
ESRF
The European Synchrotron

Frontiers of High Pressure Research at the European Synchrotron Radiation Facility

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OUTLINE

- Static High Pressure Research: status and trends
- Dynamic Compression: recent developments, future plans
- The EBS
- Extreme Conditions Science at EBS

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High Pressure at ESRF today

ID06, ID15B, ID27: X-ray Diffraction – Structure, Crystallography, Strain, Deformation, …

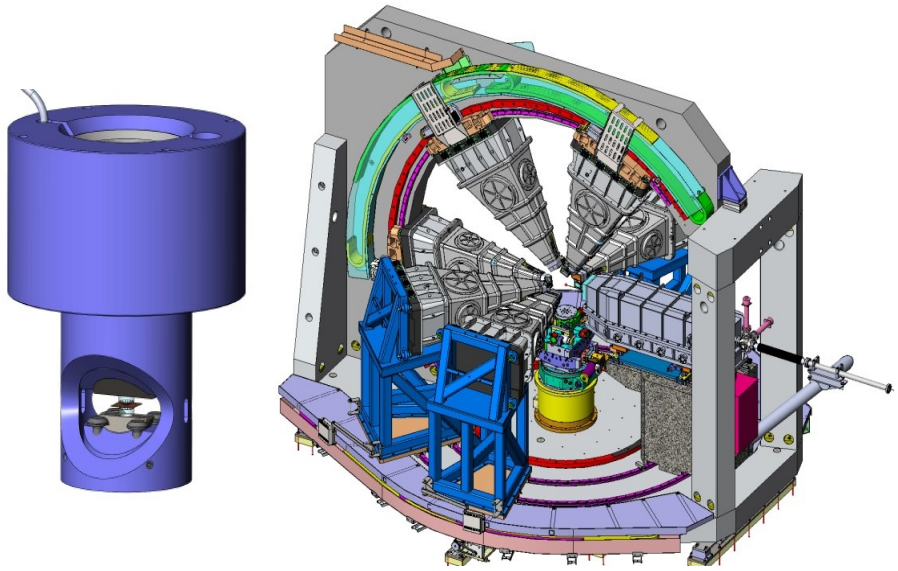
ID18: Nuclear Resonance Scattering - Magnetism, Phonons

ID20: Resonant Inelastic X-ray Scattering - Electronic and Magnetic Structure

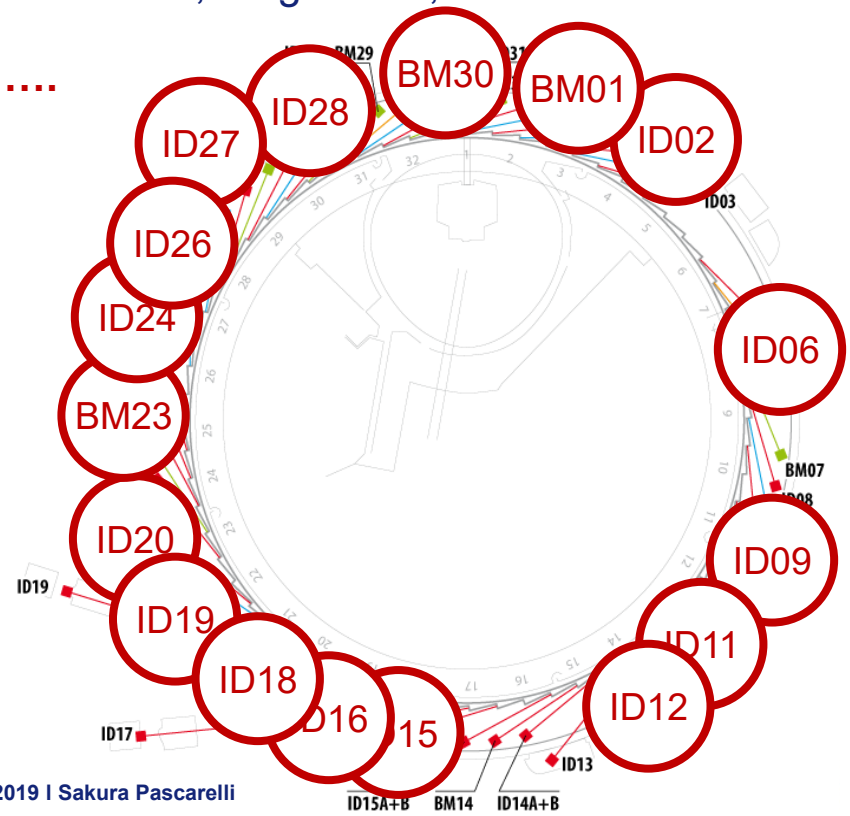
ID28: Inelastic X-ray Scattering, Diffuse Scattering – Phonons

ID12, BM23, ID24: XAS, XMCD - Local and electronic structure, Magnetism,…

ID02, ID26, ID11, ID16B, BM01, BM30, ID09B, ID19, ….

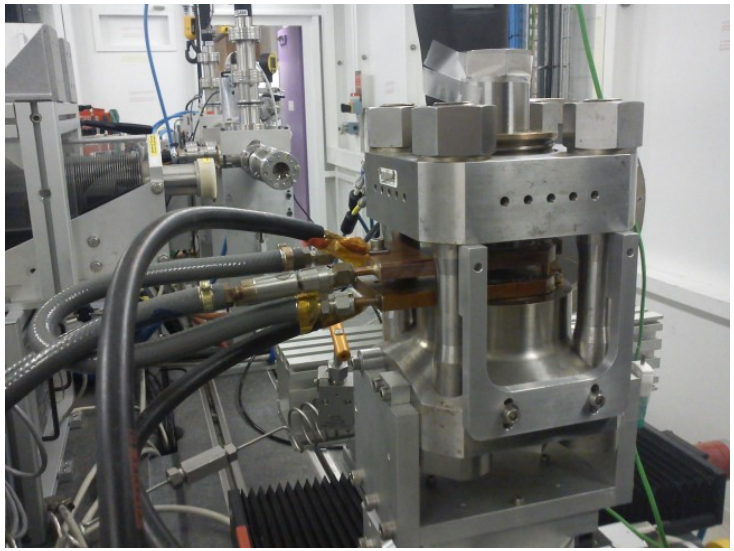


ID20: 72 Analysers and Panoramic DAC



Static High Pressure Facilities

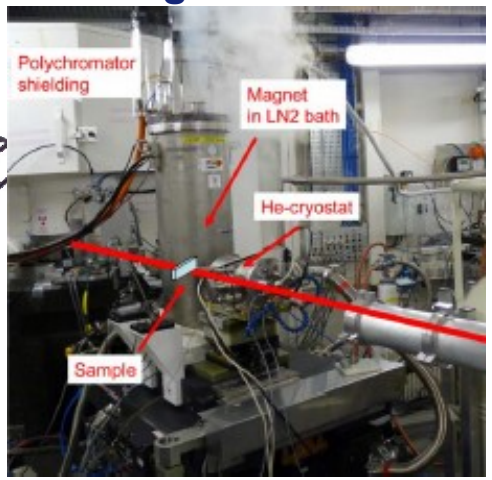
- Diamond Anvil Cell - P < 300 GPa (1 TPa)
- Paris-Edinburgh Press (ID27 and BM23) – 2 mm^3 – P < 17 GPa, T < 1800 K
- Large Volume multi-anvil Press (ID06) – 50 mm^3 – P < 20 GPa, T < 2500 K



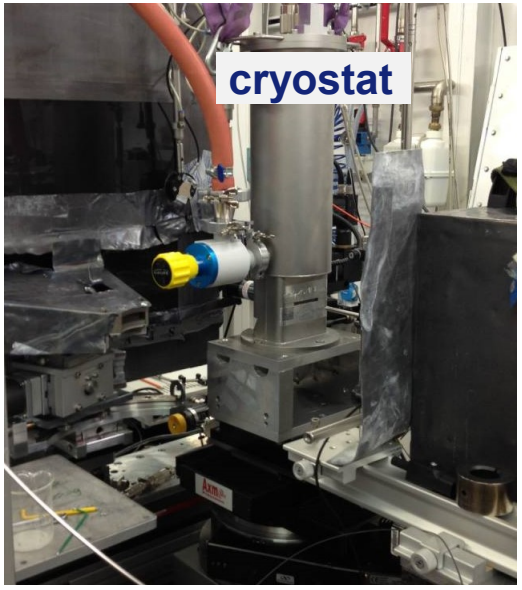
Static High Pressure Facilities

Static field - 8 T

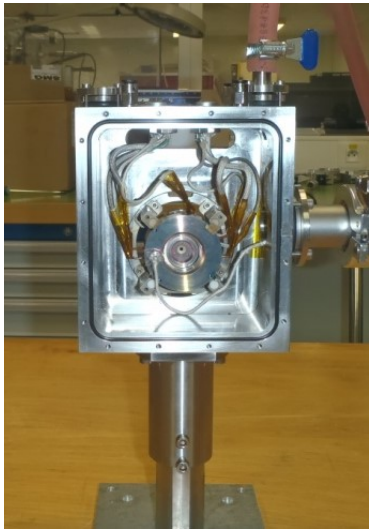
Pulsed Magnetic field - 30 T



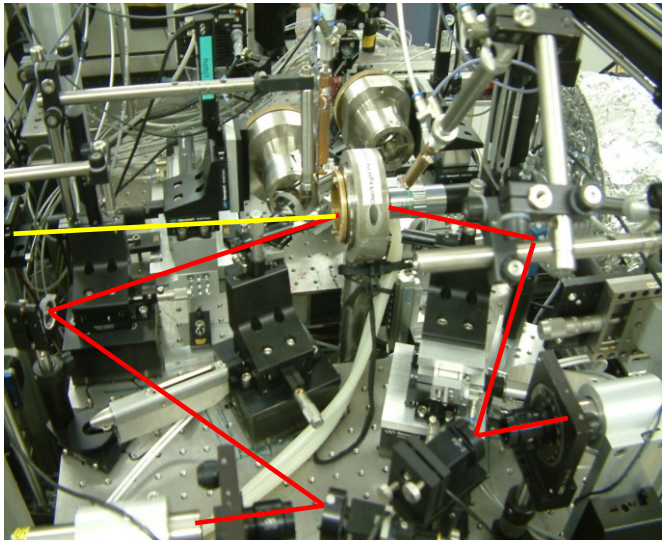
Low T - 2 K



Resistive heating - 1300 K

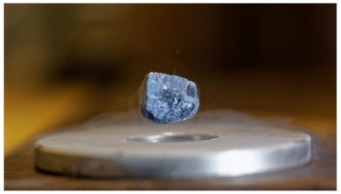


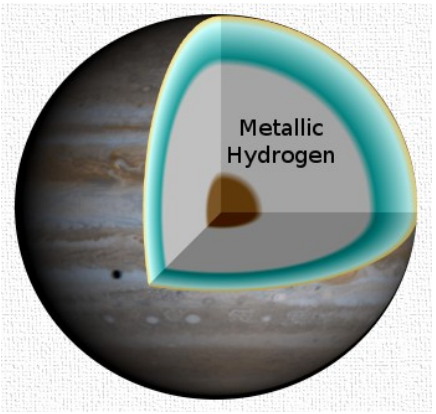
Laser heating - 5000 K



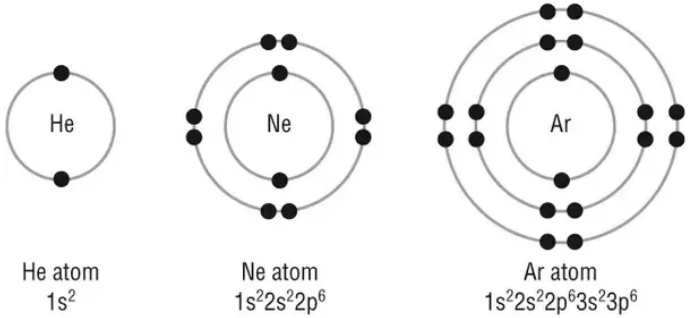
SOME OUTSTANDING QUESTIONS IN HIGH PRESSURE RESEARCH

1. The quest for metallic solid Hydrogen

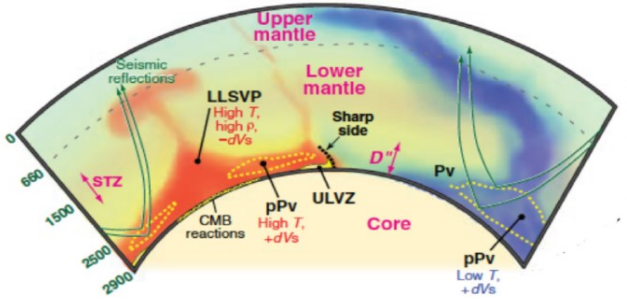




2. Pressure-induced reactivity of rare gases



3. Superplumes at the Core-Mantle Boundary



NRS OBSERVES APPEARANCE OF SUPERCONDUCTIVITY IN H_2S AT HP

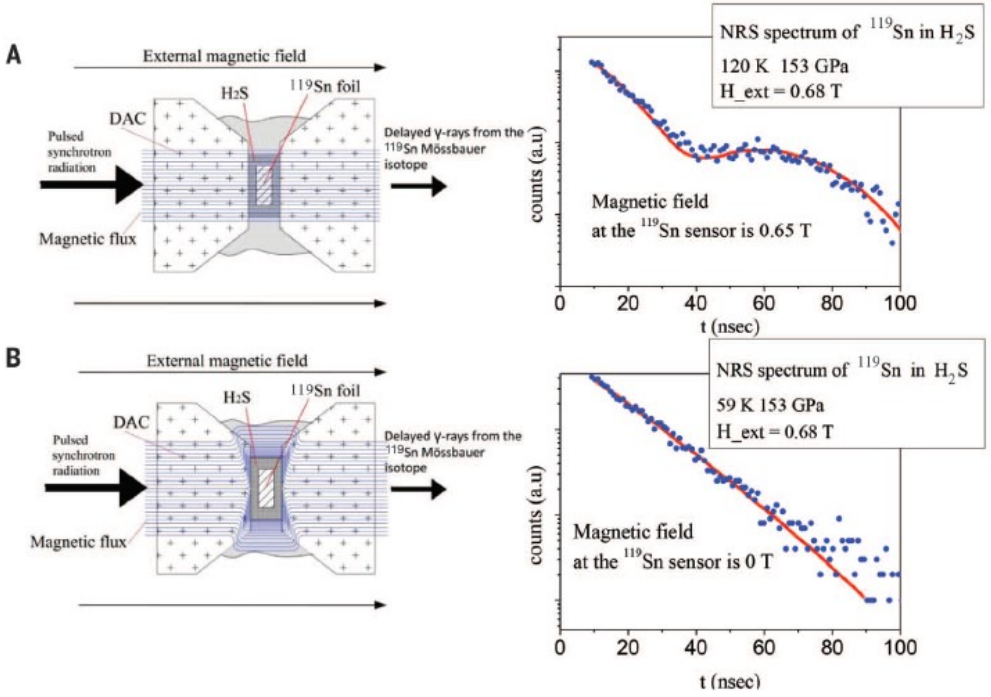
ID18

REPORTS

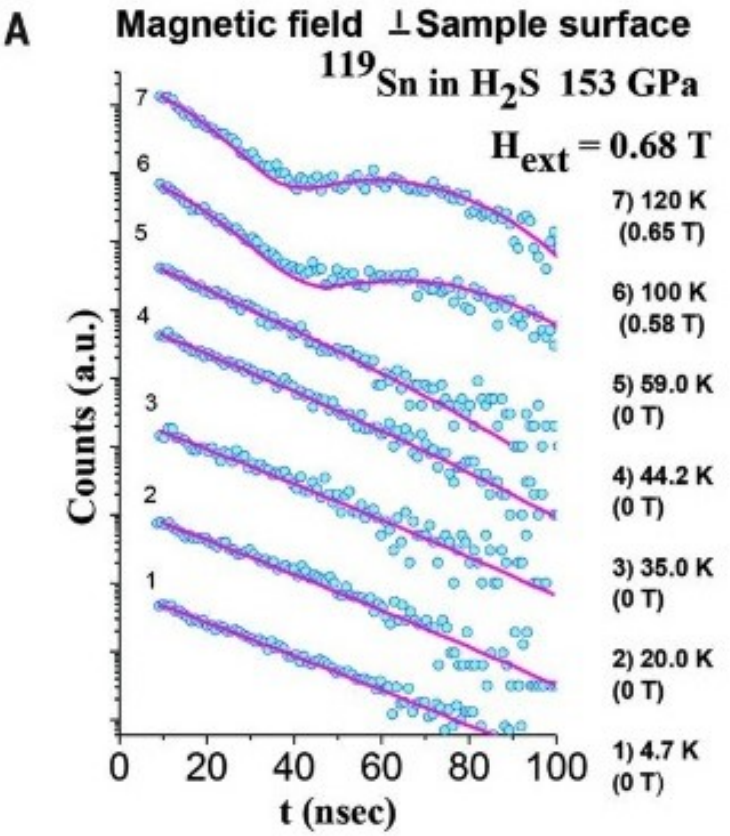
SUPERCONDUCTIVITY

Observation of superconductivity in hydrogen sulfide from nuclear resonant scattering

Ivan Troyan,[1,2*†] Alexander Gavriliuk,[2,3†] Rudolf Rüffer,[4] Alexander Chumakov,[4,5] Anna Mironovich,[2] Igor Lyubutin,[2] Dmitry Perekalin,[6] Alexander P. Drozdov,[1] Mikhail I. Eremets[1]



Direct observation of Meissner effect in H_2S compressed to 153 GPa



Troyan Science 2016

Expulsion of magnetic field in H_2S by monitoring NRS from ^{119}Sn sensor

FeH_5 : AN ANALOGUE TO ATOMIC H

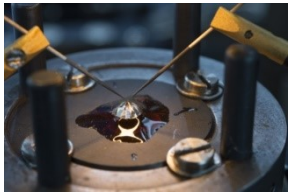
ID27

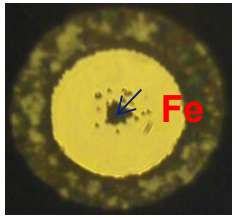
RESEARCH

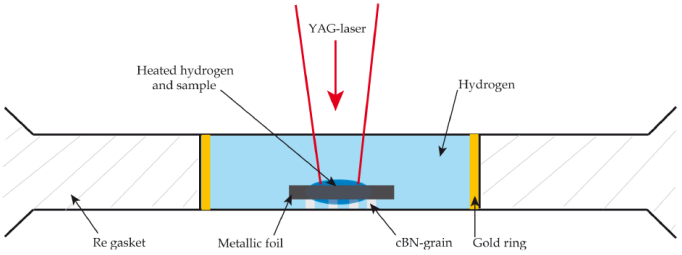
HIGH-PRESSURE PHYSICS

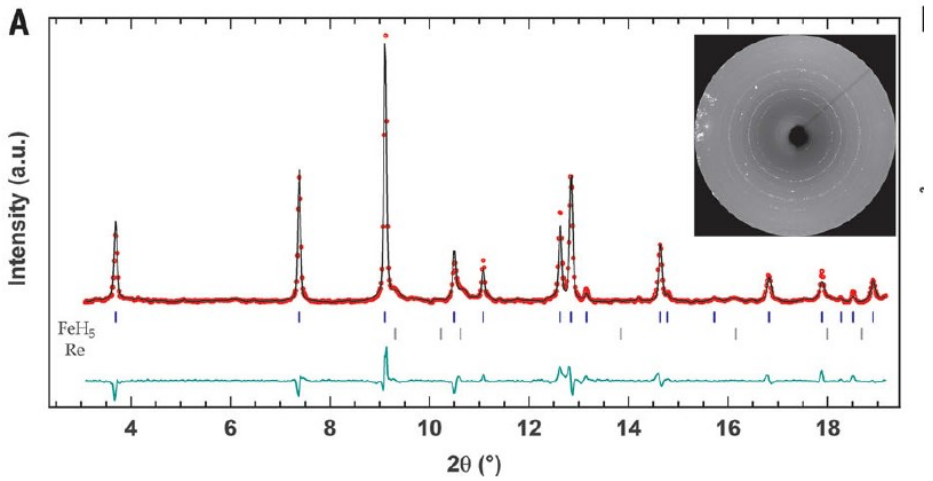
Synthesis of FeH_5: A layered structure with atomic hydrogen slabs

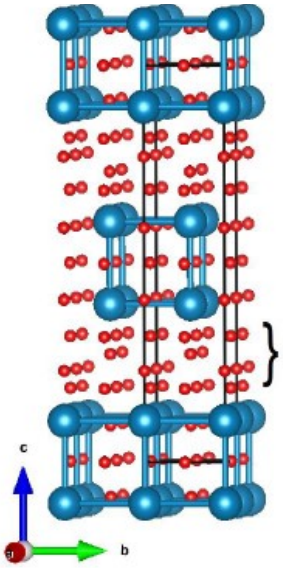
C. M. Pépin,[1,2]* G. Geneste,[1] A. Dewaele,[1] M. Mezouar,[3] P. Loubeyre[1]*



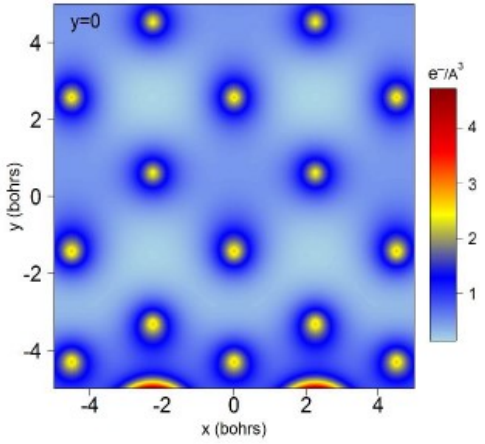








Atomic hydrogen slabs



Pépin Science 2017

- Planes of atomic H
- Potential high Tc superconductor

CHEMISTRY OF XENON AT MEGABAR PRESSURE

BM23

ID27

nature chemistry ARTICLES
PUBLISHED ONLINE: 30 MAY 2016 | DOI: 10.1038/NCHEM.2528

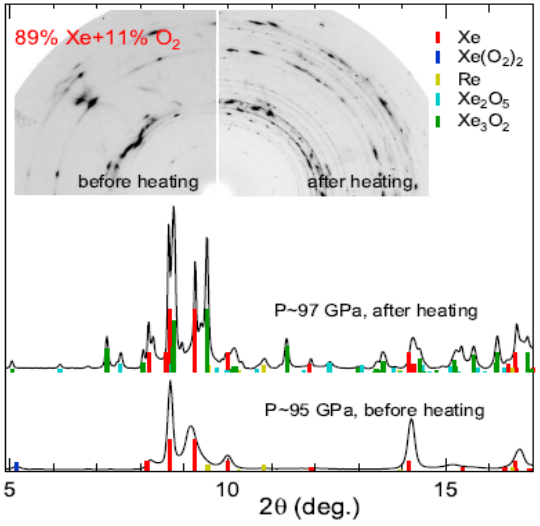
Synthesis and stability of xenon oxides Xe_2O_5 and Xe_3O_2 under pressure

Agnès Dewaele[1]*, Nicholas Worth[2], Chris J. Pickard[3,4,5], Richard J. Needs[2], Sakura Pascarelli[6], Olivier Mathon[6], Mohamed Mezouar[6] and Tetsuo Irifune[7,8]

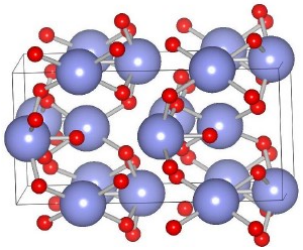
Xe [Kr] $4d^{10}5s^2\ 5p^6$

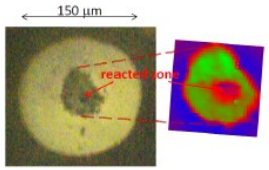
nature chemistry AR
PUBLISHED ONLINE: 11 NOVEMBER 2012 | DOI: 10.10

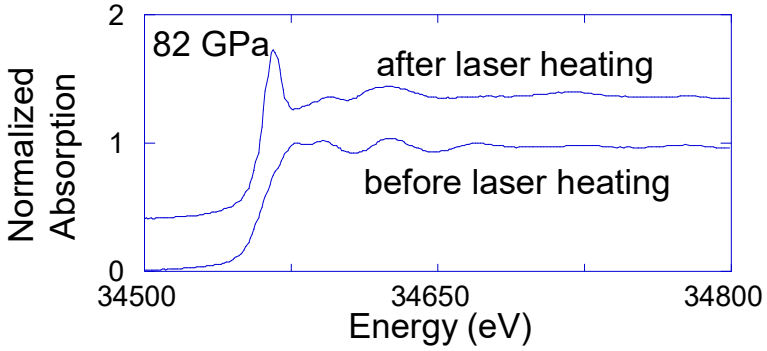
Stability of xenon oxides at high pressures

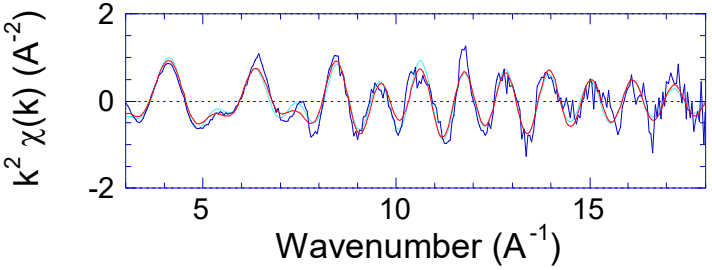


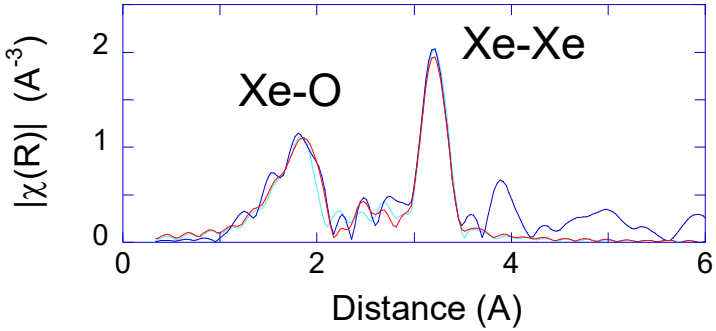
Xe_2O_5
P4/ncc space group











Dewaele Nature Chemistry 2016

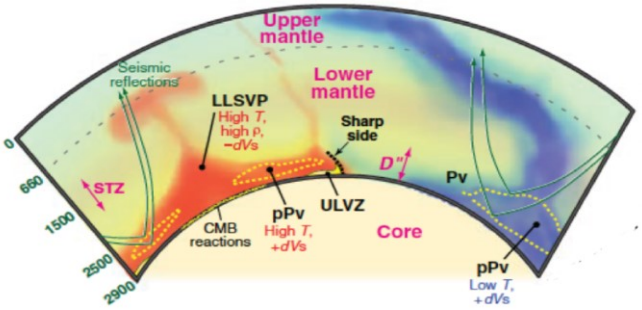
Structural changes in SiO_2 down to the Core Mantle boundary

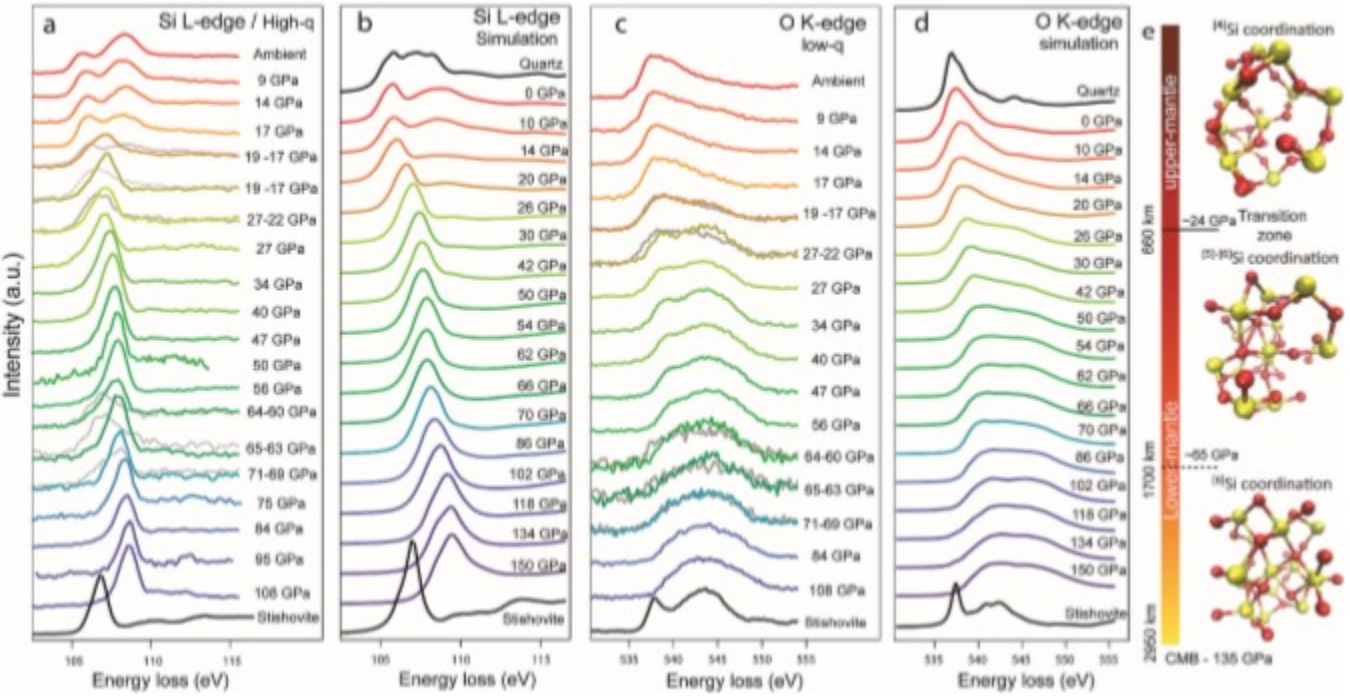
ID20

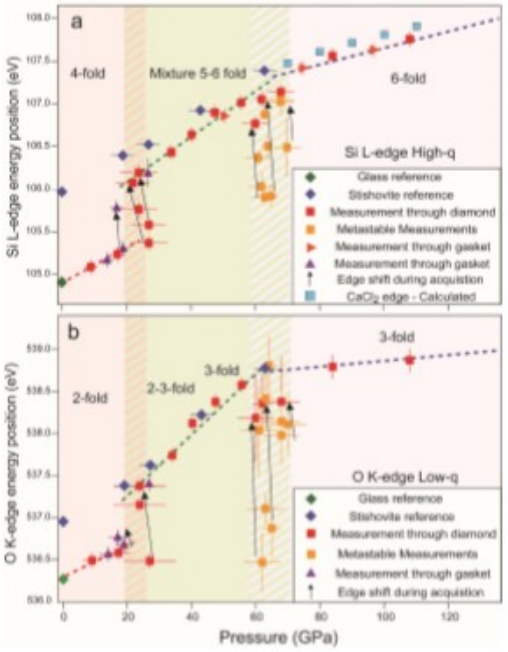
Geochemical Perspectives Letters

Magma properties at deep Earth's conditions from electronic structure of silica

S. Petitgirard[1*], C.J. Sahle[2], C. Weis[3], K. Gilmore[2], G. Spiekermann[4], J.S. Tse[5], M. Wilke[4], C. Cavallari[2], V. Cerantola[2], C. Sternemann[3]



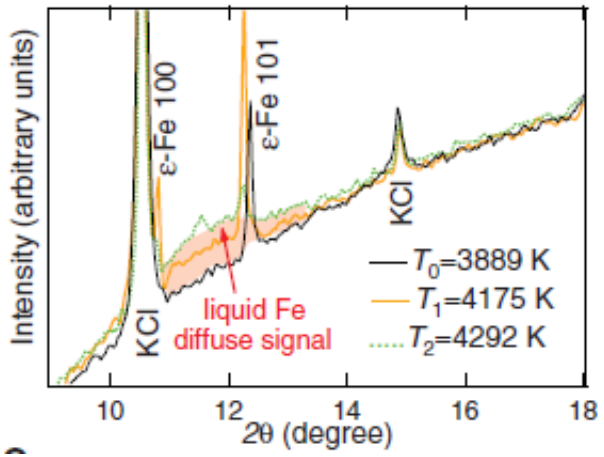




Petitgirard Geochemical Research Letters 2018

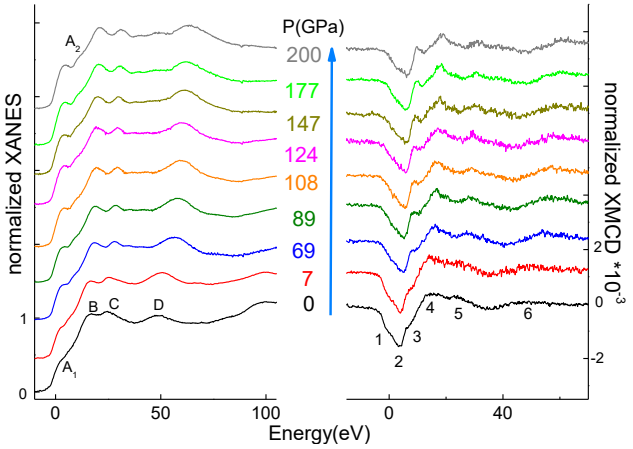
STATIC COMPRESSION AT SYNCHROTRONS TODAY

XRD 1.3 Mbar 4300K



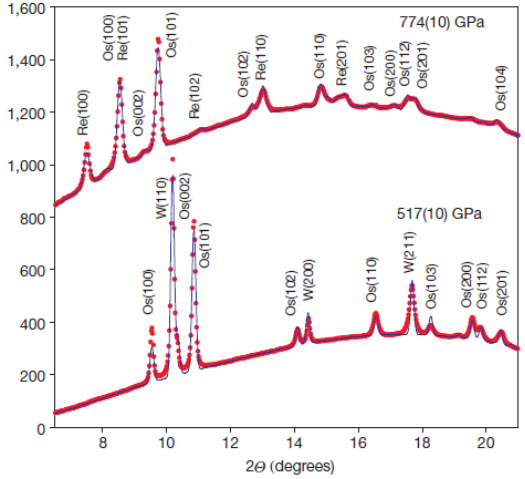
Anzellini Science 2013

XMCD 2 Mbar



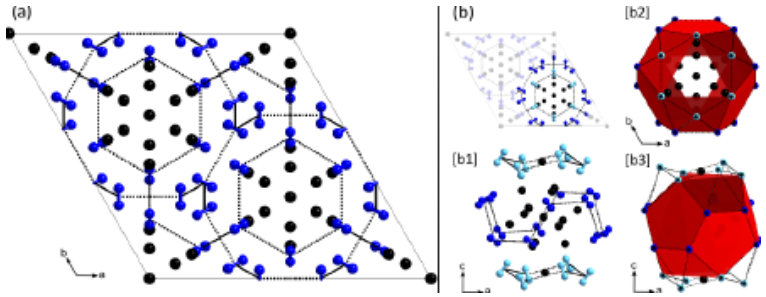
Torchio PRL 2011

XRD 7 Mbar



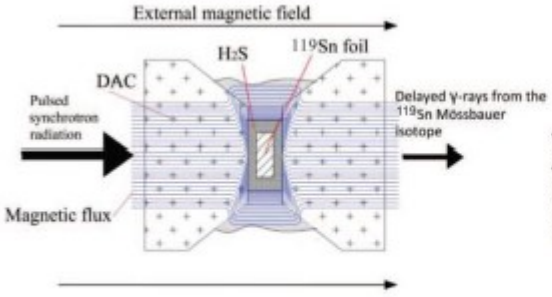
Dubrovinsky Nature 2015

Single Crystal XRD

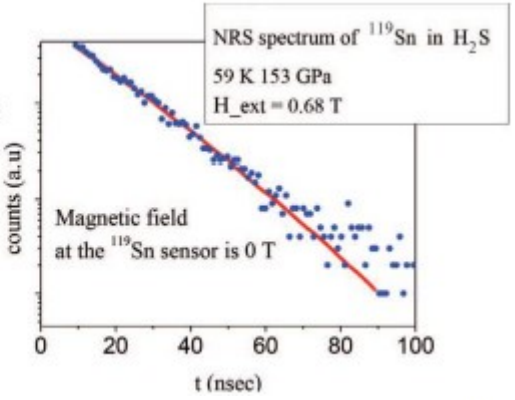


Spaulding Nature Comm. 2014

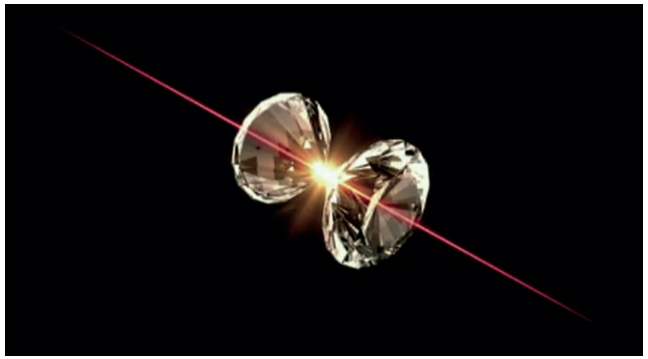
Nuclear Resonance Scattering



Troyan Science 2016

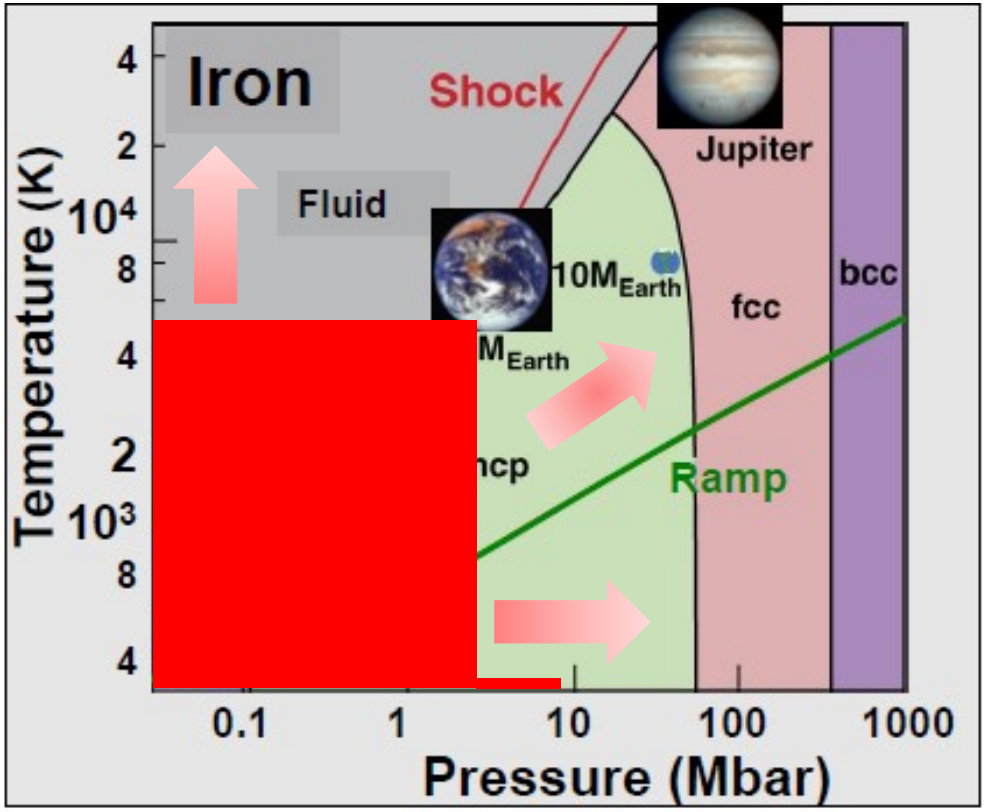


GOING BEYOND THE LIMIT OF STATIC COMPRESSION



Static compression with LH–DAC covers Earth's core conditions

~ 360 GPa, 5500 K



1. What is the stability limit of hcp phase in solid Fe ?
2. What is the local structure in the liquid ?
3. What is the nature of ion-ion correlations in the WDM regime ?

Can we create and probe WDM at the synchrotron, with data quality as "at ambient" ?

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PUSHING THE FRONTIERS OF HIGH PRESSURE RESEARCH

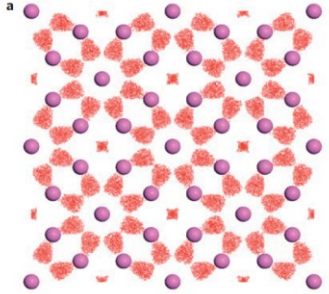
❑ Go more extreme → TPa & eV

Conditions beyond those existing in our planet → Input for planetary models

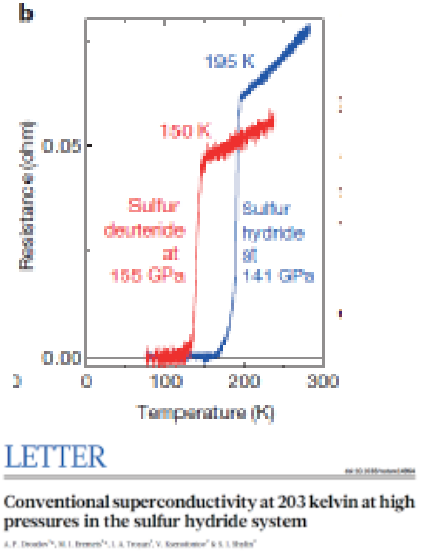
Synthesis of novel materials

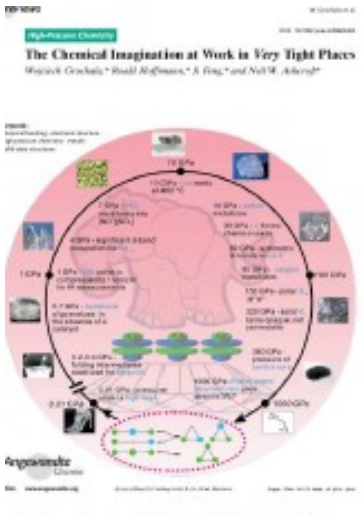
Reveal new physical chemistry

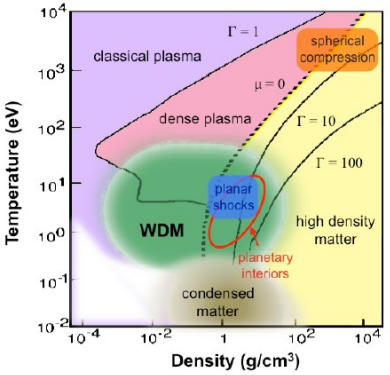
Al @ 1TPa



Pickard and Needs 2010







Particle ejection

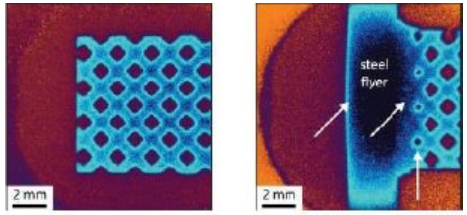
ENSMA Poitiers

❑ Explore the time scale of high pressure phenomena → ns

Dynamic behavior of matter and materials under high strain rates

- Mechanisms and nucleation of phase transitions
- Yield strength (dynamics of dislocations)
- Nanostructuration, amorphisation, metastable phases

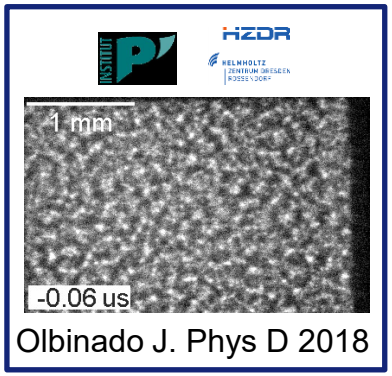
Heterogeneous media



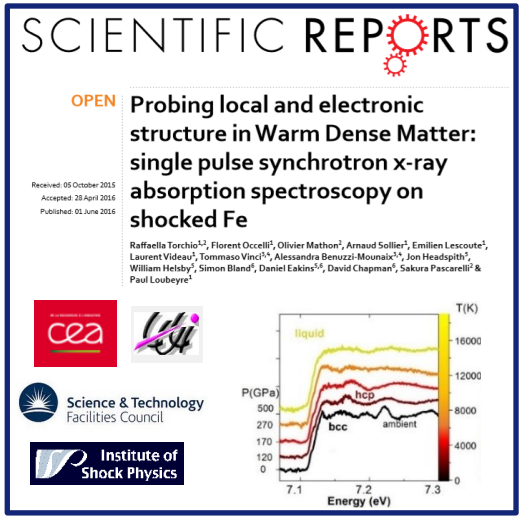
Pre-shot 2.4 μs

ISP Imperial London

A SYNERGETIC APPROACH TO DYNAMIC COMPRESSION AT ESRF

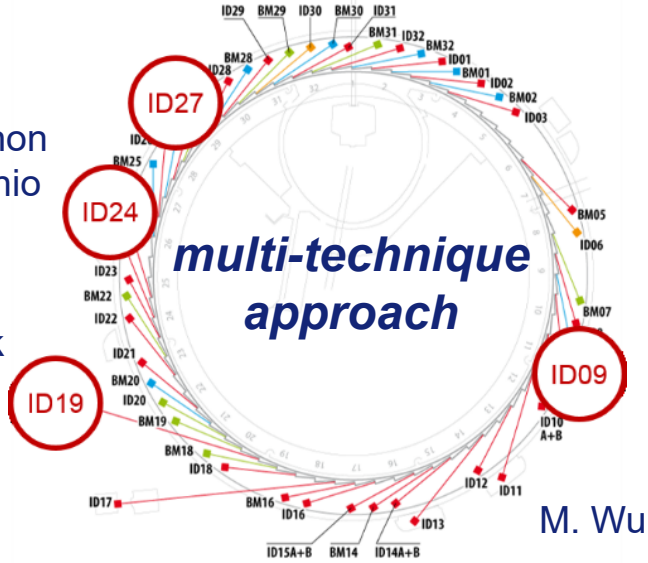


Olbinado J. Phys D 2018

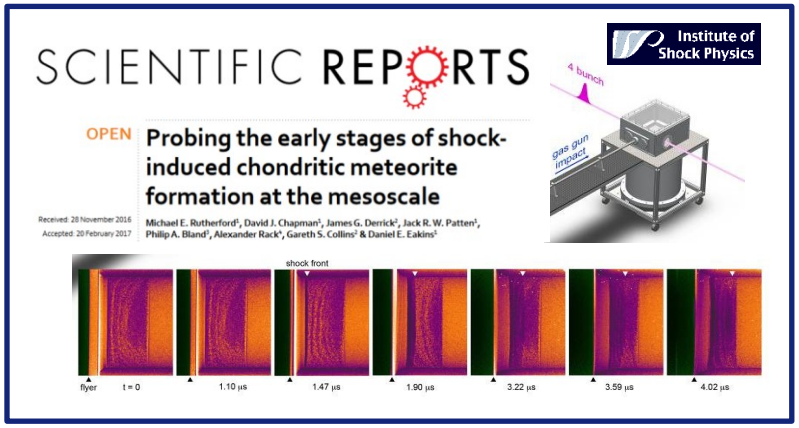


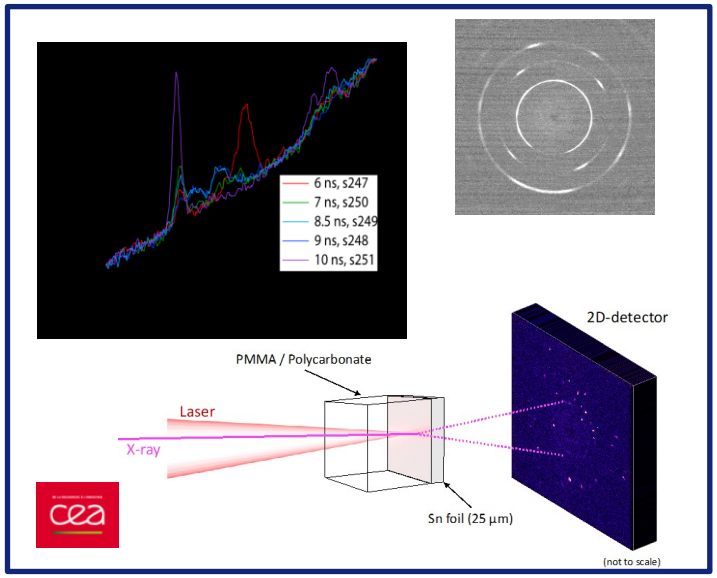
O. Mathon
R. Torchio

A. Rack



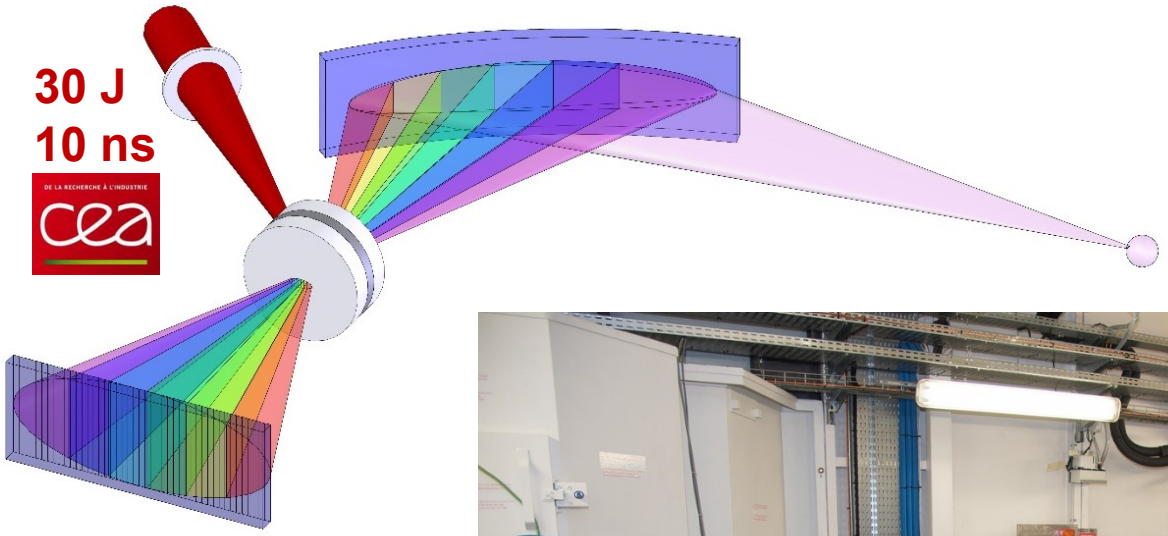
M. Wulff



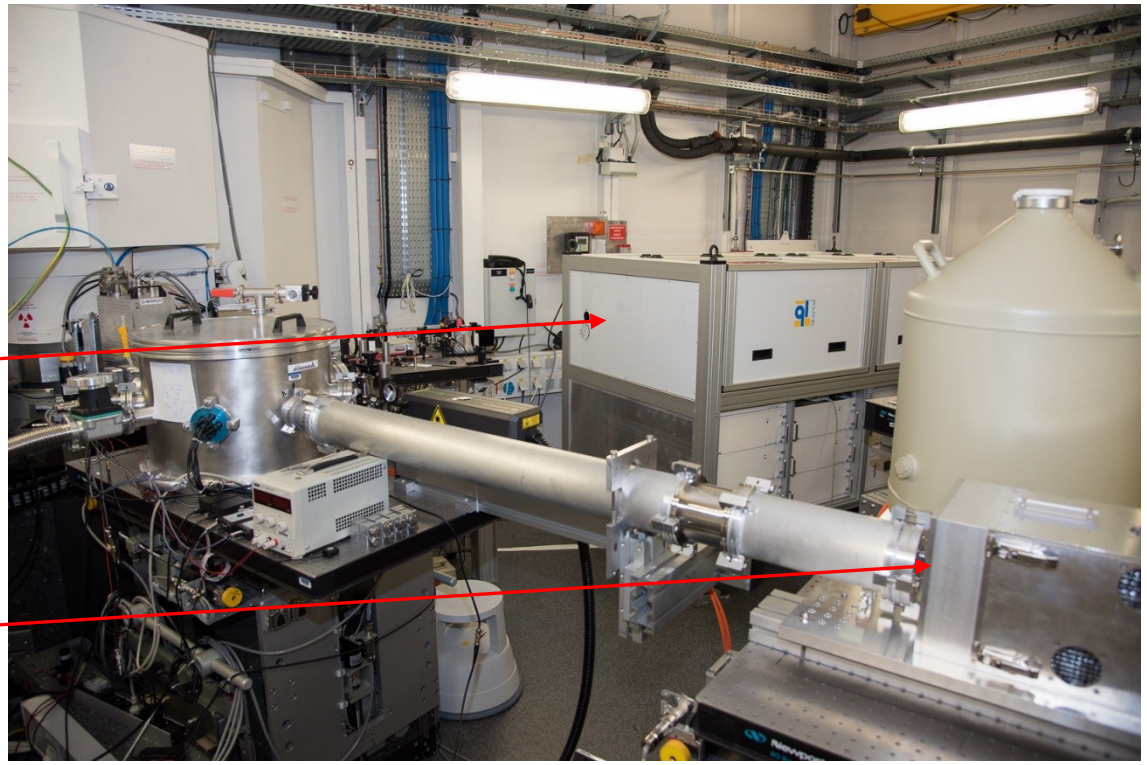


First dynamic compression experiments using high power lasers

ID24



ID24, ESRF



GCLT 30J 10 ns

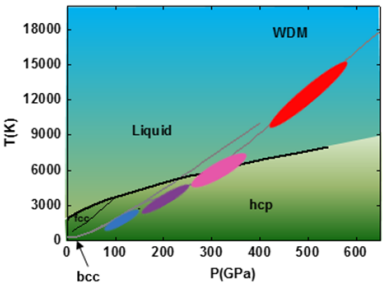
XH detector

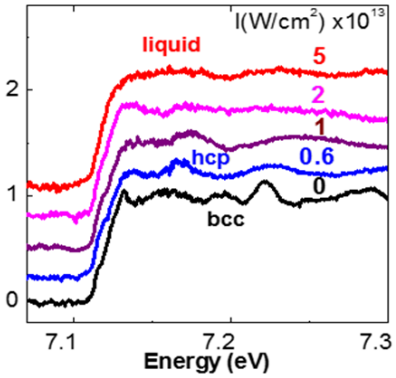
First experiments on ID24 (2014-2016)

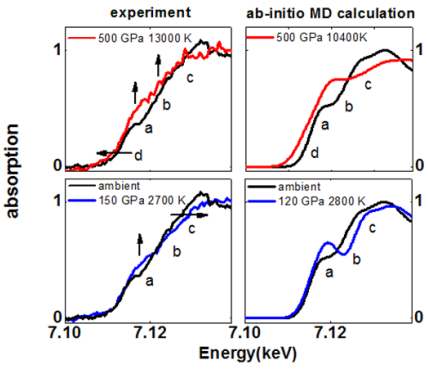
Local and electronic structure in warm dense Fe

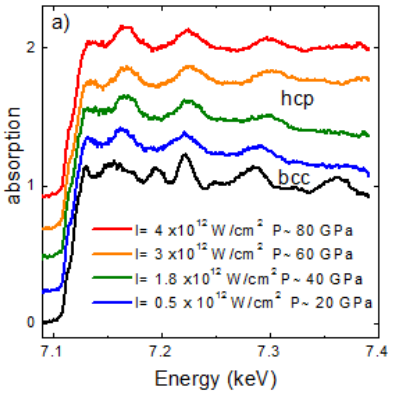
R. Torchio et al. Sci. Rep. 2016

500 GPa, 12000 K



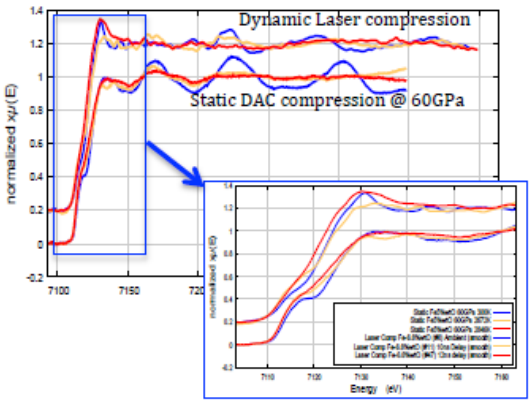






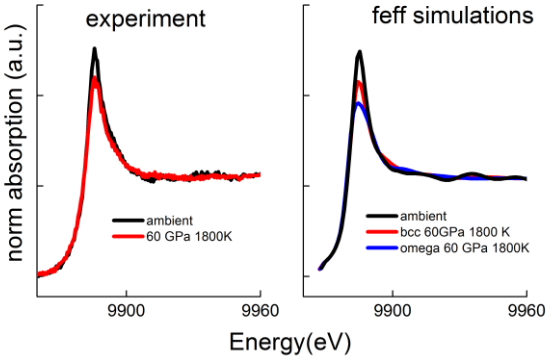
Local structure of laser shocked Fe-6.6%wtO

M. Harmand et al.



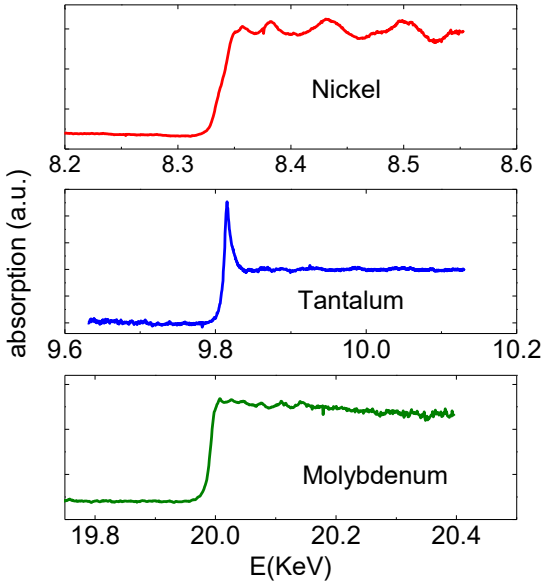
Electronic structure of laser shocked Ta

A. Sollier et al



Single bunch XAS at Ni, Ta and Mo

O. Mathon et al., HPR 36, 404 (2016)



The High Power Laser Facility at the ESRF

HPLF-I (2018-2021)

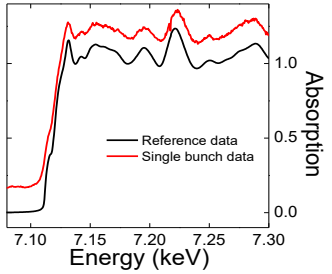
Couple a 100 J (upgradable to 200 J) ns-shaped laser to XAS on ID24

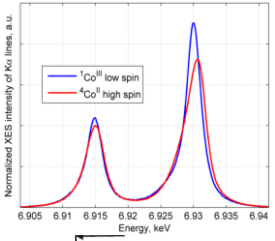
In Construction

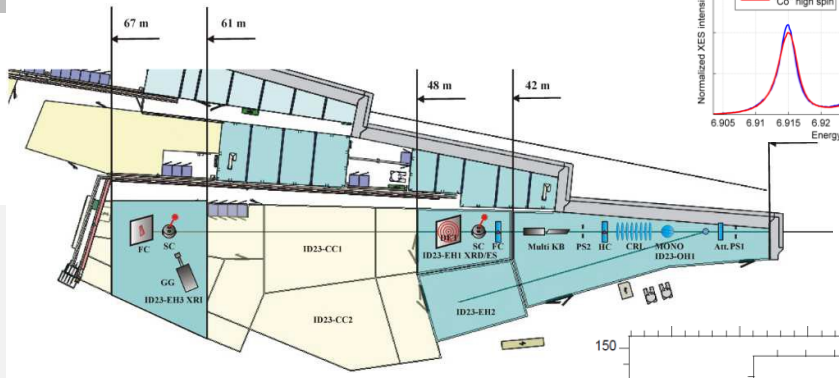
HPLF-II (from 2023)

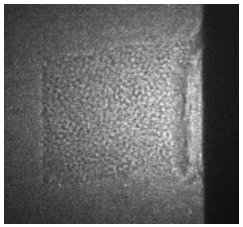
Extend to XRD, XRI, XES on ID23
Laser upgrade

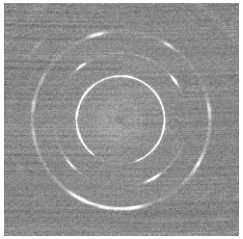
EBS Beamline program

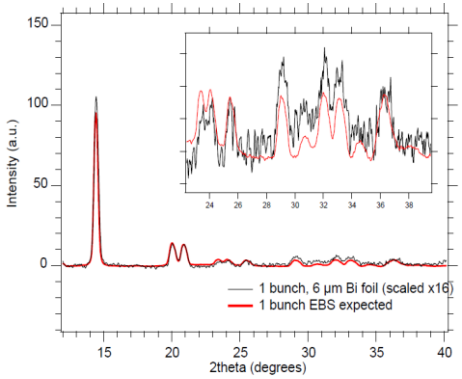












HPLF – I : PLANNING

2018: Delivery & commissioning of the 15J laser front end

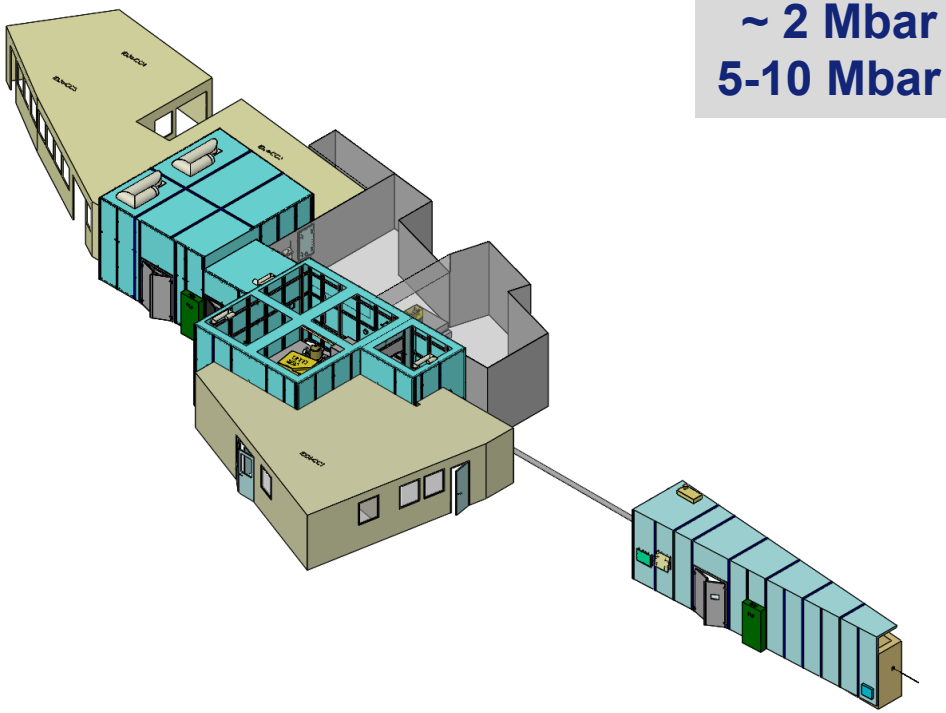
2019: EBS shutdown

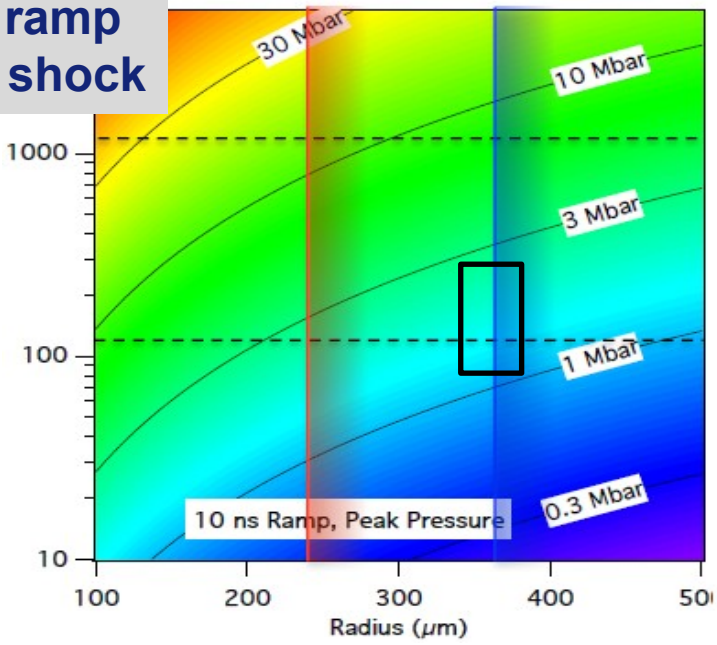
2020: 100J laser delivery

2021: User operation

15J, 10ns
40-200 GPa

100J, 6ns
~ 2 Mbar in ramp
5-10 Mbar in shock



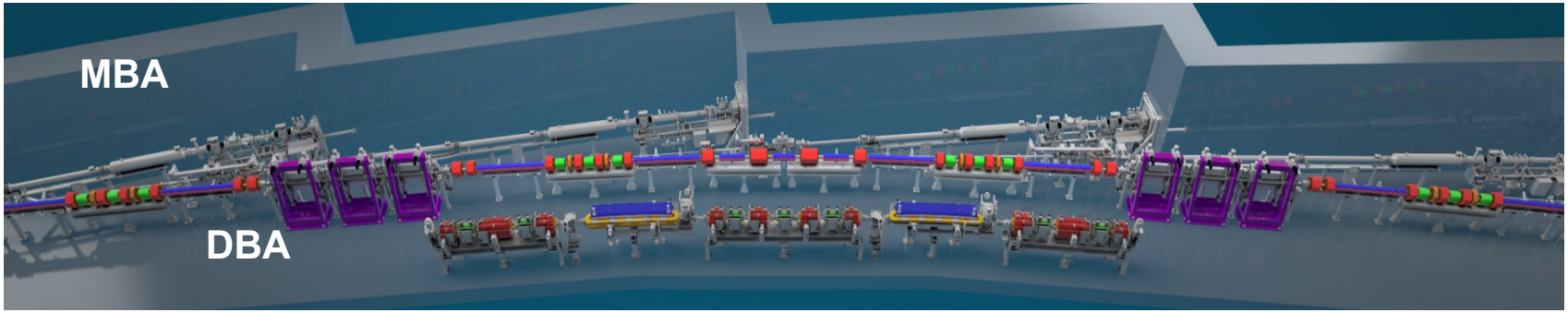


Fratanduono 2011

OUTLINE

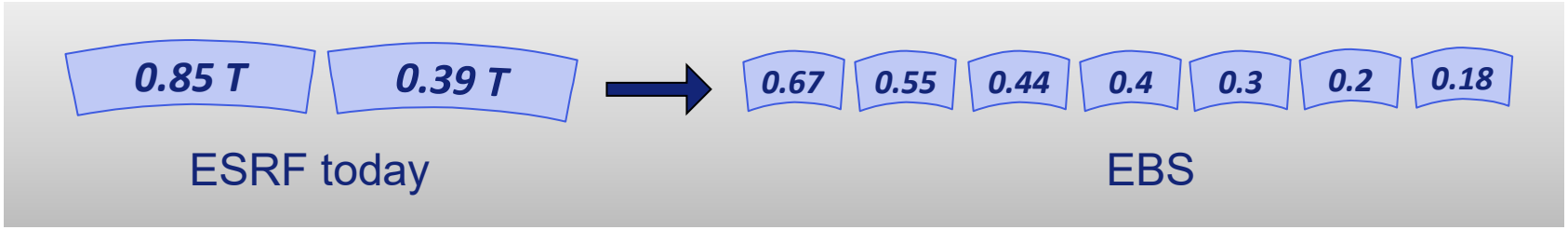
- Static High Pressure Research: status and trends
- Dynamic Compression: recent developments, future plans
- The EBS
- Extreme Conditions Science at EBS

DIFFRACTION LIMITED STORAGE RINGS



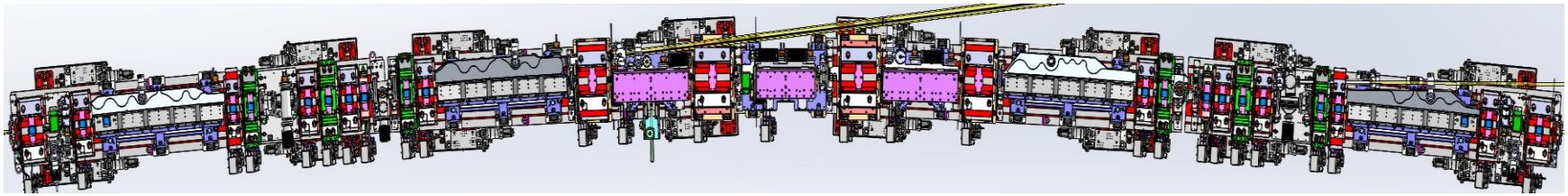
GOAL: REDUCE EQUILIBRIUM HORIZONTAL EMITTANCE

$$\varepsilon \propto \frac{E_e^2}{\left(N_{sect} \cdot N_{dipole}\right)^3}$$



EBS lattice

- Hybrid 7 Bend Achromat = (4 dipoles + 3 dipole-quad + 24 quad., sext., oct.) per cell
- ID length = 5 m

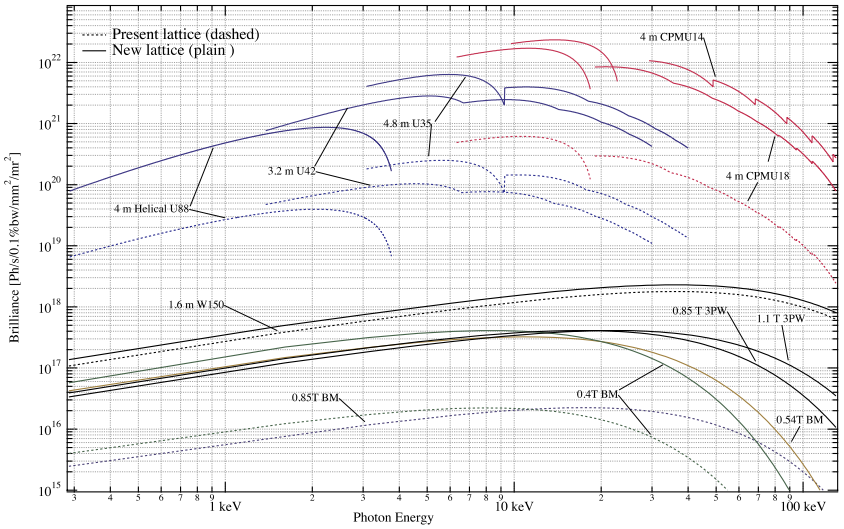


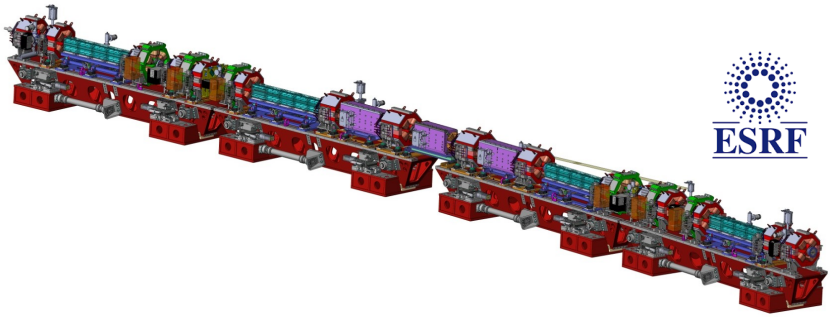
31 magnets per cell instead of currently 17

32 cells (arcs) with 4 girders each

4TH GENERATION SYNCHROTRON SOURCES

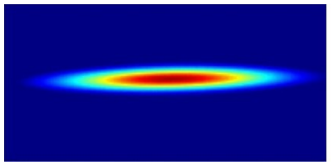
Brilliance



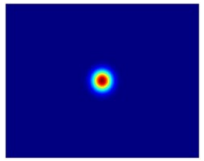


Horizontal emittance

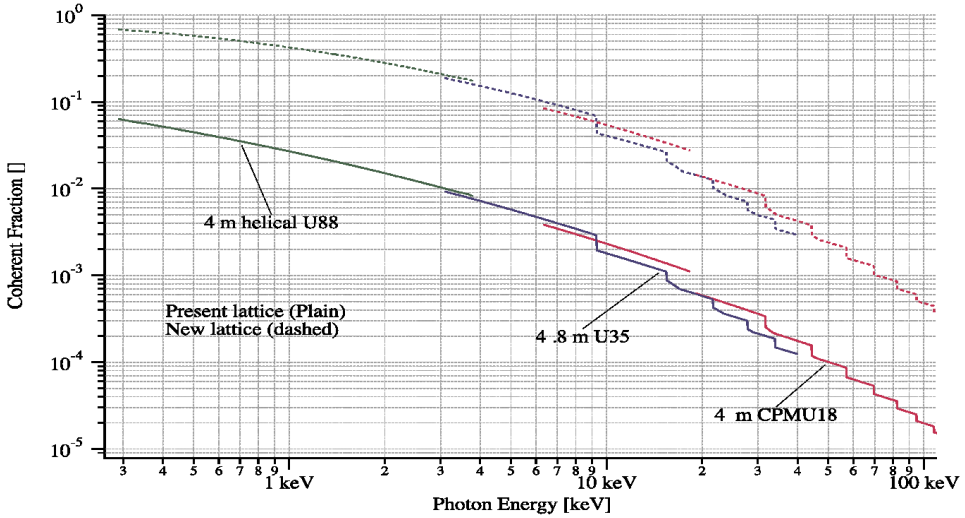
ε_x= 4 nm



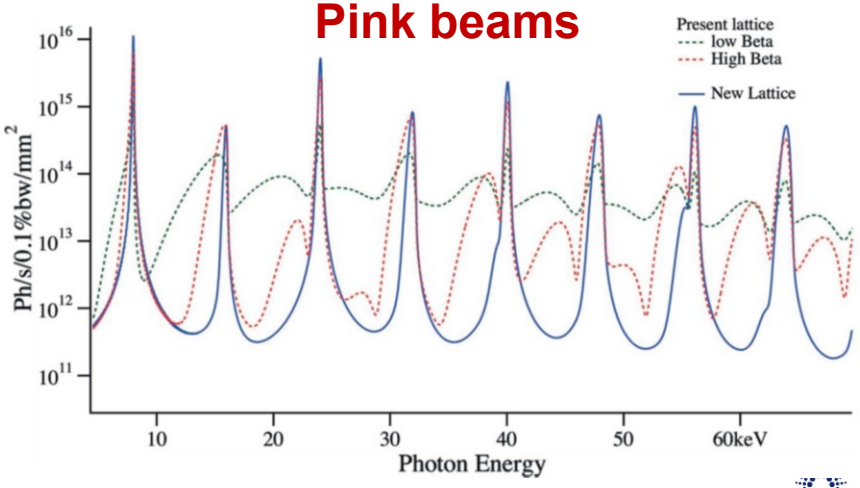
ε_x= 0.15 nm



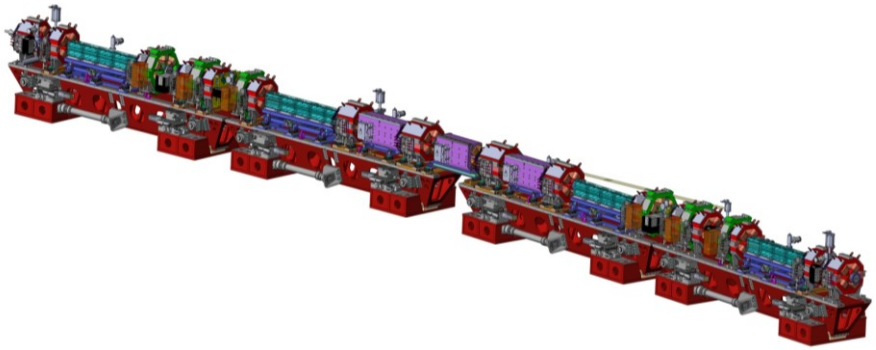
Coherent Fraction



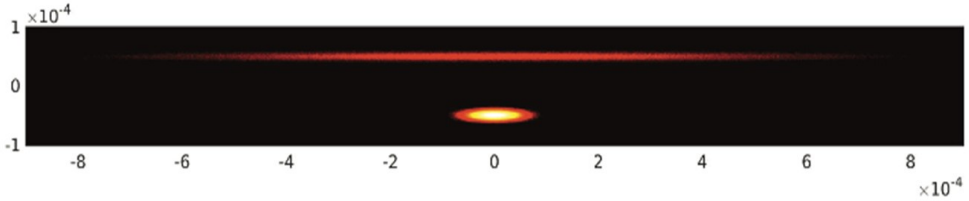
Pink beams



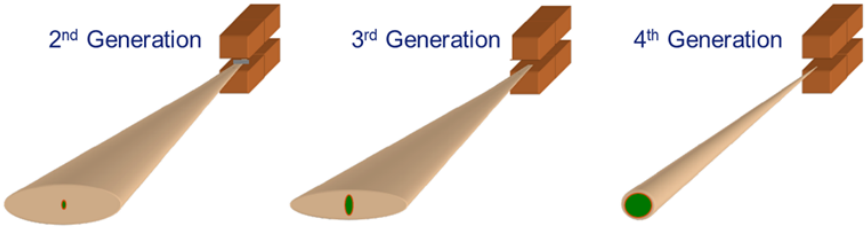
THE ESRF EXTREMELY BRILLIANT SOURCE (ESRF-EBS)

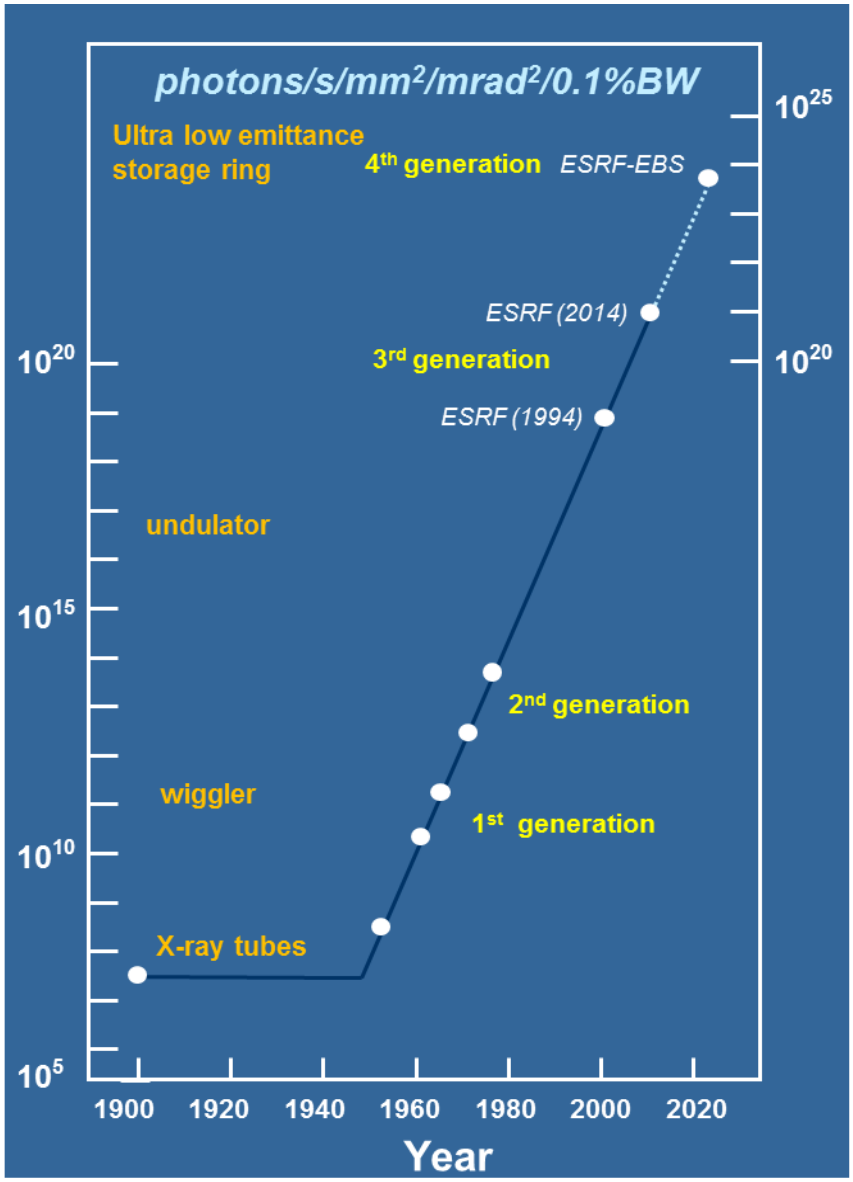


before and after EBS



- photon source **brilliance** (**x100**)
- **coherent** fraction of the photon beam (**x50**)



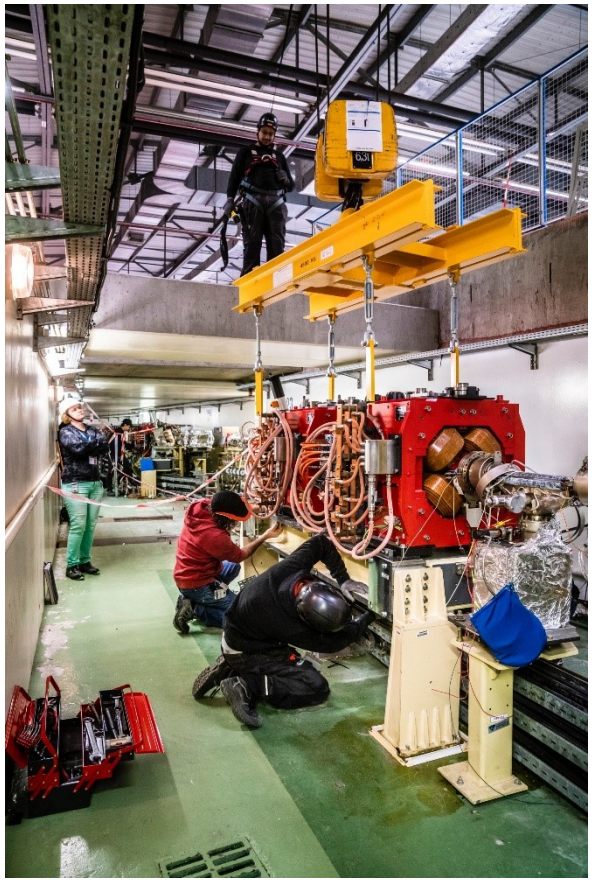


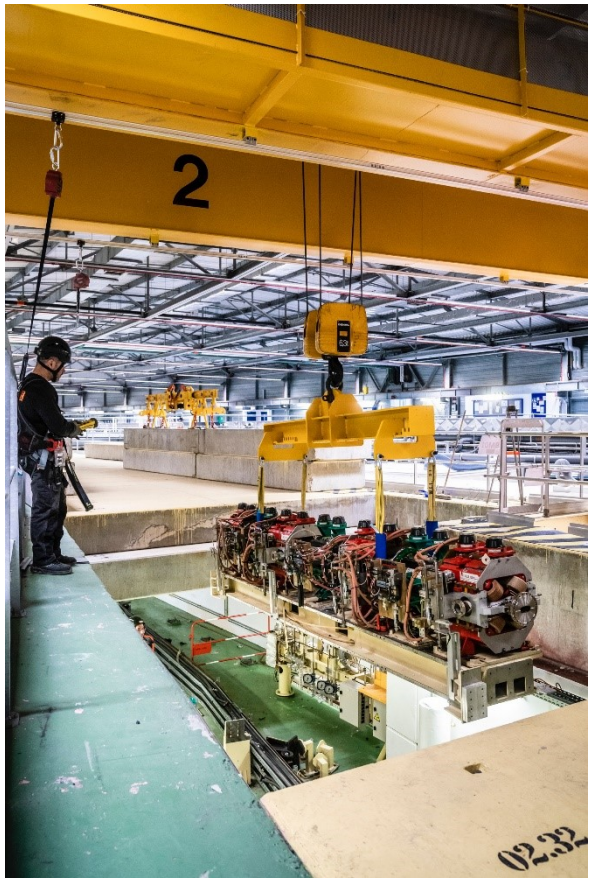
THE EBS PROGRAMME

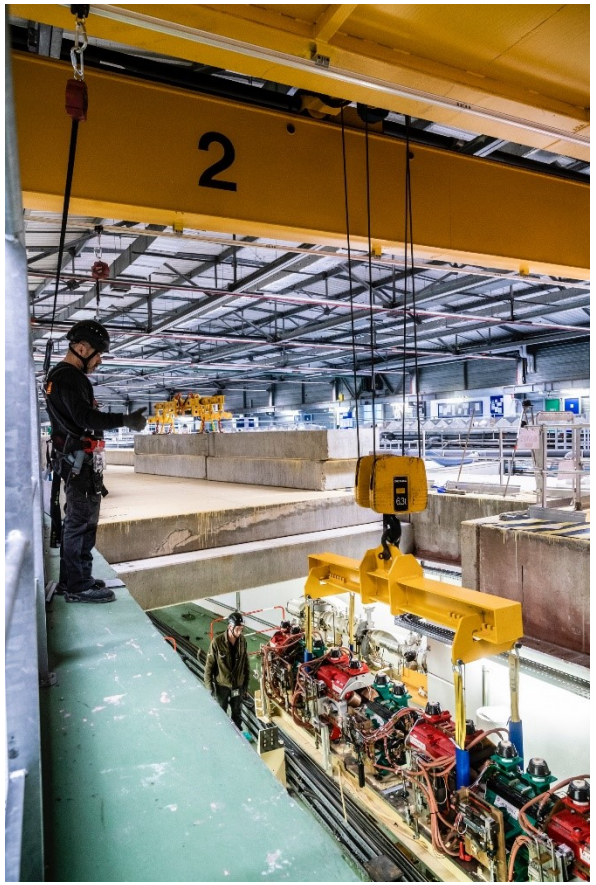
SCHEDULE:

2017-2018	Delivery of the components, testing, and pre-assembly
10th Dec 2018	End of USM and start of the shutdown
Jan – March 2019	Dismantling of the storage ring
April – Nov 2019	New storage ring installation
Dec 2019 – March 2020	Accelerator commissioning
March – Aug 2020	Beamline restart and commissioning
25th August 2020	Back to full User Operation

DISMANTLING THE HISTORICAL ESRF STORAGE RING







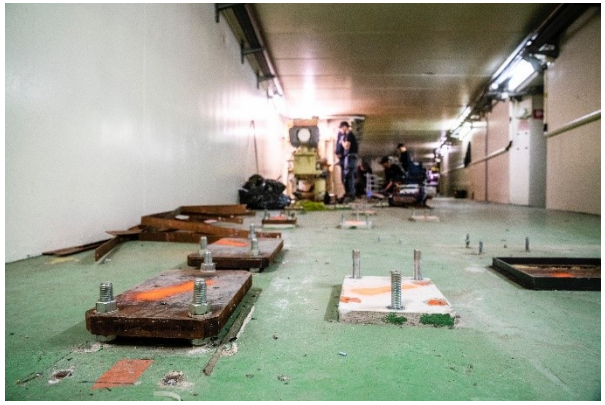
TEMPORARY STORAGE OF THE ESRF HISTORICAL RING

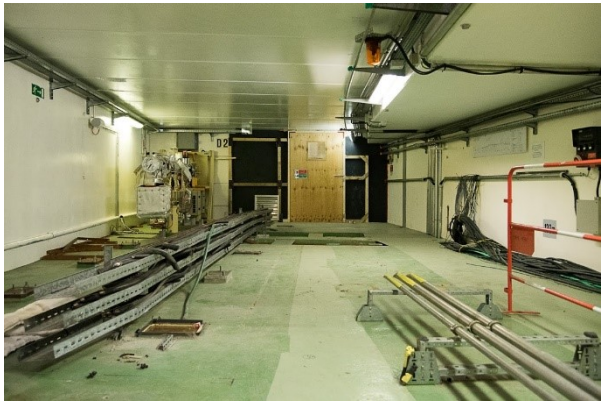


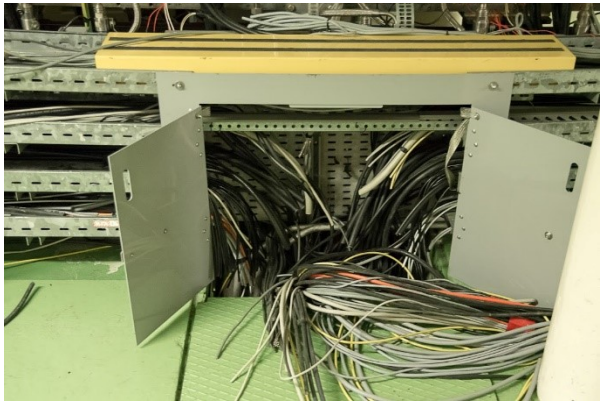



1700 TONS OF MATERIAL AND 200 KM CABLES

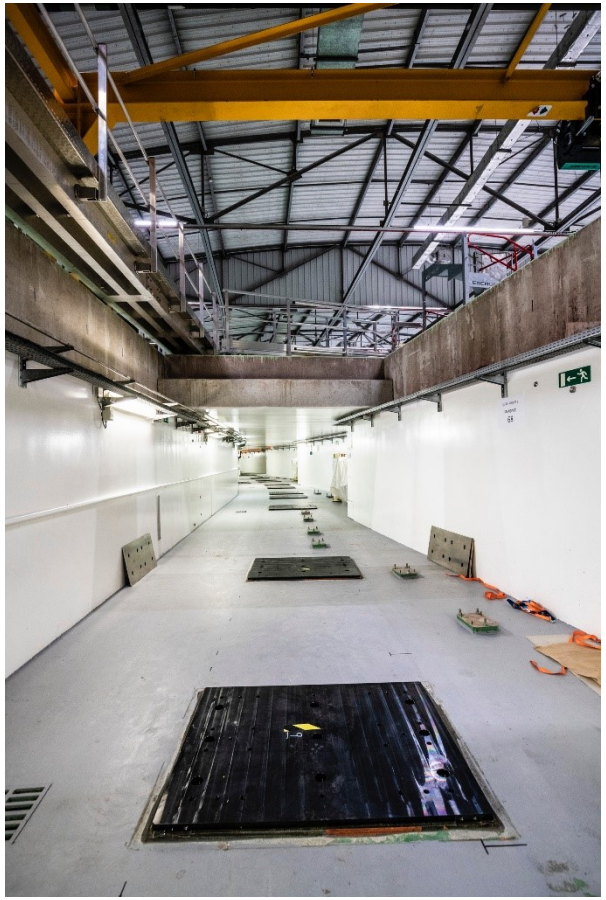
CIVIL WORK AND PREPARATION OF THE TUNNEL



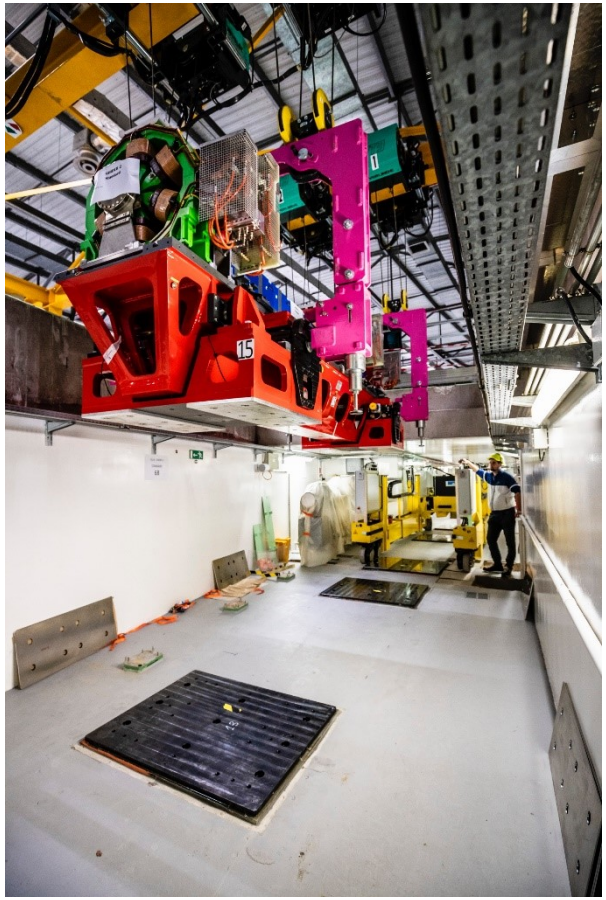


CIVIL WORK AND PREPARATION OF THE TUNNEL



INSTALLATION OF THE NEW GIRDERS



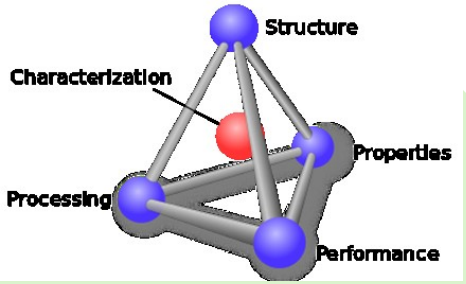
THE EBS STORAGE RING STARTS TO TAKE ITS SHAPE

EBS – SCIENCE CASE(S) IN BRIEF

- In-situ or operando characterisation
- Coherence based techniques
- Spatial resolution
- Temporal resolution

- New focusing/collimation schemes
- Horizontal diffraction/scattering planes
- Penetration
- Radiation damage

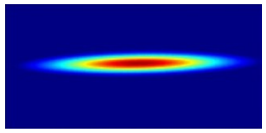
BRILLIANCE

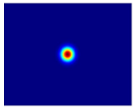


COHERENCE

FLUX

HARD X-RAYS





FROM AVERAGED TO SINGLE OBJECT INFORMATION

+

DYNAMICS

+

REAL SYSTEMS

EXPERIMENTAL PROGRAM OVERVIEW: 3 INTERCONNECTED SUB-PROGRAMS

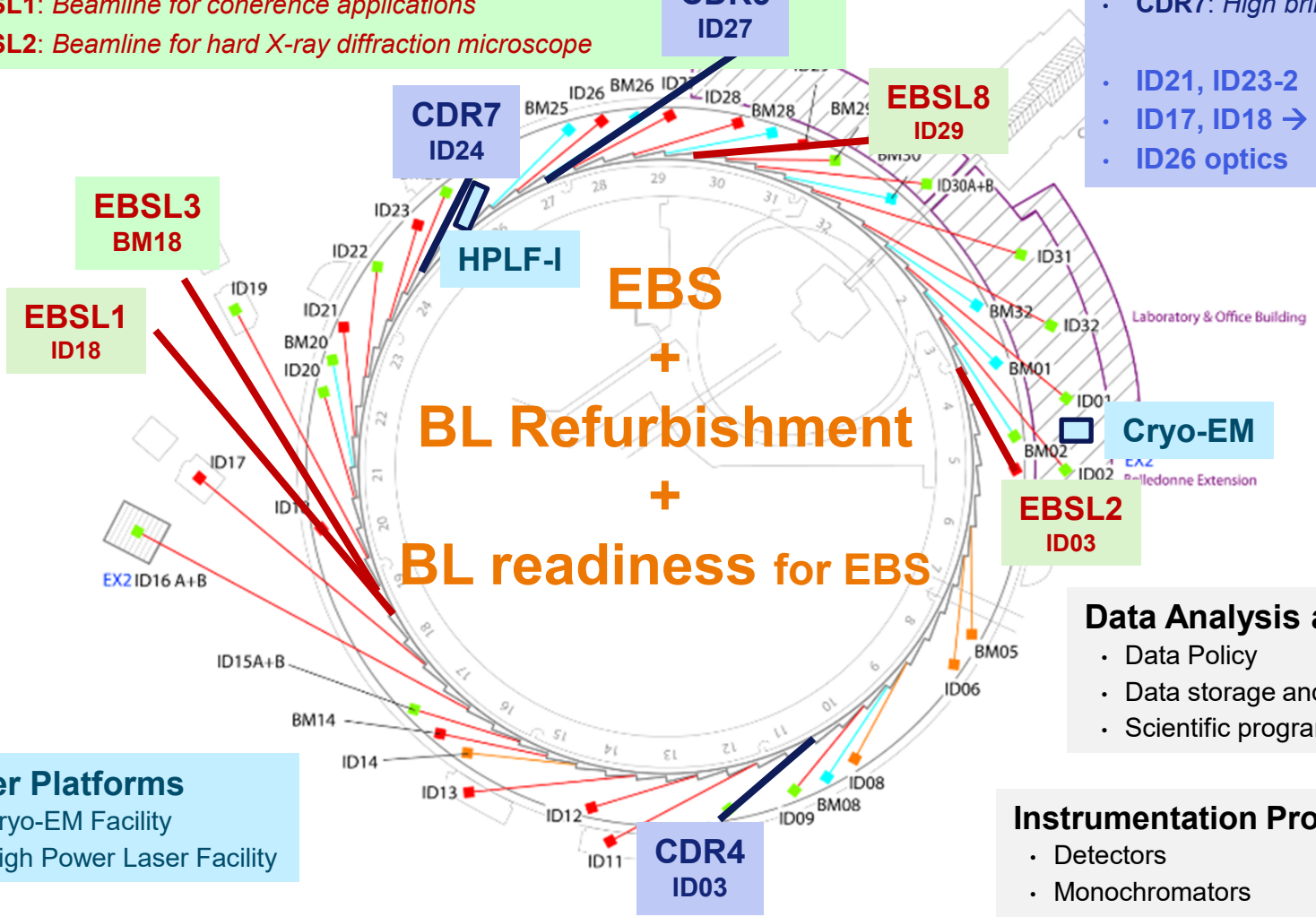
EBS Beamlines

- **EBSL3**: *High throughput large field phase-contrast tomography beamline*
- **EBSL8**: *Serial crystallography beamline*
- **EBSL1**: *Beamline for coherence applications*
- **EBSL2**: *Beamline for hard X-ray diffraction microscope*

Refurbishment Programme

- **CDR4**: *Surface science*
- **CDR5**: *Extreme conditions*
- **CDR7**: *High brilliance XAS*

- **ID21, ID23-2**
- **ID17, ID18 → ID14**
- **ID26 optics**



Data Analysis as a Service

- Data Policy
- Data storage and archiving
- Scientific programming

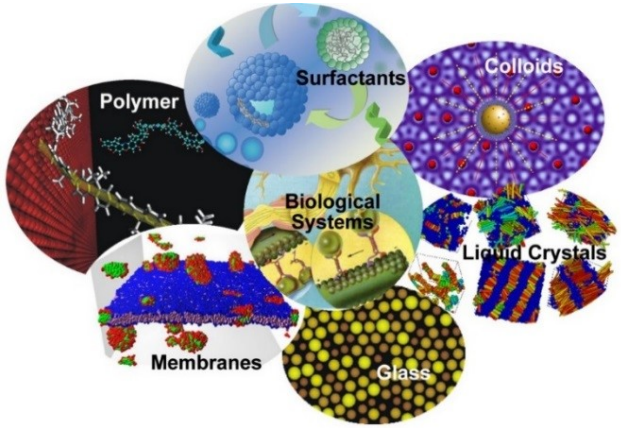
User Platforms

- Cryo-EM Facility
- High Power Laser Facility

Instrumentation Programme

- Detectors
- Monochromators
- BL control system

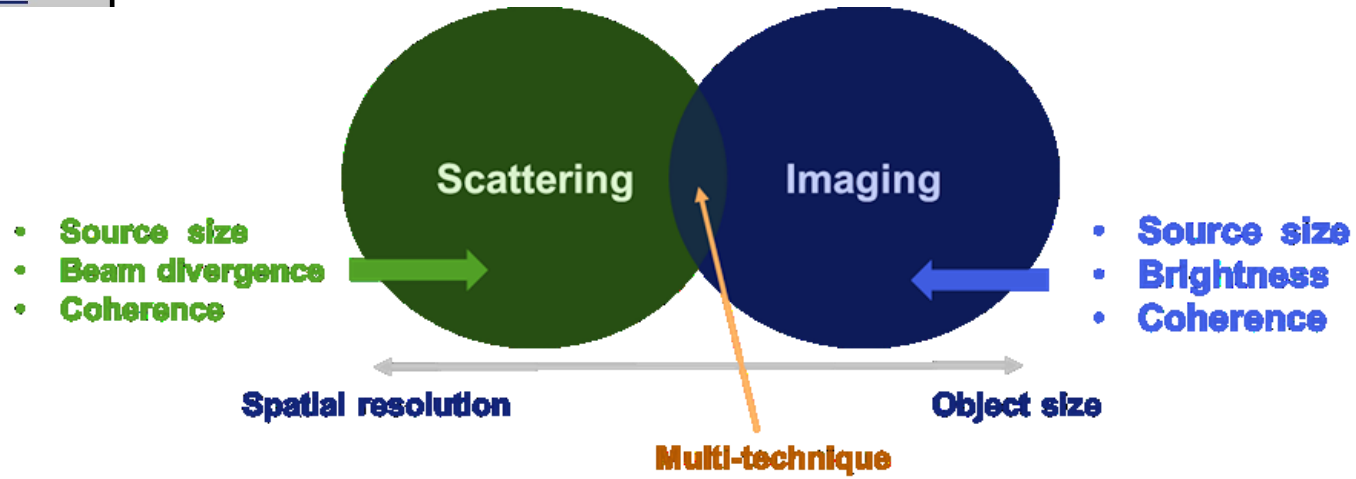
EBSL1 – COHERENCE APPLICATIONS (ID18)



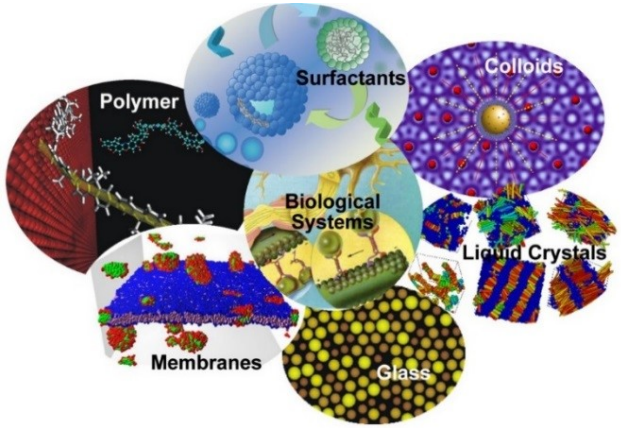
$$Fastest\ time\ scale:\ \tau_{min} \propto \frac{1}{B^2}$$

$$Coherent\ flux:\ F_{coh} \propto \frac{B\lambda^2}{}$$

- Unrivalled **X-ray coherent flux at high energies** with **temporal resolution, down to 100ns and potential spatial resolution below 1nm**
- **Techniques: XPCS, CDI, far field ptychography on a very long beamline (~ 200m)**
- **Research applications:**
  - **dynamics and structure in biological & soft systems**
  - **bio-mineralisation processes**
  - **image formation in photonic devices**
  - **domain fluctuations in high-Tc superconductors**
  - **glasses & melts under real conditions**



EBSL1 – COHERENCE APPLICATIONS (ID18)



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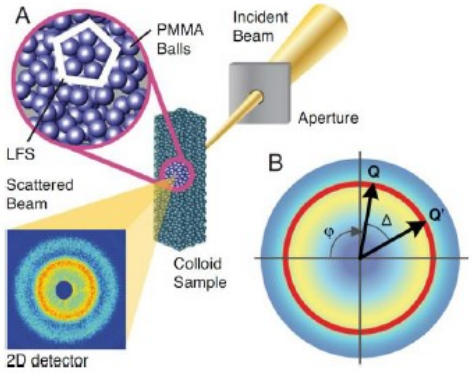
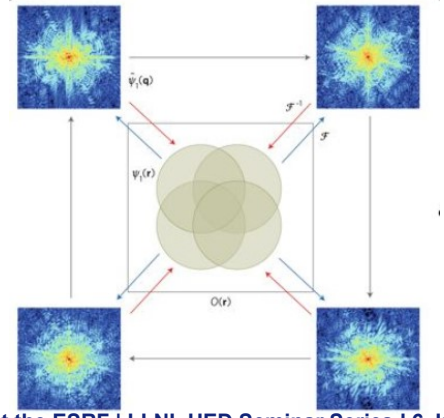
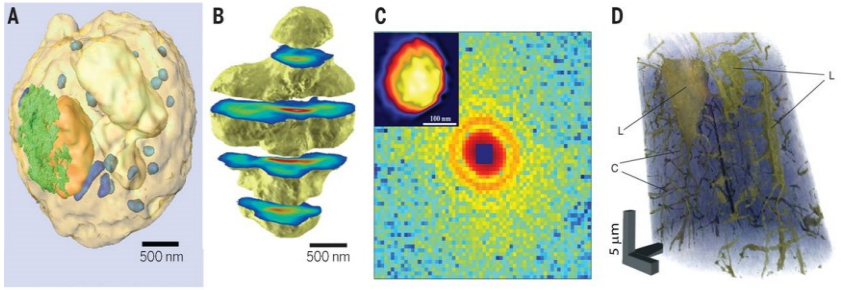






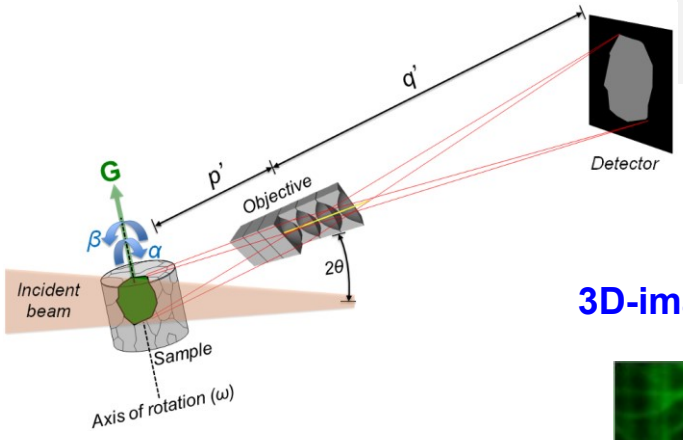
J. Miao, et al. SCIENCE 2015 • 348 6234

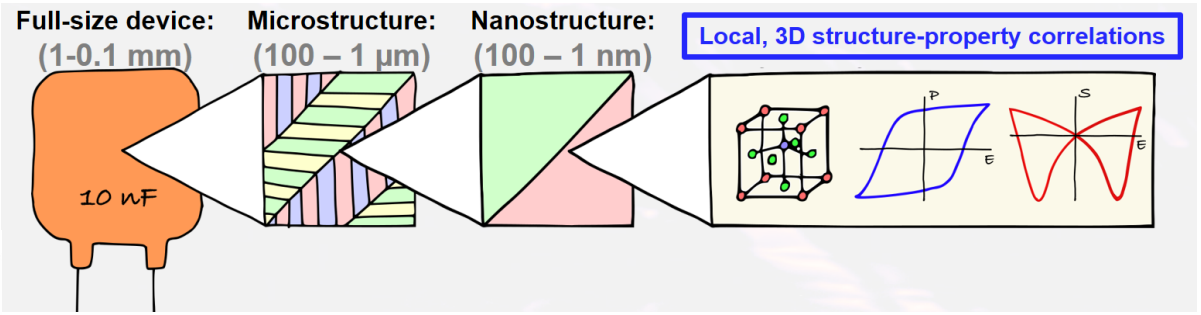
EBSL2 – HARD X-RAY DIFFRACTION MICROSCOPE (ID03)

Multiscale 3D Characterisation of Materials with Hard X-ray Diffraction Microscopy

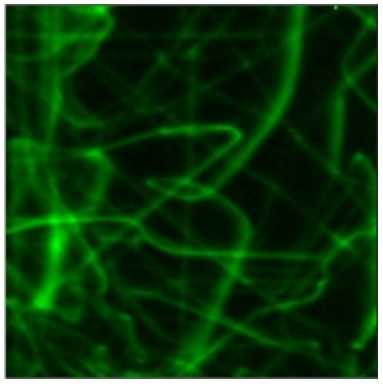
Hard x-ray diffraction microscopy

full-field imaging using a lens between sample and detector

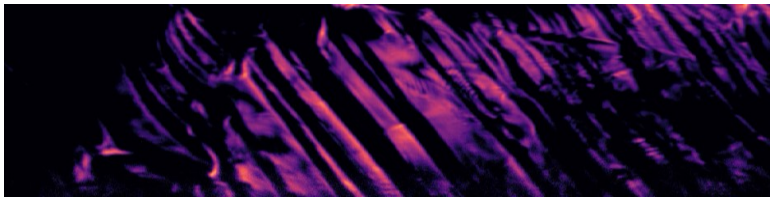




3D-imaging of dislocations



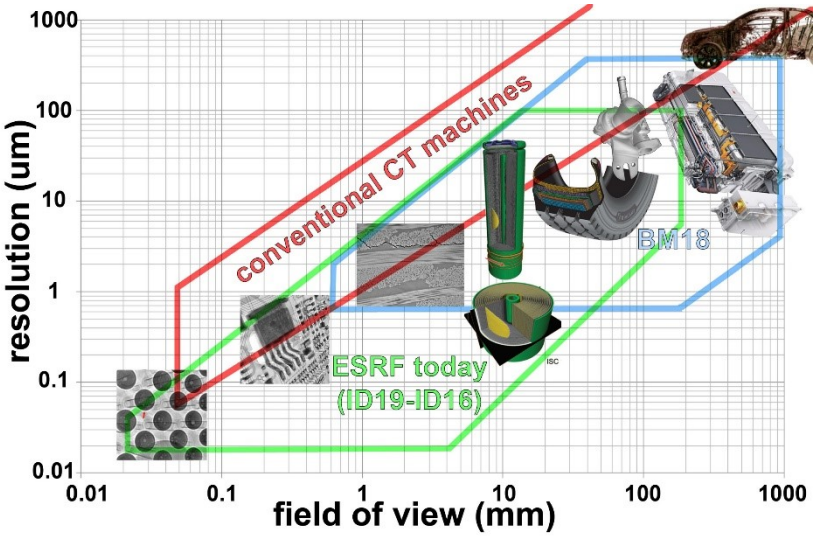
Time-resolved mapping of electric fields



H. Simons *et al.*, *Nat. Comm.* 2015, **6**, 6098; *MRS Bull.* 2016, **41**, 454.

EBSL3 – HIGH THROUGHPUT LARGE FIELD PHASE-CONTRAST TOMOGRAPHY BEAMLINE (BM18)

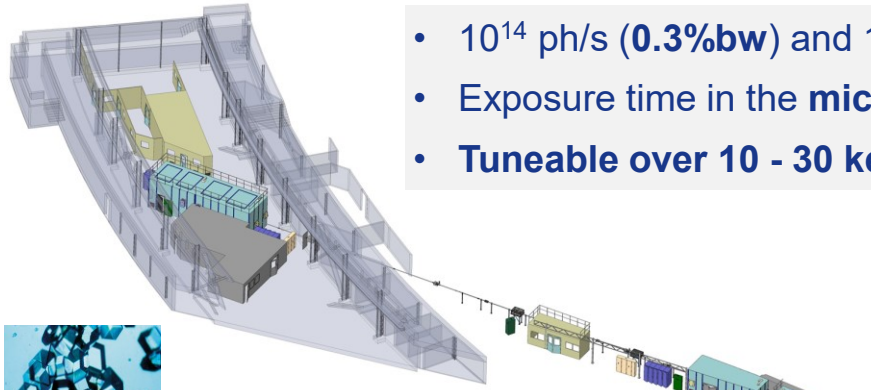
Multiscale phase-contrast tomography for large objects



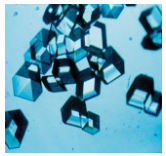
up to 40 m of phase propagation @ 400 keV
Object size: 700 mm x 2500 mm

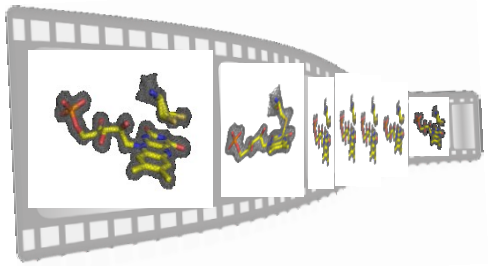
Tomographic reconstruction of an ancient aegyptian mummified crocodile

EBSL8 – SERIAL CRYSTALLOGRAPHY (ID29)



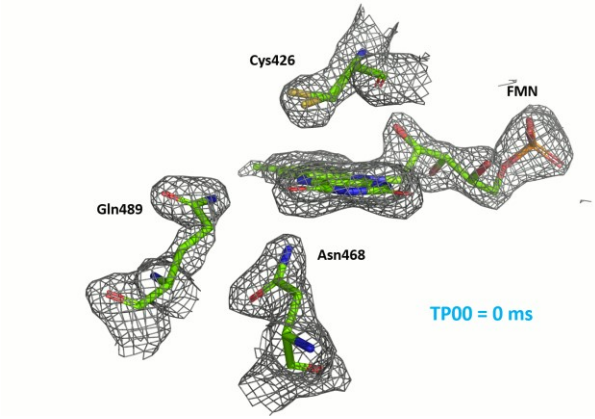
- 10^{14} ph/s (**0.3%bw**) and 10^{16} ph/s (**2%bw**) in **0.5x0.5** µm^2 or **0.5x10** µm^2 beam
- Exposure time in the **micro-** to **millisecond** range
- **Tuneable over 10 - 30 keV** energy range





high flux + submicron X-ray probe + microfluidics to

- **Determine room temperature structure of biologically relevant enzymes using SSX data collection methods**
- **Determine time-resolved structural changes to capture "on-the-fly" enzyme intermediate**
- **Investigate *in-crystallo* non reversible enzymatic reactions and receptor signal transduction**
- **Exploit high dose-rate and high energy data collection to outrun radiation damage**



phototropin 2 from Arabidopsis thaliana

EBSL8 – SERIAL CRYSTALLOGRAPHY (ID29)

- 10^{14} ph/s (**0.3%bw**) and 10^{16} ph/s (**2%bw**) in **0.5x0.5** µm^2 or **0.5x10** µm^2 beam
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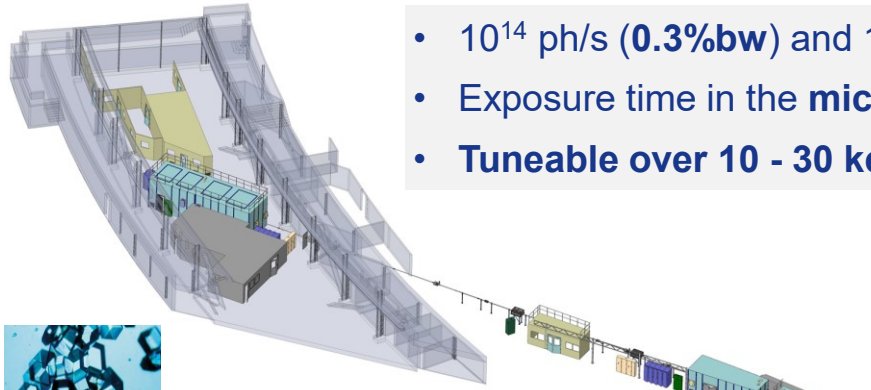
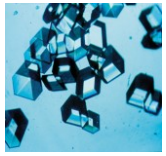
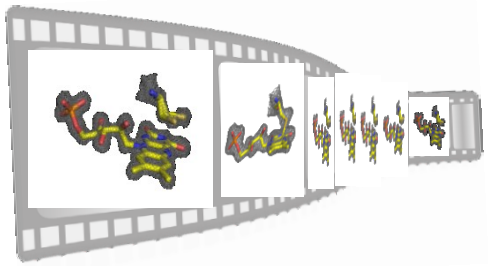
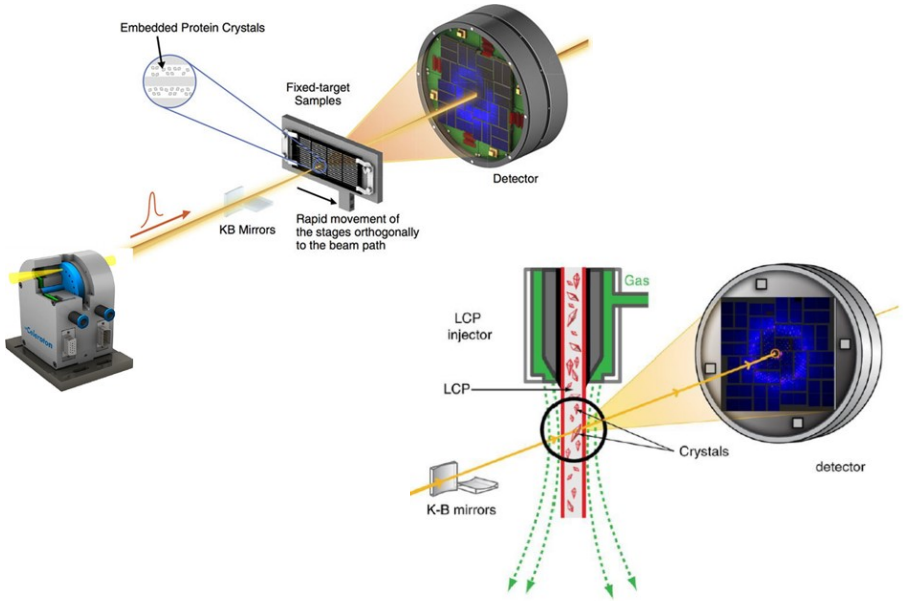
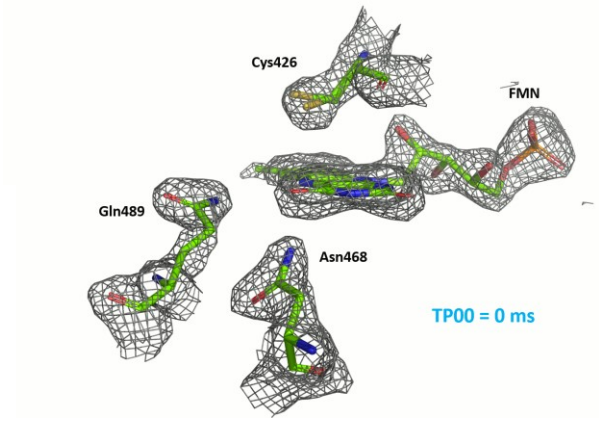










phototropin 2 from Arabidopsis thaliana

OUTLINE

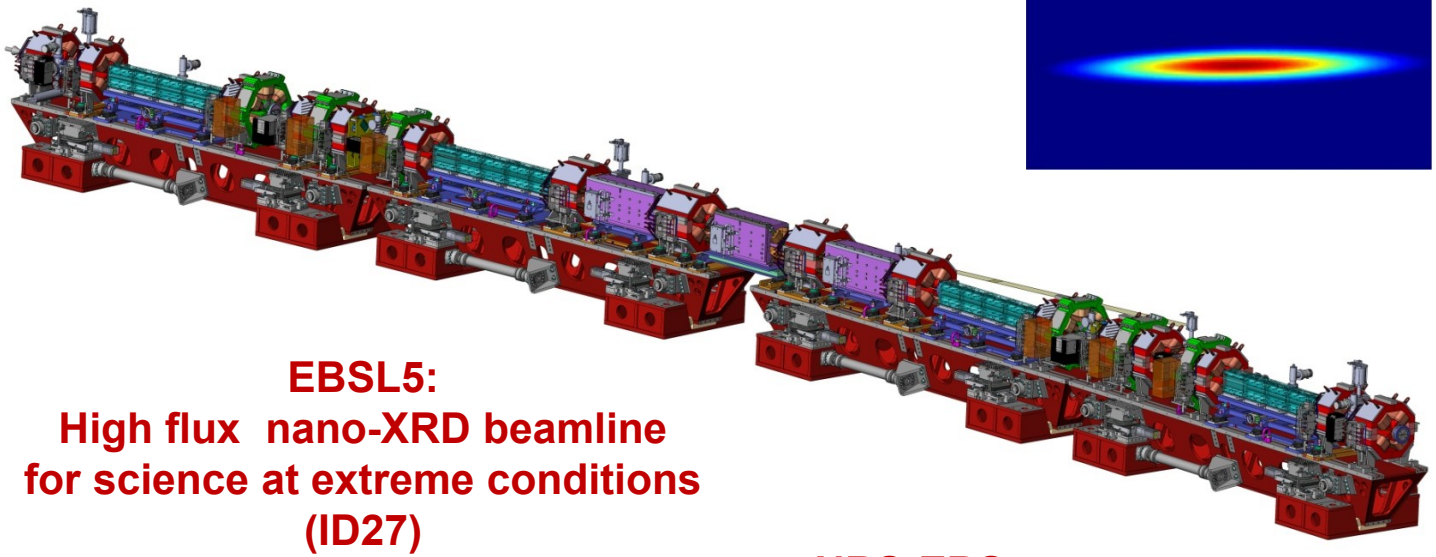
- Static High Pressure Research: status and trends
- Dynamic Compression: recent developments, future plans
- The EBS
- Extreme Conditions Science at EBS

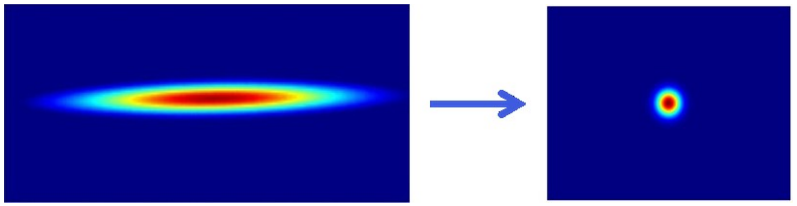
EXTREME CONDITIONS RESEARCH WITH ESRF-EBS

Horizontal emittance

ε_x= 4 nm

ε_x= 0.15 nm





EBSL5:
High flux nano-XRD beamline for science at extreme conditions (ID27)

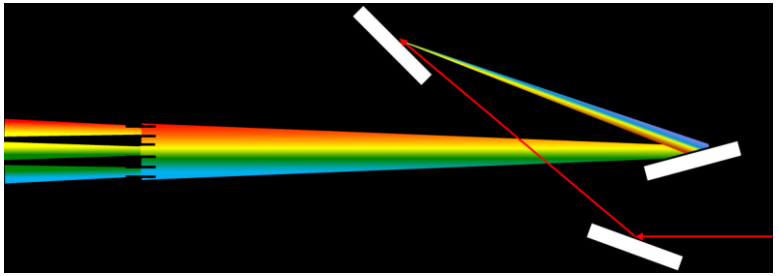
NRS-EBS:
Pushing the limits of NRS in energy and spatial resolution (ID14)

CDR6:
A worldwide unique facility for XRD, XRI, XES, XAS dynamic compression studies (ID23 and ID24)

EBSL7:
Towards sub-μm, high brilliance EXAFS (ID24)

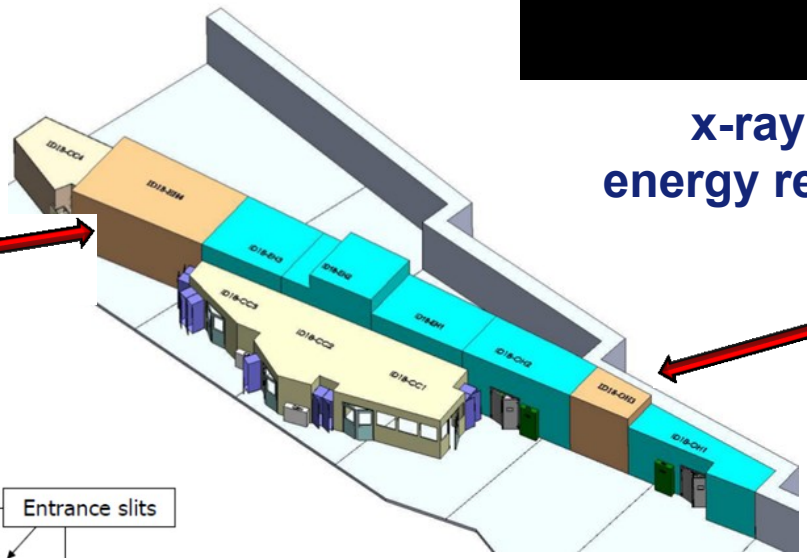
NRS-EBS - NUCLEAR RESONANCE REFURBISHMENT PLANS (→ ID14)

Super-Earth planets interiors



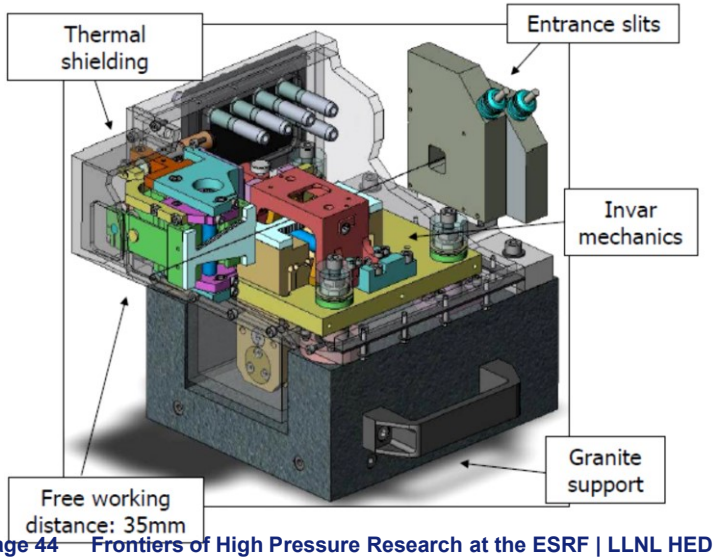
x-ray spectrograph: energy resolution of 40 μeV

New EH4

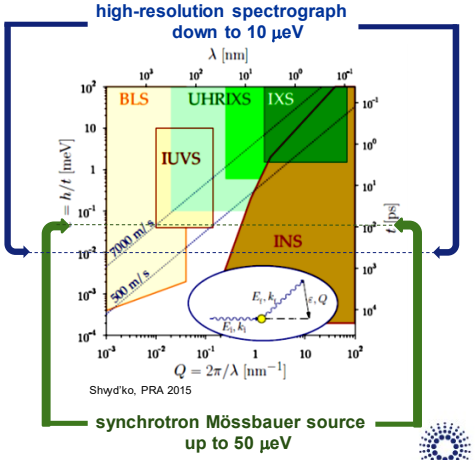


New OH3

KB system: 0.2 μm × 0.2 μm



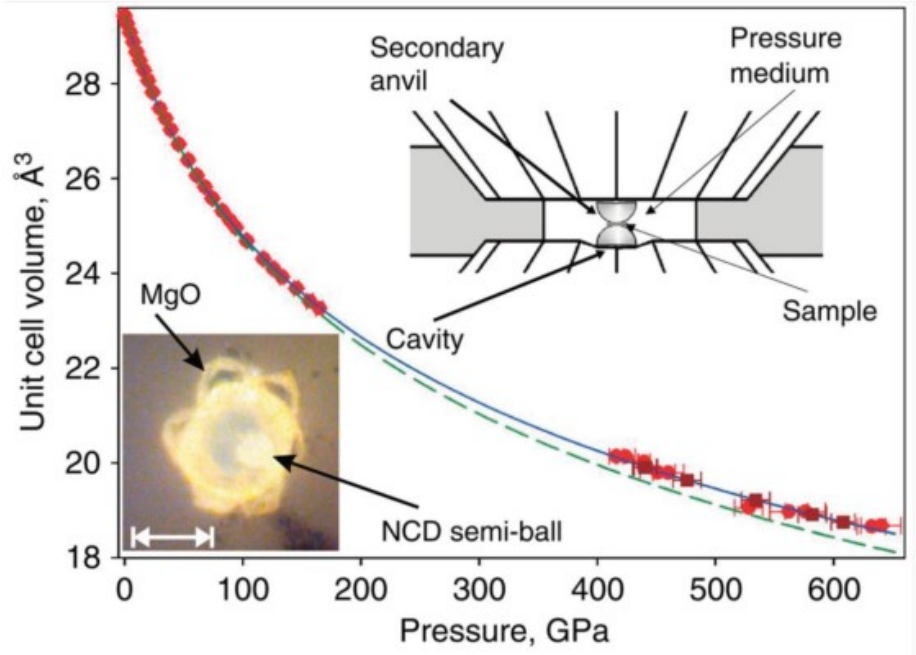
Elastic properties, sound velocity at Megabars



EBSL5 - SCIENCE UNDER EXTREME CONDITIONS (ID27)

Materials at and beyond the current limits of static P and high T

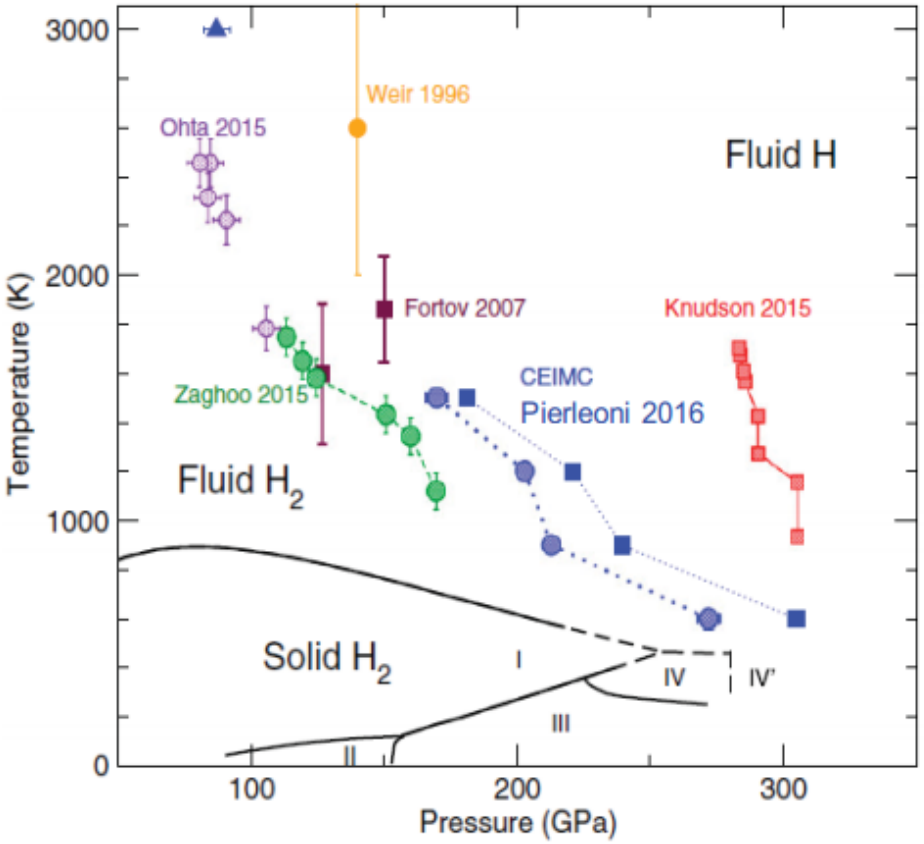
Double stage Diamond Anvil Cell



Pressure to 1000 GPa

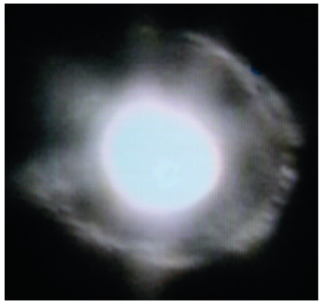
Dubrovinsky Nat. Comm. 2013

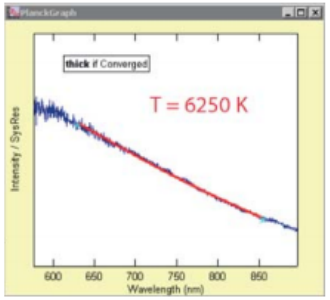
Solving the fluid H_2 to fluid H transition

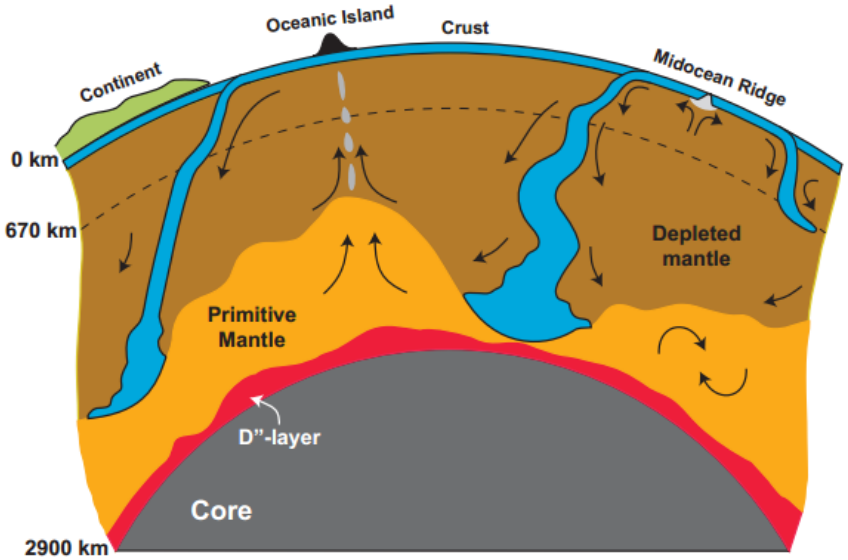


EBSL5 - Structure and chemistry of low Z melts and glasses

Fast melting, kinetics of chemical reactions at extreme conditions



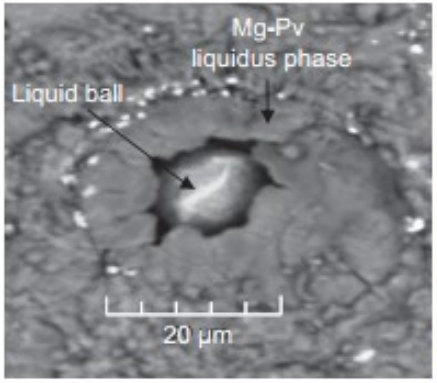


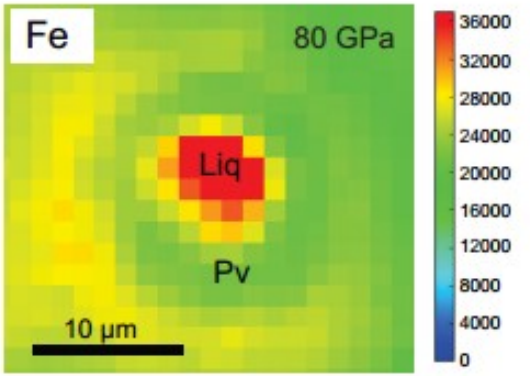


Derived from Kellog et al. 1999

Exploring extreme temperature states using laser heating

In situ chemical analysis of complex systems by combining nano-XRD and XRF

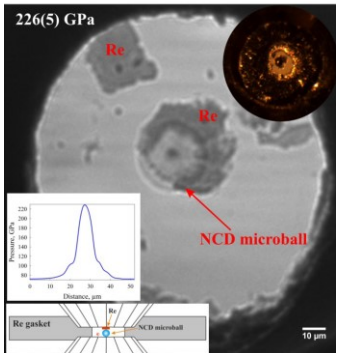




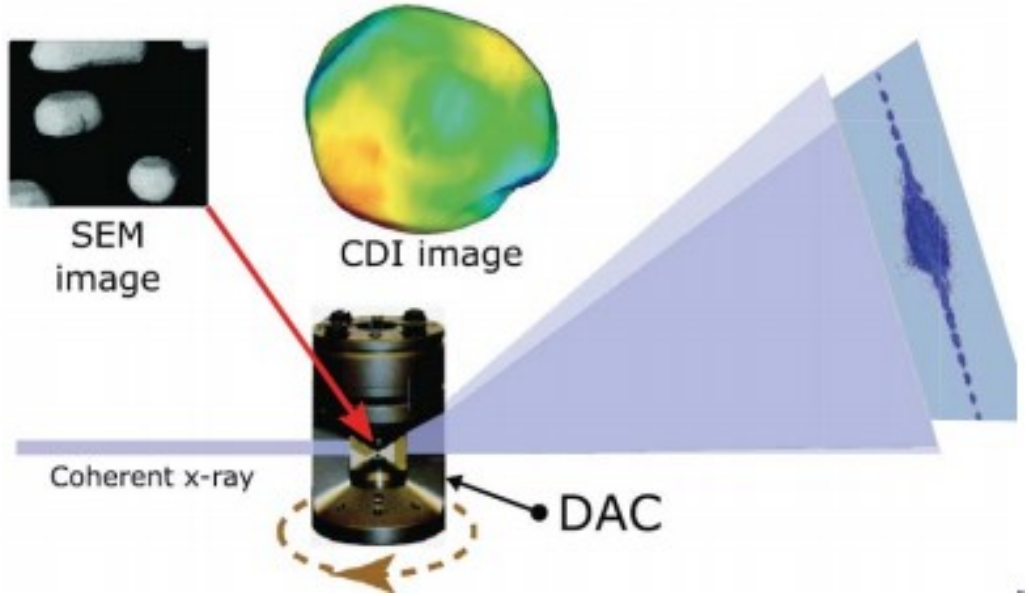
EBSL5 - RHEOLOGY OF MATERIALS AT EXTREME CONDITIONS

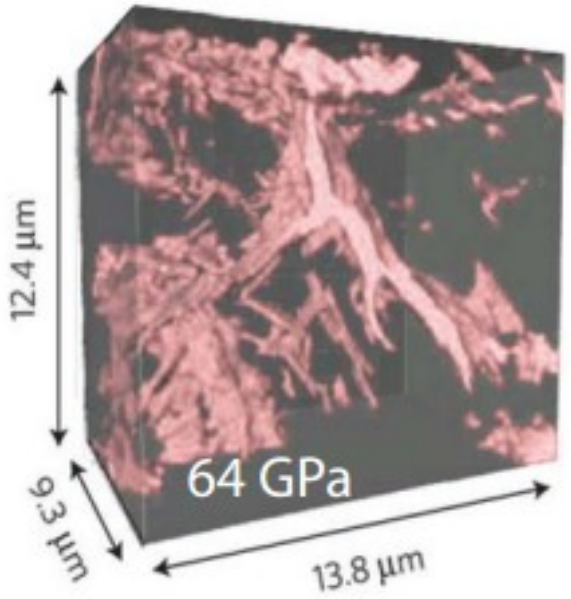
X-ray phase contrast imaging with EBS

- new type of experiments: tomography in the DAC
- rheology of materials under extreme conditions
- highly detailed images of sample environment
- valuable information for extending limits of static pressure generation



ID06: courtesy of N. Dubrovinskaia

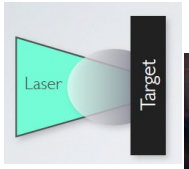


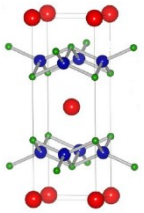


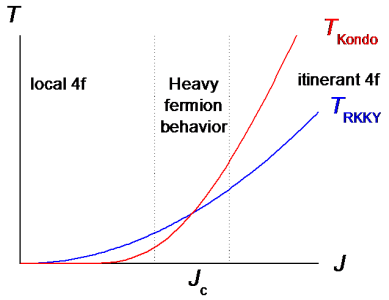
EBSL7 – Time Resolved and Extreme Conditions XAS

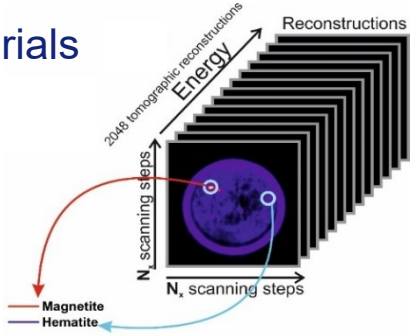
Time resolved & extreme conditions XAS (ID24_ED)

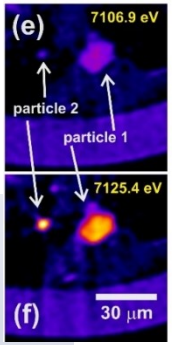
- extreme conditions for geophysics, planetary science, new materials
- magnetic response in the MegaGauss regime
- 3D spatially resolved chemical speciation

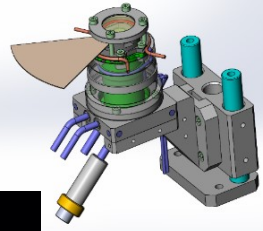


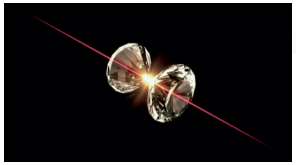






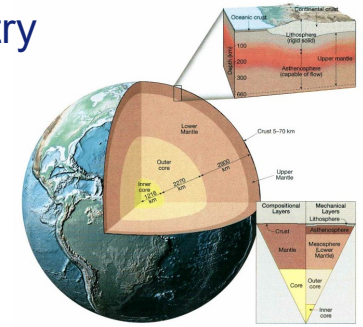


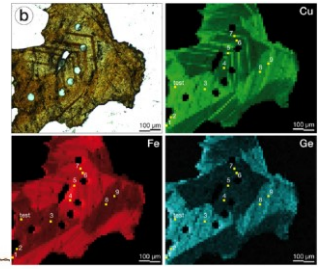


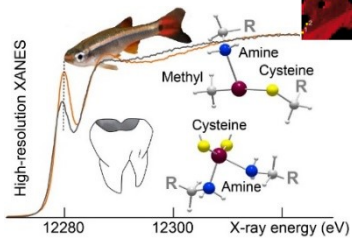


Sub-µm, high brilliance EXAFS (ID24_DCM)

- *in situ* and *operando* time resolved chemistry
- environmental science and mineralogy
- high pressure, earth and planetary science



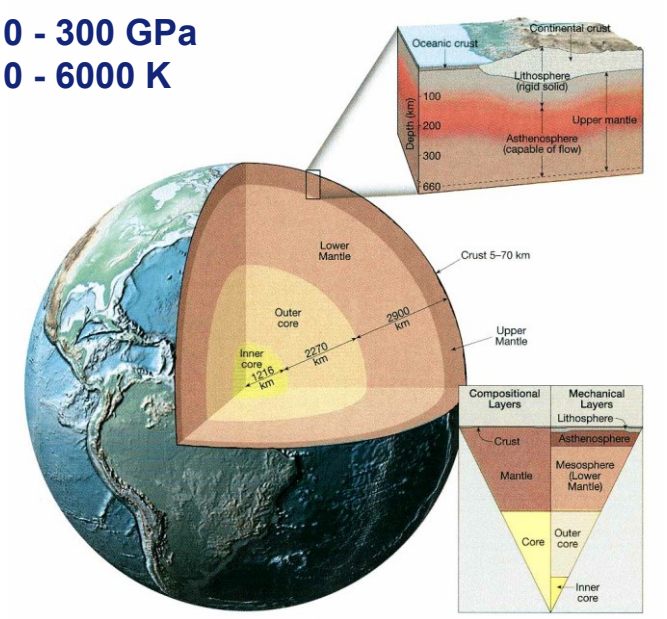


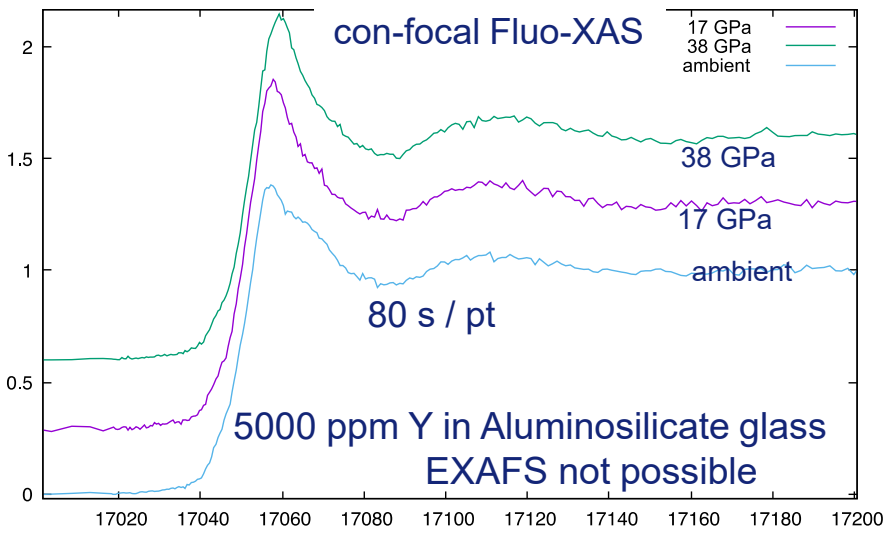


EBSL7 - EXTREME CONDITIONS XAS

Understanding the inaccessible Earth: interactions of minerals with aqueous fluids

0 - 300 GPa
0 - 6000 K





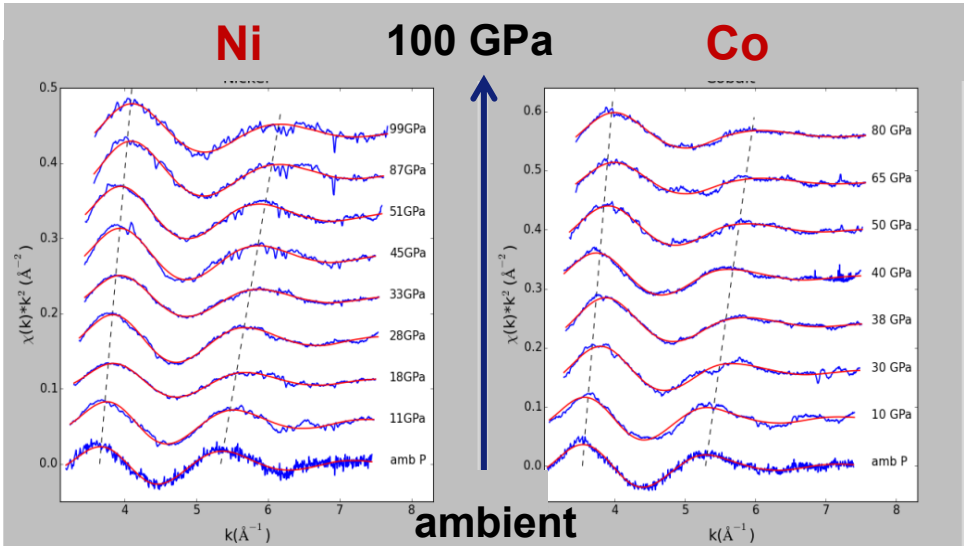
M. Wilke and A. Rosa (Potsdam, Germany), BM23

Challenges

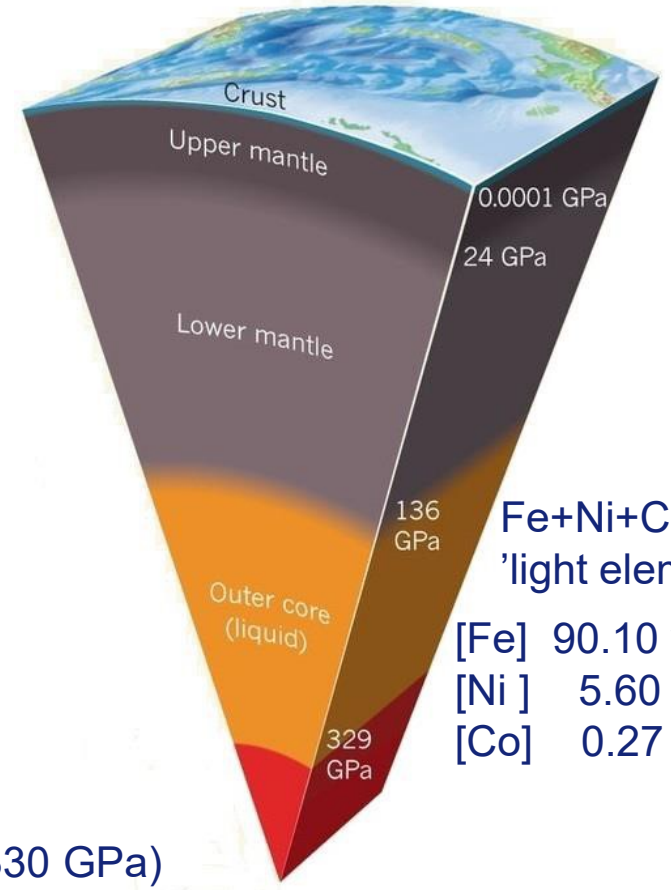
- μ-EXAFS
- naturally relevant concentrations: 1 - 100 ppm
- micrometric sample size at P > 100 GPa and T > 3000 K
- stability of sample during LASER-heating (**1s**)

EBSL7 - EXTREME CONDITIONS XAS

Understanding the inaccessible Earth: Local structure of melts at the Inner Core Boundary



S. Boccato, PhD thesis, ID24



Fe+Ni+Co+
'light elements'

[Fe] 90.10 wt %
[Ni] 5.60 wt%
[Co] 0.27 wt %

Challenges

- **1 μm** size spot to reach Inner Core boundary (330 GPa)
- fast acquisition (**1s**) for pulsed laser heating in the DAC (> 6000 K)
- extended XAFS w/ high S/N on melts with low absorber content

CONCLUSIONS

- High Pressure Research is, since 25 years, a very important part of the scientific program at ESRF
- Static compression methods are now offered on more than half of the beamlines.
- ESRF offers highly specialized beamlines for studies of matter at extreme P and T, allowing to probe long range order, local environment, electronic, magnetic vibrational properties, charge ordering, …
- We are observing a trend from our user community to push towards dynamic compression, to go to higher P, T values & to start exploring the time scale of high pressure phenomena.
- Dynamic compression methods are being developed on several beamlines, including ID24 (XAS), ID19 (XRI) and ID09 (XRD).
- The EBS will offer orders of magnitude higher flux and brilliance, and will allow us to address outstanding questions in high pressure research that are out of reach today.

ACKNOWLEDGMENTS

M. Wulff (ID09)

A. Chumakov (ID18)

M. Olbinado, A. Rack (ID19)

C. Sahle (ID20)

O. Mathon, A. Rosa, N. Sevelin, R. Torchio (BM23&ID24)

G. Garbarino, M. Mezouar, V. Svitlyk (ID27)

THANKS

Thank you for your attention