ESRF | The European Synchrotron

Frontiers of High Pressure Research at the European Synchrotron Radiation Facility

Sakura Pascarelli

European Synchrotron Radiation Facility

sakura@esrf.fr

OUTLINE

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

OUTLINE

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

HIGH PRESSURE AT ESRF TODAY

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

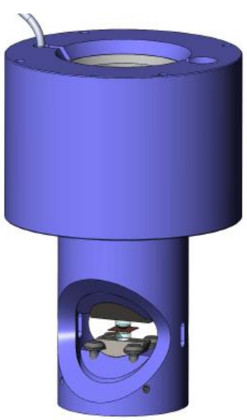
ID18: Nuclear Resonance Scattering - Magnetism, Phonons

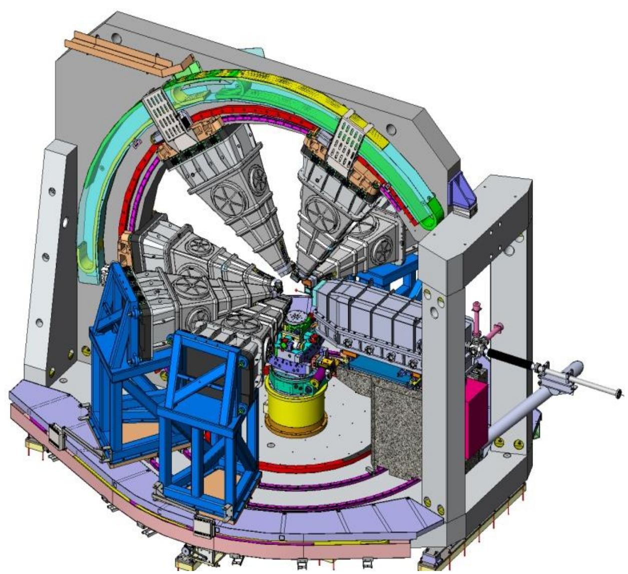
ID20: Resonant Inelastic X-ray Scattering - Electronic and Magnetic Structure

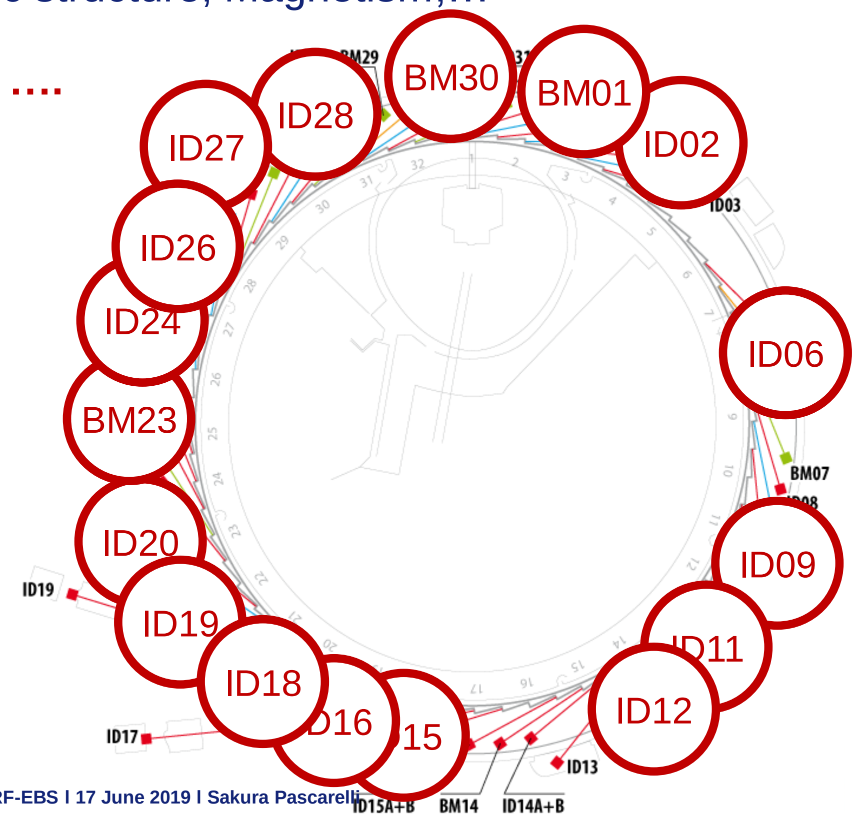
ID28: Inelastic X-ray Scattering, Diffuse Scattering – Phonons

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

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



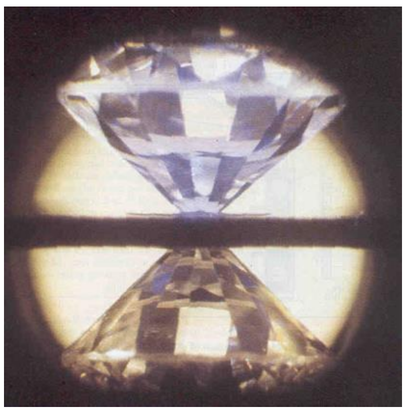


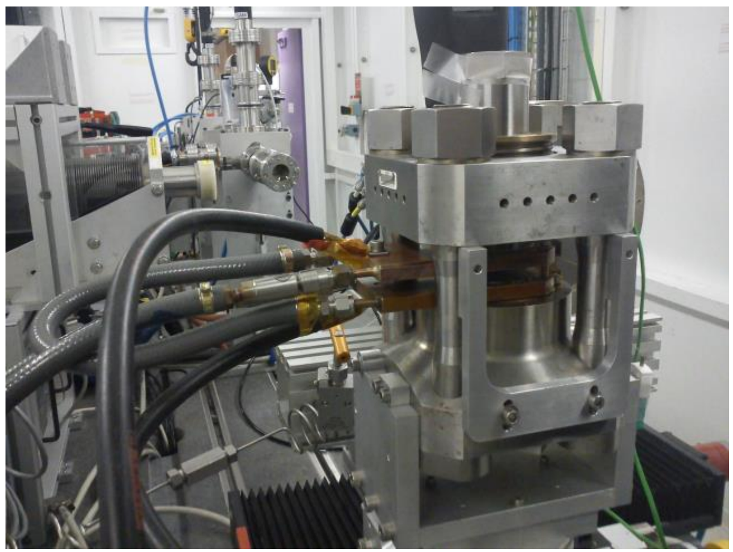


ID20: 72 Analysers and Panoramic DAC

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





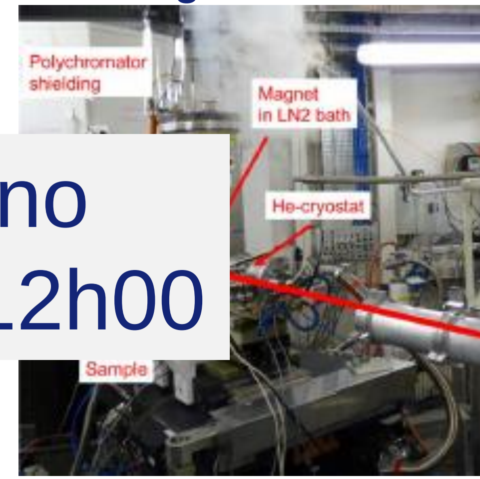
Wilson Crichton
Wednesday 19 June 9h45

Static High Pressure Facilities

Static field - 8 T

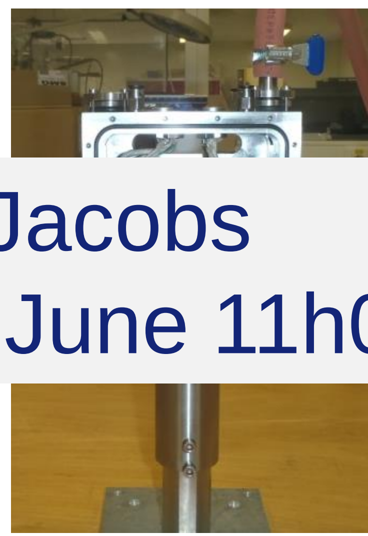
Gaston Garbarino
Tuesday 18 June 12h00

Pulsed Magnetic field - 30 T



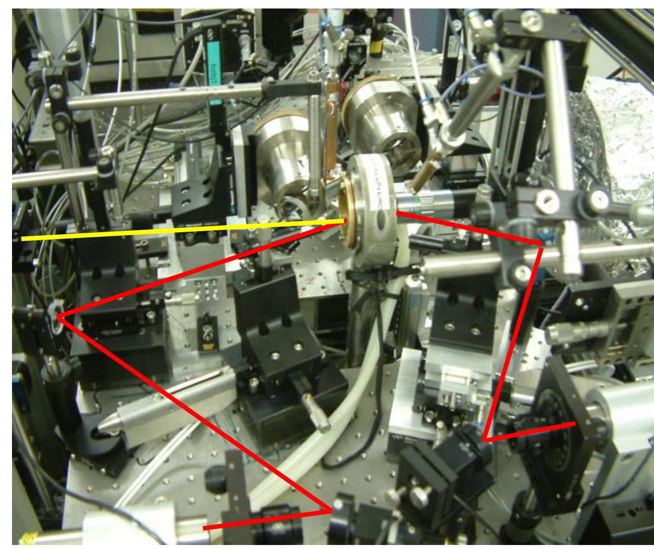
Low T - 2 K

Resistive heating - 1300 K



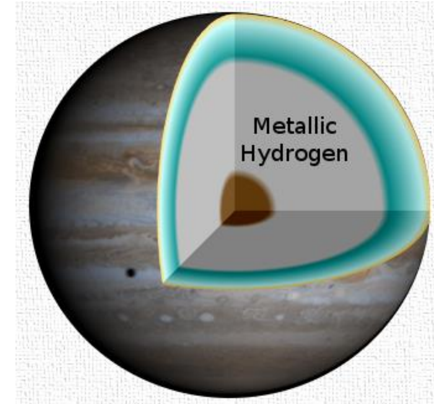
Jeroen Jacobs
Thursday 20 June 11h00

Laser heating - 5000 K

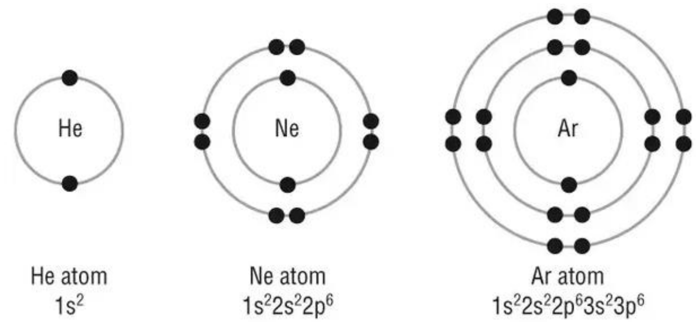


Some outstanding questions in high pressure research

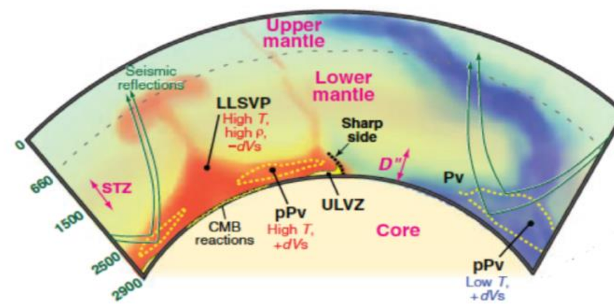
1. The quest for metallic solid Hydrogen



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

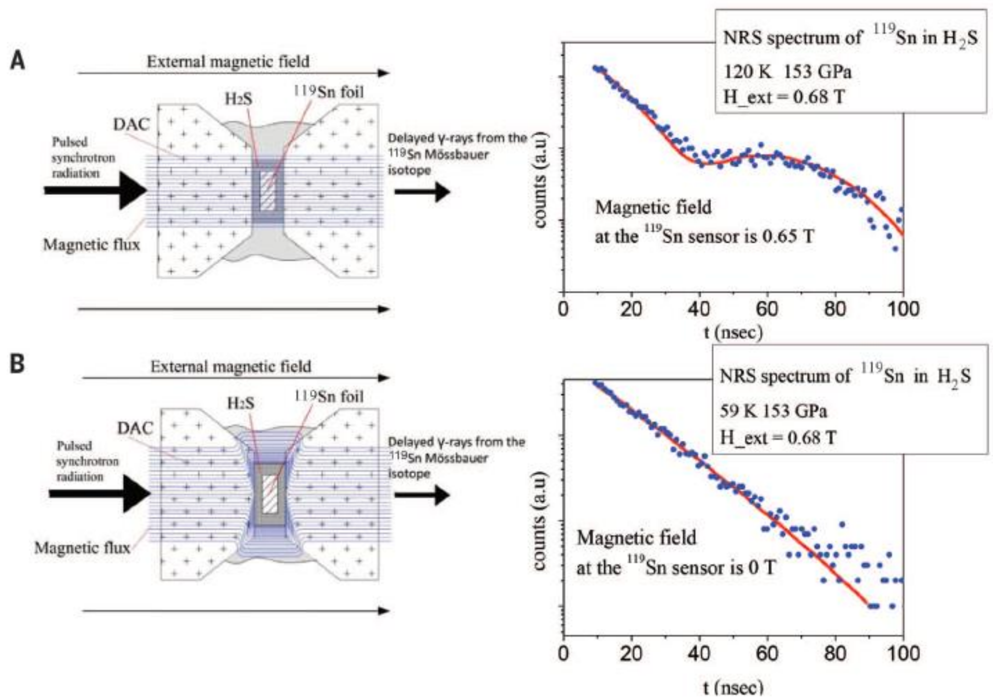
REPORTS

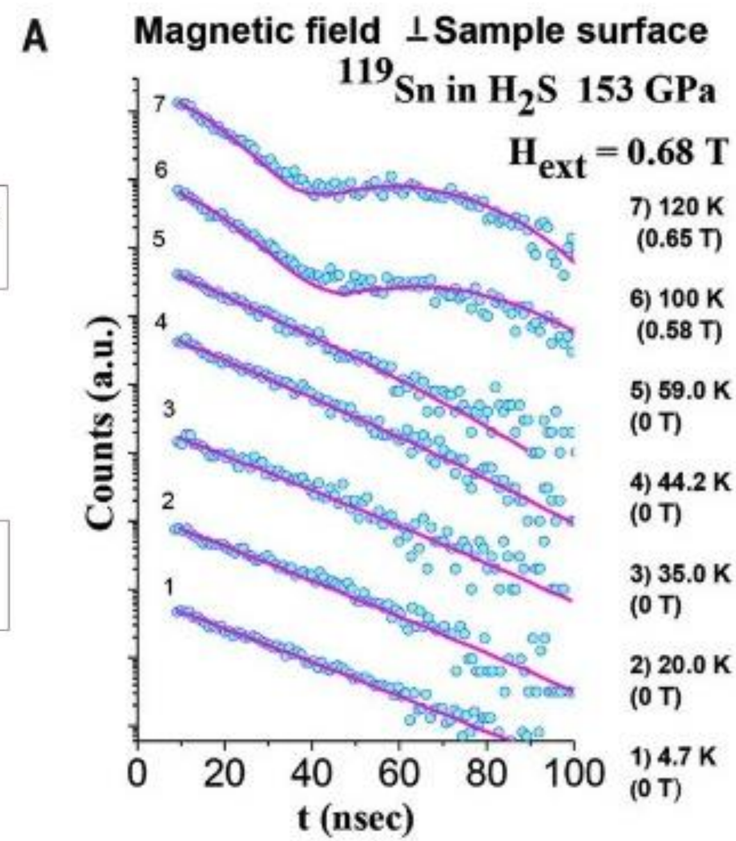
SUPERCONDUCTIVITY

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,[3] Igor Lyubutin,[3] 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

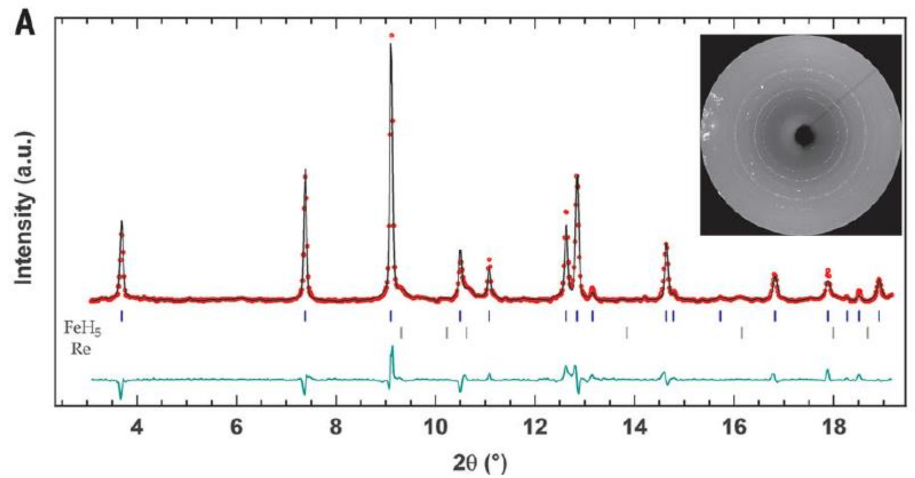
ID27

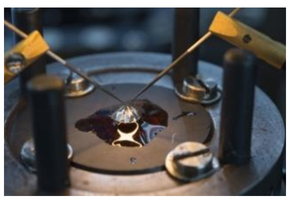
RESEARCH

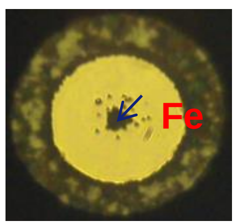
HIGH-PRESSURE PHYSICS

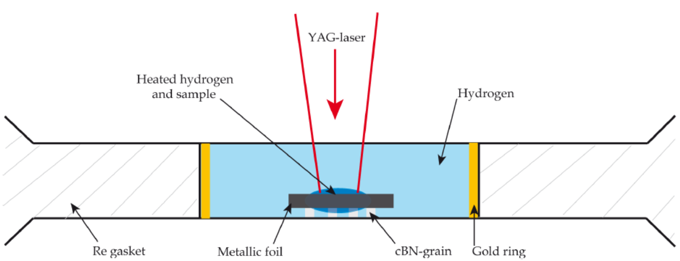
Synthesis of FeH_5: A layered structure with atomic hydrogen slabs

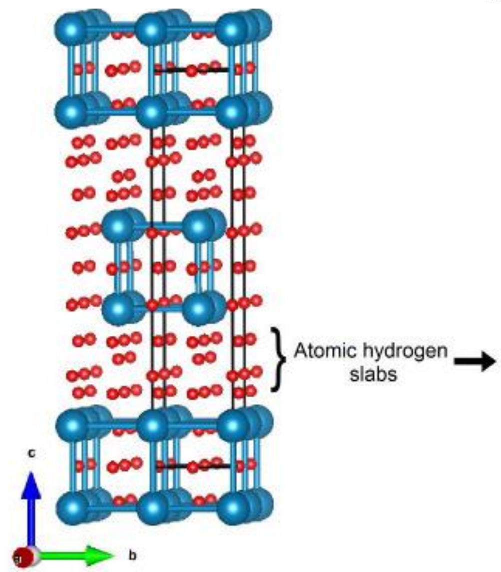
C. M. Pépin,[1,2*] G. Geneste,[1] A. Dewaele,[1] M. Mezouar,[3] P. Loubeyre[1*]

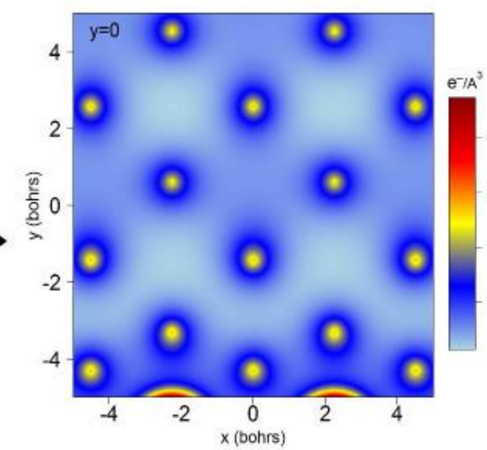












Pépin Science 2017

- 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

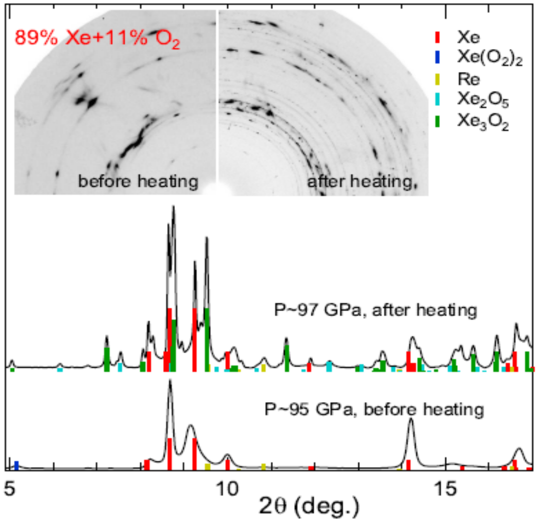
Synthesis and stability of xenon oxides Xe_2O_5 and Xe_3O_2 under pressure

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]

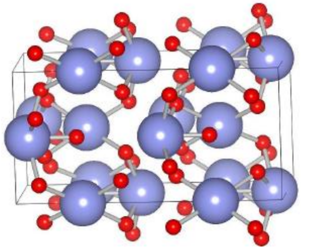
Xe [Kr] $4d^{10}5s^2\ 5p^6$

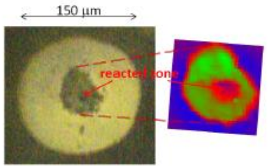
nature chemistry AR
PUBLISHED ONLINE: 11 NOVEMBER 2012 | DOI: 10.10

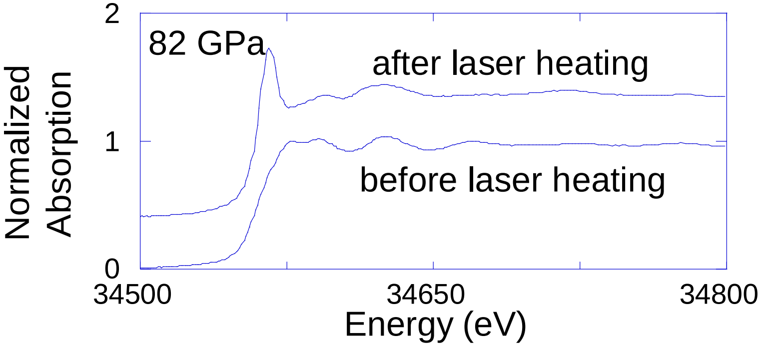
Stability of xenon oxides at high pressures

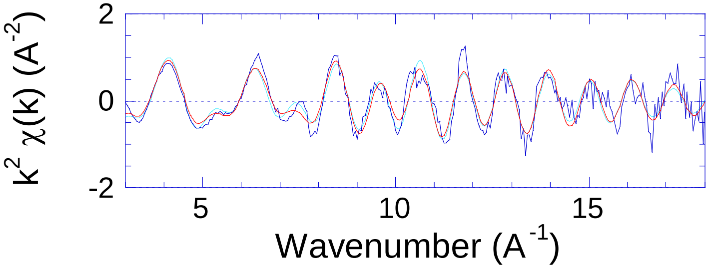


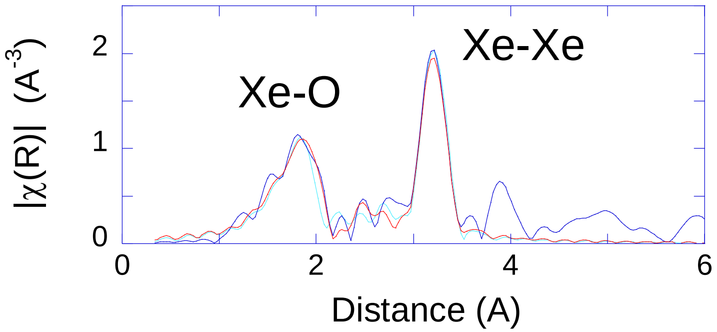
Xe_2O_5
P4/ncc space group











Dewaele Nature Chemistry 2016

STRUCTURAL CHANGES IN SiO_2 DOWN TO THE CORE MANTLE BOUNDARY

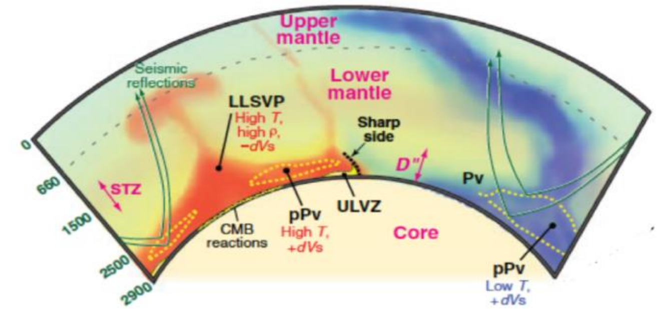
ID20

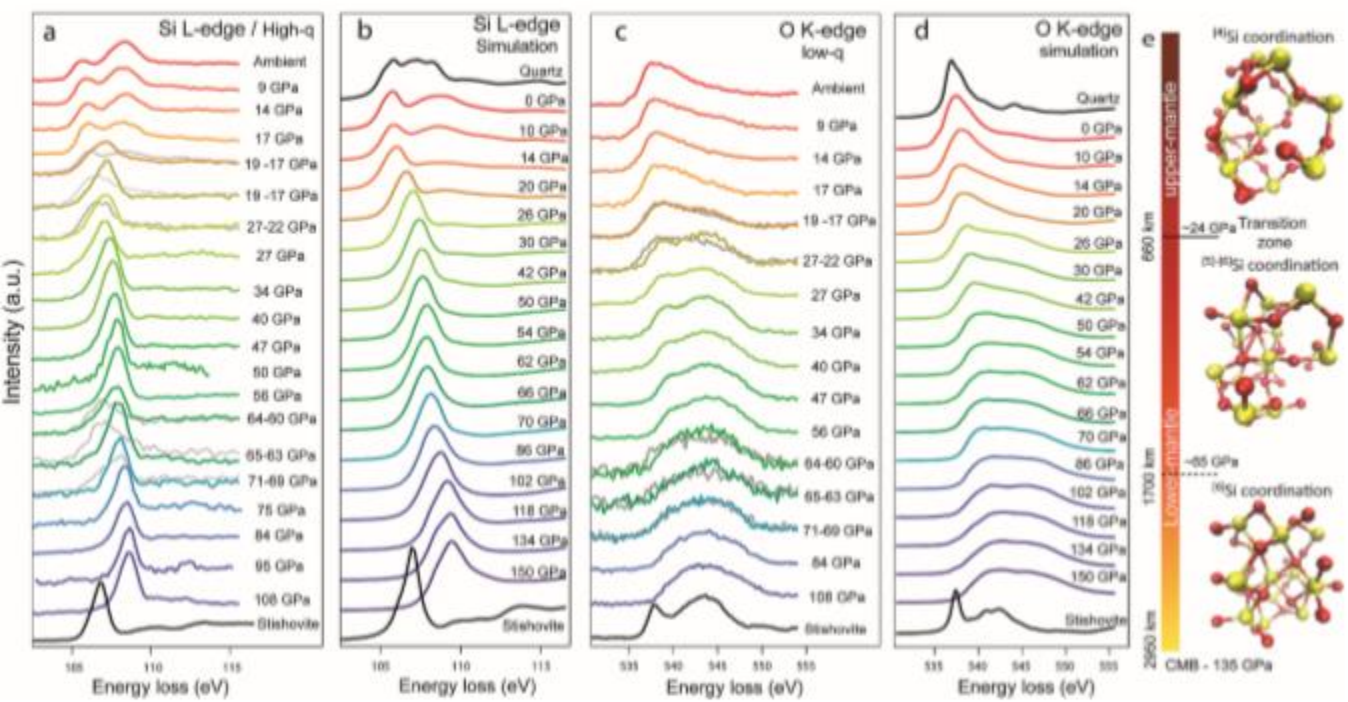
Geochemical Perspectives Letters

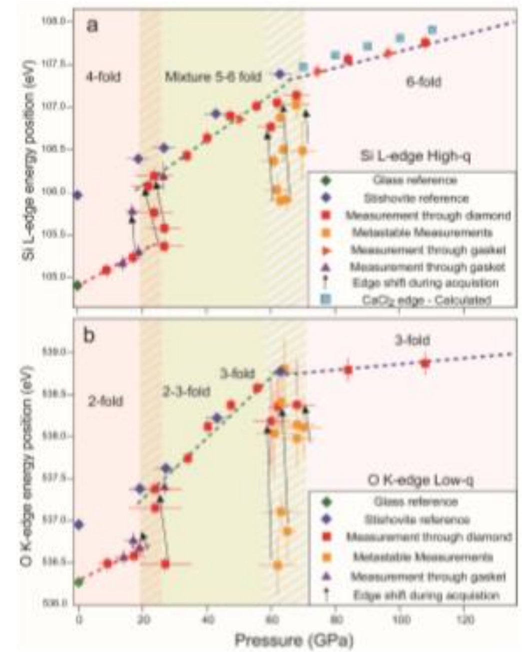
© 2019 The Authors
Published by the European Association of Geochemistry

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]



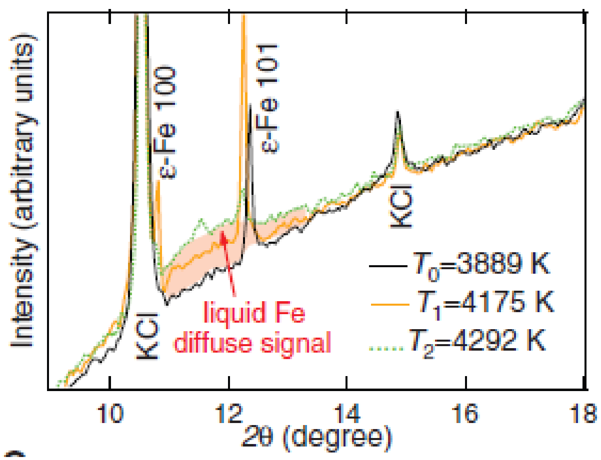




Petitgirard Geochemical Research Letters 2018

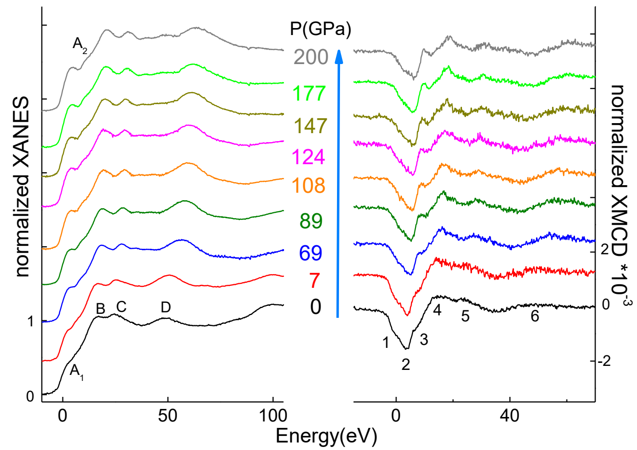
STATIC COMPRESSION AT SYNCHROTRONS TODAY

XRD 1.3 Mbar 4300K



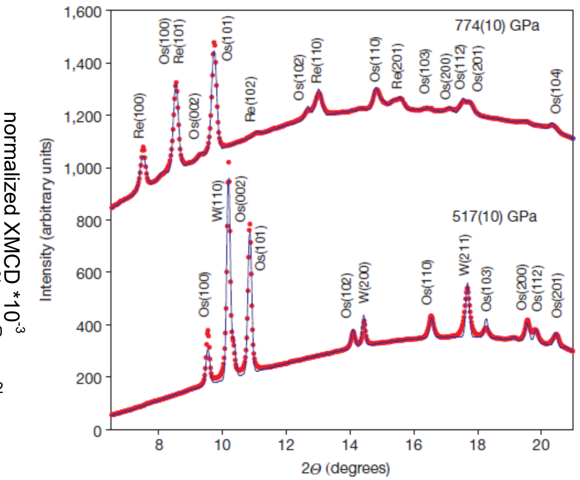
Anzellini Science 2013

XMCD 2 Mbar



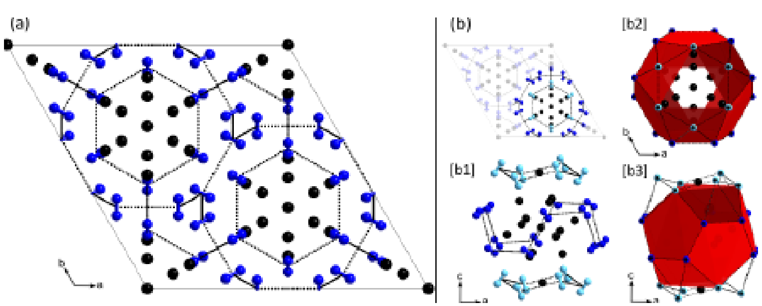
Torchio PRL 2011

XRD 7 Mbar



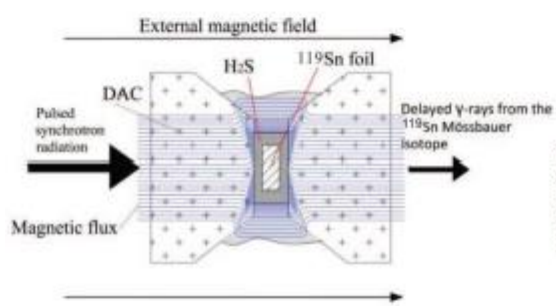
Dubrovinsky Nature 2015

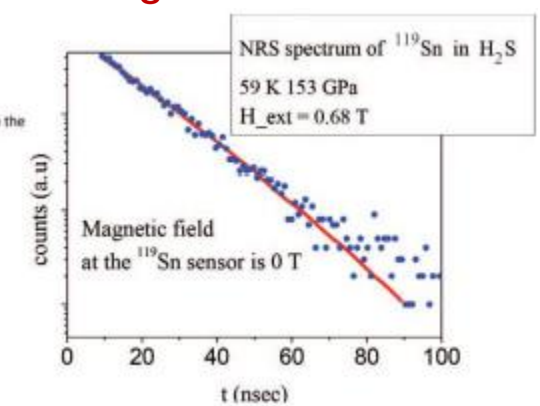
Single Crystal XRD



Spaulding Nature Comm. 2014

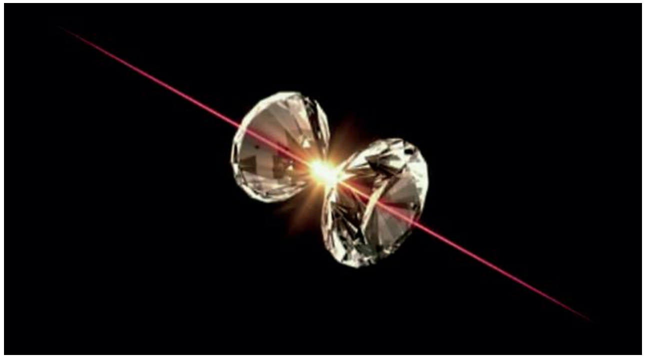
Nuclear Resonance Scattering

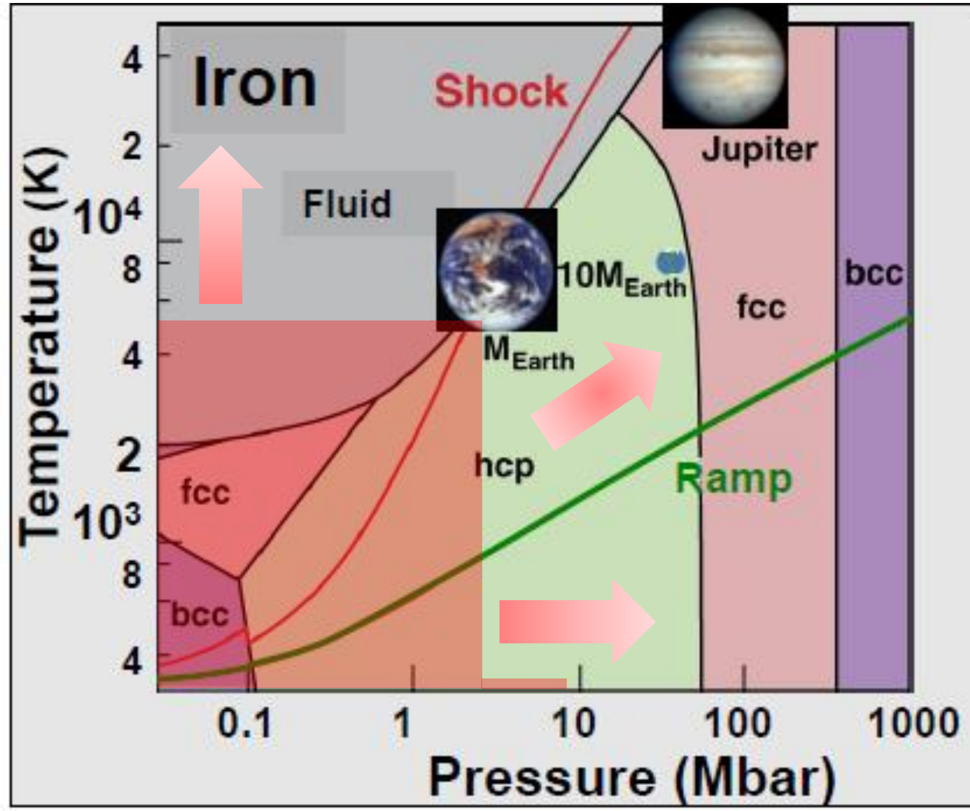




Troyan Science 2016

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 ?

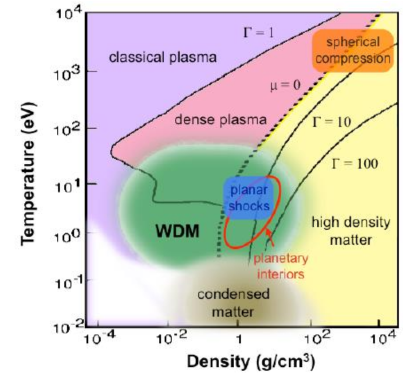
Can we create and probe WDM at the synchrotron, with data quality as "at ambient" ?

OUTLINE

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

PUSHING THE FRONTIERS OF HIGH PRESSURE RESEARCH

❑ Go more extreme → TPa & eV

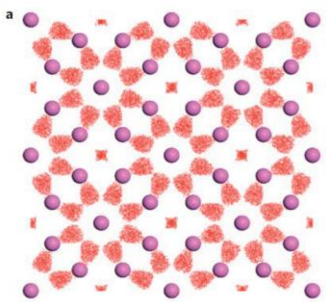


Conditions beyond those existing in our planet → Input for planetary models

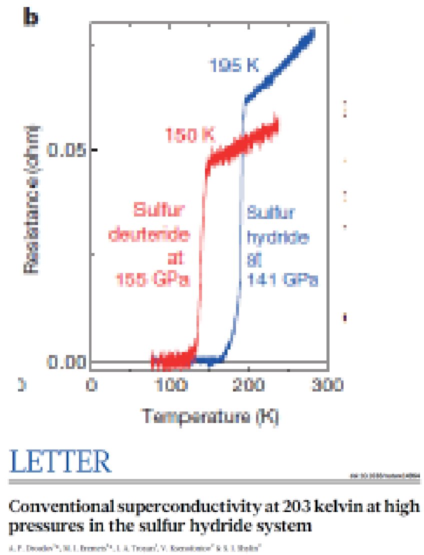
Synthesis of novel materials

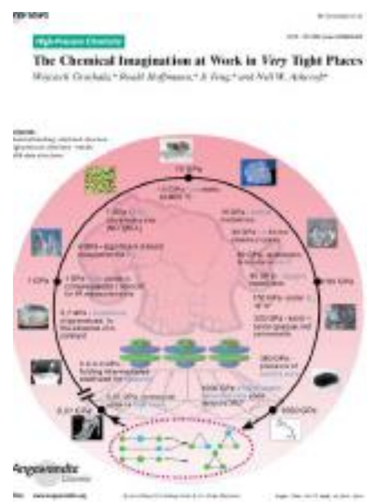
Reveal new physical chemistry

Al @ 1TPa



Pickard and Needs 2010





Particle ejection

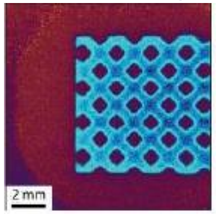
ENSMA Poitiers

❑ Explore the time scale of high pressure phenomena → ns

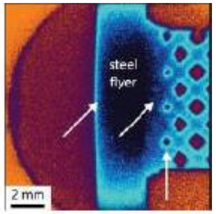
Dynamic behavior of matter and materials under high strain rates

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

Heterogeneous media



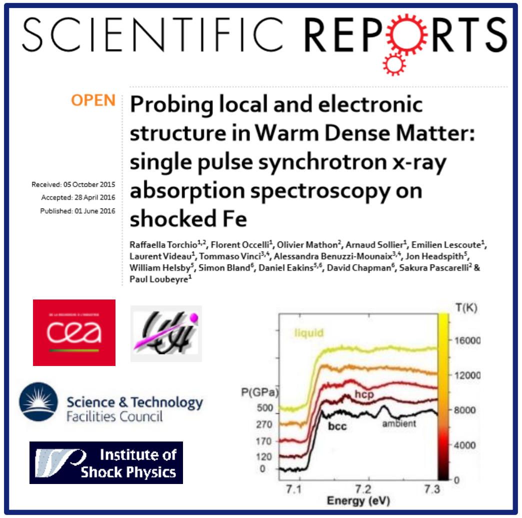
Pre-shot



2.4 µs

ISP Imperial London

A SYNERGETIC APPROACH TO DYNAMIC COMPRESSION AT ESRF


SCIENTIFIC REPORTS

OPEN

Probing local and electronic structure in Warm Dense Matter: single pulse synchrotron x-ray absorption spectroscopy on shocked Fe

Received: 05 October 2015
Accepted: 28 April 2016
Published: 01 June 2016

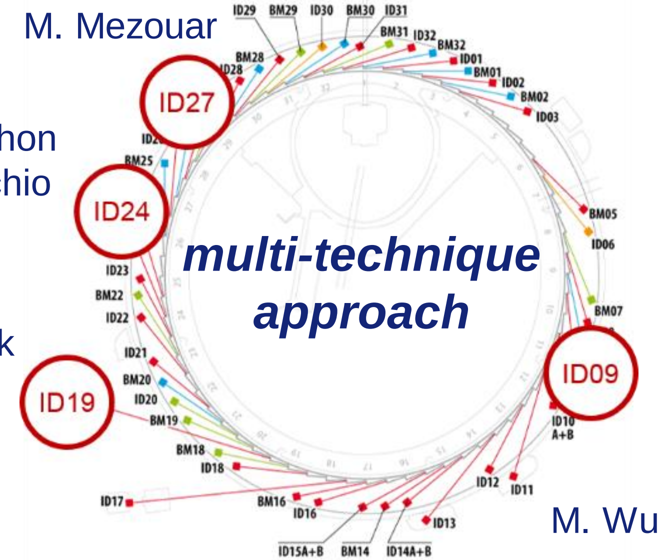
Raffaella Torchio[1,2], Florent Occelli[1], Olivier Mathon[2], Arnaud Sollier[1], Emilien Lescoute[1], Laurent Videau[1], Tommaso Vinci[3,4], Alessandra Benuzzi-Mounaix[3,4], Jon Headspith[5], William Helsby[5], Simon Bland[6], Daniel Eakins[5,6], David Chapman[6], Sakura Pascarelli[2] & Paul Loubeyre[1]

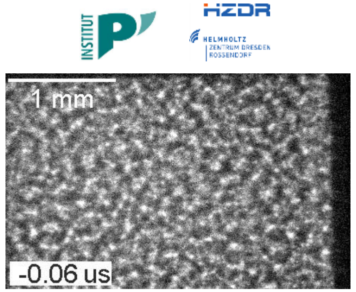
M. Mezouar

O. Mathon
R. Torchio

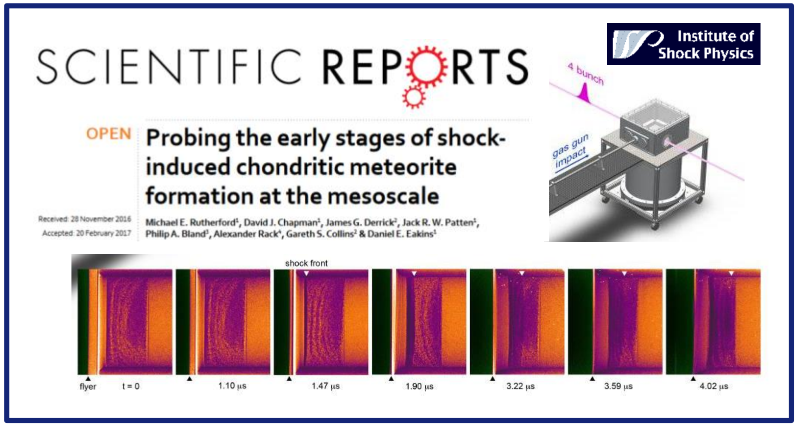
A. Rack

M. Wulff





Olbinado J. Phys D 2018

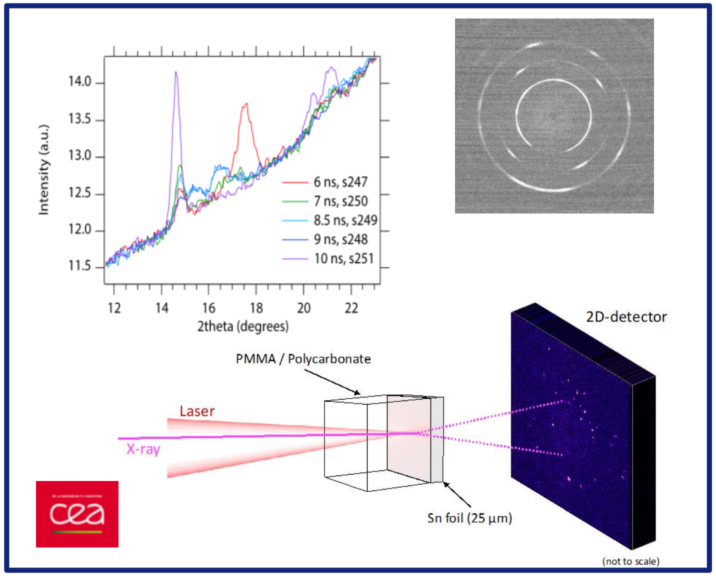

SCIENTIFIC REPORTS

OPEN

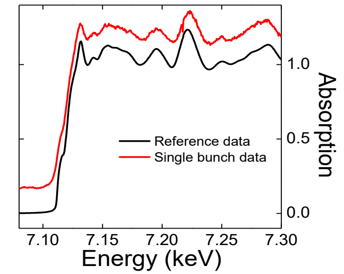
Probing the early stages of shock-induced chondritic meteorite formation at the mesoscale

Received: 28 November 2016
Accepted: 20 February 2017

Michael E. Rutherford[1], David J. Chapman[1], James G. Derrick[1], Jack R. W. Patten[1], Philip A. Bland[3], Alexander Rack[4], Gareth S. Collins[2] & Daniel E. Eakins[1]



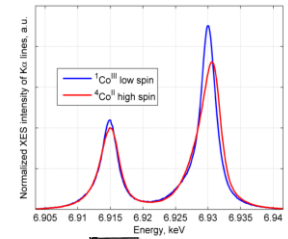
THE HIGH POWER LASER FACILITY AT THE ESRF

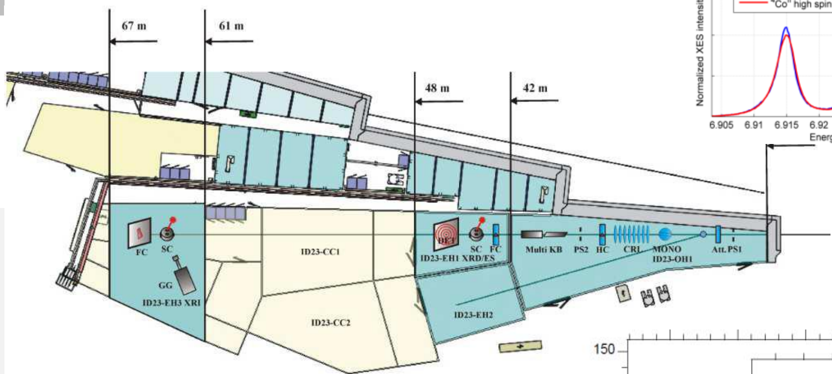


HPLF-I (2018-2021)

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

In Construction

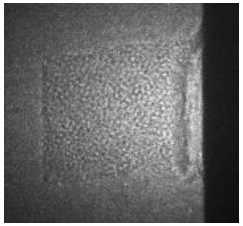


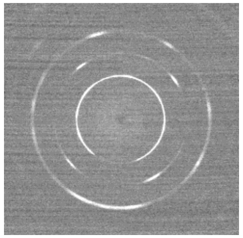


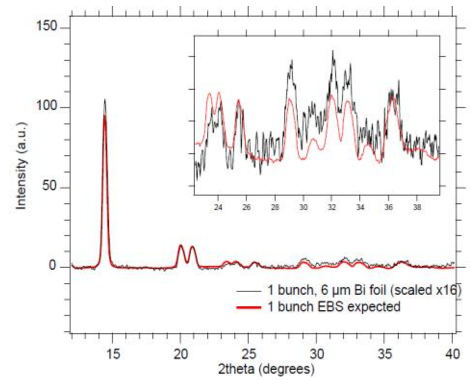
HPLF-II (from 2023)

Extend to XRD, XRI, XES on ID23

Laser upgrade





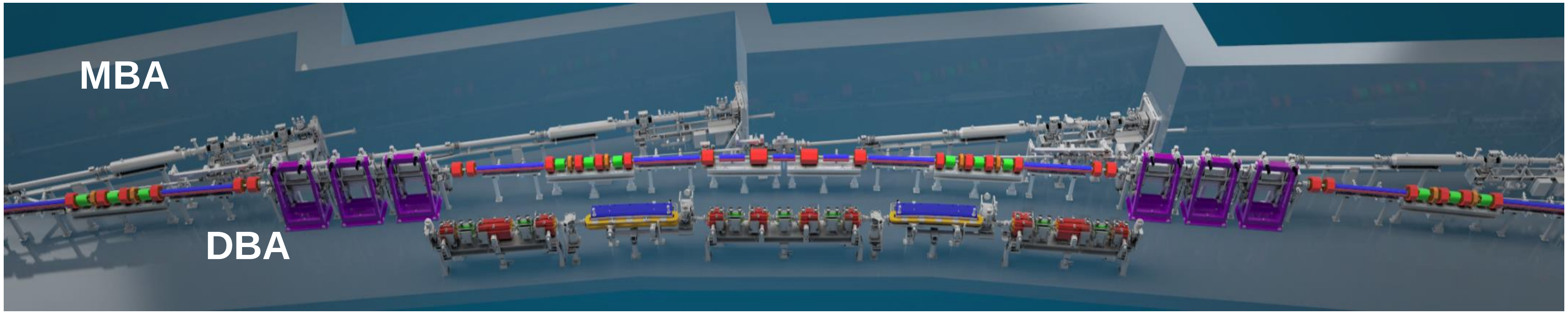


EBS Beamline program

OUTLINE

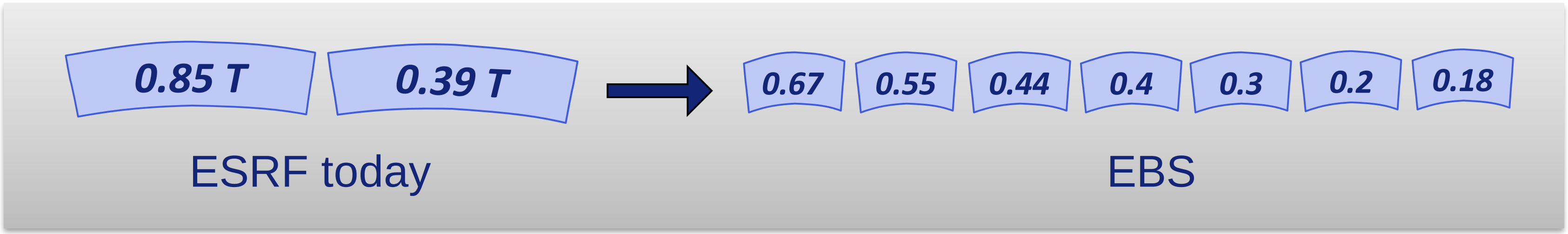
- ❑ Static High Pressure Research: status and trends
- ❑ Dynamic Compression: recent developments, future plans
- ❑ The EBS
- ❑ Extreme Conditions Science at EBS

DIFFRACTION LIMITED STORAGE RINGS



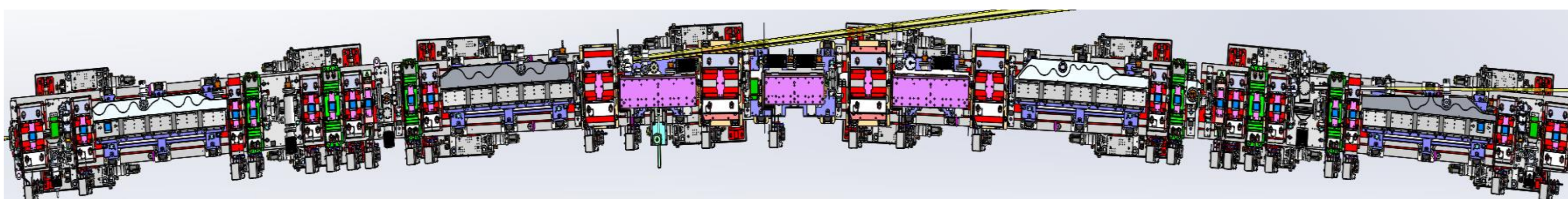
GOAL: REDUCE EQUILIBRIUM HORIZONTAL EMITTANCE

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



EBS lattice

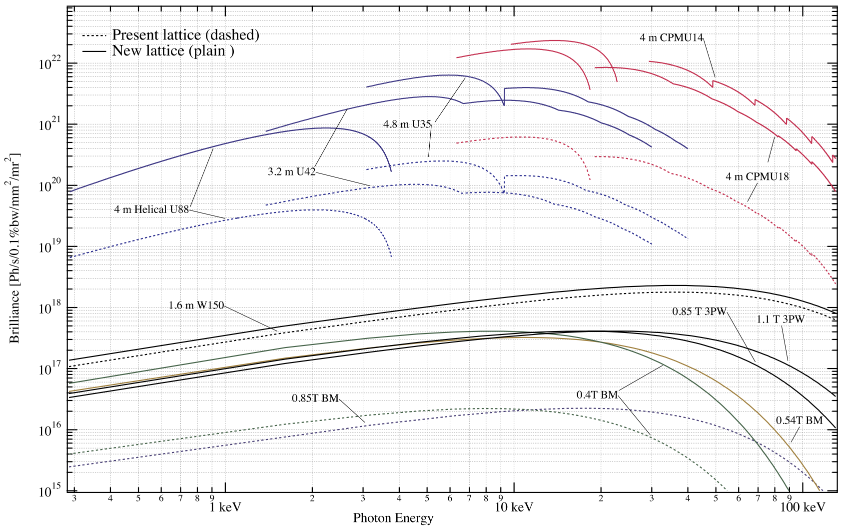
- Hybrid 7 Bend Achromat = (4 dipoles + 3 dipole-quad + 24 quad., sext., oct.) per cell
- ID length = 5 m

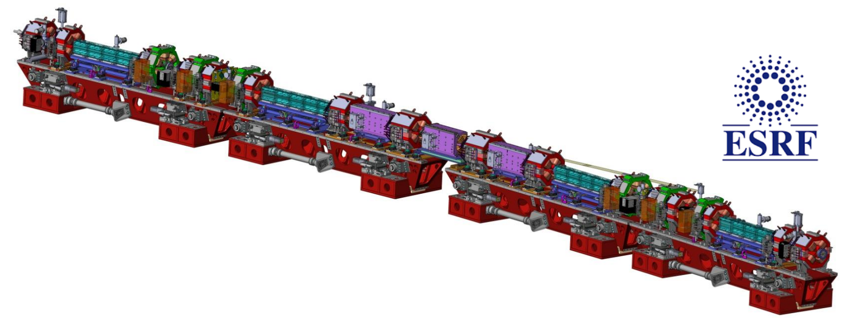


31 magnets per cell instead of currently 17

32 cells (arcs) with 4 girders each

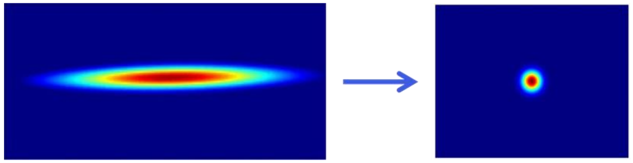
Brilliance



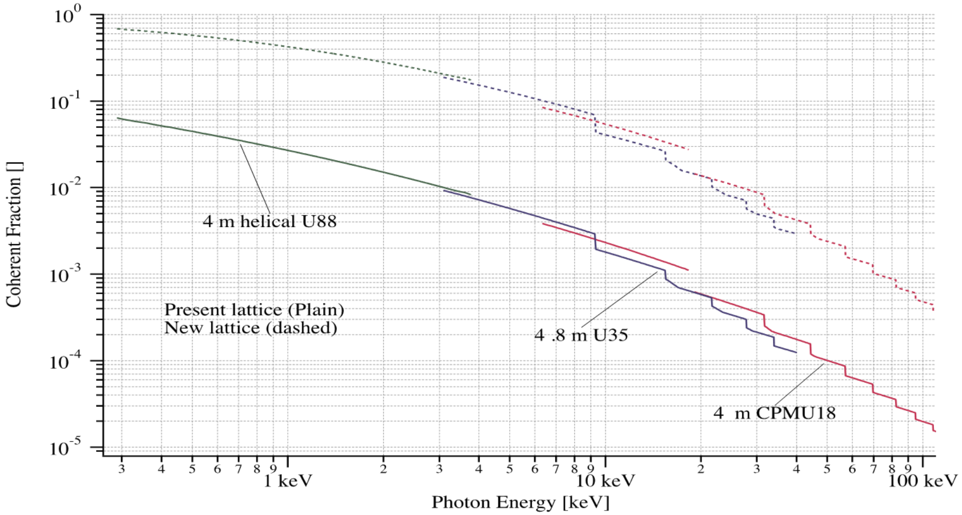


Horizontal emittance

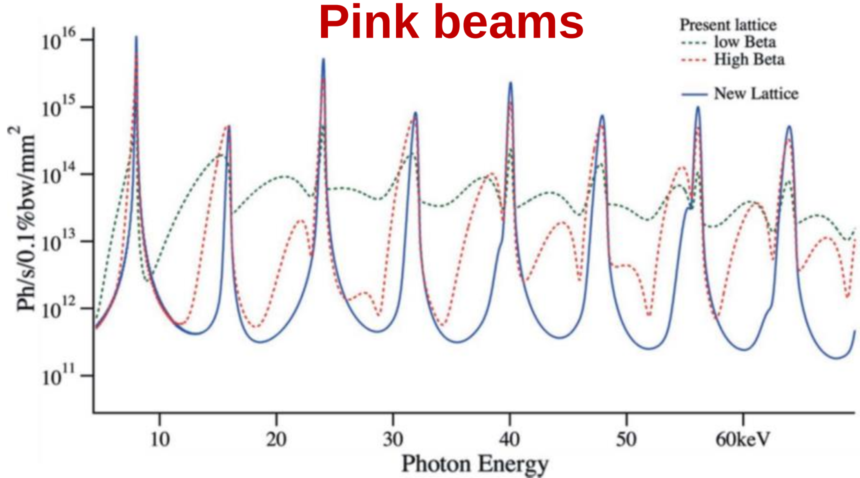
ε_x= 4 nm ε_x= 0.15 nm



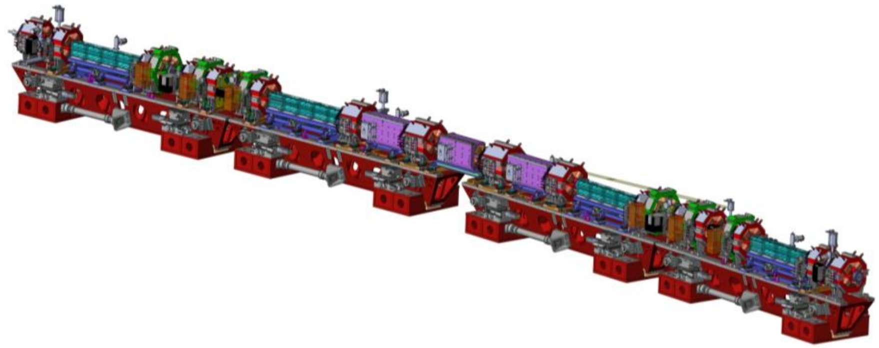
Coherent Fraction



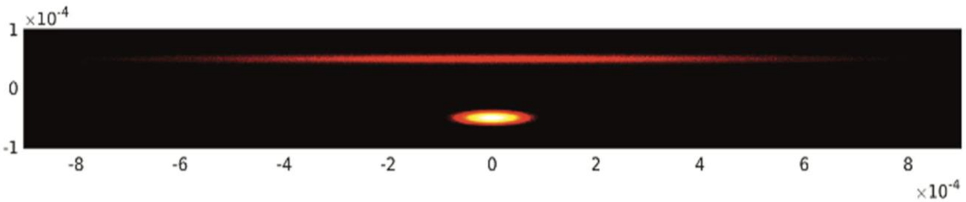
Pink beams



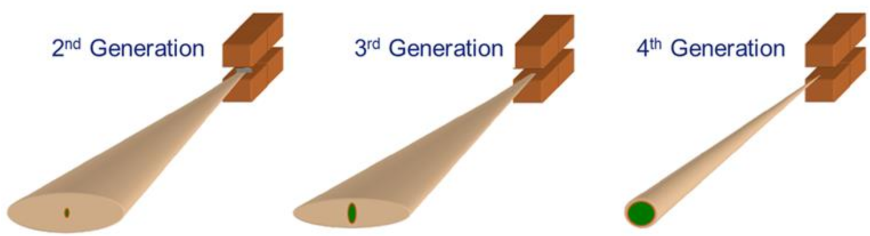
THE ESRF EXTREMELY BRILLIANT SOURCE (ESRF-EBS)

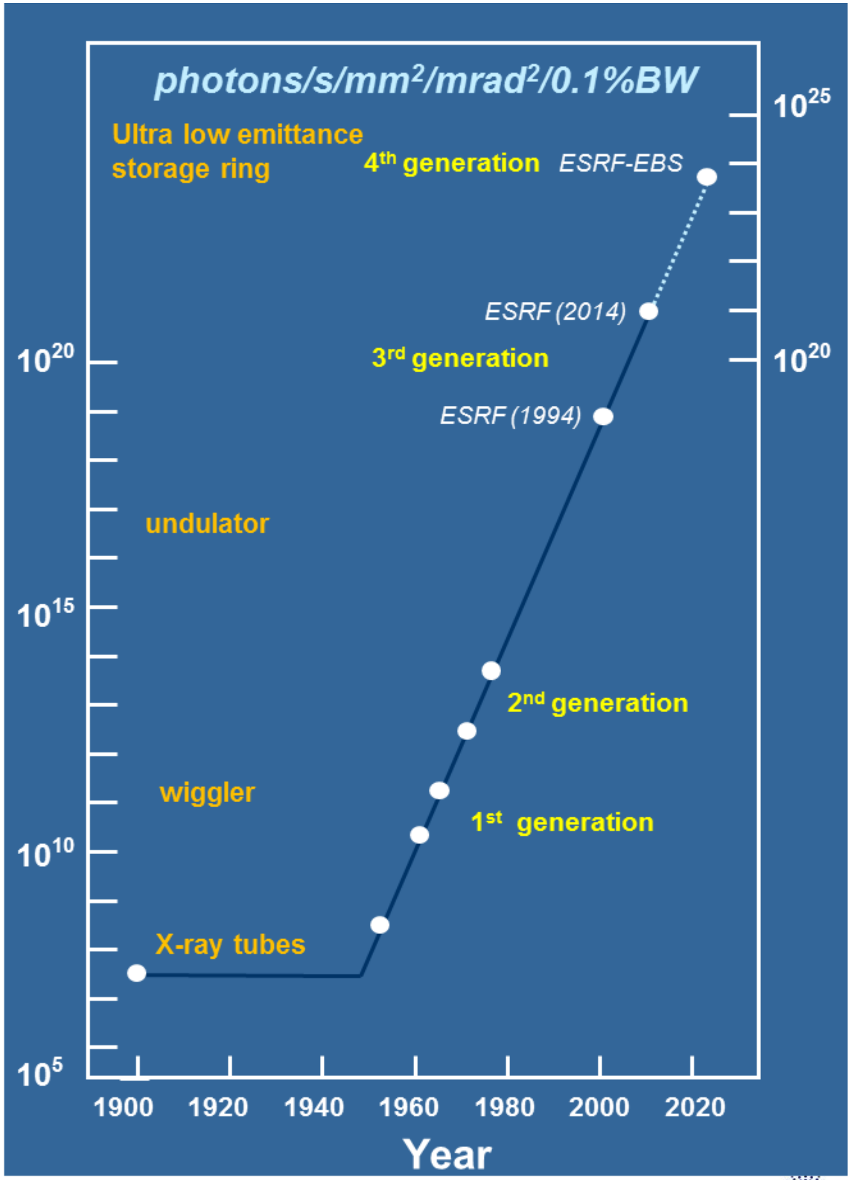


before and after EBS



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



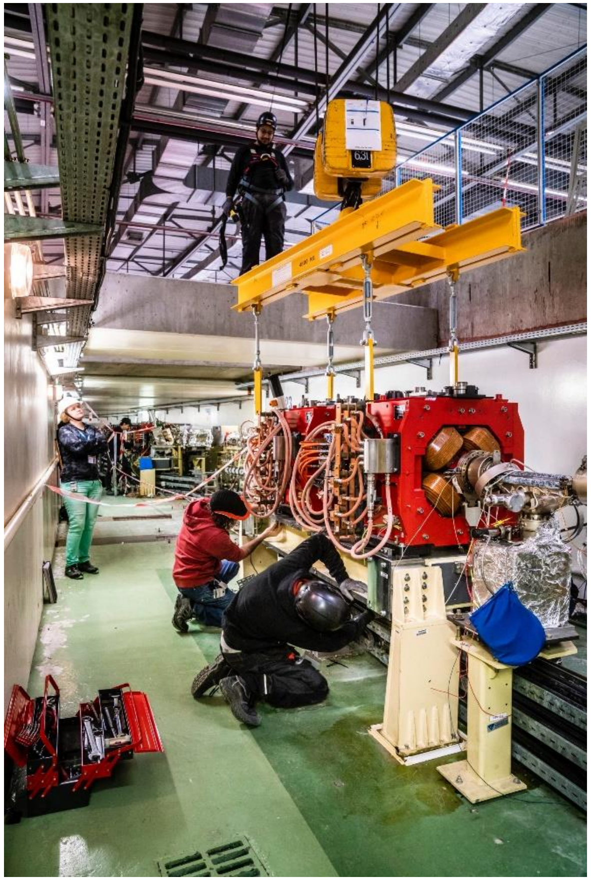


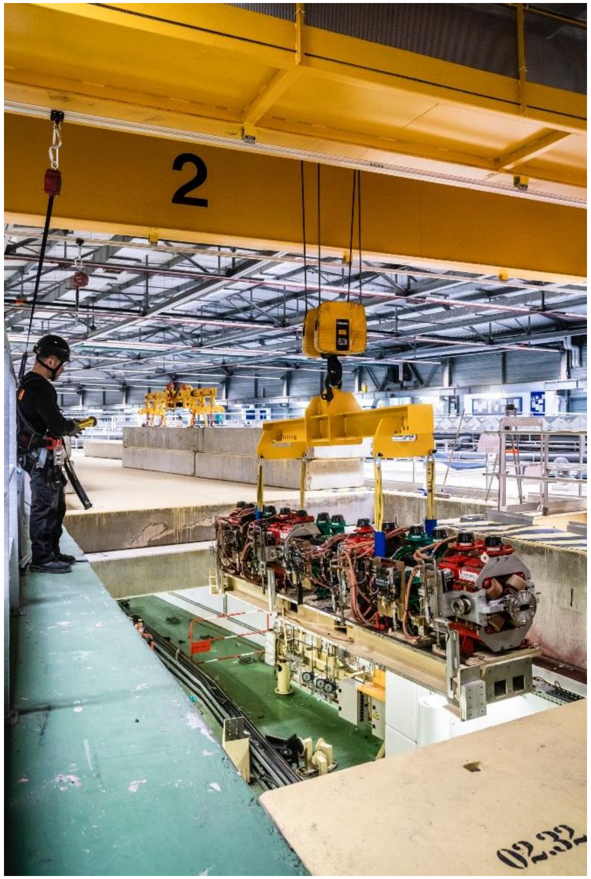
THE EBS PROGRAMME

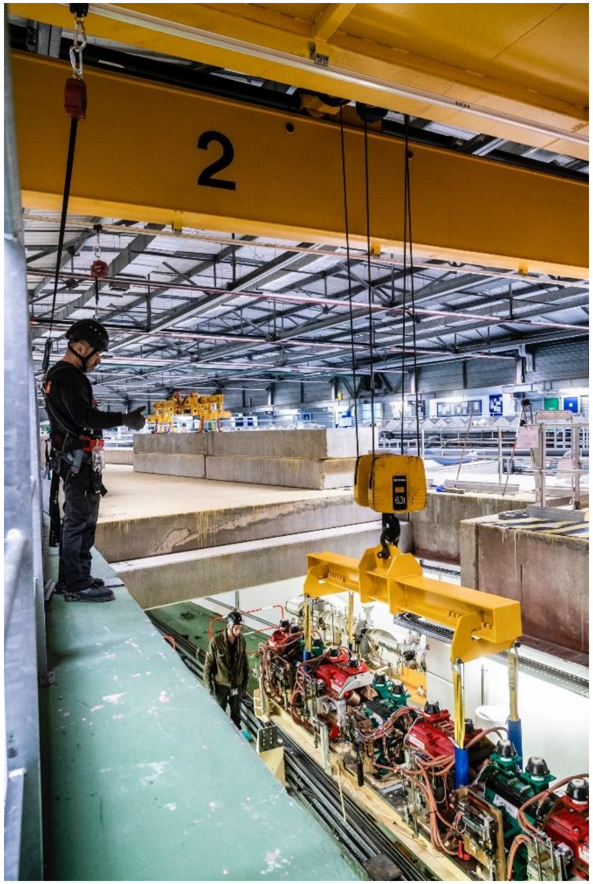
SCHEDULE:

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







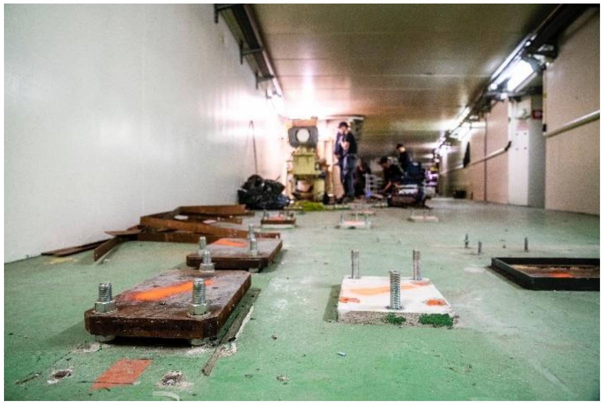
TEMPORARY STORAGE OF THE ESRF HISTORICAL RING

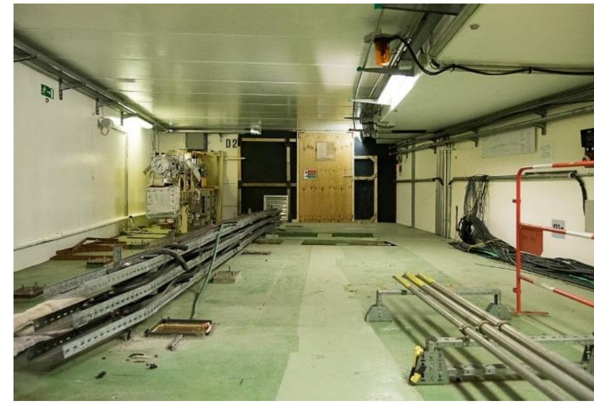




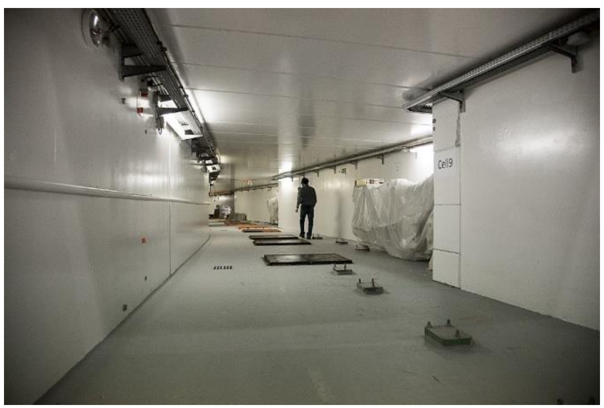

1700 TONS OF MATERIAL AND 200 KM CABLES

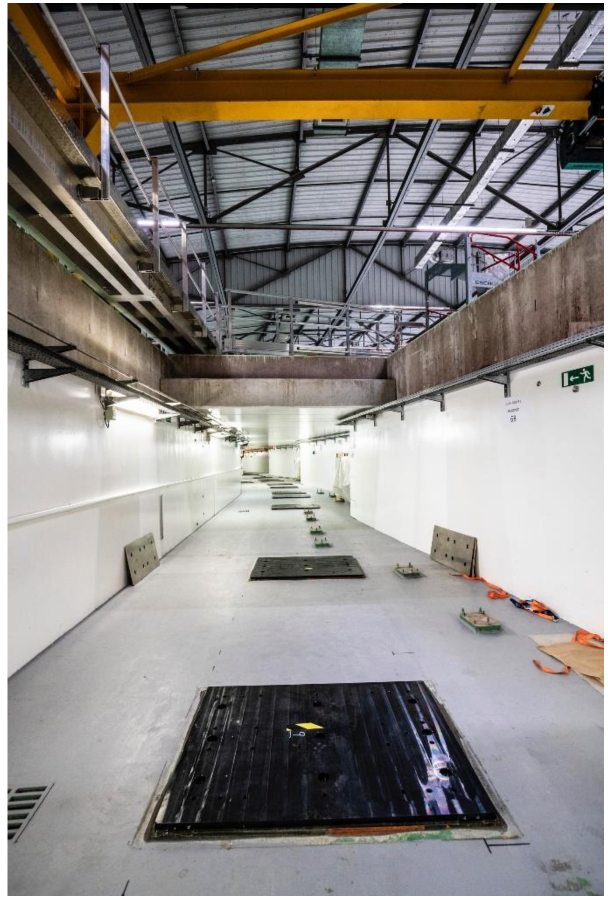
Civil work and preparation of the tunnel



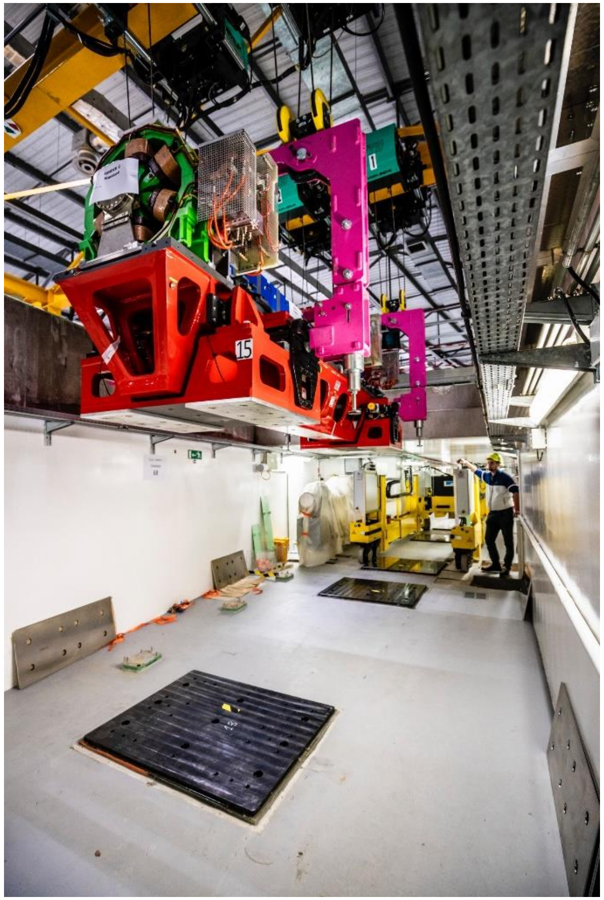


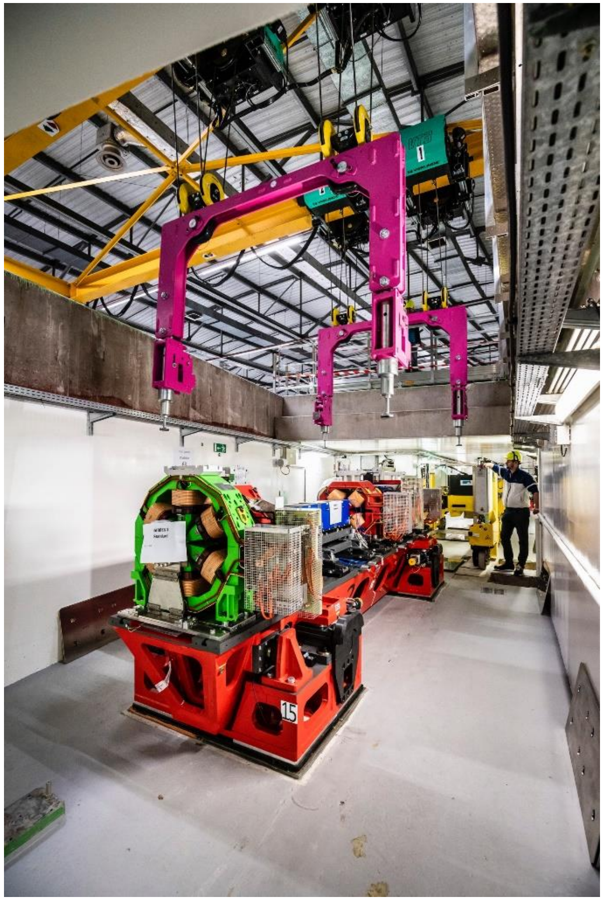
CIVIL WORK AND PREPARATION OF THE TUNNEL





INSTALLATION OF THE NEW GIRDERS

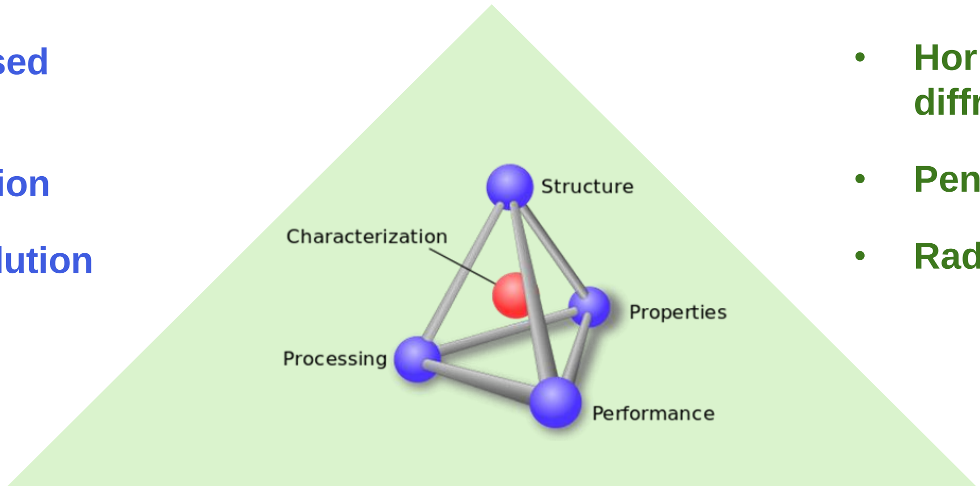




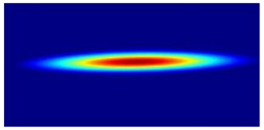
THE EBS STORAGE RING STARTS TO TAKE ITS SHAPE

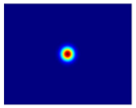
EBS – SCIENCE CASE(S) IN BRIEF

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



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





FROM AVERAGED TO SINGLE OBJECT INFORMATION

+

DYNAMICS

+

REAL SYSTEMS

EXPERIMENTAL PROGRAM OVERVIEW: 3 INTERCONNECTED SUB-PROGRAMS

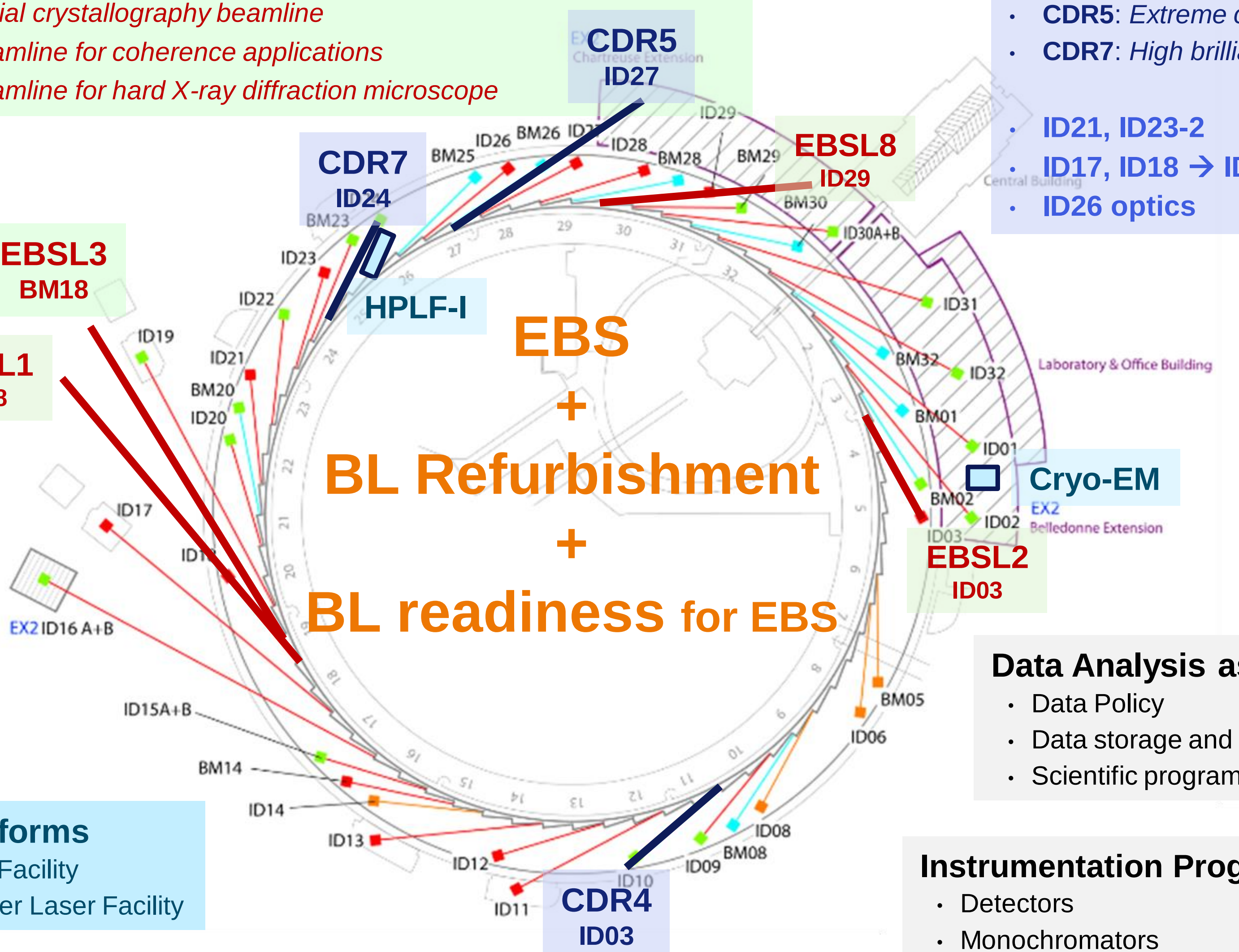
EBS Beamlines

- **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

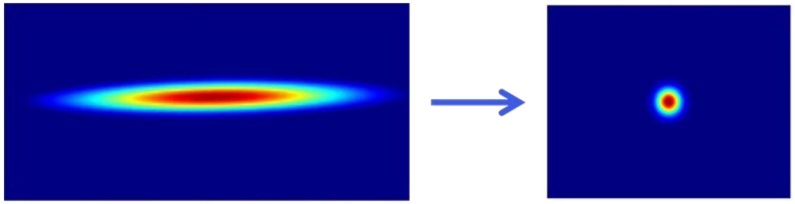
OUTLINE

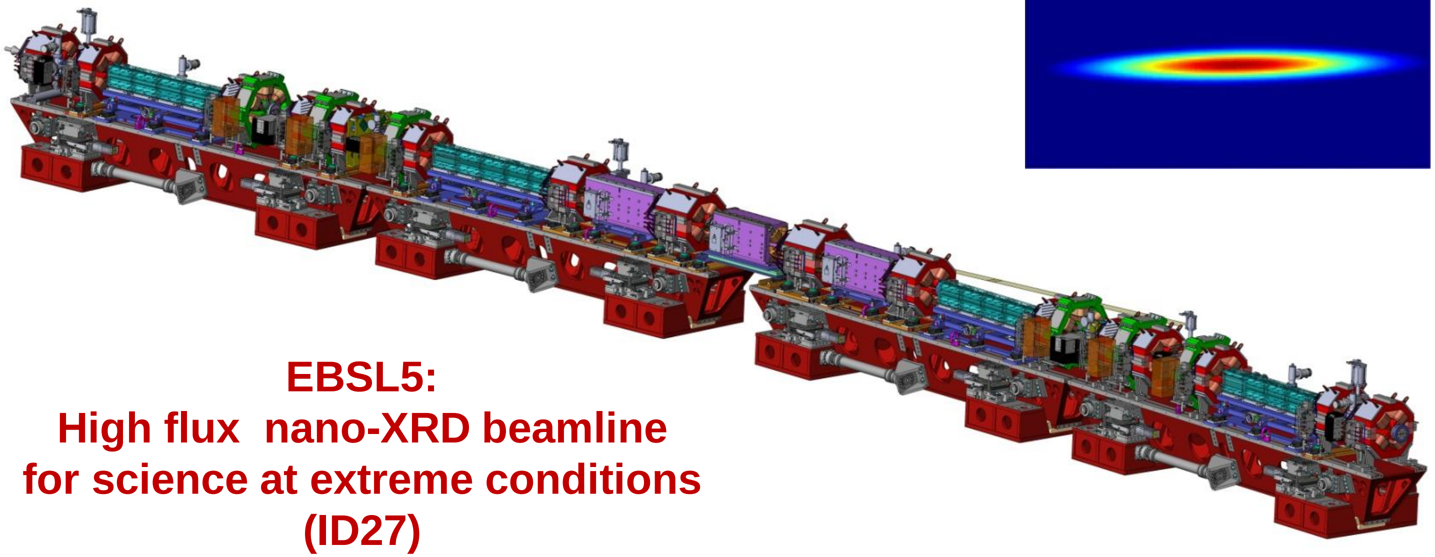
- ❑ Static High Pressure Research: status and trends
- ❑ Dynamic Compression: recent developments, future plans
- ❑ The EBS
- ❑ Extreme Conditions Science at EBS

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)

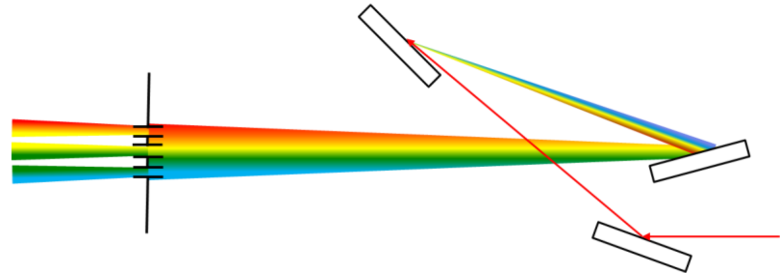
NRS-EBS:
Pushing the limits of NRS in energy and spatial resolution (ID14)

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

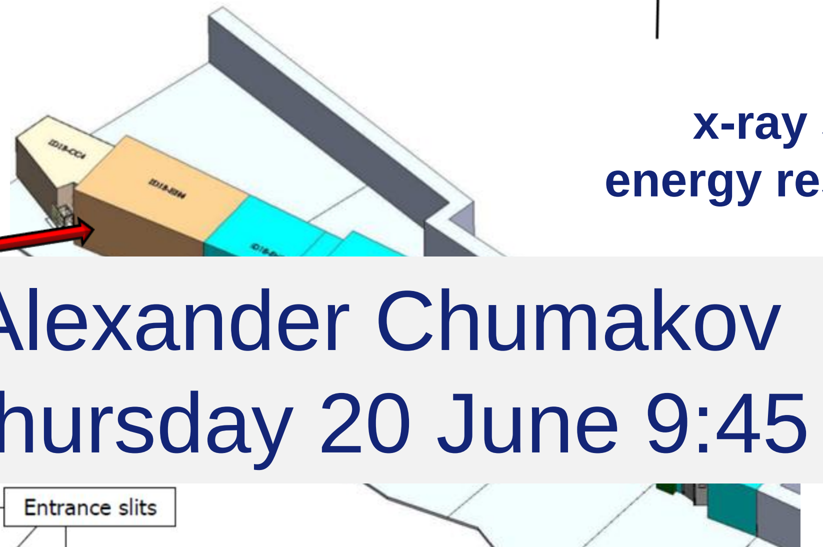
EBSL7:
Towards sub-μm, high brilliance EXAFS (ID24)

NRS-EBS - NUCLEAR RESONANCE REFURBISHMENT (→ ID14)

Super-Earth planets interiors



x-ray spectrograph: energy resolution of 40 μeV

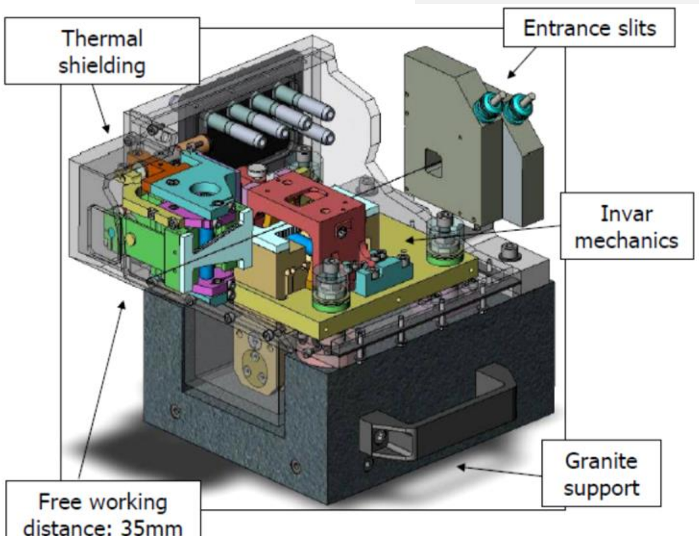


New EH4

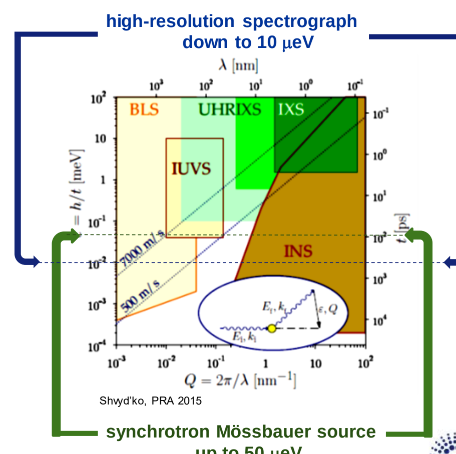
New OH3

Alexander Chumakov
Thursday 20 June 9:45

KB system: 0.2 μm × 0.2 μm

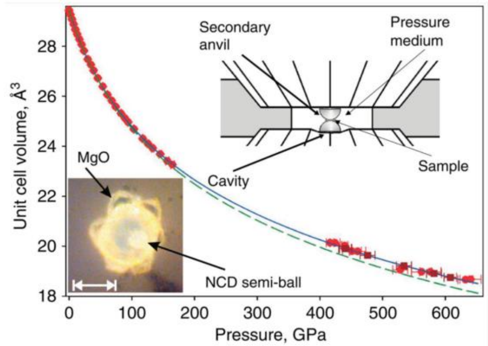


Elastic properties, sound velocity at Megabars



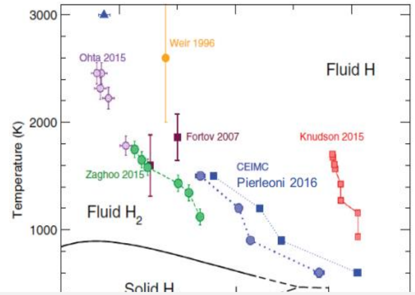
EBSL5 - SCIENCE UNDER EXTREME CONDITIONS (ID27)

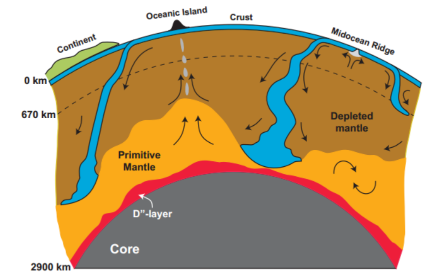
- **Materials at and beyond the current limits of static P and high T**



Double stage Diamond A

Solving the fluid H_2 to fluid H transition



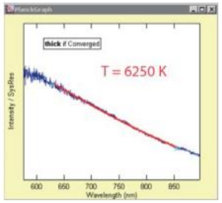


Fast melting, kinetics of chemical reactions at extreme conditions

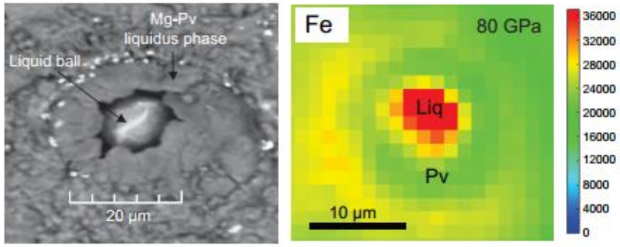
Mohamed Mezouar
Tuesday 18 June 9:45

- **Structure and che**

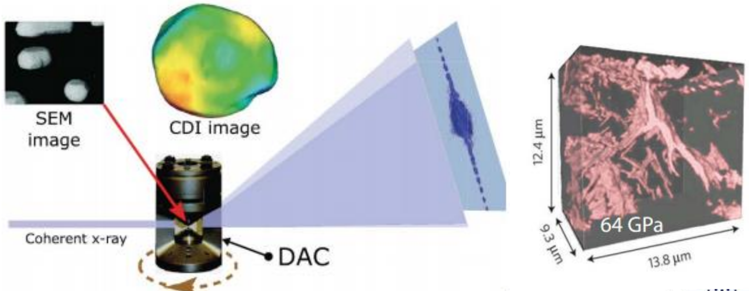
Exploring extreme temperature states using laser heating



In situ chemical analysis by nano-XRD and XRF



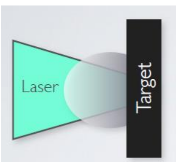
- **Rheology of materials at extreme conditions**

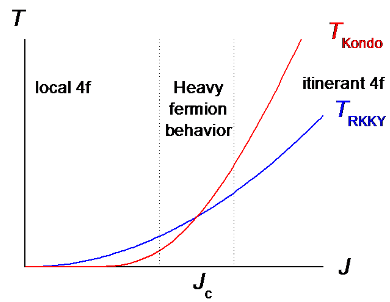


EBSL7 – Time Resolved and Extreme Conditions XAS

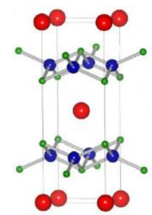
Time resolved & extreme conditions XAS (ID24_ED)

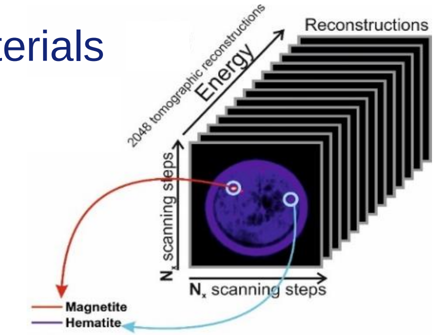
- extreme conditions for geophysics, planetary science, new materials
- magnetic response in the MegaGauss regime
- 3D spatially resolved chemical speciation

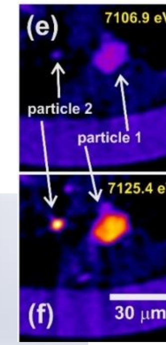


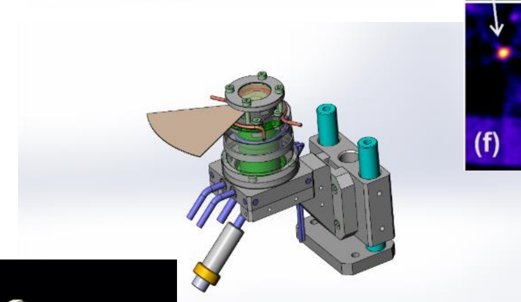


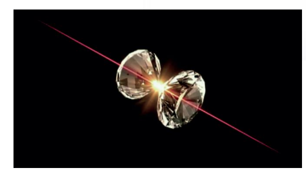
LNCMI





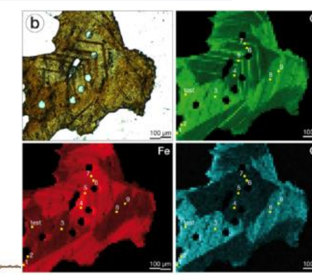


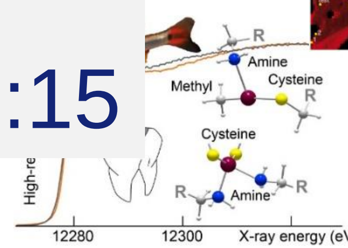




Sub-μm, high brilliance EXAFS (ID24_DCM)

- *in situ* and *operando* time resolved chemistry
- environmental scienc
- high pressure, earth a





Angelika Rosa
Tuesday 18 June 12:15

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