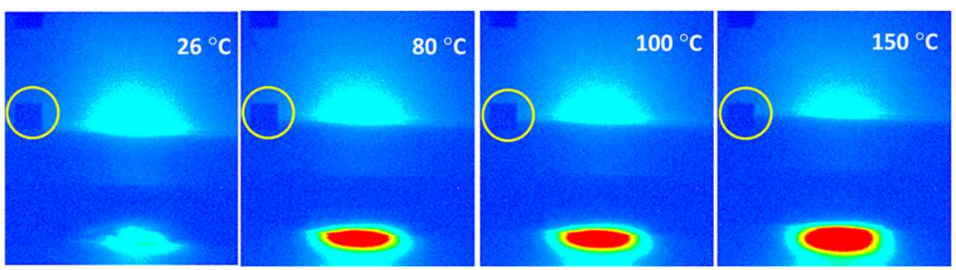
Catalysis

(high pressure reactor – up to 200 bar)

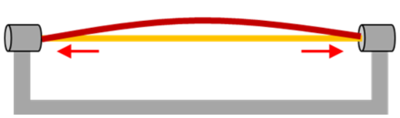
A. Bansode, G. Guilera, V. Cuartero, L. Simonelli, M. Avila and A. Urakawa, Submitted to Rev. Sci. Instrum.

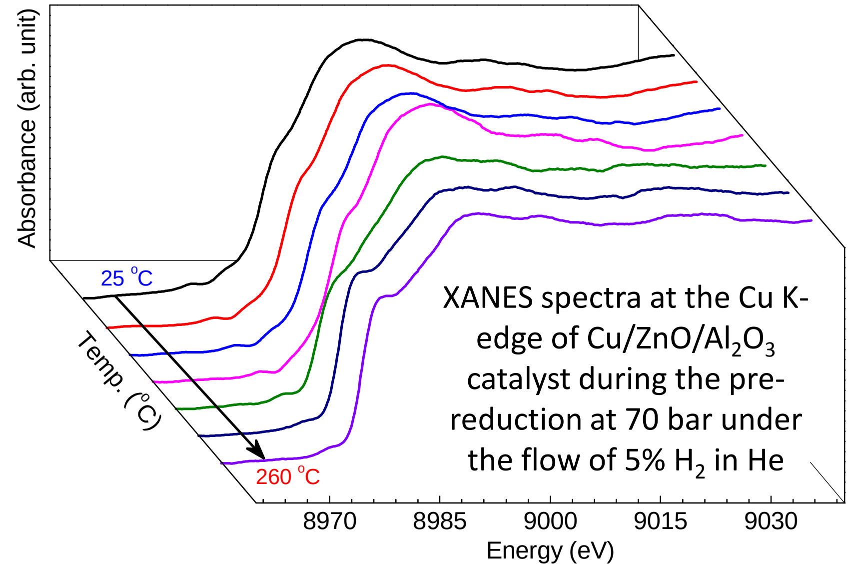




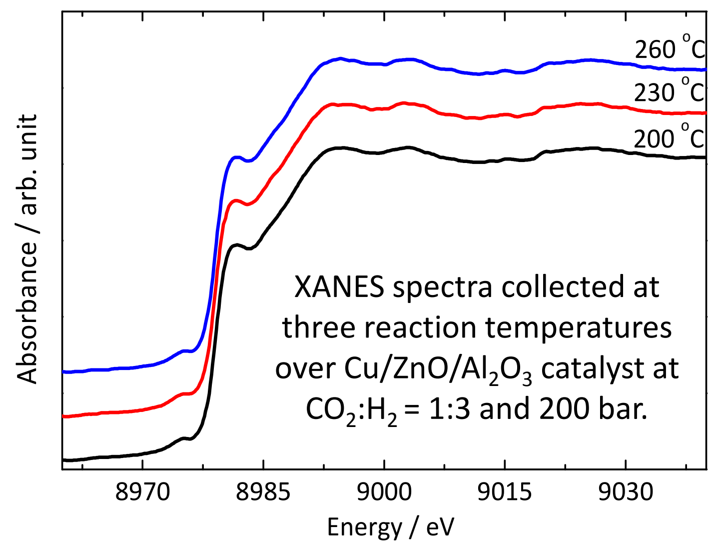


High-Pressure Enhanced Thermal Expansion



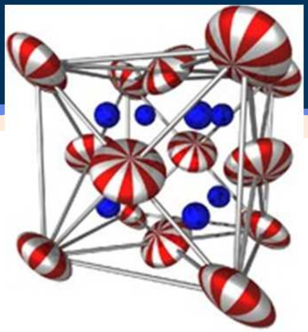


XANES spectra at the Cu K-edge of $Cu/ZnO/Al_2O_3$ catalyst during the pre-reduction at 70 bar under the flow of 5% H_2 in He



XANES spectra collected at three reaction temperatures over $Cu/ZnO/Al_2O_3$ catalyst at CO_2:H_2 = 1:3 and 200 bar.

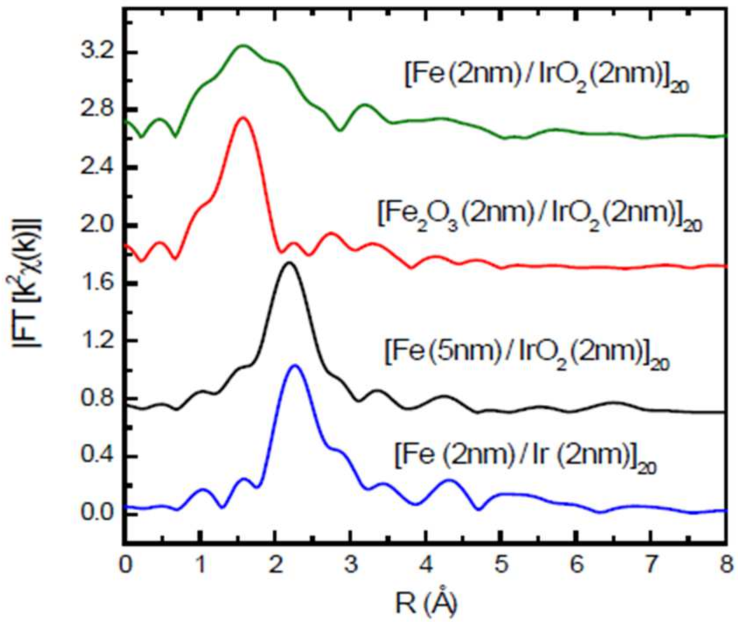
Users research



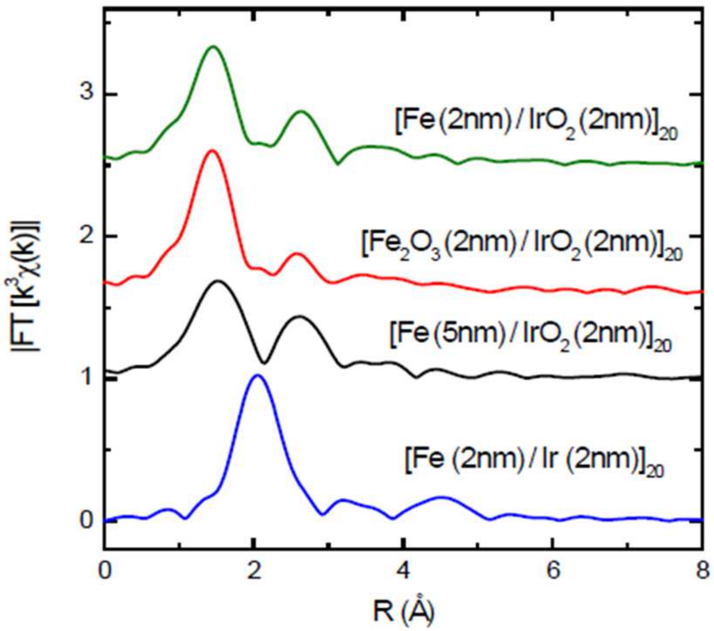
Courtesy of Maria Ángeles Laguna Marco

XAS study on the crystal and electronic structures of $Fe(FeO_x)/Ir(IrO_2)$ multilayers.

Solid State Physics



FT of the Ir L_3 edge EXAFS



FT of the Ir L_3 edge EXAFS

The results indicate a fully oxidized Fe layer with a very similar crystal structure in all the samples. The Ir shell, on the other hand, evolves from pure metallic to a mixture of metal and oxide phases and finally to pure IrO_2.

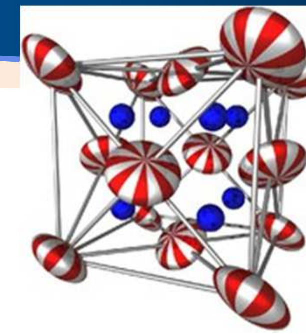
Differences in the M(H) behavior can be associated to the state of the Ir layer.

IHR

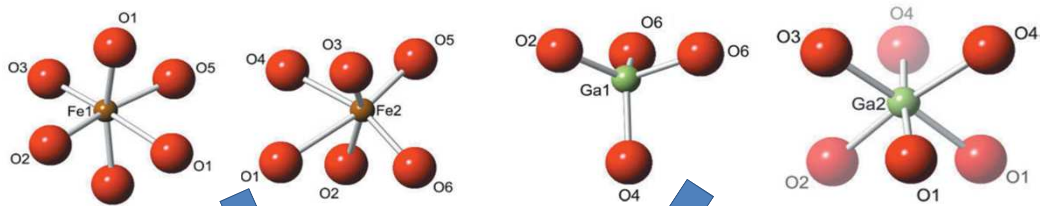
$GaFeO_3$

Combined analysis of **Fe** and **Ga** local environment

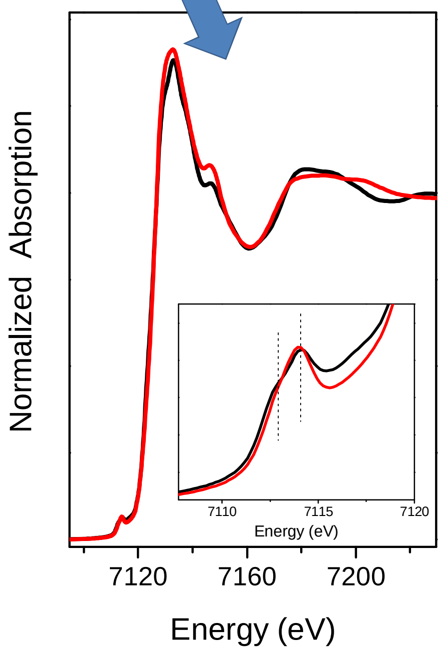
Local structure determination

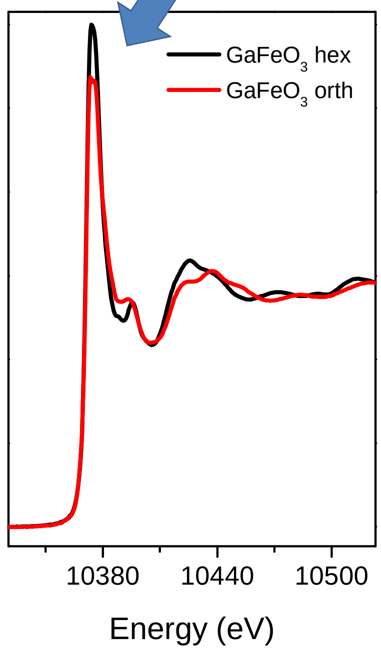


Solid State Physics

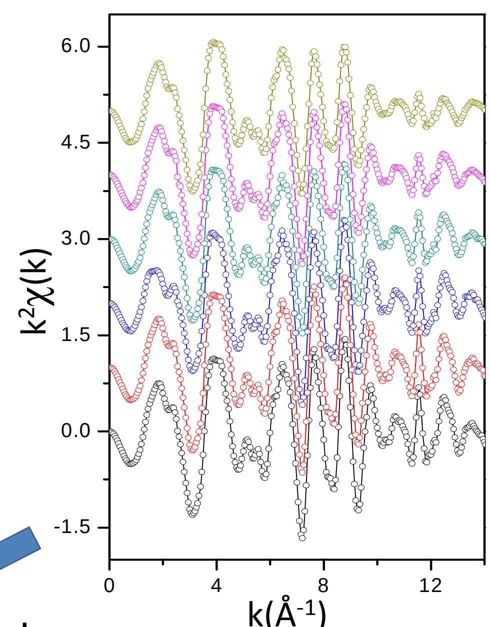


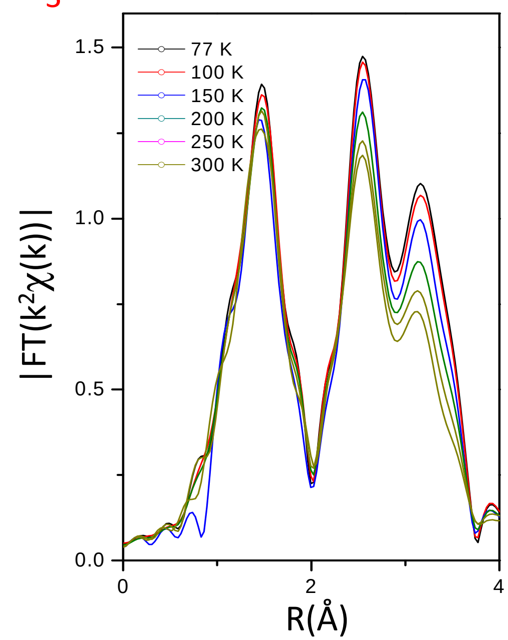
W. Olszewski et al., in preparation





$GaFeO_3$ hex

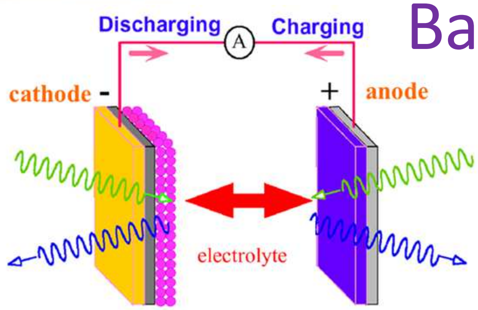


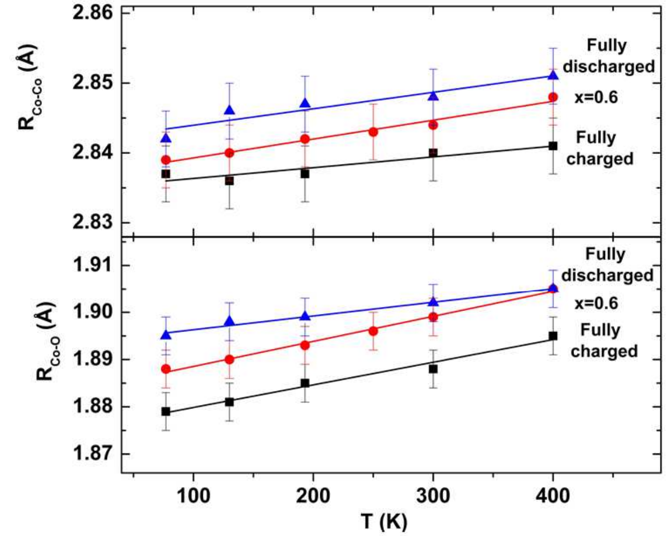


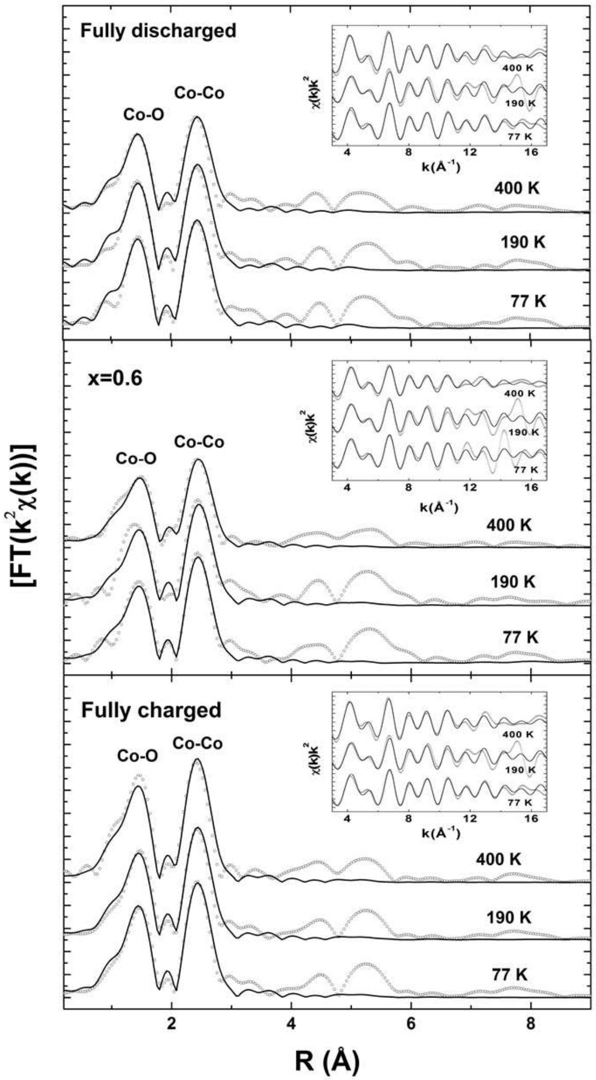
Fe-Ga local force constant

M. Avila et al., in preparation

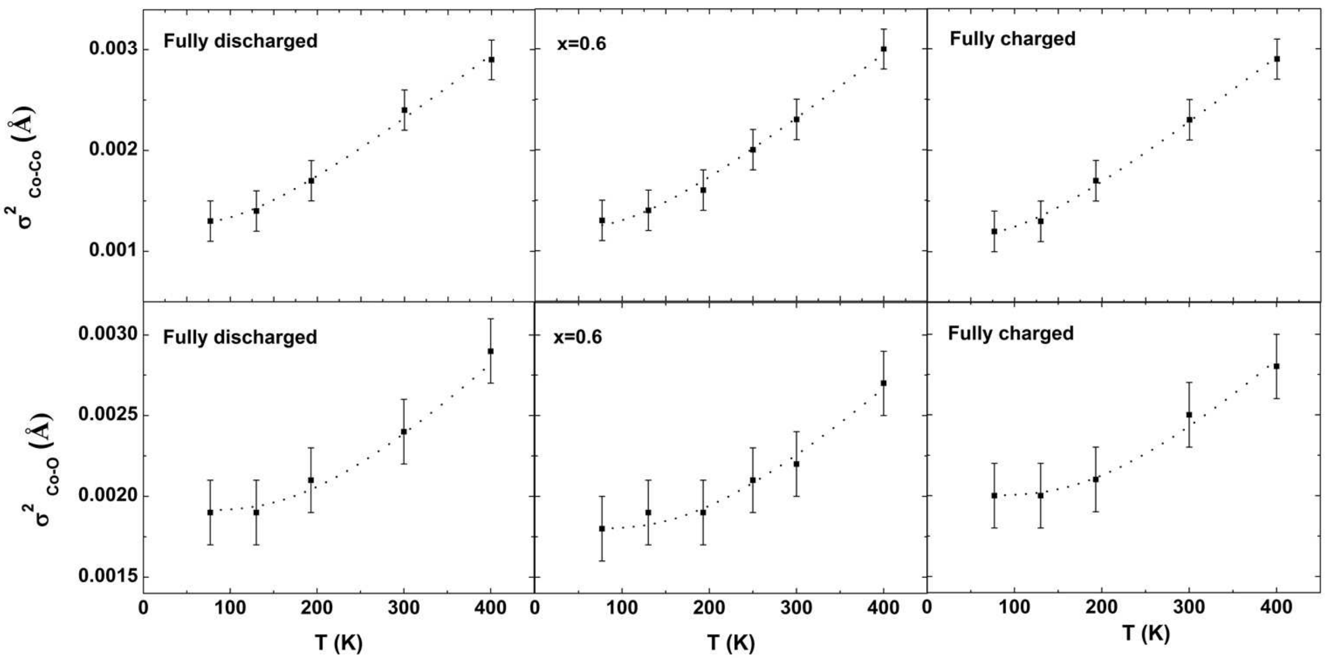
Batteries







Study of the Co-O / Co-Co local force strength as a function of sodiation/desodiation



BL22 Status

Flux and stability

(≈ 10^{12} ph/s – beam displacement in ±10-15 μm)

Energy range

(2.4 – 70 keV)

Safety

(external gas cabinet – integrated gas detectors)

Experimental setups

(remotely controlled temperature: 80-700 °C
Solid-gas reactor: 80-700°C / A.P.-8 bar)

Scan mode

(Continuous scan in transmission and fluorescence mode)

CLEAR

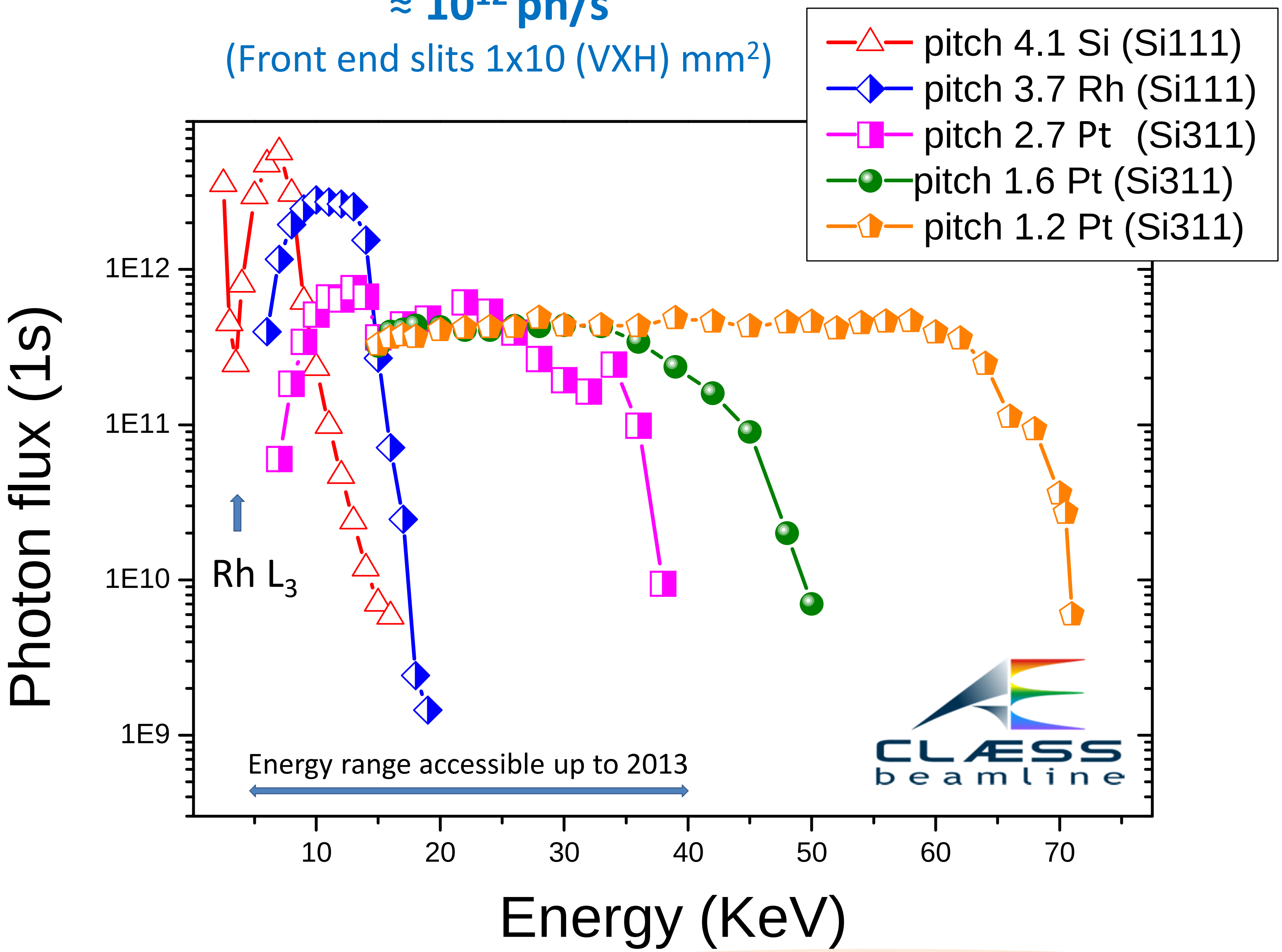
(under commissioning)

Beam flux and energy range

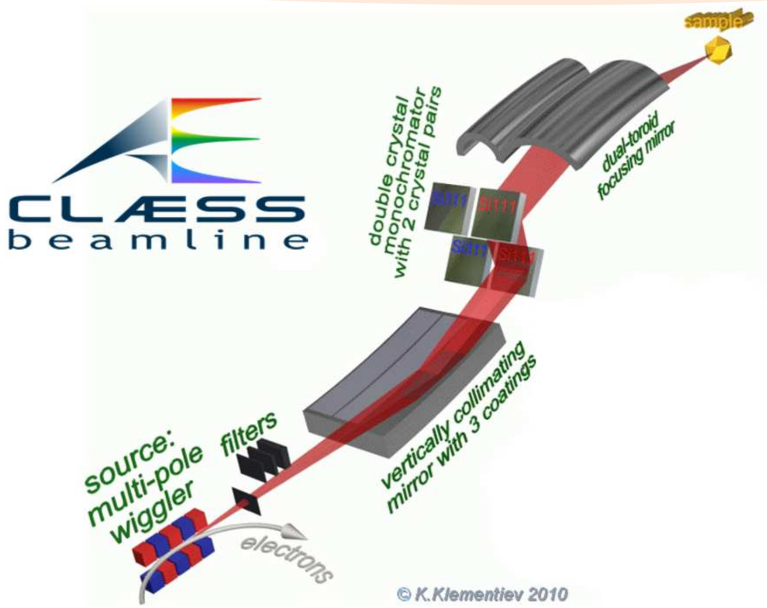
Flux at the sample from **2.4 to 70 keV**

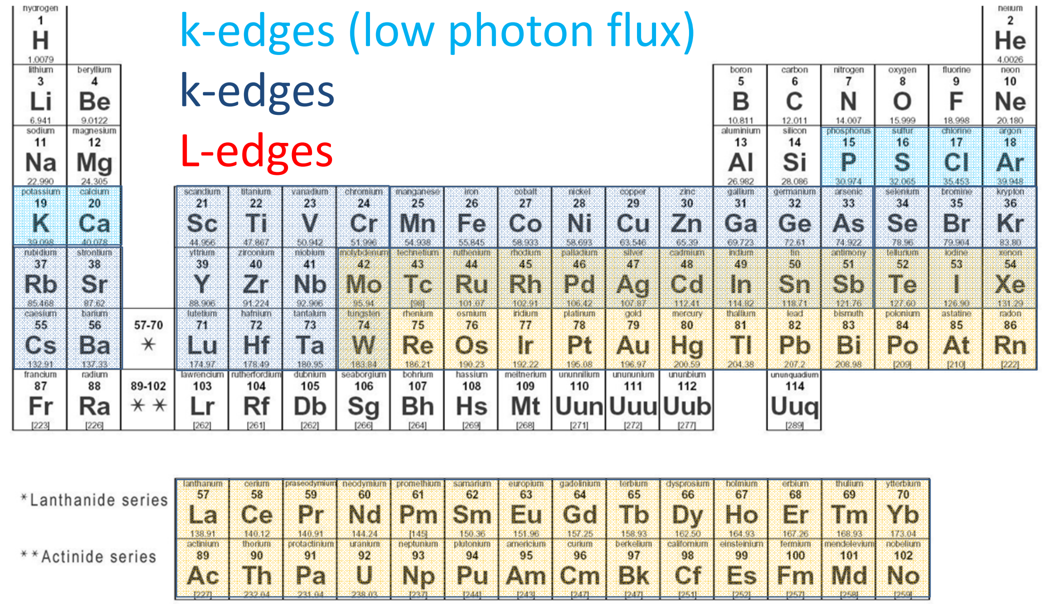
≈ 10^{12} ph/s

(Front end slits 1x10 (VXH) mm^2)

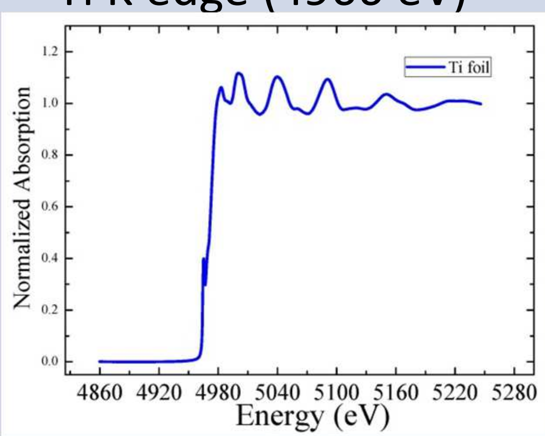


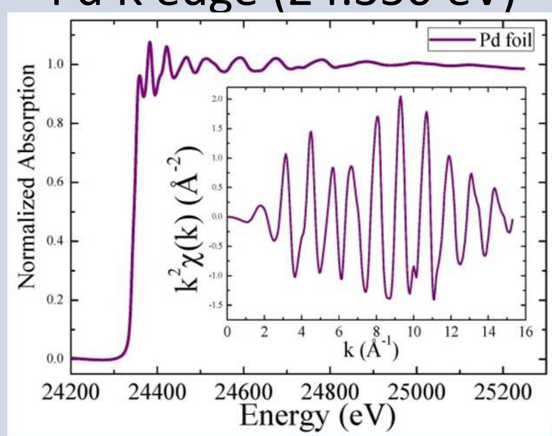
Beam flux and energy range

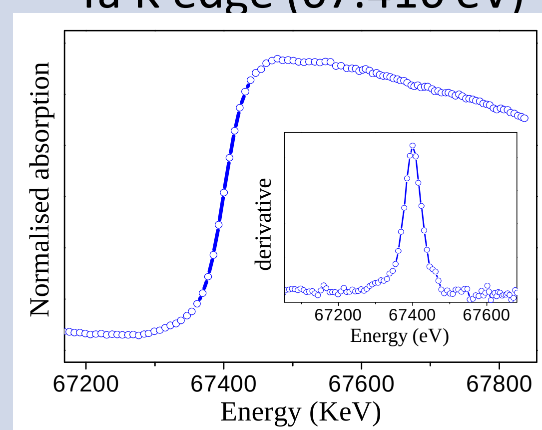




Low Energies	Middle Range	High Energies
Ti K edge (4966 eV)	Pd K edge (24.350 eV)	Ta K edge (67.416 eV)





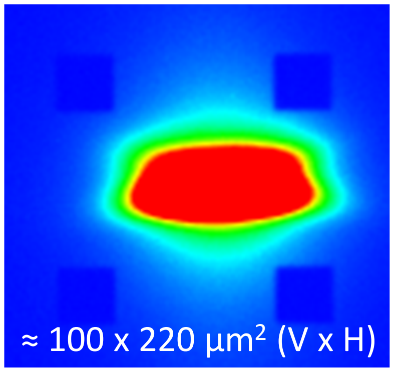


BL22 Status

Beam displacement during a 1.1 keV scan (up to ±7 µm)

(investigated at different energies and with both Si(111) and Si(311) monochromator)

MOCO in position mode



	Si(311)	Si(111)	MOCO
Mn	25±5 µm	20±5 µm	ON
Mn	360±10 µm	270±10 µm	OFF
Co	20±5 µm	60±5 µm	ON
Co	440±10 µm	220±10 µm	OFF
Ni	15±5 µm	35±5 µm	ON
Ni	310±10 µm	200±10 µm	OFF
Fe	30±5 µm	25±5 µm	ON
Fe	330±10 µm	360±10 µm	OFF
Cu	20±5 µm	35±5 µm	ON
Cu	310±10 µm	200±10 µm	OFF

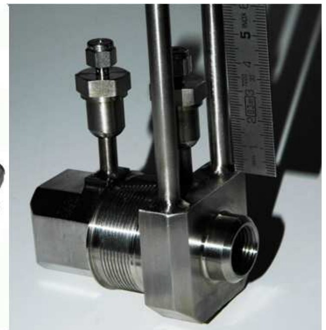
BL22 Status

Safety

New gases cabinet, CO and flammable dedicated detectors installed outside and inside the experimental hutch

Experimental setups

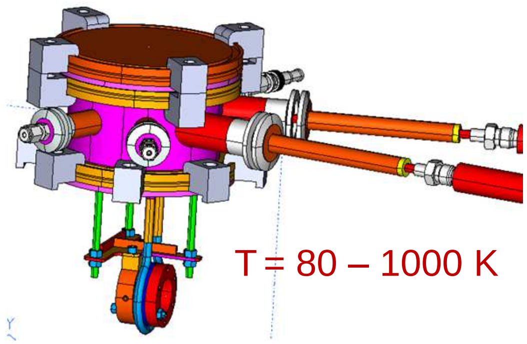
In-situ cell for catalysis



T = 300 – 1000 K
P_{max} = 8 bar
Fluo. & transm.

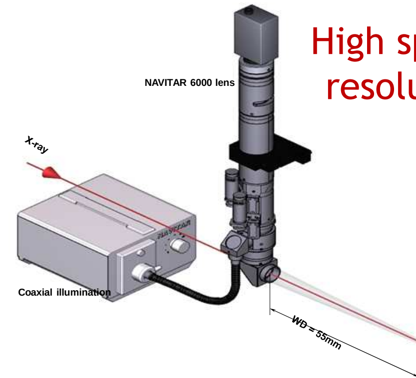
T = 80 – 1000 K
P_{max} = 4 bar
Transmission

LN2/furnace system

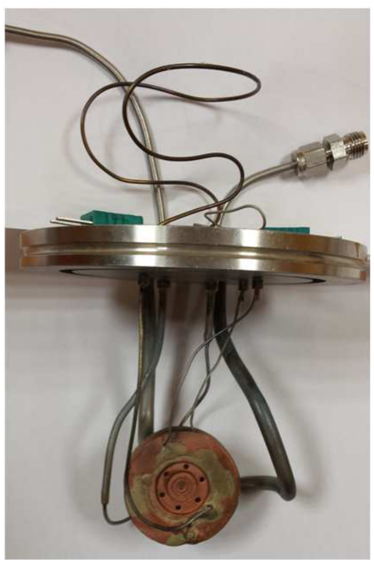


T = 80 – 1000 K

In line NAVITAR camera (I. Sics)



High spatial resolution



New prototype of in-situ cell for catalysis:

- Transmission and fluorescence geometry
- T = 80 - 1000 K
- In vacuum (low energy accessible)
- Expected pressure range below 20 bar

Iulian Preda et. al, working in progress

XAS scan mode

Continuous scan in transmission mode
E < 30 keV
≈ 5 (10) min per scan XANES (EXAFS)
Up to 4000 points

Step scan in transmission and fluorescence mode
2.4 < E < 70 keV
≈ 10 (60) min per scan XANES (EXAFS)

Continuous scan in fluorescence mode
E < 30 keV
In commissioning since June 2014

BL22 Status

CLEAR

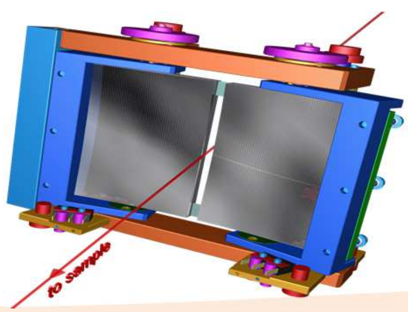
x-ray emission spectrometer (2-22 keV)

In commissioning from May 2014

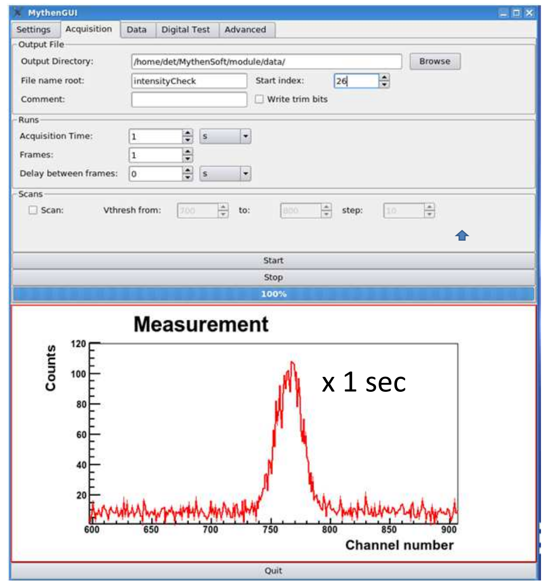

CLEAR installation on CLÆSS
April 2014

First fixed radius analyzer prototype in commissioning – first users on **Dec. 2014**

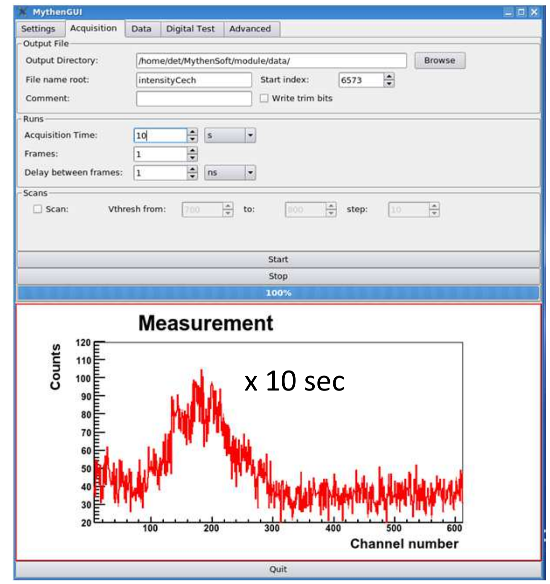
Final analyzer bender project ready for **Feb. 2015**



Si(555) reflection on Mythen



Verbeni's Si(555) diced
$3\cdot10^3$ cps



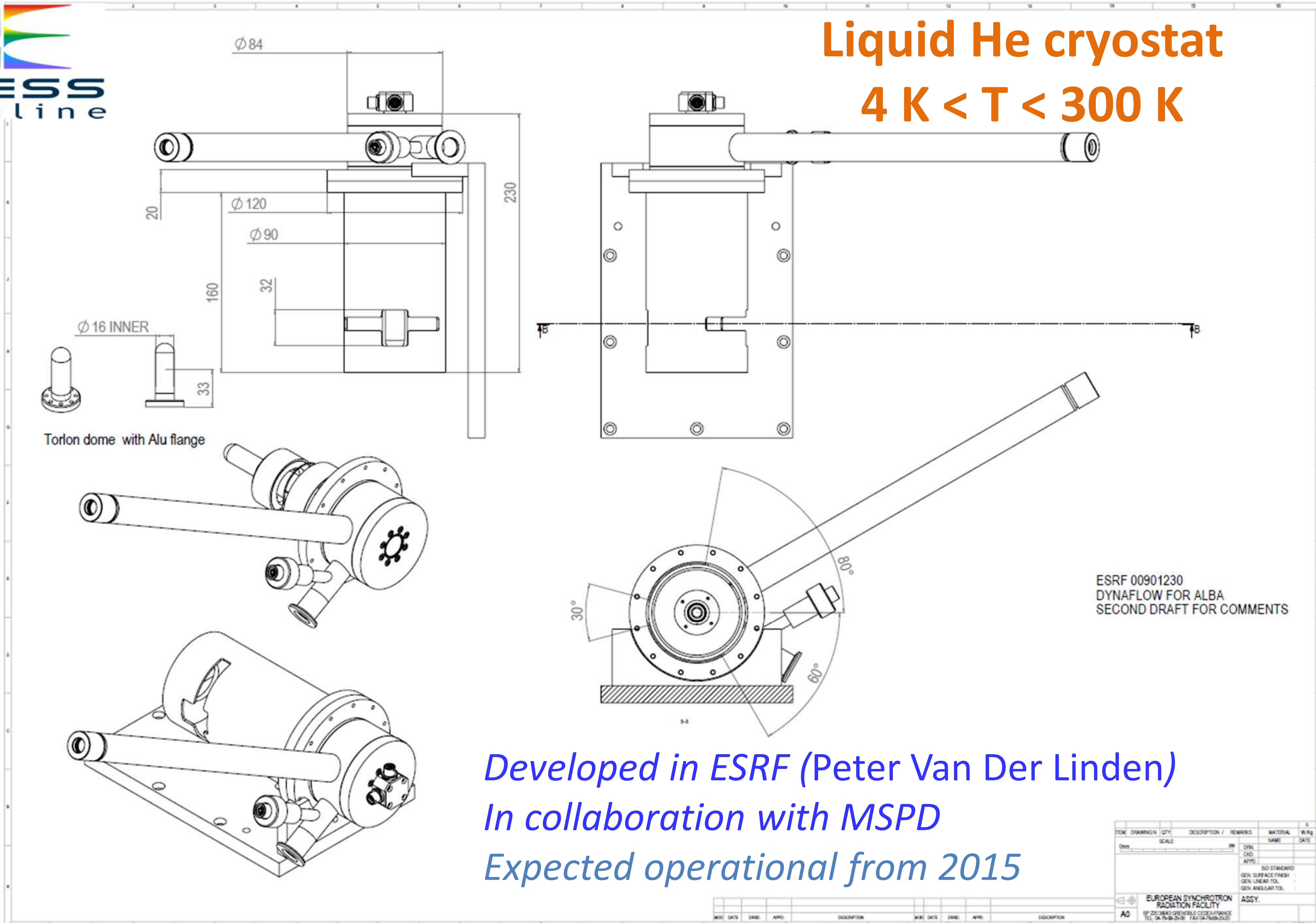
5 Si(555) diced stripes
660 cps → $4\cdot10^3$ cps for a full Verbeni diced analyser

CLÆSS beamline

Future and perspective

Liquid He cryostat

4 K < T < 300 K

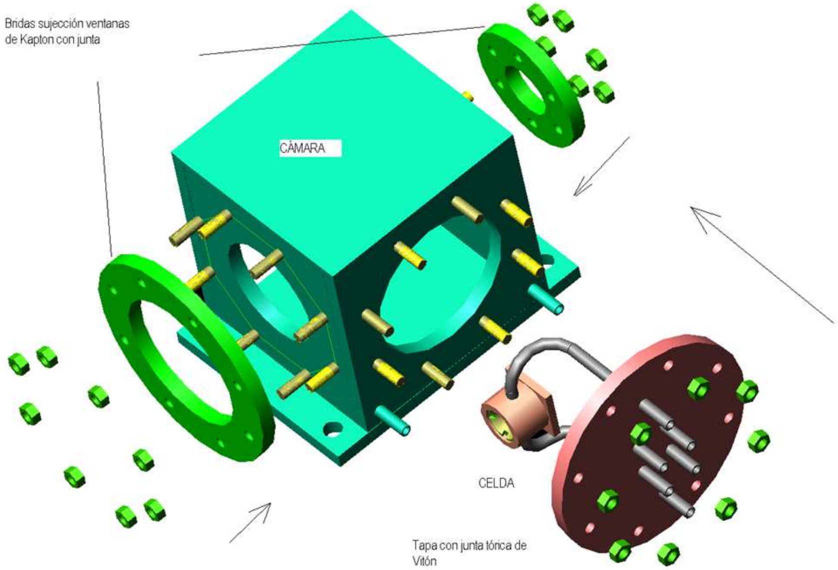


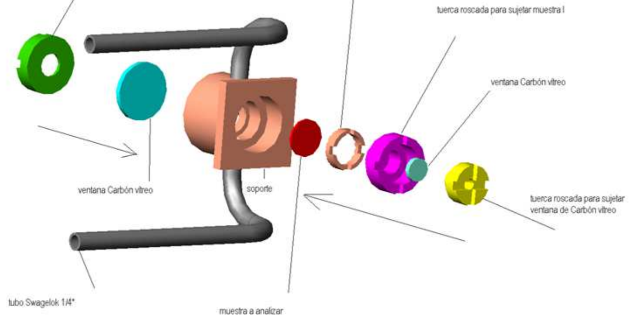
*Developed in ESRF (*Peter Van Der Linden*)*

In collaboration with MSPD

Expected operational from 2015

Future and perspective

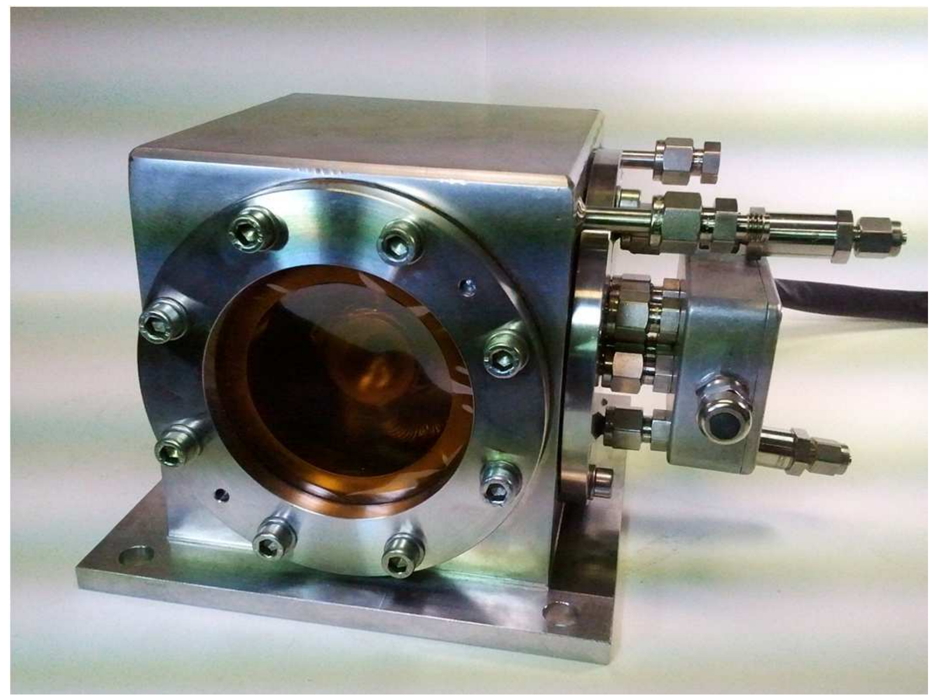




New liquid-solid-gas reactor cell

300 K < T < 1000 K

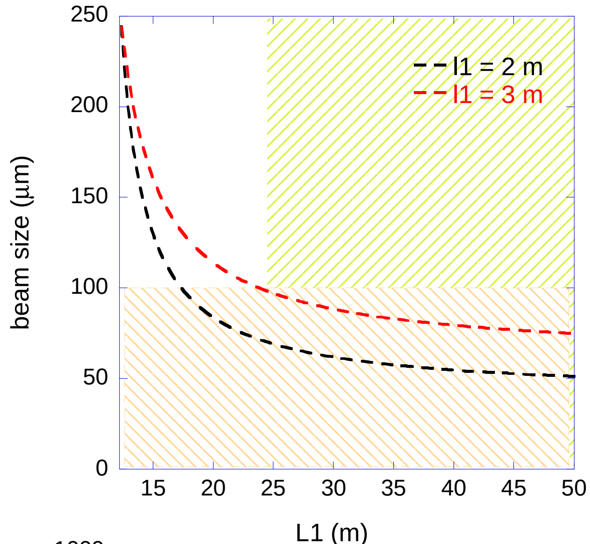
P < 20 bar

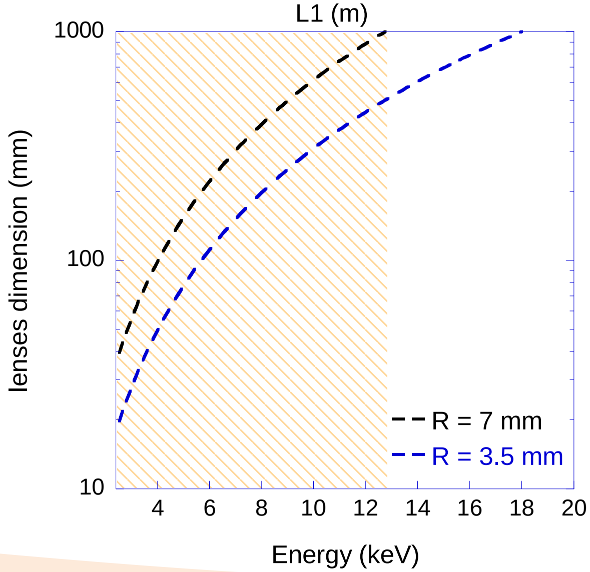


Developed in the ITQ in collaboration with MSPD

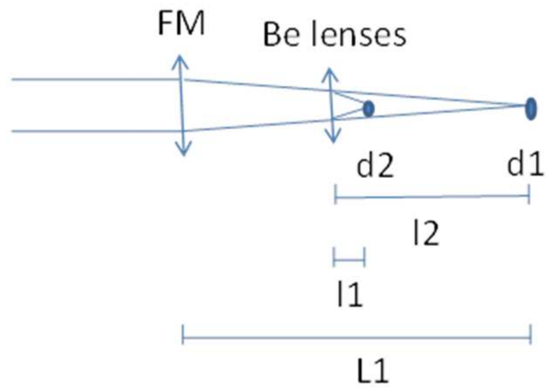
Expected operational from 2015

Beam size reduction – Be lenses ?





VERTICAL PLANE

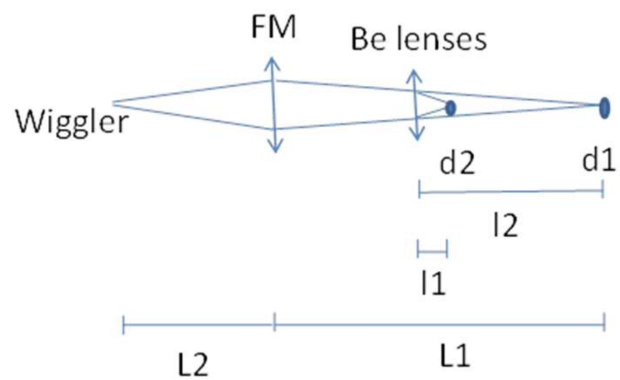


$1/L1 = 2/(R\cdot\sin\theta)$; $\theta \sim 0.002$ rad

$l1 \sim 2$ or 3 m

$M_{CRL}/M_{OF} = (L1 \cdot l1)/[(L1-l2+l1) \cdot l2]$

HORIZONTAL PLANE



$1/L1 + 1/L2 = 2\cdot\sin\theta/\rho$; $\theta \sim 0.002$ rad

$l1 \sim 2$ or 3 m

$M_{CRL}/M_{OF} = (L1 \cdot l1)/[(L1-l2+l1) \cdot l2]$

IMAGING ?

High-PRESSURE ?

BL22-CLÆSS: XAS & XES

CLÆSS beamline

XAS Chemical, electronic, and structural information **(2.4 - 70 keV)**

XES electronic, magnetic, and structural information **(2 - 22 keV)**

Beamline performance:

1. Continuous scan in transmission mode (5-10 min scan)
2. XAS in transmission and fluorescence detection mode
3. XES – *first analyser prototype expected operational from 2015*
4. Stable beam-position (±10 μm) and beam-size (200-500 μm) for 2.4 < E < 70 keV

Experimental set-ups:

1. Remote temperature controller from 80 K to 800 K
2. In-situ cell for solid-gas reactions (80 K – 700 K)
3. Low temperature cryostat (4 K – 300 K) - *Expected operational from 2015*

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