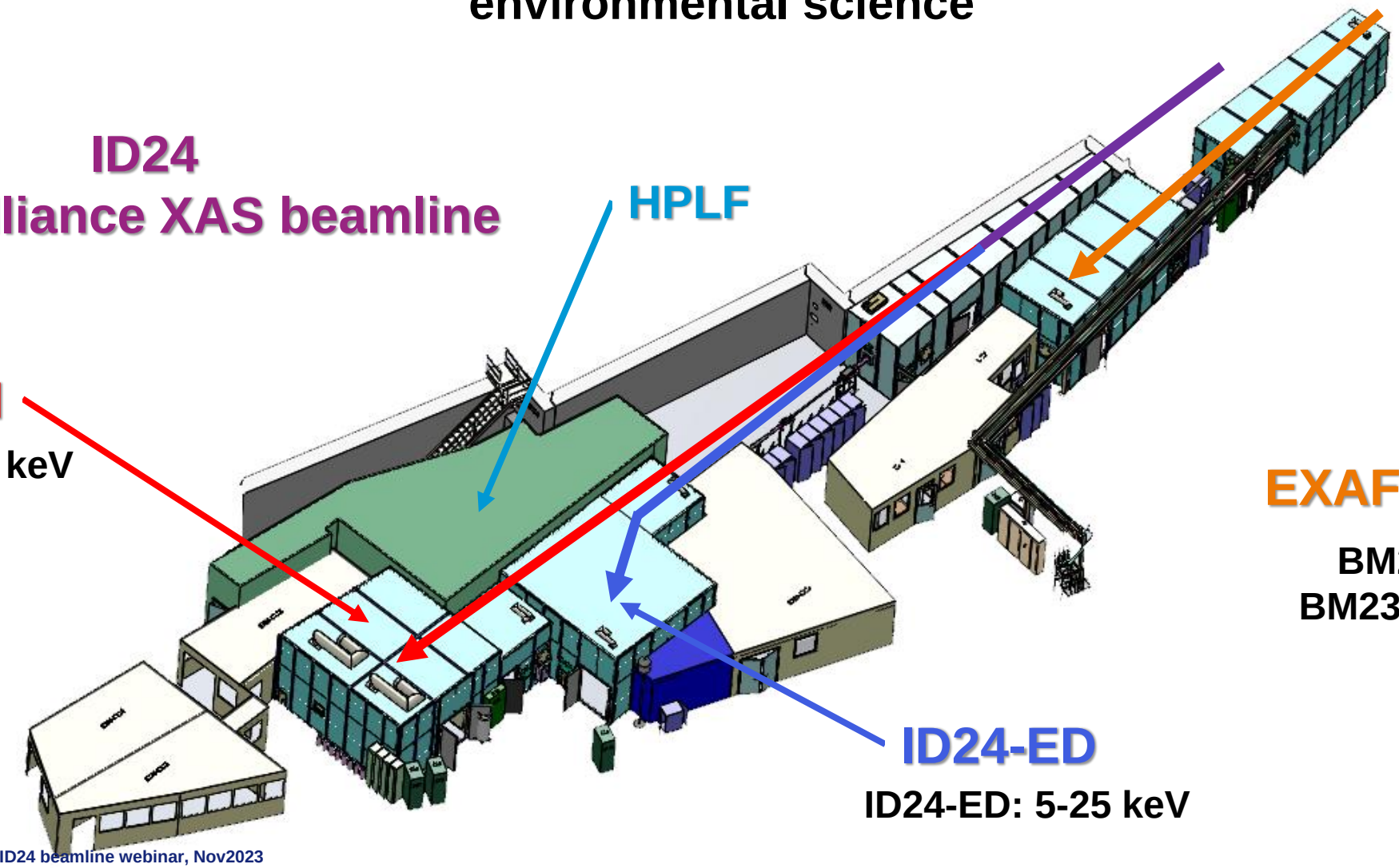
New scientific opportunities at BM23 and ID24 XAS beamlines

ID24/BM23 unit:
J.-A. Hernandez, K.A. Lomachenko, O. Mathon
A.D. Rosa, R.Torchio

BM23-ID24

Two beamlines dedicated to X-ray Absorption Spectroscopy
For *in situ/operando* chemistry, matter under extreme conditions (P, T, H) and environmental science



ID24
High Brilliance XAS beamline

HPLF

ID24-DCM

ID24-DCM 5-45 keV

BM23
EXAFS beamline

BM23 5-80 keV
BM23 HP: 5-45 keV

ID24-ED

ID24-ED: 5-25 keV

ESRF

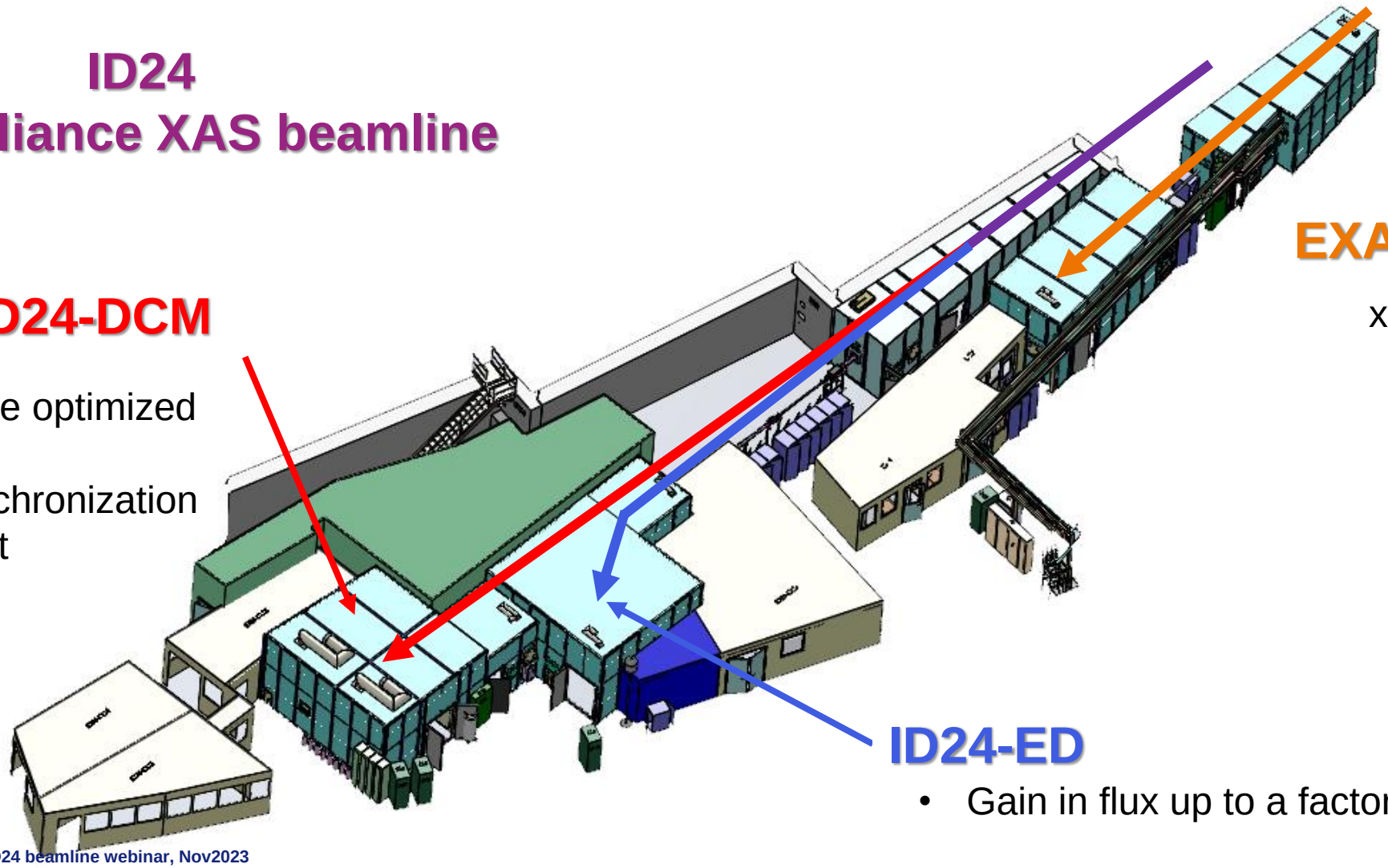
BM23-ID24 : ESRF-EBS

ESRF-EBS at BM23-ID24

ID24
High Brilliance XAS beamline

ID24-DCM

- Simple beamline optimized for EBS
- Gap scans synchronization
- Small focal spot
- High flux



BM23
EXAFS beamline

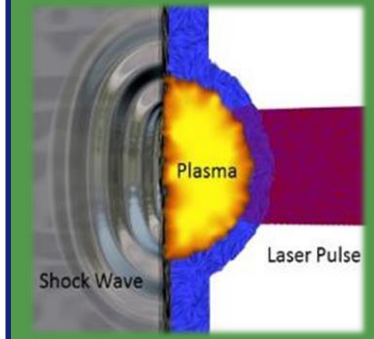
x3 gain in flux

ID24-ED

- Gain in flux up to a factor x10

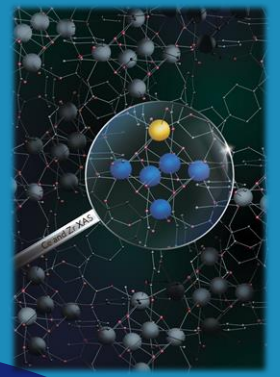
BM23/ID24

Matter at extremes



- Physics and chemistry of matter at extreme P,T
- Warm Dense Matter
- Synthesis of new materials
- Materials under high pulsed magnetic field
- Dynamic behavior of matter

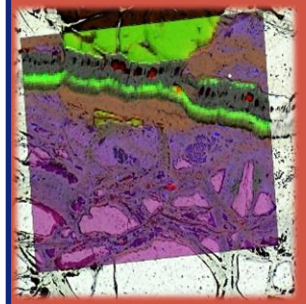
Structure of novel materials



- Batteries and fuel cells
- Nanoparticles
- Gas sensors and separators
- Drugs

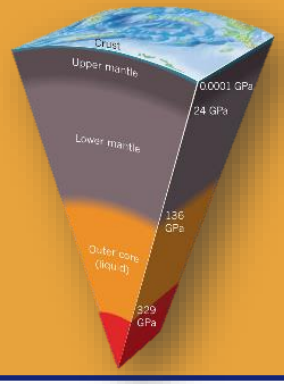
Physics and chemistry of complex materials under relevant conditions

Environmental science



- Geo-resources
- Biogeochemical processes
- Impact of human activity on our environment

Geo and Planetary Science



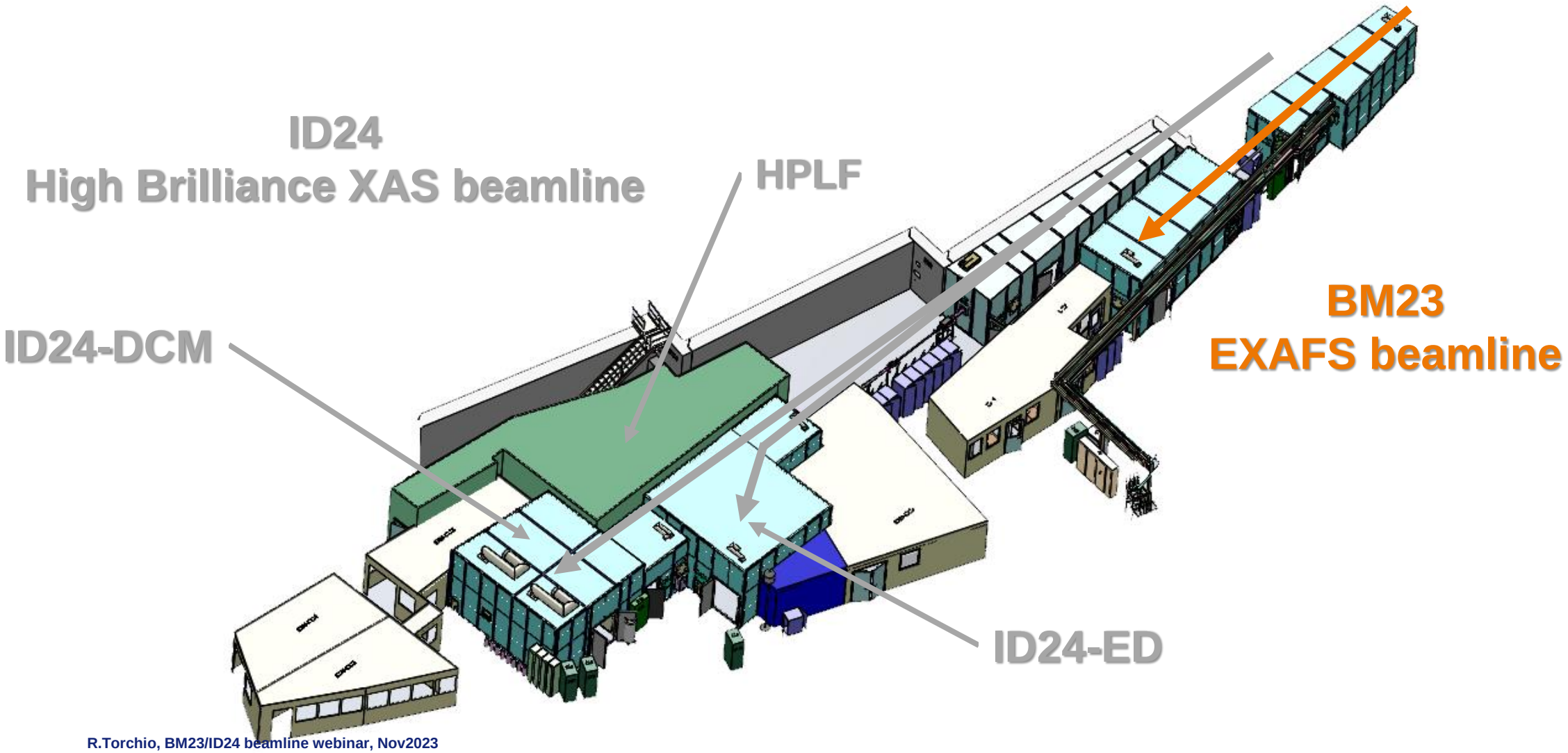
- Planetary interiors
- Melting curves
- global element cycles and geodynamical processes in the deep Earth's interior

In situ and *operando* chemistry

- Catalysis
- Synthesis
- Electrochemistry
- Photochemistry

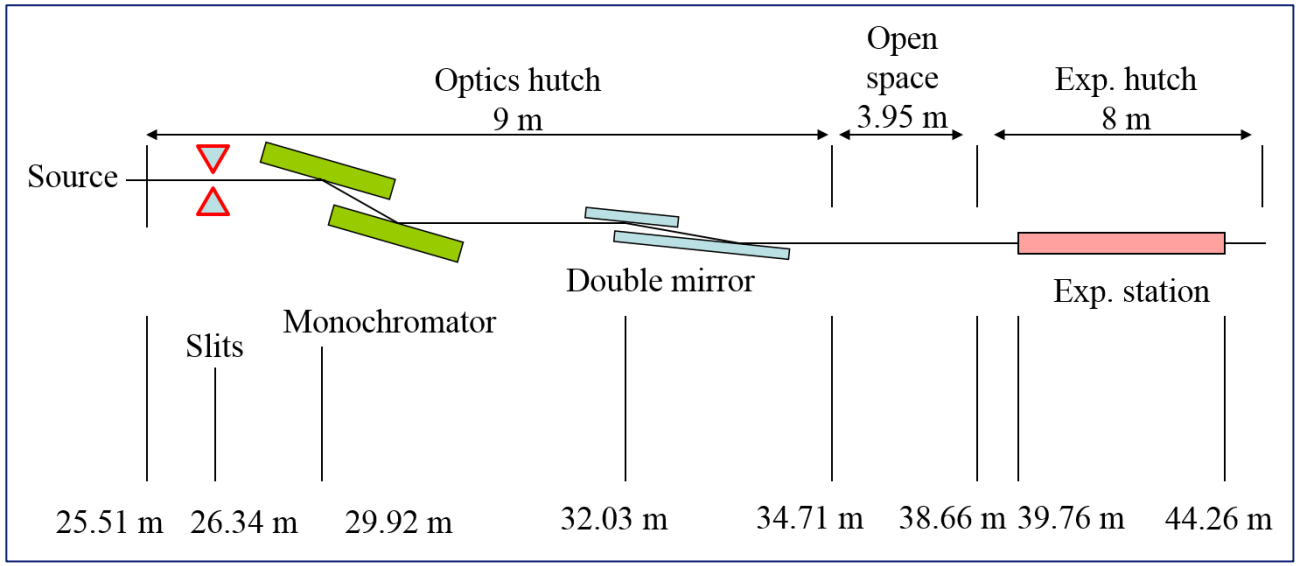
BM23-ID24

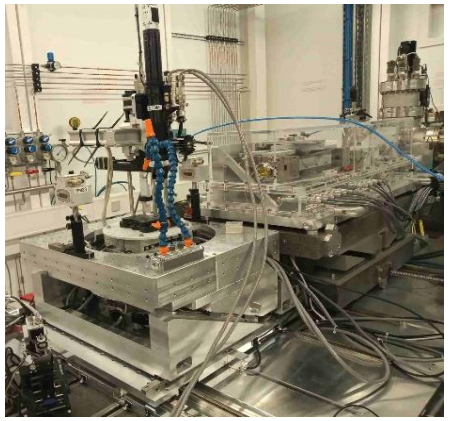
Two beamlines dedicated to X-ray Absorption Spectroscopy
For *in situ/operando* chemistry and matter under extreme conditions (P, T, H)

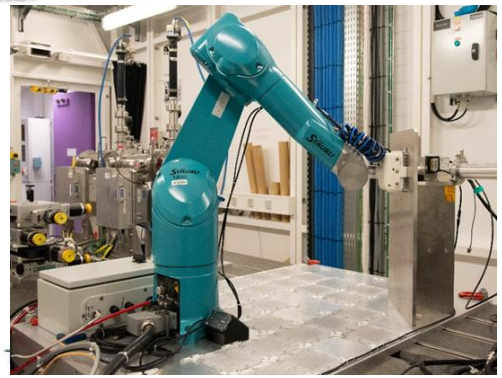


BM23: ESRF GENERAL PURPOSE EXAFS BEAMLINE

A simple optical scheme dedicated to high quality EXAFS

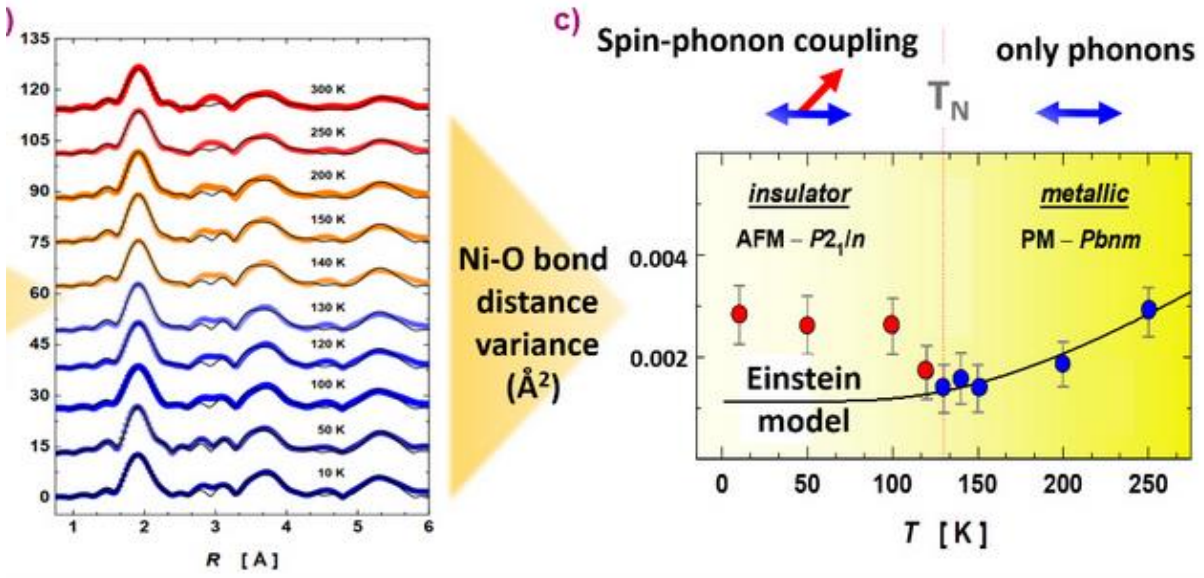


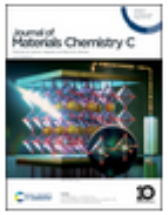




- Available energy range: 4-75 keV
- 10^{10} - 10^{9} ph/s
- Excellent signal-to-noise ratio over a large k-range
- Transmission and fluorescence modes
- Versatility and high automation level, robot
- μXAS station 3x3 μm^2 5-40 KeV
- Sample environments: He cryostats, ovens, XRD
  High pressure PE cell, DAC, RH-DAC, LT-DAC
  Chemistry: XAS/DRIFTS/MS setup

Unveiling the spin-phonon coupling in $PrNiO_3$ Nickelate, a promising spintronic material





From the journal:
Journal of Materials Chemistry C

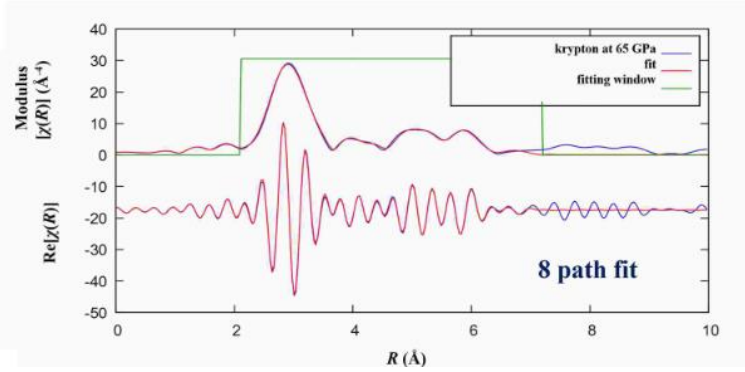
J.E.Rodriguez et al. 2023

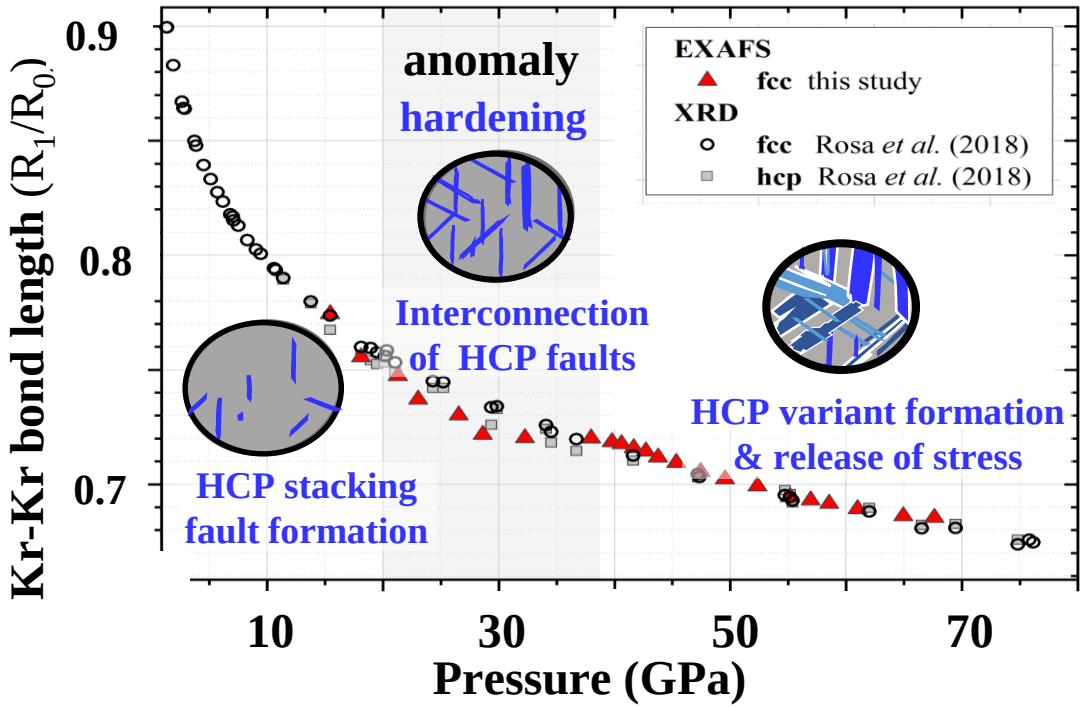
The martensitic transformation in solid Kr and Xe

PHYSICAL REVIEW B

A. D. Rosa, A. Dewaele, G. Garbarino, V. Svitlyk, G. Morard, F. De Angelis, Mathon, and M. A. Bouhifd

Phys. Rev. B **105**, 144103 – Published 1

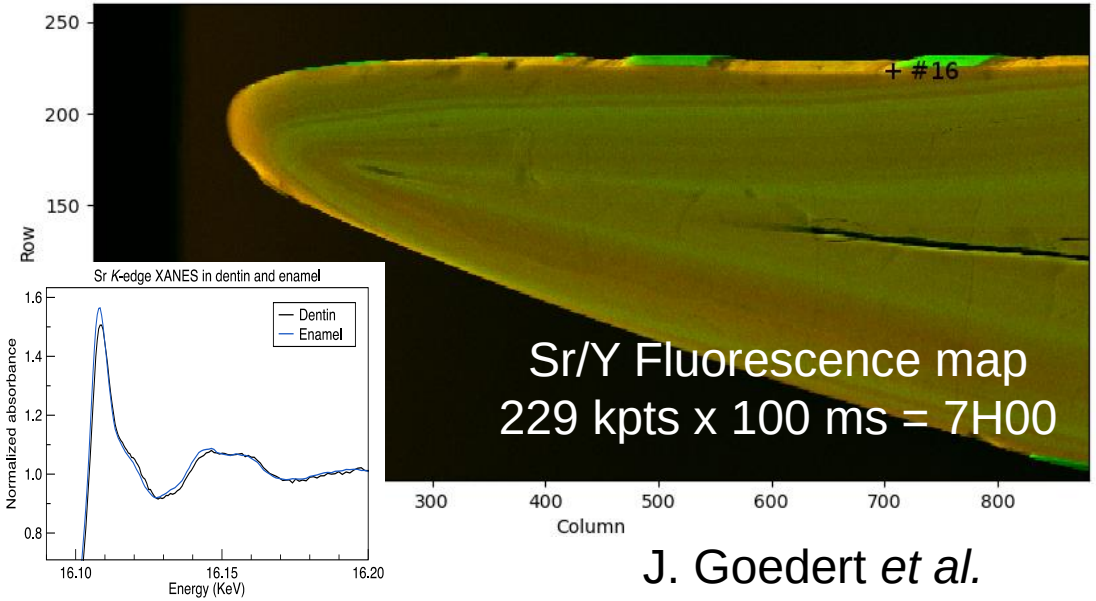




CULTURAL HERITAGE AND PALEONTOLOGY

Theropodous Teeth, Early Cretaceous (140 my) Deposit of Angeac-Charente (France)

- Proxies for *de vivo* elements (Sr) and elements acquired *post-mortem (Y)*
- Assess the degree of preservation of *de vivo* elements in bio-apatite

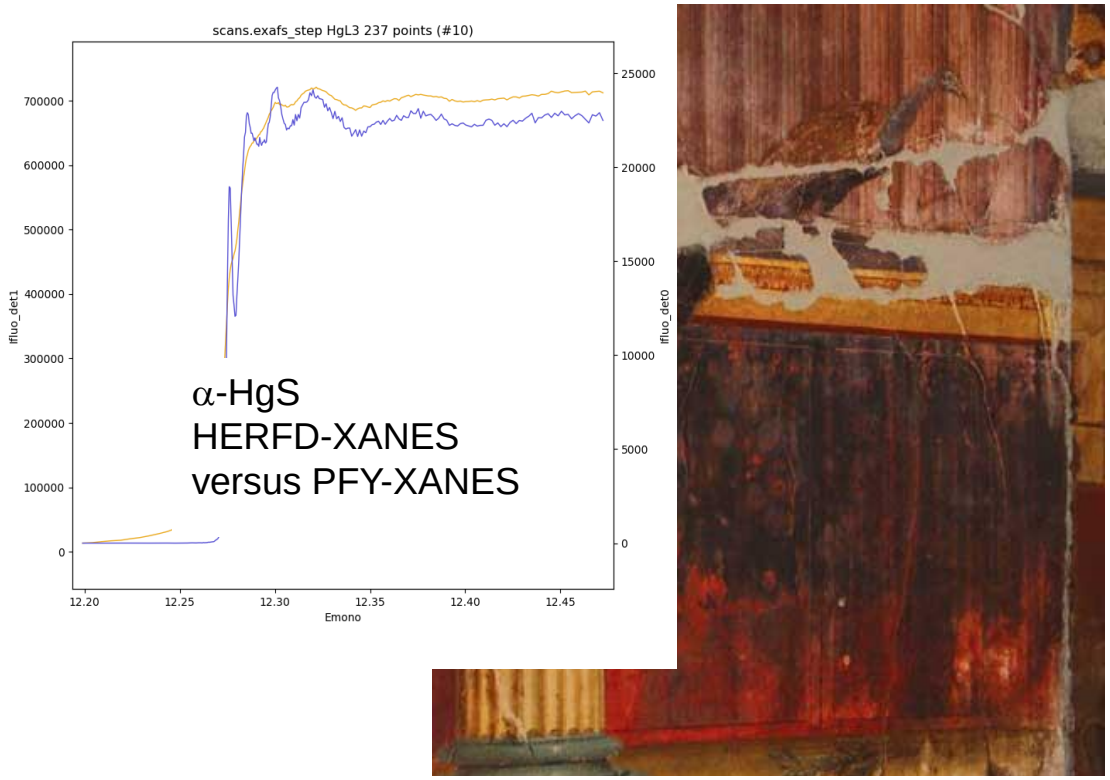


J. Goedert *et al.*

Unravel the transformation of cinnabar in the wall paintings of Pompeii

M. Maguregui *et al.*

6th Inter. Congress Chemistry for Cultural Heritage (2022)



A wide variety of Hg species. For the first time Hg_0 has been identified clearly in an historical painting.

5-crystal analyser moved to ID24-DCM

CHEMISTRY

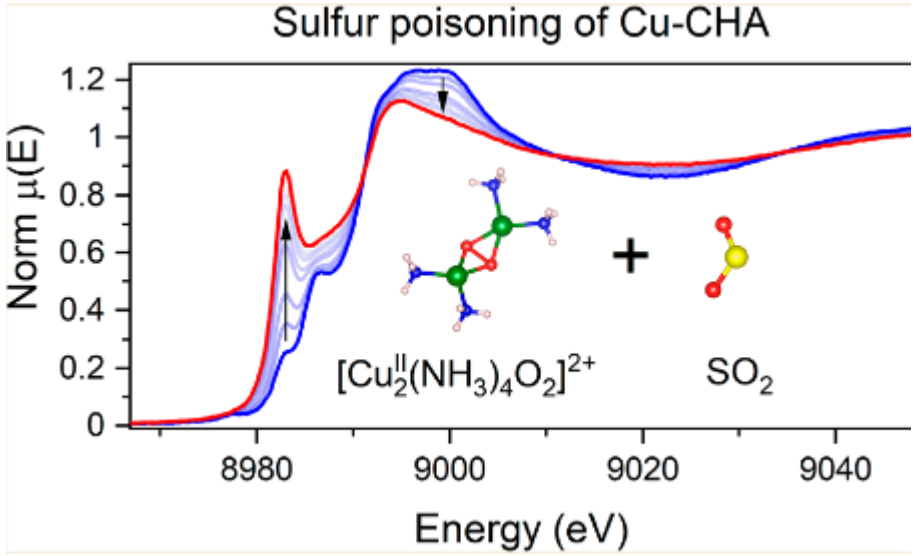
JACS Au
AN OPEN ACCESS JOURNAL OF THE AMERICAN CHEMICAL SOCIETY

pubs.acs.org/jacsau Letter

SO_2 Poisoning of Cu-CHA deNO_x Catalyst: The Most Vulnerable Cu Species Identified by X-ray Absorption Spectroscopy

Anastasia Yu. Molokova, Elisa Borfecchia, Andrea Martini, Ilia A. Pankin, Cesare Atzori, Olivier Mathon, Silvia Bordiga, Fei Wen, Peter N. R. Vennestrøm, Gloria Berlier, Ton V. W. Janssens,* and Kirill A. Lomachenko*

Cite This: *JACS Au* 2022, 2, 787–792 Read Online

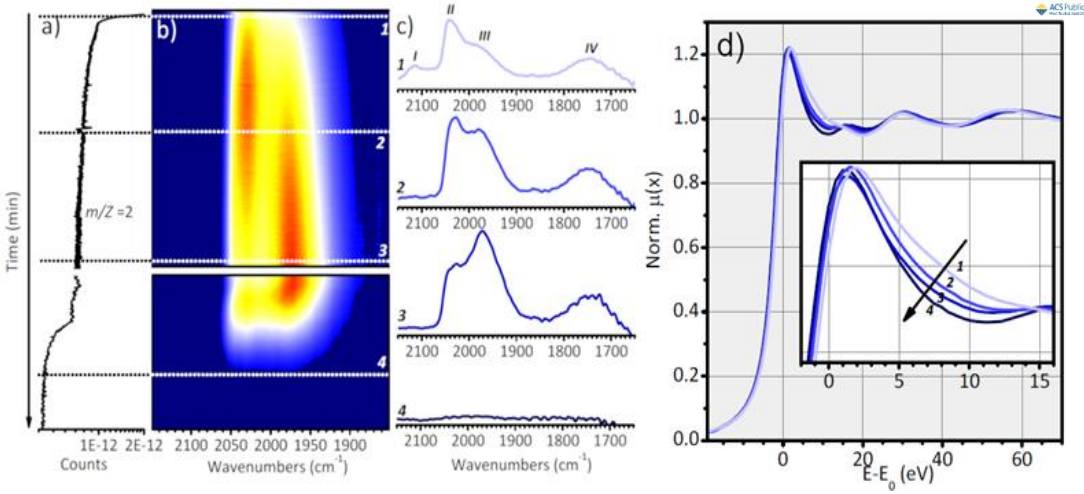


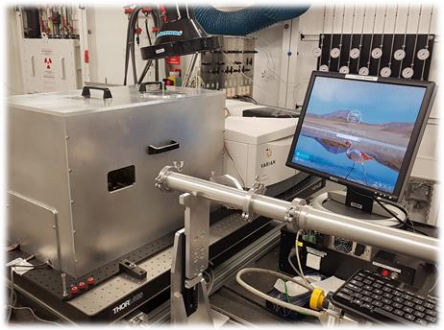
CuI and CuII species with different ligants under exposures to SO_2 using insitu XAS

SO_2mostly affects the low-temperature activity of Cu-CHA catalysts

Multi-edge and multi-technique studies

M. Carosso, *et al.*, *ACS Catal.* **2019**, *9*, 7124



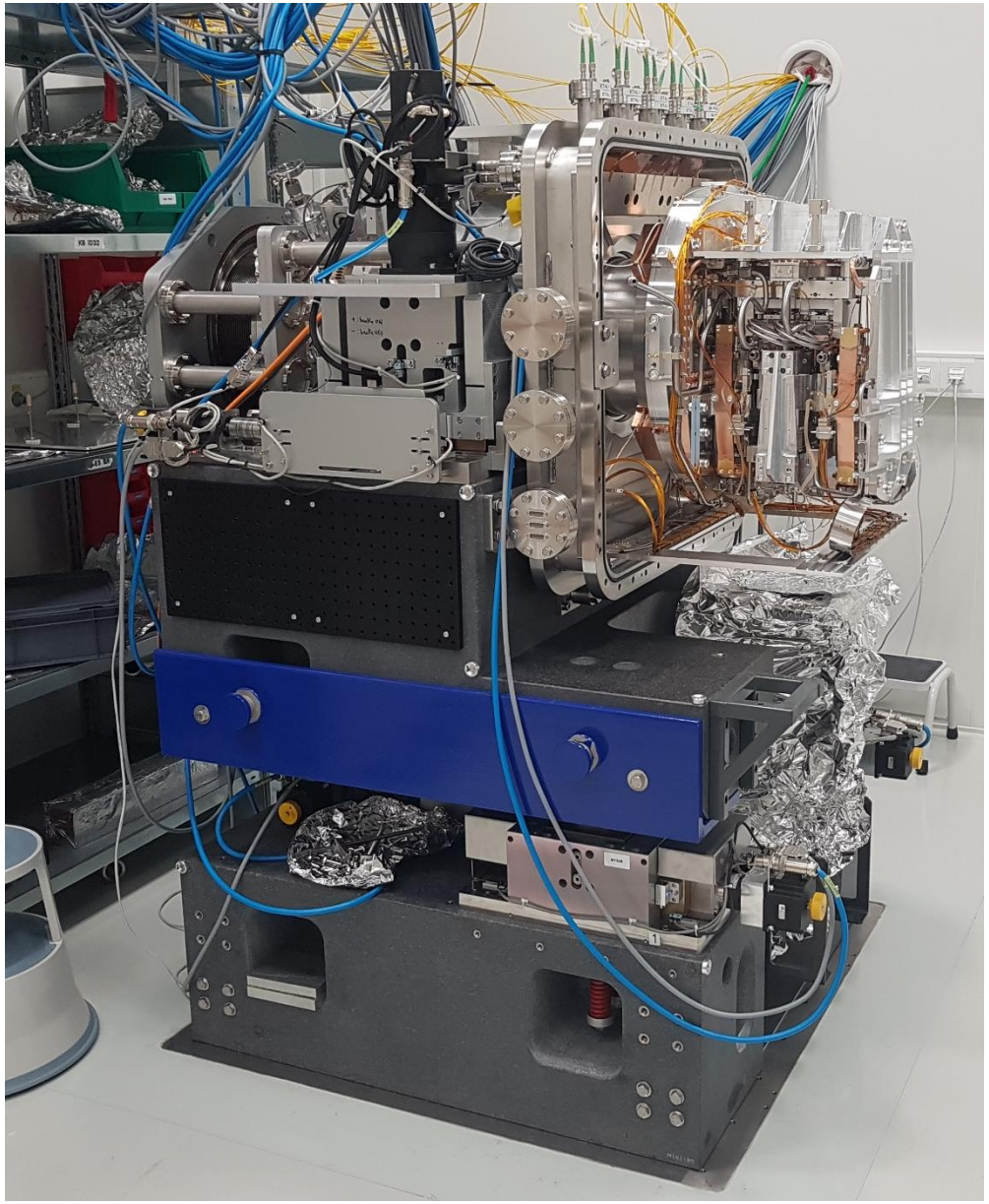


XAS+DRIFTS+MS

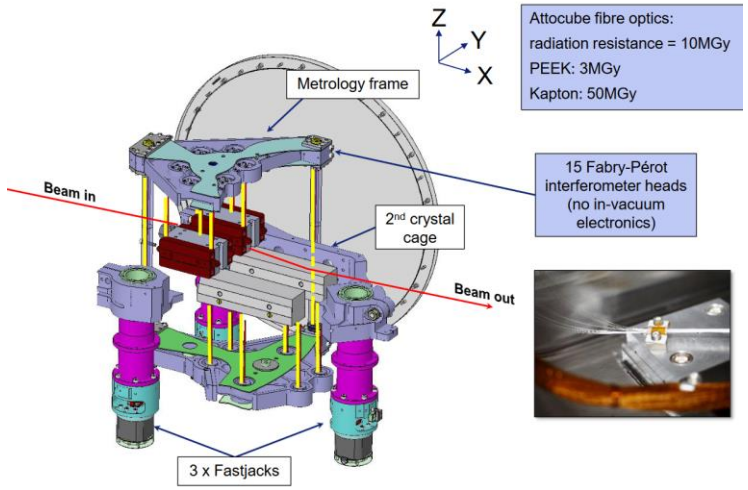
complete characterization of the surface Pt-hydride species on Pt/Al_2O_3 catalyst under different hydrogenation/dehydrogenation conditions. Surface Pt-hydrides play a fundamental role, to maintain the activity of Pt nanoparticles.

The European Synchrotron | ESRF

BM23 - NEXT STEP: Installation of the new ESRF-DCM (Winter 2023/2024)



DCM with a new technology: stability not only based on mechanics but also on real-time feedback loops



Development of a new DCM for spectroscopy by the ESRF with

- **Continuous acquisition** mode as default mode
- Perform full EXAFS spectra at the **Hz level**
- Unprecedented **energy stability (<2 meV)**
- Unprecedented **beam position stability (ΔR_y = 10 nrad rms)**

BM23-ID24

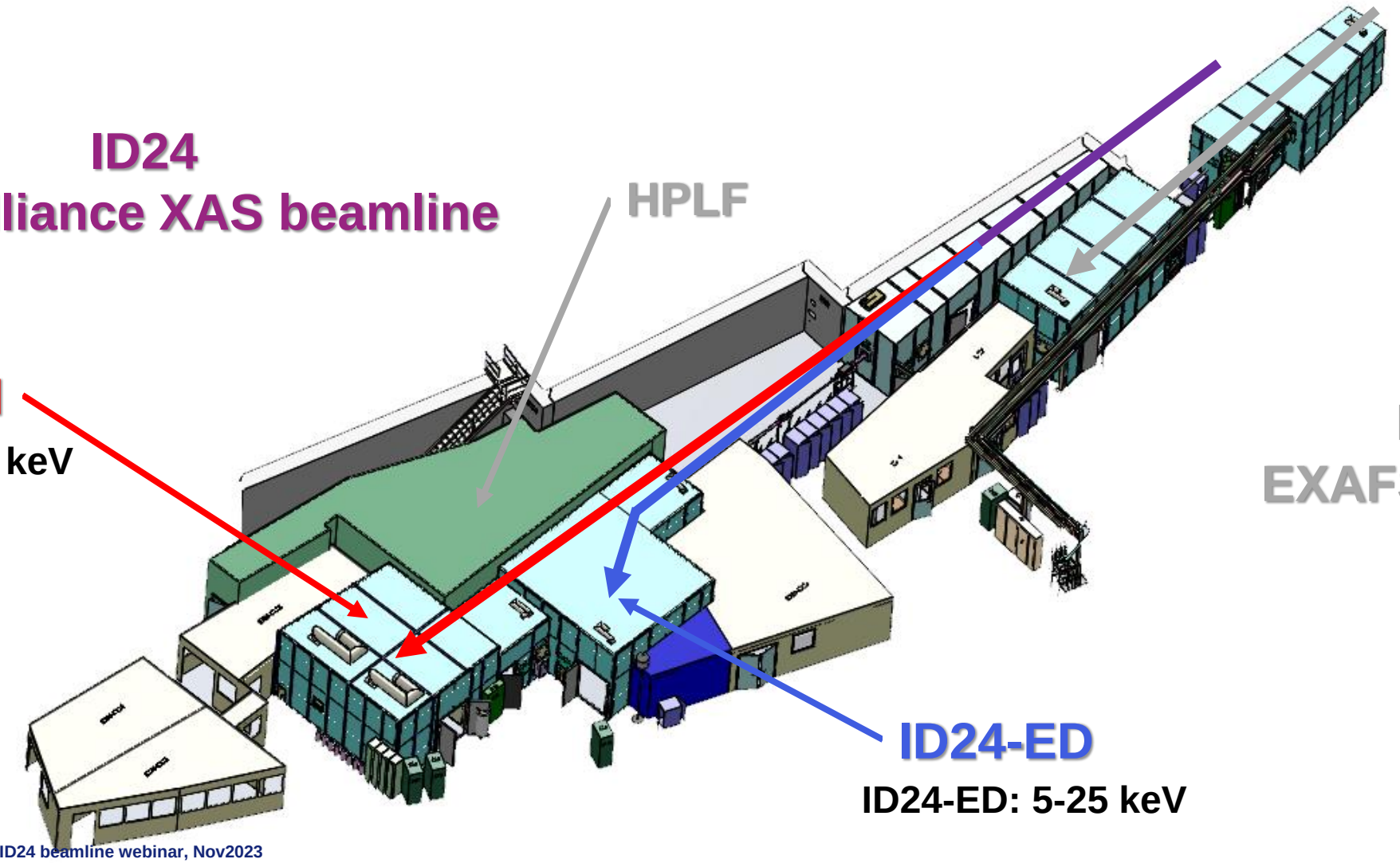
Two beamlines dedicated to X-ray Absorption Spectroscopy
For *in situ/operando* chemistry and matter under extreme conditions (P, T, H)

ID24
High Brilliance XAS beamline

HPLF

ID24-DCM

ID24-DCM 5-45 keV



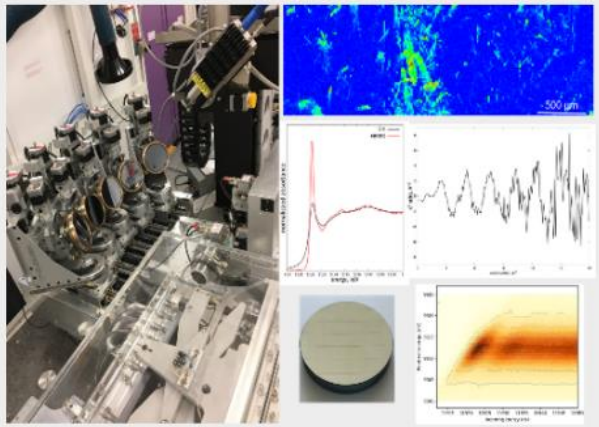
BM23
EXAFS beamline

ID24-ED

ID24-ED: 5-25 keV

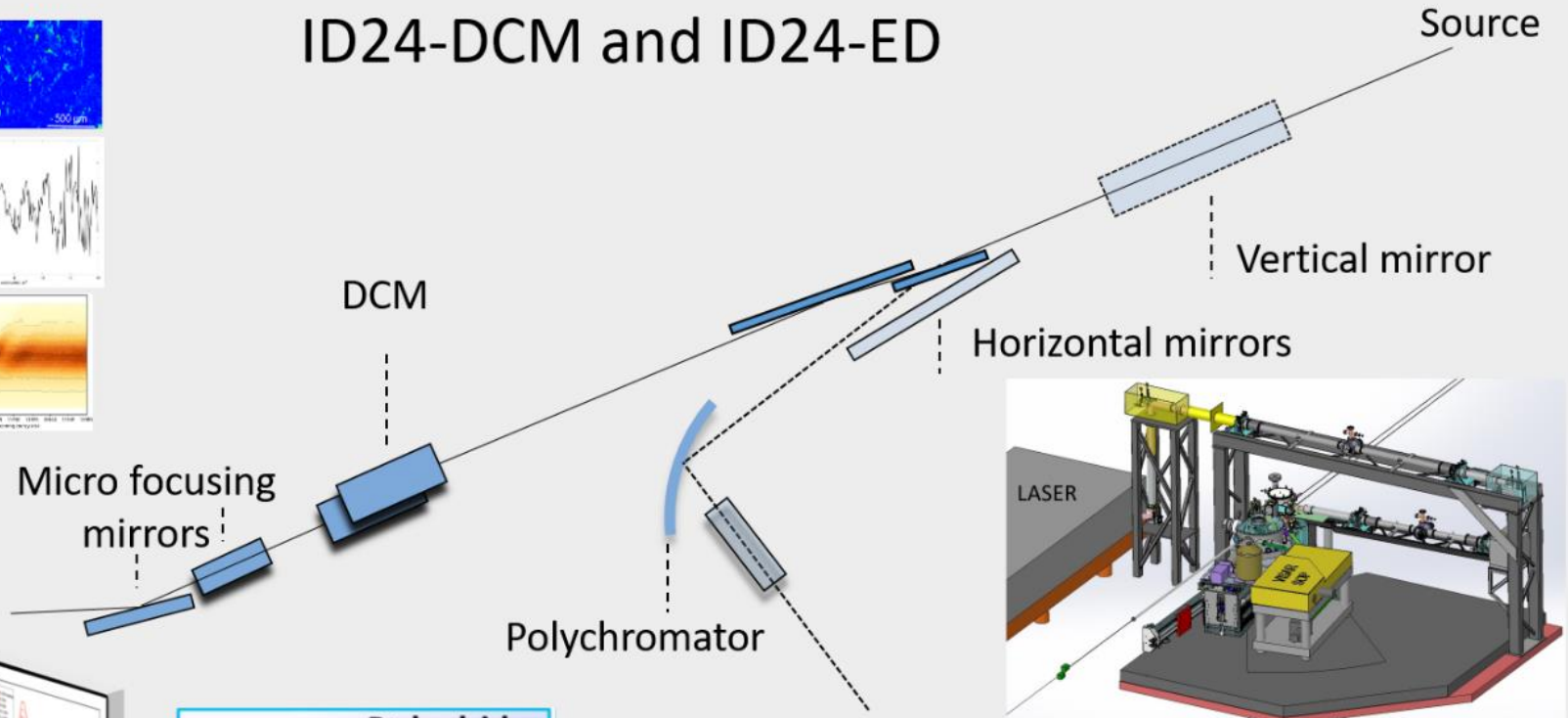
ESRF

ID24: HIGH BRILLIANCE X-RAY ABSORPTION SPECTROSCOPY BEAMLINE



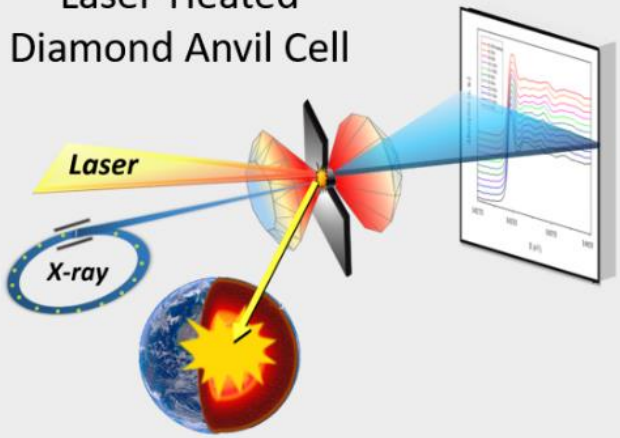
Micro-XAS/XES

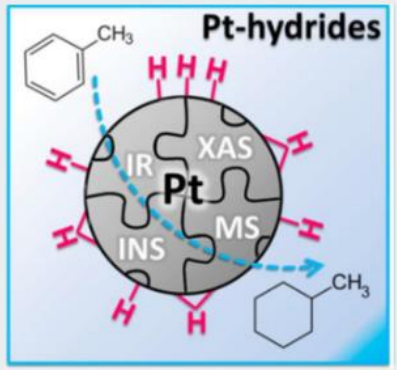
ID24-DCM and ID24-ED



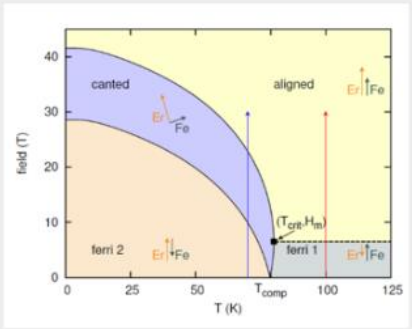
High Power Laser dynamic compression

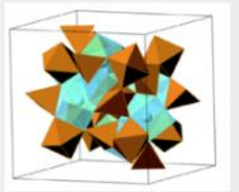
Laser-Heated Diamond Anvil Cell





In Situ XAS/DRIFTS/MS





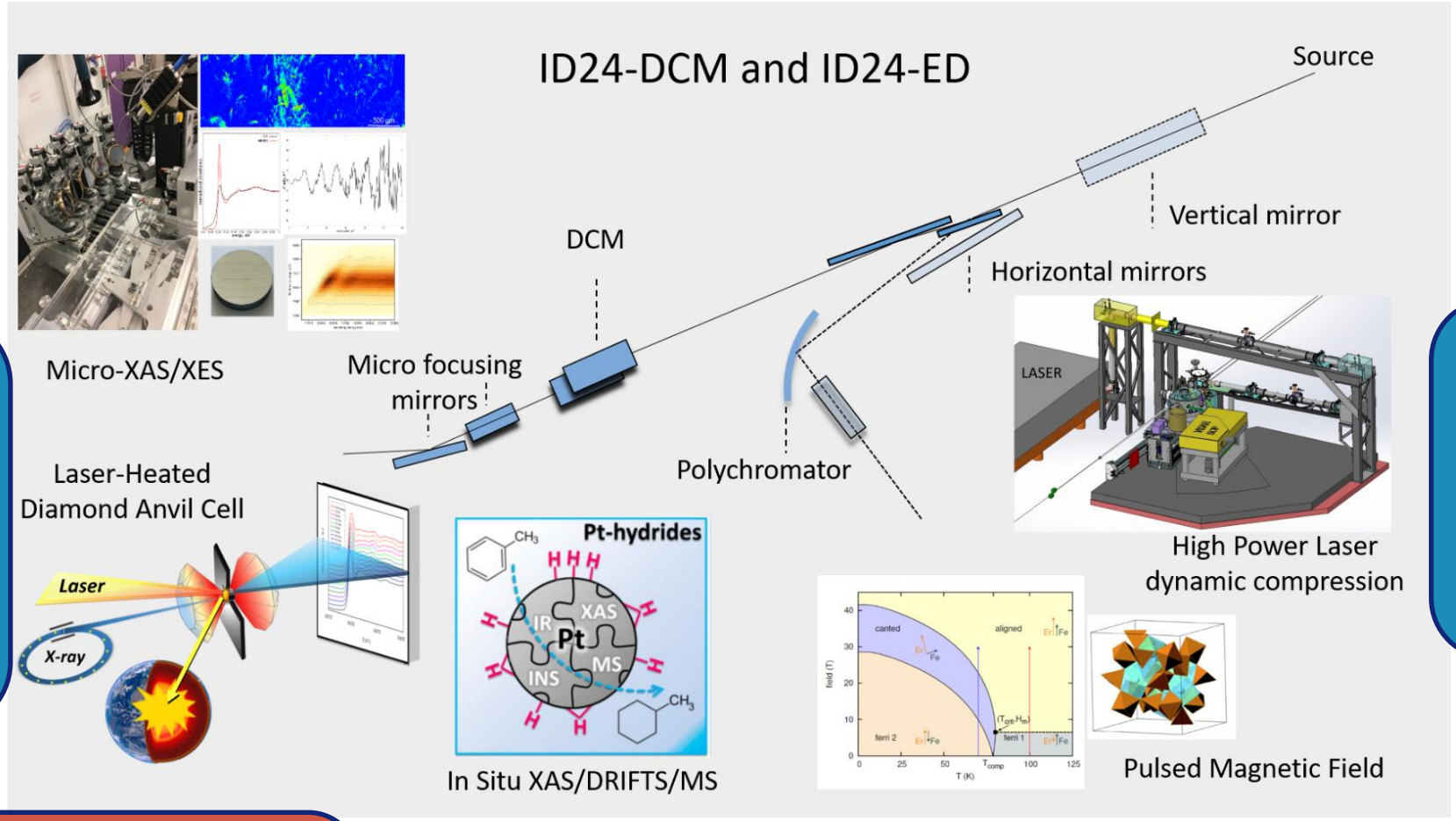
Pulsed Magnetic Field

ID24: HIGH BRILLIANCE X-RAY ABSORPTION SPECTROSCOPY BEAMLINE

ID24- DCM

- XAS, XES
- XRD, DRIFTS, MS, UV-Vis, …

- *5-40 keV*
- *focused and unfocused beam (0.5x0.5 μm^2 – 1x1 mm^2)*
- *up to 10^{13} ph./s*
- *1s/EXAFS*



ID24- ED

- XAS, XMCD

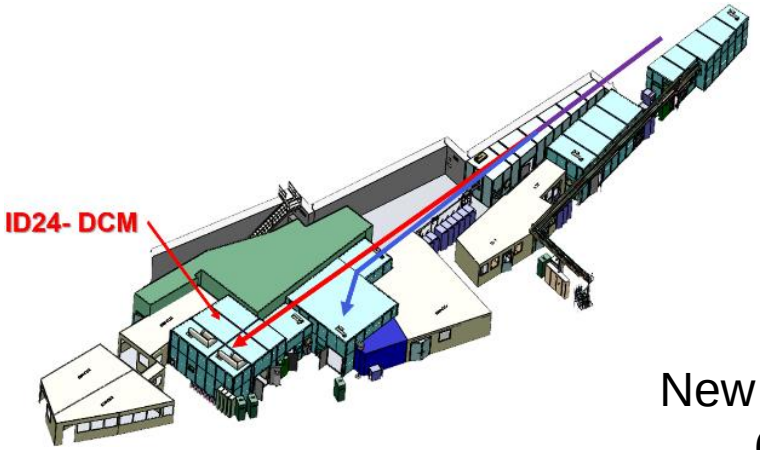
- *5-25 keV*
- *5x5 - 100x100 μm^2 E dep*
- *up to 10^{13} ph./s*
- *100 ps*
- *windowless*

- *LH-DAC setup for static compression*
- *MicroEXAFS/MicroXES setup*
- *5 crystal analyser*
- *Operando chemistry facilities*

two complementary beamlines

- *High Power Laser Facility*
- *Pulsed Magnetic Field (P,T)*
- *Stopped Flow Cell*

ID24-DCM



Source: 2xU27 + Rev U27/32

MH2, MH3 at 30.6 m

New ESRF-DCM
61.15 m

KB - 65.3m

Sample
65.8–66.3 m

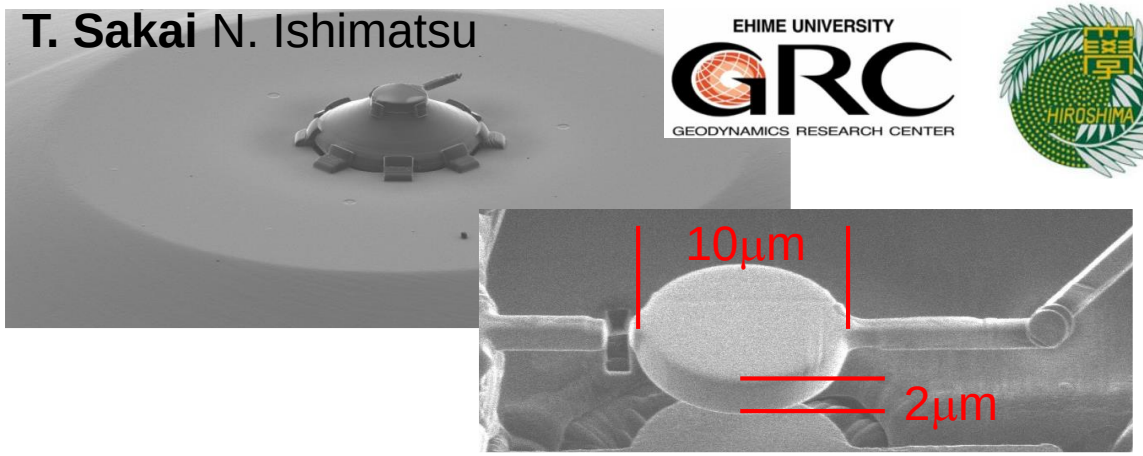
Open to users since February 2023

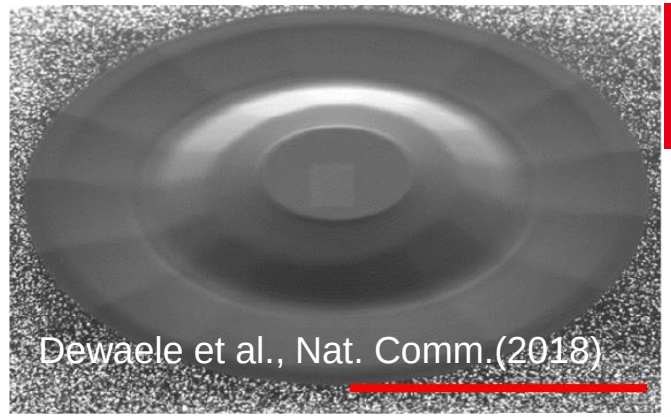
EBS allows designing a micro/nano XAS station with:

- Simple optical scheme (no secondary source)
- Small focal spot < 0.5x0.5 μm^2 FWHM
- Large area for sample and sample environment
- Short beamline
- High flux up to 10^{13} ph/s (Undulators), allowing:
  - Photon-hungry technics like μXES
  - Time resolved acquisitions (1 EXAFS/s)

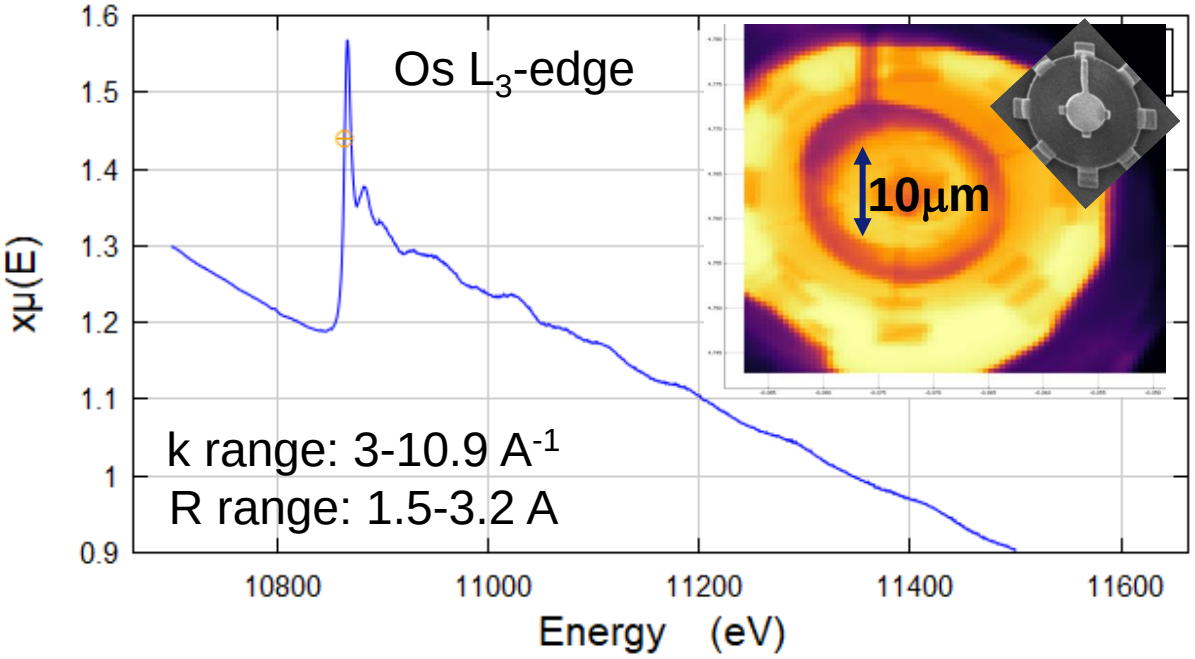
ID24-DCM – SUBMICRON BEAM FOR ULTRA HIGH STATIC PRESSURE

T. Sakai N. Ishimatsu

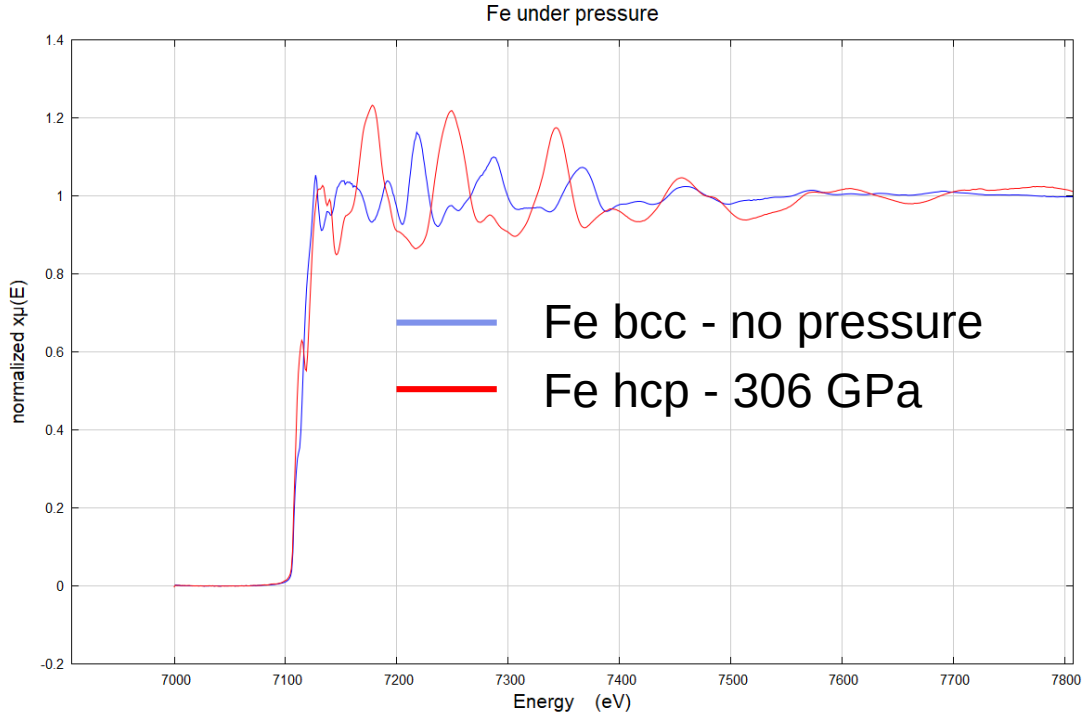




F. Occelli and **P. Loubeyre** T. Irifune



jump =0.18 , P=253 GPa

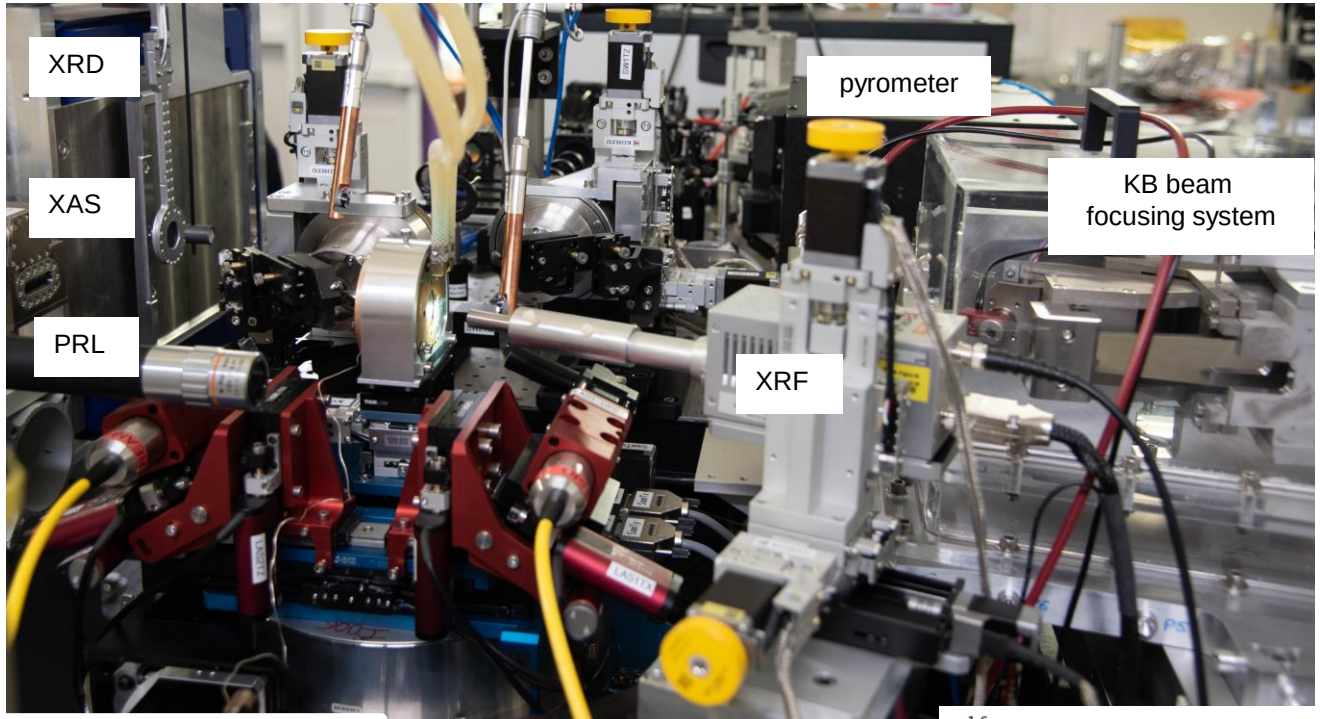


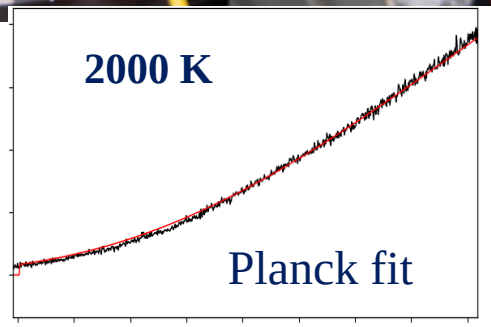
DCM SAMPLE ENVIRONMENTS: LH-DAC FOR EXTREME HP HT

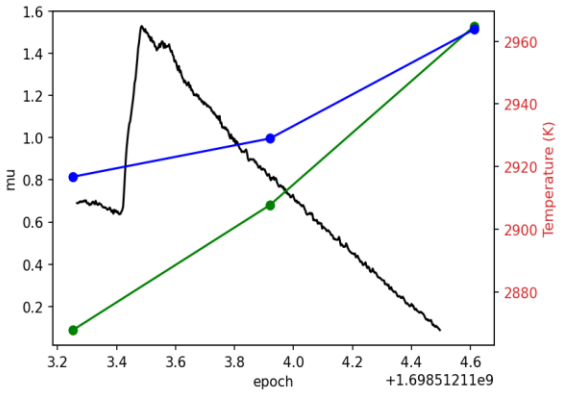
***P/T* range:** 0-2 Mbars, 6000 K **Time-resolution:** 1 sec

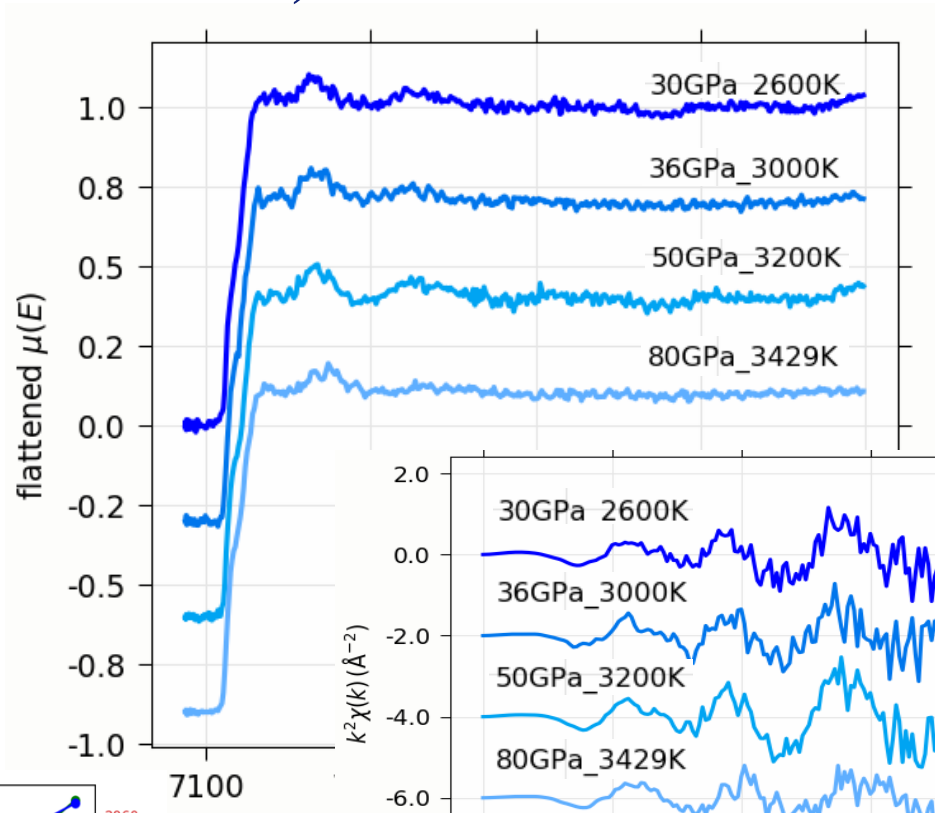
Multi detection: nano-XAS, XRF, XRD (XES foreseen in 2024)

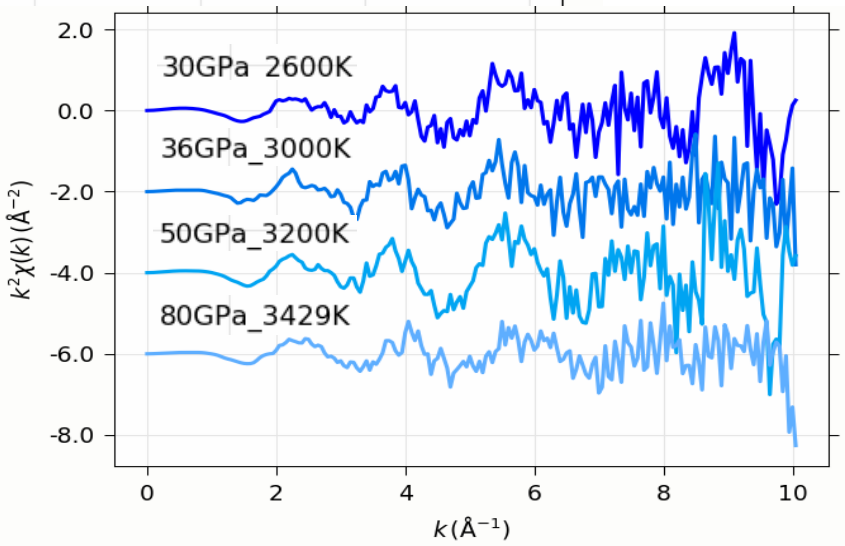
A. Rosa







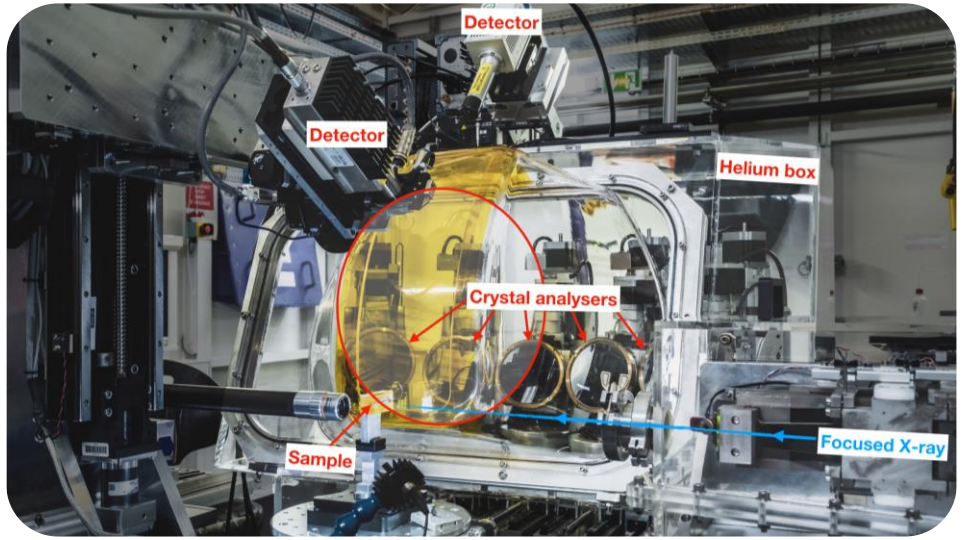




S.Balugani et al.

SAMPLE ENVIRONMENTS: 5-CRYSTAL ANALIZERS FOR ENVIROMENTAL SCIENCE

5 spherically bent crystals + fluorescence detector positioned in a **Rowland geometry**

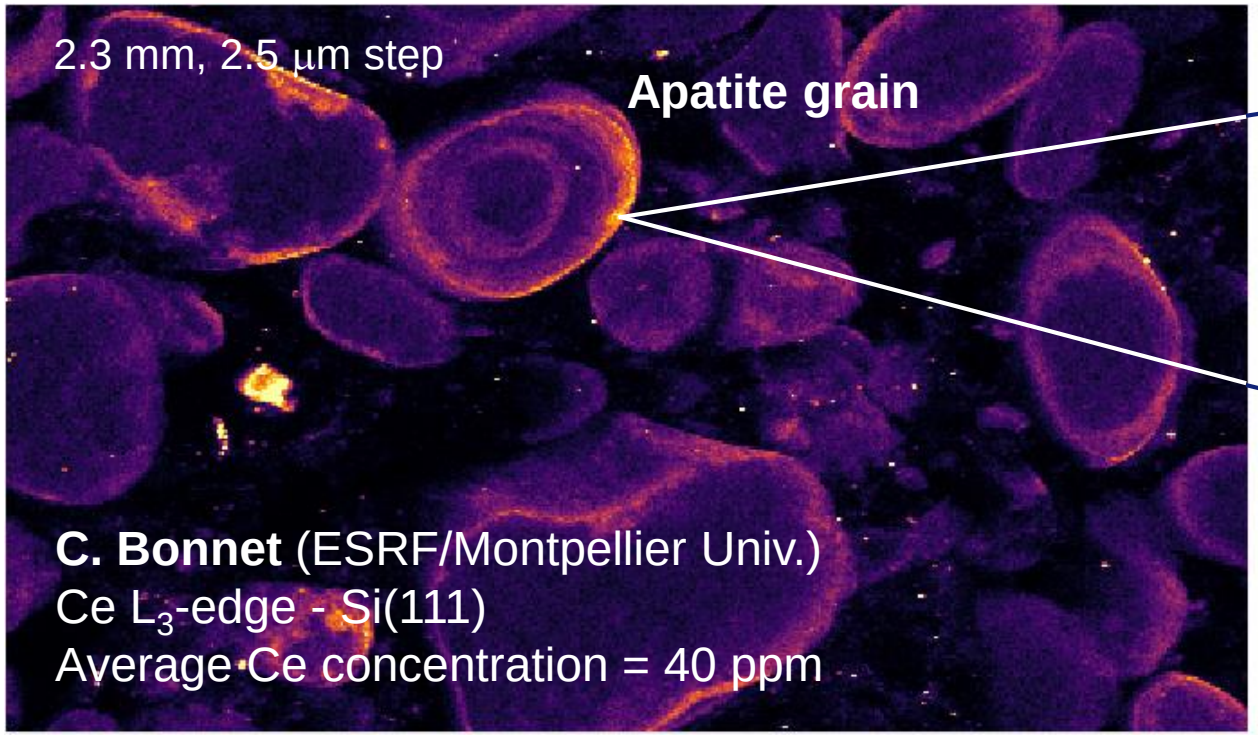


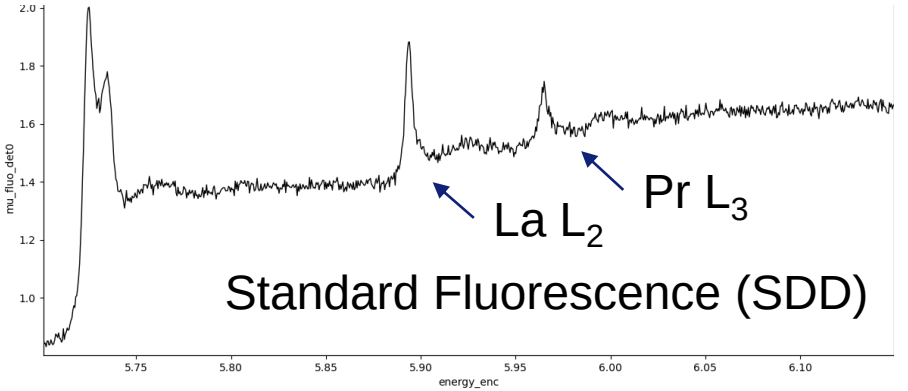
A **worldwide unique setup:**

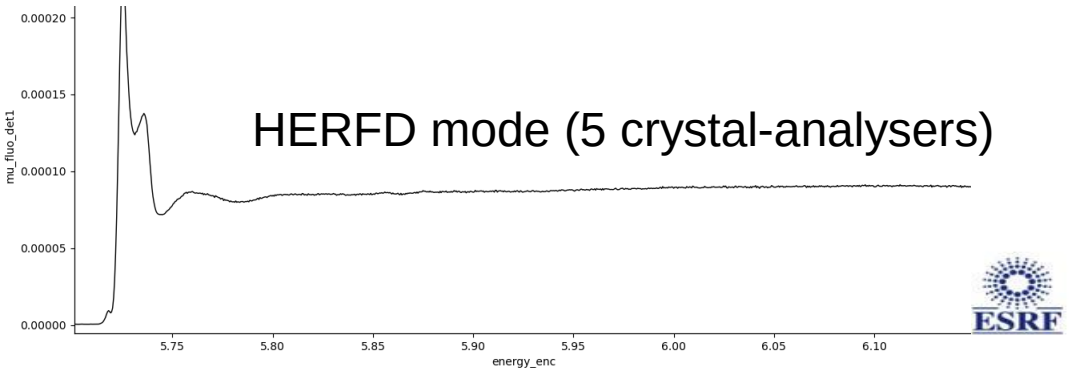
- Reveals hidden fluorescence lines, improved contrast between phases
- Improved S/N ratio for XAS, sensitivity to lower concentration

→ diluted elements in complex/natural matrixes

- E range **5 to 25 keV,** E resolution **0.5 – 5 eV**

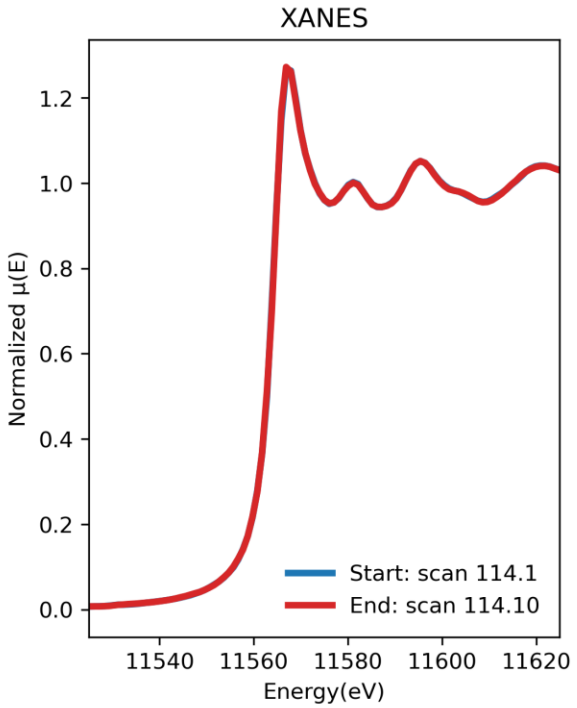


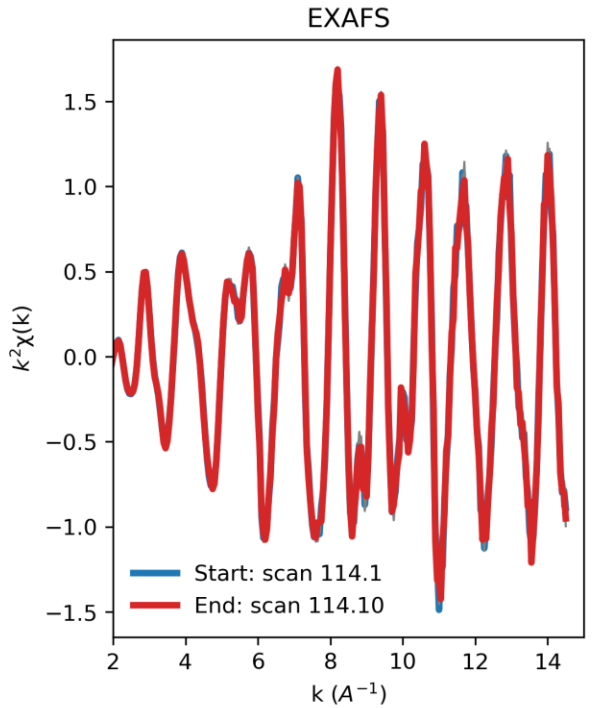


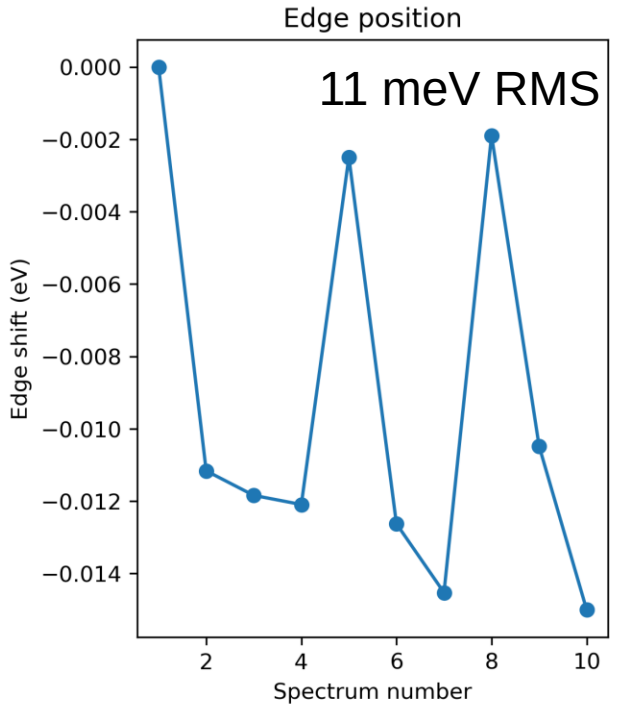


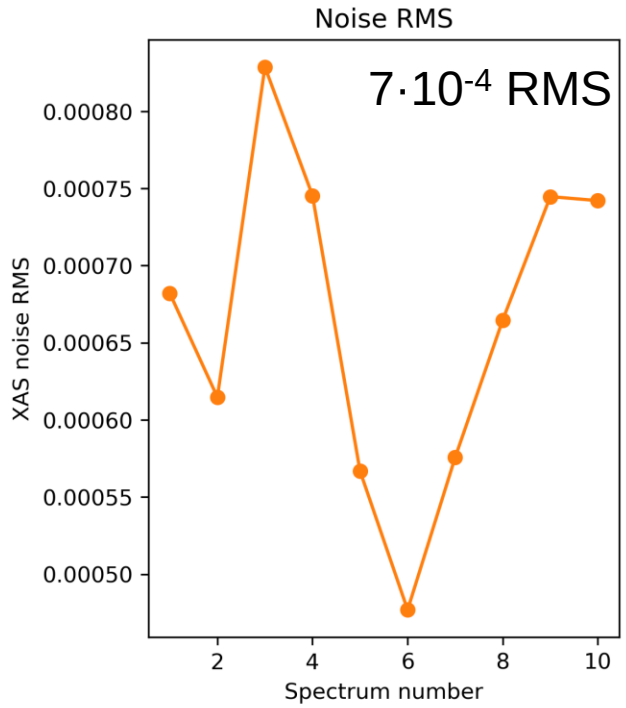
ID24-DCM - CHEMISTRY

Pt L_3-edge EXAFS until k = 15 A^{-1} (1000 pts, 2 ms/pt)
Integration time **2s per spectrum**
10 consecutive EXAFS scans in 57 seconds









Data quality is very good, **but**:

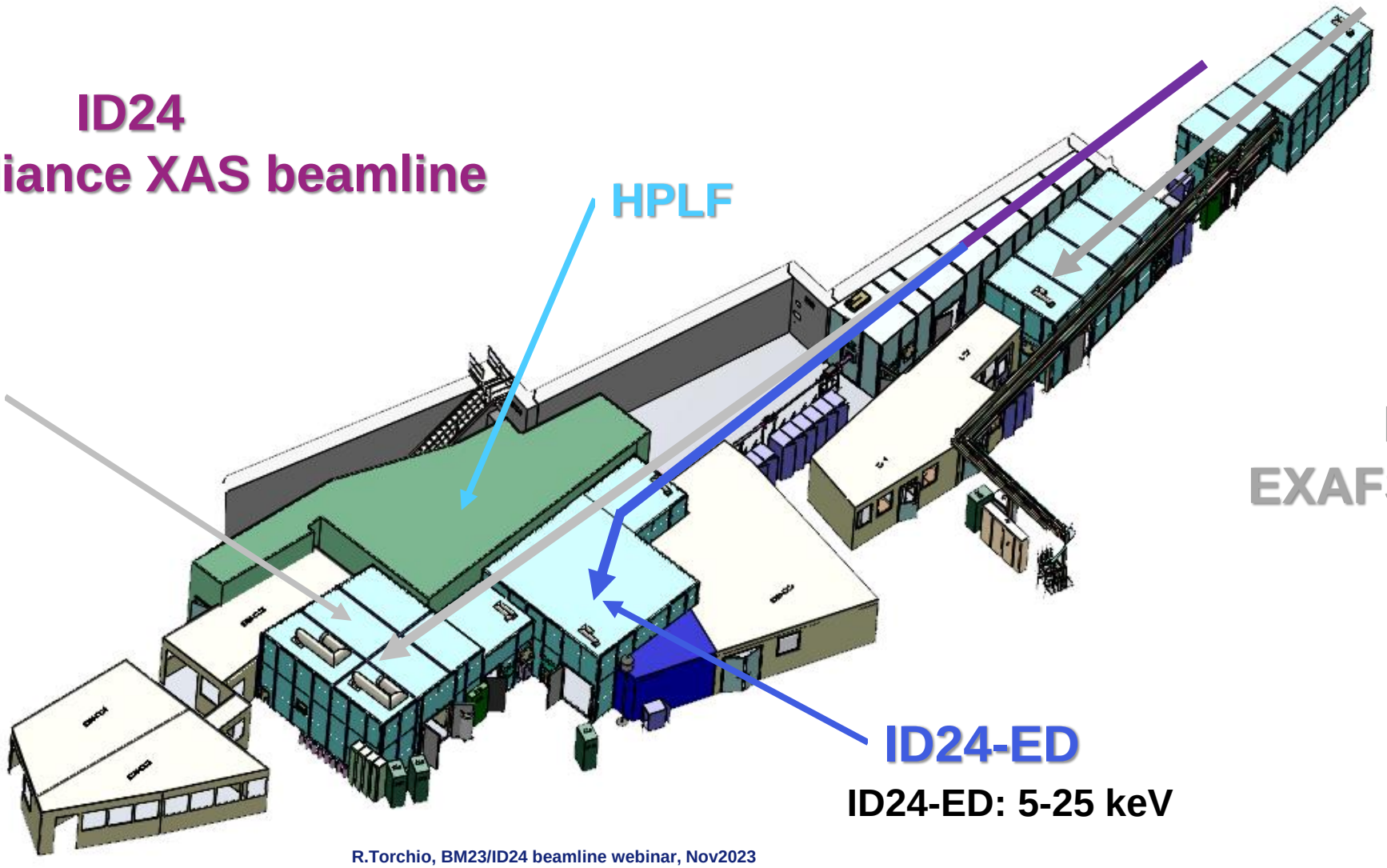
- Dead time between scans is 3.7 seconds. To be improved: significant contribution of software
- Synchronization with the undulator to be improved: critical for fast scans

	ID24 DCM	BM23
µXAS	5-40 keV (µXES 4 - 25 keV)	
	up to 20 $Å^{-1}$, $\Delta E/E = 2.10^{-4}$, $N/S = 5.10^{-5}$	
Smallest spot size	0.5*0.5 μm^2	3*3 μm^2
Flux ph/s	$8*10^{11} – 2*10^{13}$	$2*10^{9} – 2*10^{10}$
Time resolution	Down to 1s/EXAFS	
XRF / XES	With spatial resolution	

BM23-ID24

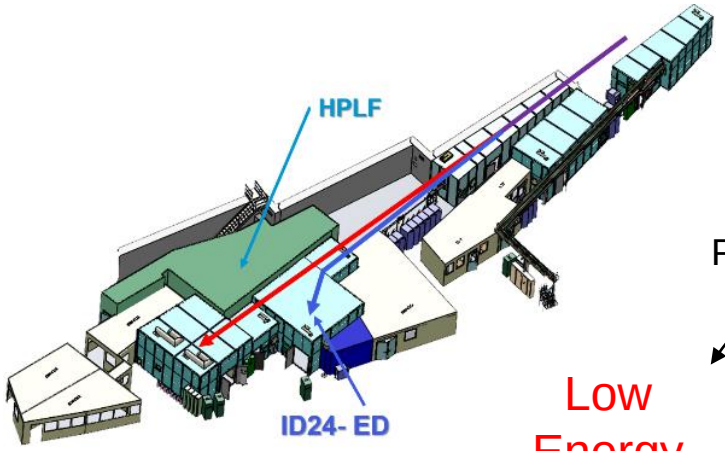
Two beamlines dedicated to X-ray Absorption Spectroscopy
For *in situ/operando* chemistry and matter under extreme conditions (P, T, H)

ID24
High Brilliance XAS beamline



ID24-ED: 5-25 keV

ID24-ED/HPLF



Dispersive setup:
Allows Ultrafast XAFS down to the single bunch time resolution (100ps)

High Energy

Low Energy

Polychromator

$\lambda = 2d \sin\theta$

Sample

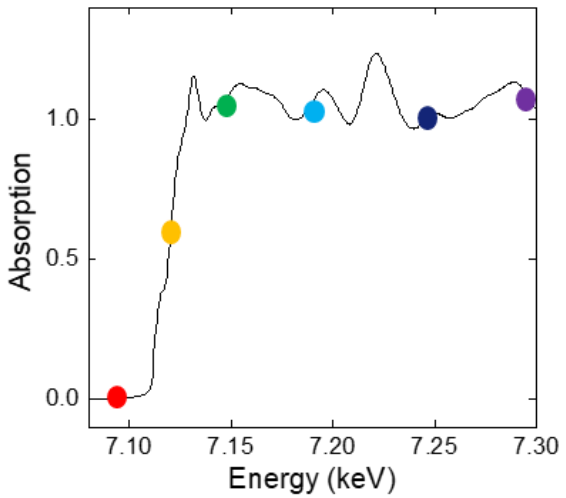
Pink X-rays from undulators

XH Ge detector
int time: 100 ns

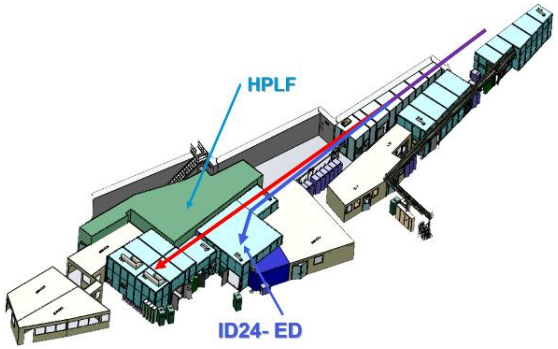
Science & Technology Facilities Council

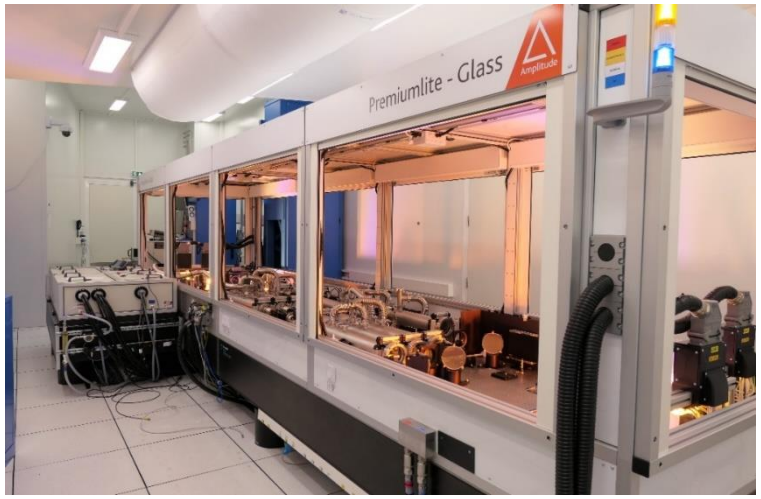
- 5-25 keV
- Beam size: 5^2 – 100^2 μm^2
- E range: 150-600 eV
- up to 10^{13} ph/s
- $\Delta E/E = 10^{-4}$

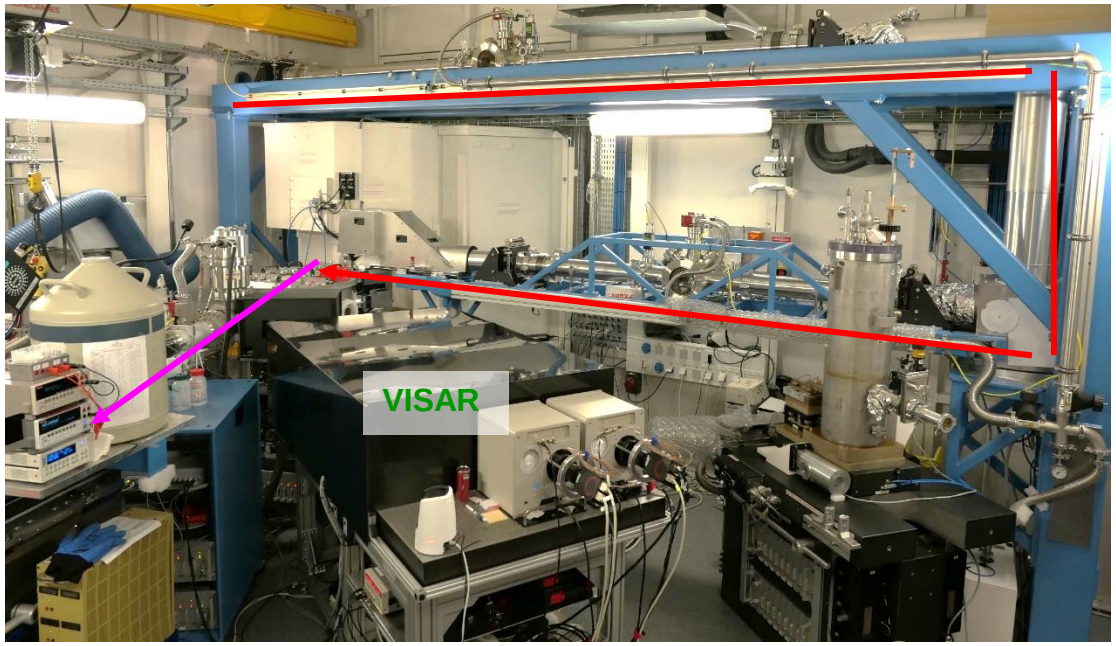
E dep

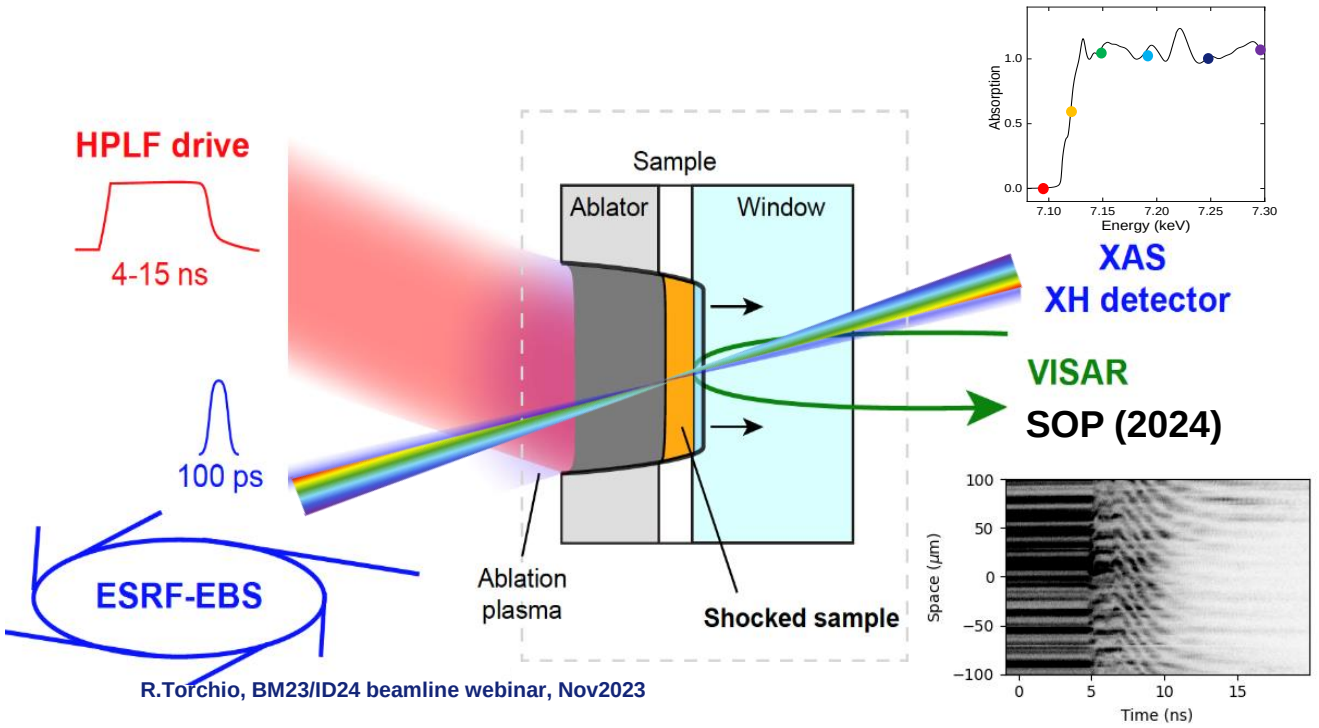


THE HIGH POWER LASER FACILITY FOR DYNAMIC COMPRESSION









Pump-probe exp. with 1 pump and 2 probe beams ns time scale, single bunch XAS

photo of the shock event @ HPLF

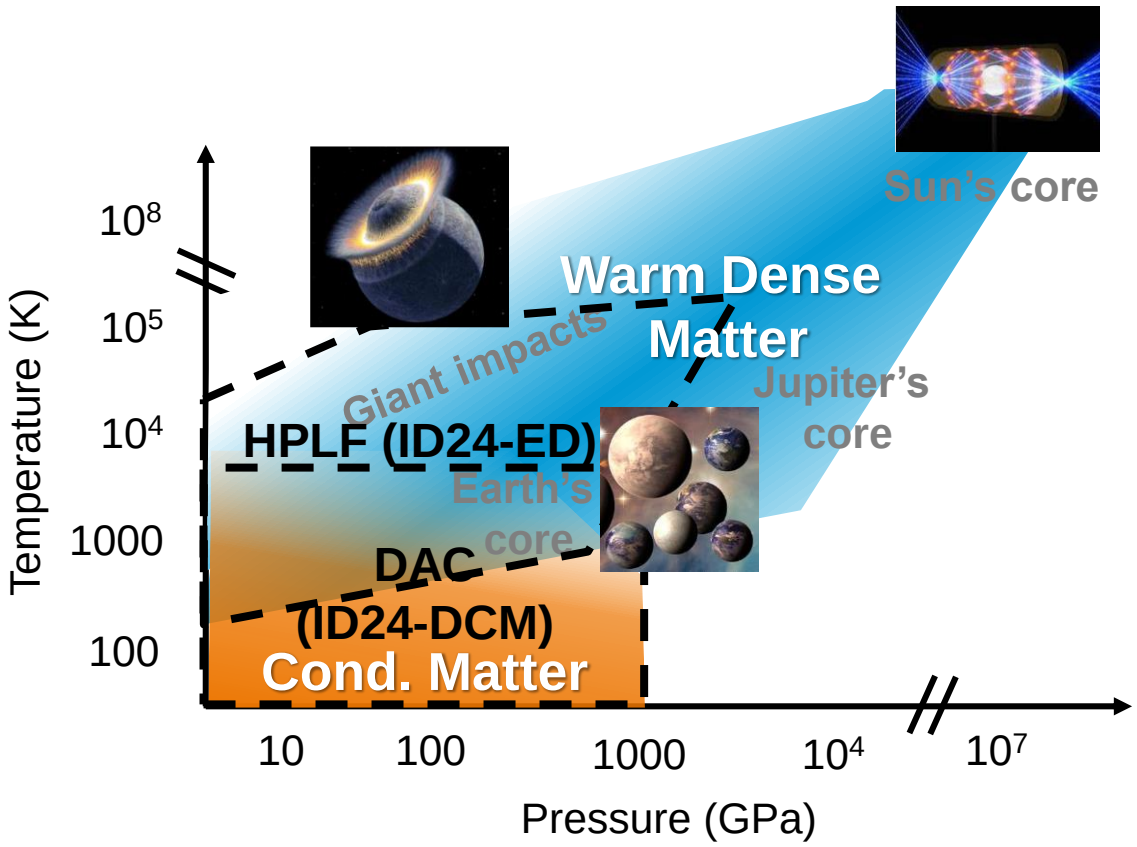
WHY DYNAMIC COMPRESSION

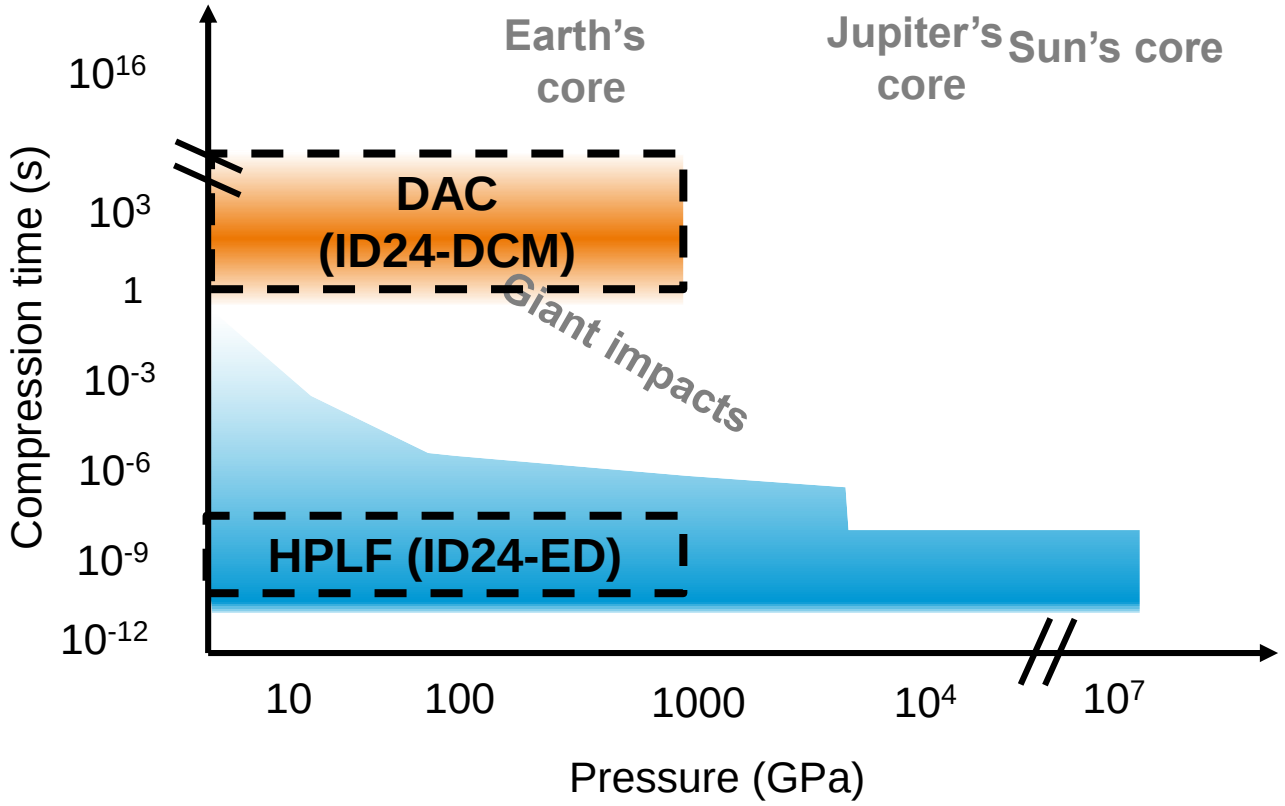
P/T range

Static

Dynamic

Timescale



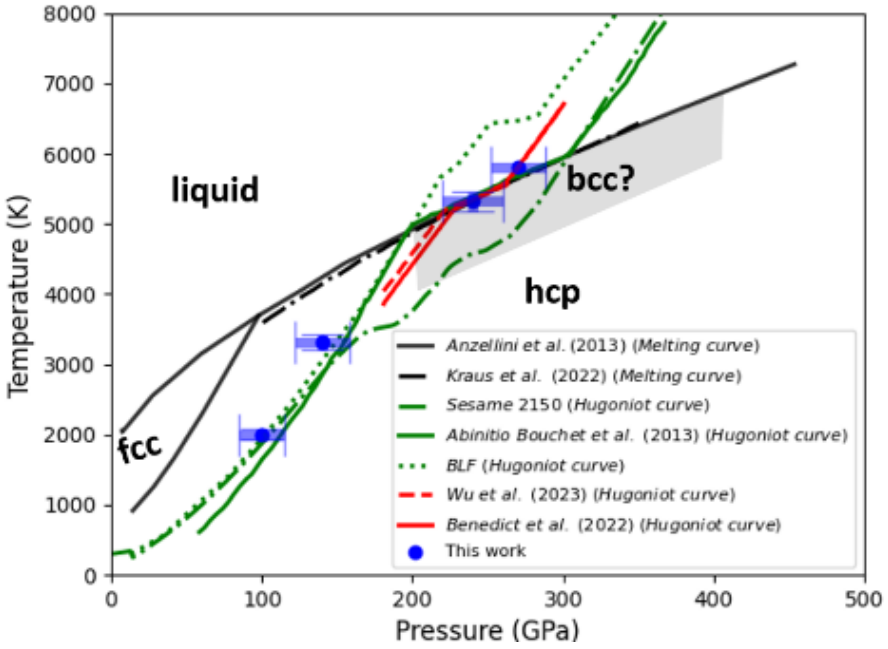


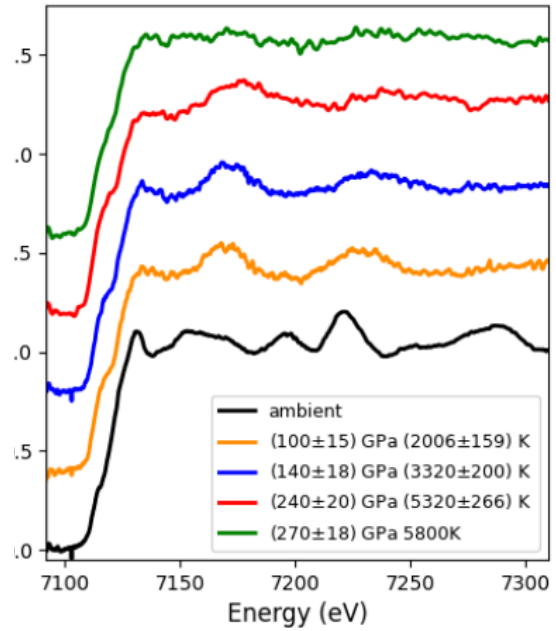
Earth and Planetary Science

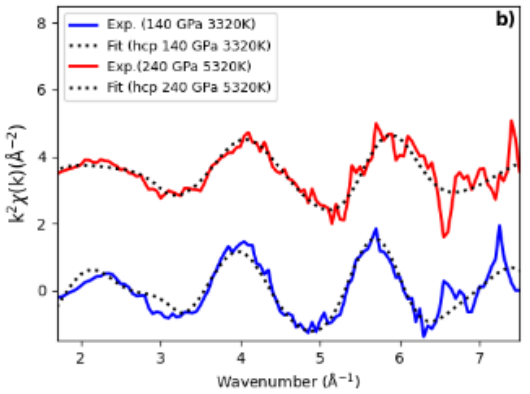
Fundamental Physics and Chemistry

Materials and energy science

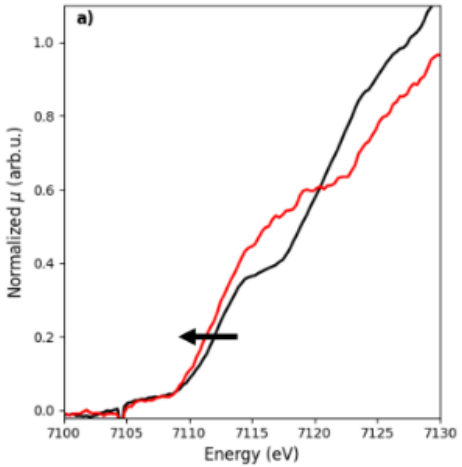
UNVEILING WARM DENSE MATTER OF 3DMETALS BY XAS



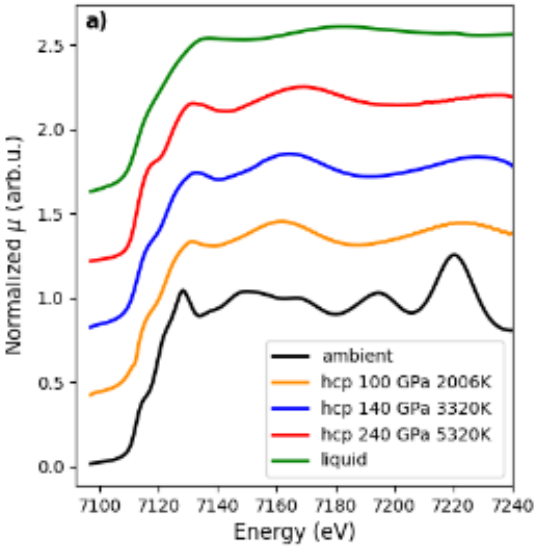


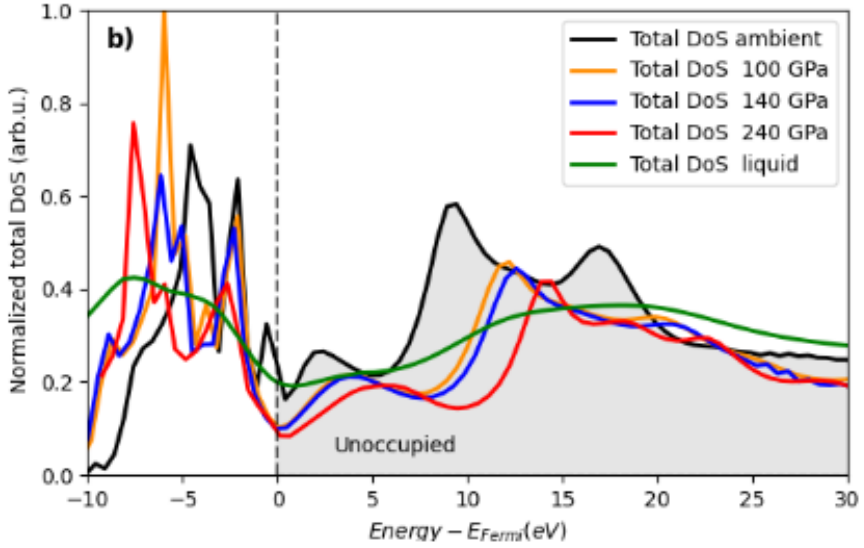


S. Balugani et al.



- Shock melting of Fe around 240 GPa
- exclusion of bcc phase
- derivation of r and T
- Electronic structure

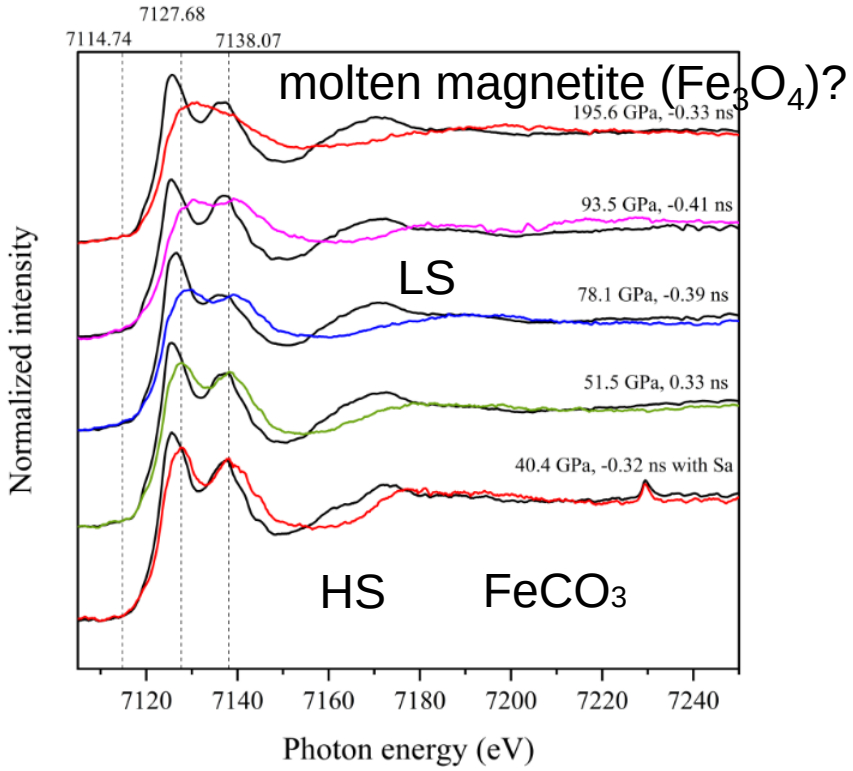




GEO AND PLANETARY SCIENCE / MATERIALS SCIENCE

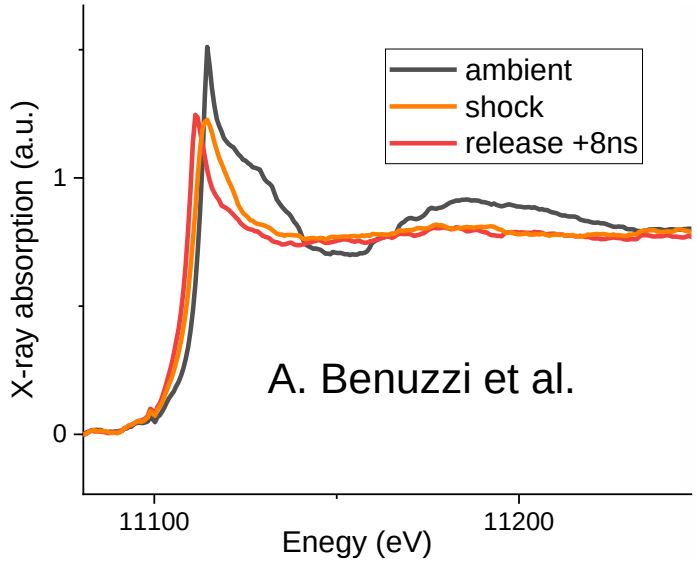
Shock formation of Magnetite from Siderite

A. Dwiviedi,
T. De resseguier et al.

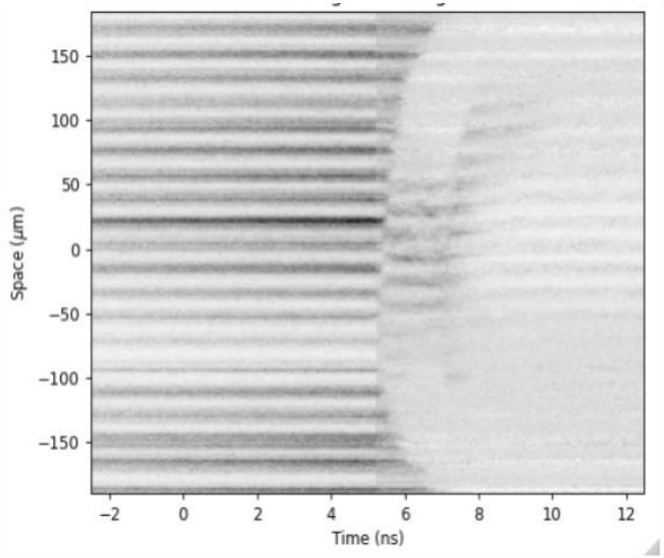


R.Torchio, BM23/ID24 beamline webinar, Nov2023

Glassy GeO2 up to 250 GPa



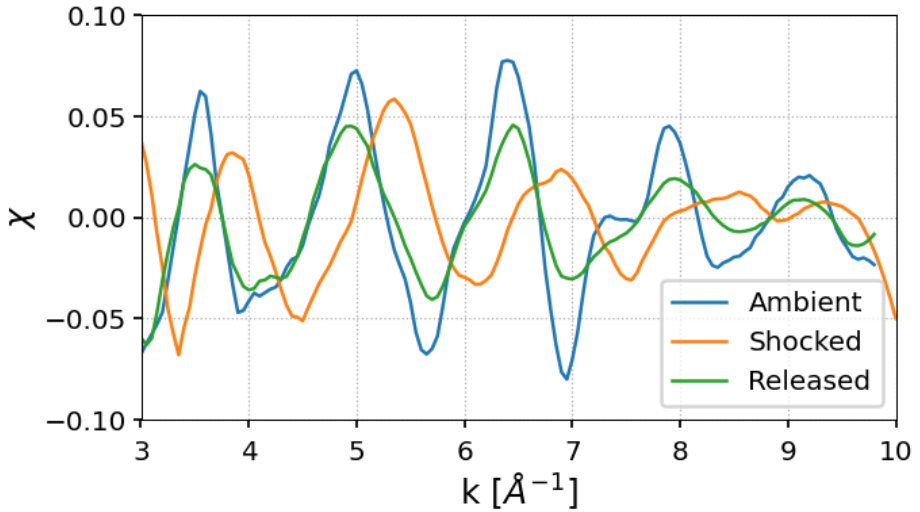
A. Benuzzi et al.



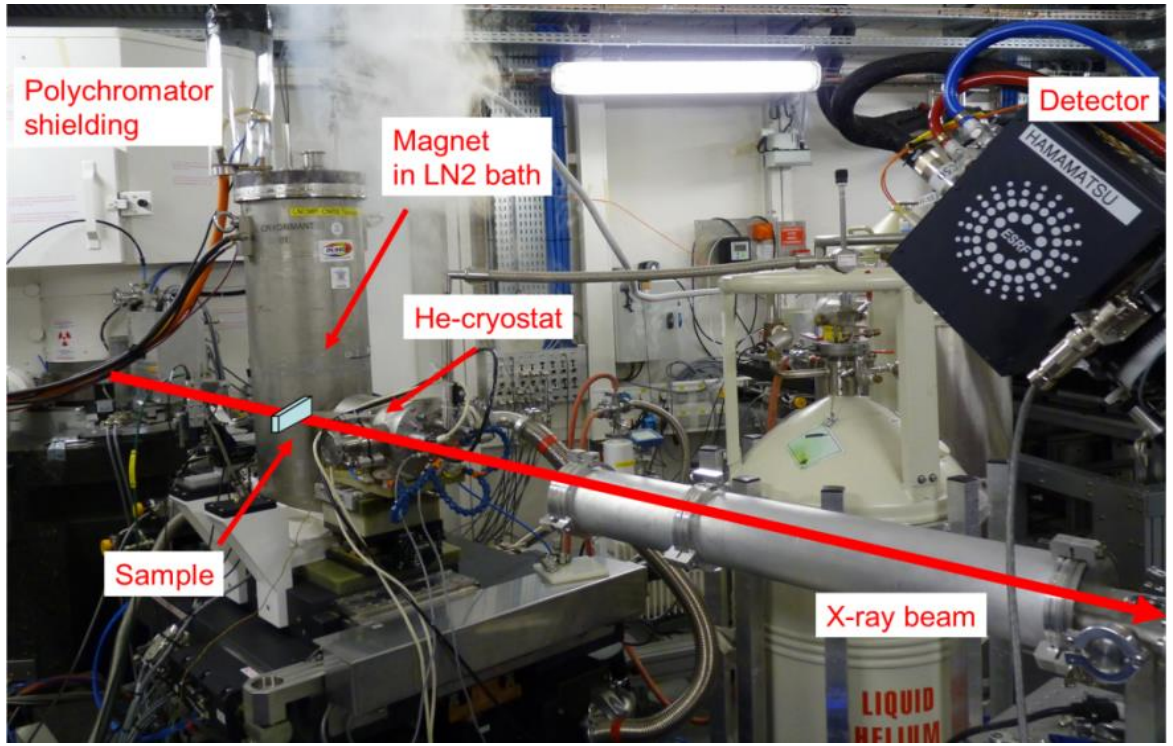
Laser-Shocked nanopourous Cu

A. Krygier et al.

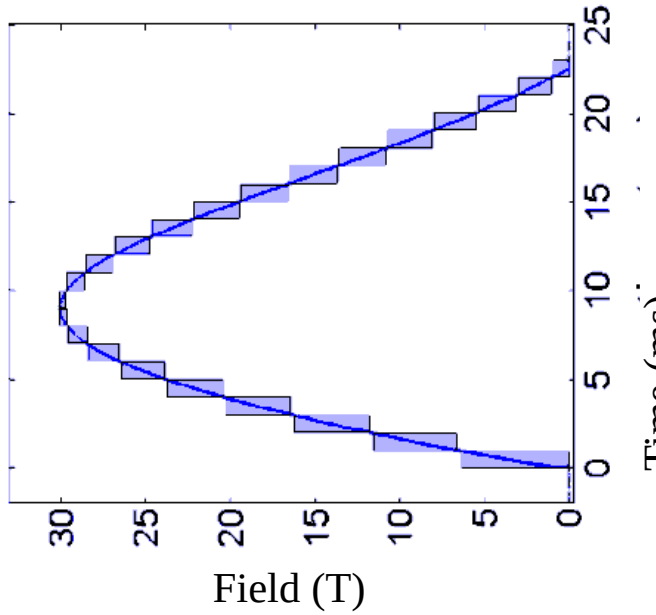
Lawrence Livermore National Laboratory



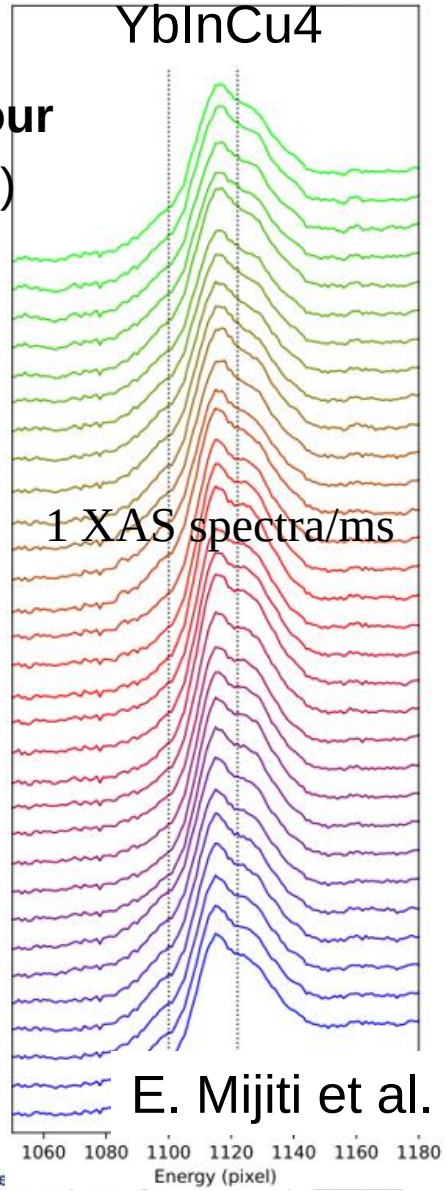
PULSED MAGNETIC FIELD



- Maximum field **30 T (50T)**
- Field Pulse duration (total) **23 ms**
- Repetition rate at B_{max} **10 pulses/hour**
- X-rays detection **ms-µs** (film mode)
- Low temperature **2K**
- High pressure (few GPa)



Yb L_3-edge YbInCu4



E. Mijiti et al.

Laboratoire National des Champs Magnétiques Intenses
(F. Duc, Toulouse, France)

First Order Valence transition in YbInCu4 under multi-extreme conditions of field (0-30 T), pressure (0-3.0 GPa) and temperature (2-300 K)

SYNERGY: BM23 + ID24-DCM + ID24-ED

multipurpose **specific**

	BM23	ID24-DCM	ID24-ED
Timescale	1 s -mins	1 s	<100 ms (down to 100 ps)
Beam size	3 µm to 3 mm	0.5 µm to 1 mm	4 µm to 100 µm
Flux	up to 10^{10}	up to 10^{13}	up to 10^{13}
Target applications	- Concentrated samples, relatively slow processes - Photon-sensitive samples and processes - Multipurpose, industrial experiments	- Photon-hungry techniques - Fast processes - Multi-edge, multi-technique, multi-dimensional experiments - extreme conditions (P/T), natural (very diluted) samples (5-crystals), chemistry	Specific, ultra- fast experiments: - Laser shocks - Pulsed magnetic field

Well-equipped to help in solving pressing scientific and societal challenges

CONTACTS

Olivier Mathon
Unit coordinator
BM23 beamline responsible

BM23
EXAFS beamline

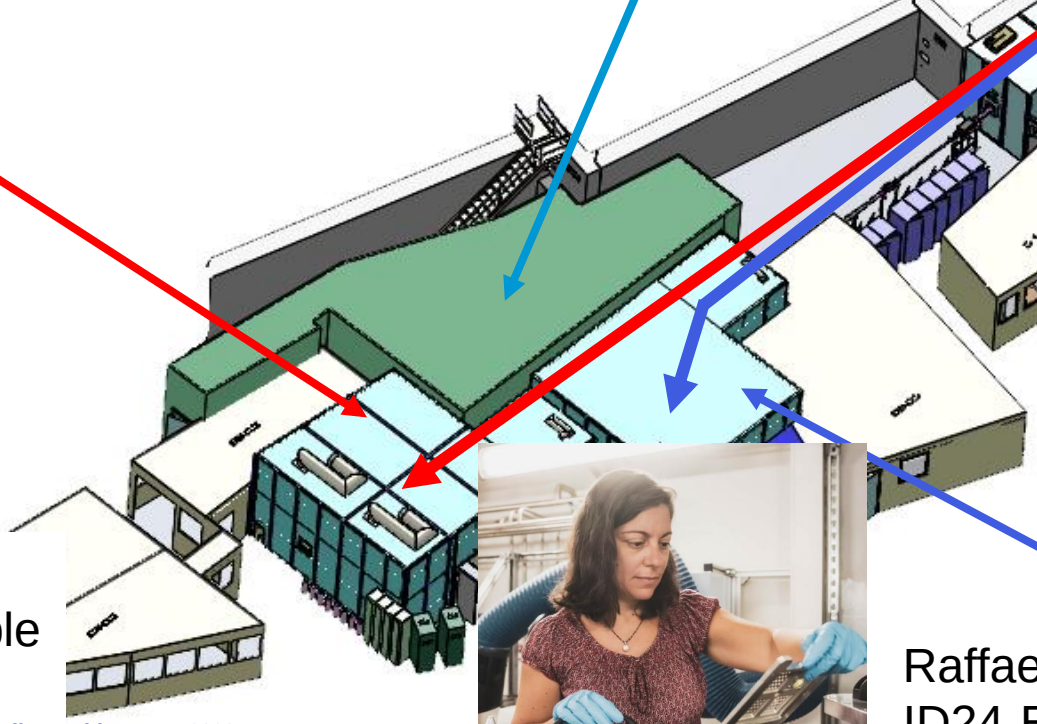
Angelika Rosa
ID24/BM23/ID15
Static High pressure

ID24
High Brilliance XAS beamline

HPLF

ID24-DCM

Kirill Lomachenko
ID24-DCM responsible
Chemistry



ID24-ED

Raffaella Torchio
ID24-ED responsible

Jean-Alexis Hernandez
Dynamic compression

ACKNOWLEDMENTS

A. Manceau	scientist ERC DEEP-SEE
E. Mijiti	Post-Doc High Pressure
A. Molokova	Post-Doc Chemistry
D. Salusso	Post-Doc Chemistry
S. Balugani	PhD Student InnovaXN HPLF
C. Bonnet	PhD Student
N. Sévelin-Radiguet	HPLF laser engineer
F. Perrin	technician electronics and control
S. Pasternak	technician mechanics
D. Bugnazet	technician mechanics

ISDD support

C. Clavel	engineer mechanics
F. Villar	engineer mechanics HPLF
A. Moyne	engineer mechanics
G. Berruyer	engineer software
S. Chazalette	technician electronics

THANKS FOR YOUR ATTENTION