Hands on! High-pressure techniques at the ESRF-EBS

ESRF Auditorium - Grenoble - France
17 – 21th of June 2019

Organizers	A. D Rosa and G. Garbarino
Assistance	E. Jahn, S. Girodon, F. Mengoni, J. Jacobs, S. Bauchau, N. Sevelin, F. Perrin, S. Pasternak
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Speakers	D. Andrault, D. Braithwaite, W. Crichton, F. Datchi, L. Dubrovinsky, G. Garbarino, J. Geck, Y. Le Godec, N. Guignot, K. de Hantsetters, N. Hilairet, T. Irifune, J. Jacobs, S. Klotz, Y. Kono, M. Louvel, M. Mezouar, G. Morard, F. Occelli, S. Pascarelli, G. Pokrovski, A.D. Rosa, C. Sahle, C. Sanloup, T. Sheppard, I. Sergeev, D. Testemale, L. Truche , F. Wilhelm, M. Wilke
Collaborator	Réseau de Technologie des Hautes Pressions

ESRF - EBS Workshop Series

ESRF-EBS
Extremely Brilliant Source

Programme
Abstracts
List of Posters
List of Participants

Programme outline

	Monday 17 June Mulit Mbar DAC	Tuesday 18 June LH-DAC	Wednesday 19 June LVP/PEP & Tomograph	Thursday 20 June Cryo DAC	Friday 21 June RH-DAC / Autoclave
9:00 - 10:30 Lectures Auditorium	Welcome 15’ S. Pascarelli 30’ S. Klotz 45’	D. Andrault 45’ M. Mezouar 15’ N. Guignot 30’	N. Hilairet 45’ W. Crichton 15’ Y. Le Godec 30’	J. Geck 45’ A. Chumakov 15’ I. Sergeev 30’	M. Wilke 45’. D. Testemale 15’ M. Louvel 30’
10:30 - 11:00	Coffee break				
11:00 - 12:30 Lectures Auditorium	K. De Hantsetters 45’ Y. Kono 45’	C. Sahle 15’ G. Morard 45’ G. Garbarino 15’ A.D. Rosa 15’	J.P. Perrillat 30’ T. Sheppard 30’ C. Sanloup 30’	J. Jacobs 15’ D. Braithwaite 45’ M. Hanfland 15’ F. Wilhelm 15’	F. Datchi 45’ L. Truche 30’ Closing remarks 15’
12:30 - 13:30	Lunch break				
13:30 - 16:30 Practicals Laboratories & beamlines	HANDS ON!	HANDS ON!	HANDS ON!	HANDS ON!	
16:30 - 17:30	Coffee break and beamline visits ID18/ BM16	ID27/ ID20	ID15B/ID06	ID24-BM23/ ID12	
17:30 - 18:15 Frontiers of high pressure research Auditorium	F. Occelli 45’	T. Irifune 45’	L. Dubrovinsky 45’	G. Pokrovski 45’	
18:15 - 18:30	Discussion				
18:30 Apero Mezzanine Central Building	Wine and Cheese POSTERS	Beer and Bretzel POSTERS	From 19:30 Workshop dinner	Vodka and pickles POSTERS	

Detailed Daily Programme

Monday 17th of June
DAC – multiMbar DAC

8:30 - 9:00	**Registration**
9:00 - 9:15	**Opening: Welcome, A.D. Rosa, G. Garbarino**
	Lecture round 1 - Chair: G. Morard
9:15 - 9:45	**S. Pascarelli – ESRF** Frontiers of High Pressure Research at the European Synchrotron Radiation Facility
9:45 - 10:30	**S. Klotz – IMPMC, Université de la Sorbonne, France** High pressure: Making it, measuring it, and avoiding pitfalls
10:30	**Coffee break**
	Lecture round 2 - Chair: N. Hilairet
11:00 - 11:45	**K. De Hanstetter – Almax, Belgium** Diamonds
11:45 - 12:30	**Y. Kono – Ehime University, Matsuyama, Japan** Synchrotron X-ray experiments for studying structure and properties of liquids and glasses at high-pressure and high-temperature conditions in large volume press
12:30 - 13:30	**Lunch**
13:30 - 16:30	Meeting point in the entrance hall of the Central Building HANDS ON!
16:30 - 17:30	**Coffee and beamline visits ID18/BM16** (Meeting point in the entrance hall)
	Frontiers lecture - Chair: D. Andrault
17:30 - 18:15	**F. Occelli, CEA Bruyères-Le-Chatel** Frontiers of high pressure research
18:15 - 18:30	**Discussion**
18:30	**Poster Session + Wine & Cheese**

Tuesday 18th of June

LH - DAC

	Lecture round 1 - Chair: JP. Perrillat
9:00 - 9:45	**D. Andrault – LMV Clermont-Ferrand, France** Phase transformations, chemical reactions and melting properties investigated *in-situ* using the laser heated diamond anvil cell
9:45 - 10:00	**M. Mezouar – ESRF, France** High flux nano-XRD beamline for Science under extreme conditions
10:00 - 10:30	**N. Guignot - PSICHÉ beamline, Synchrotron SOLEIL, France** The LH-DAC in synchrotrons: general principles and an overview of some important techniques
10:30	Coffee break + group photo
	Lecture round 2 - Chair: J. Le Godec
11:00 - 11:15	**C. Sahle, ESRF France** ID20: non-resonant inelastic X-ray scattering at extreme conditions
11:15 - 12:00	**G. Morard, ISTERRE, Grenoble, France** Phase transitions in laser heated diamond anvil cell: observations from *in-situ* and *ex-situ* analyses
12:00 - 12:15	**G. Garbarino, ESRF, France** ESRF high pressure laboratory: present and future
12:15 - 12:30	**A.D. Rosa, ESRF, France** Extreme conditions programme at BM23/ID24 after the EBS upgrade
12:30 - 13:30	**Lunch**
13:30 - 16:30	HANDS ON!
16:30 - 17:30	**Coffee and beamline visits ID27/ ID20 -** (Meeting point in the entrance hall)
	Frontiers lecture - Chair: O. Mathon
17:30 - 18:15	**T. Irifune, GRC, Ehime Univ., Matsuyama, Japan** Synthesis, features, and applications of nano-polycrystalline diamond: Toward multi-Mbar pressures in multianvil apparatus
18:15 - 18:30	**Discussion**
18:30	**Poster Session + Beer & Bretzel**

Wednesday 19th of June
LVP/PEP and Tomography

	Lecture round 1 - Chair: N. Guignot
9:00 - 9:45	**N. Hilairet, UMET, Lille , France** *In-situ* LVP experiments for investigation of materials deformation under high pressures
9:45 - 10:00	**W. Crichton, ESRF, France** Recent upgrades to beamline ID06LVP, the ESRF's large-volume press station
10:00 - 10:30	**Y. Le Godec, IMPMC, Sorbonne, Paris, France** Novel portable Paris-Edinburgh presses for synchrotron time-resolved 3-D micro-imagining under extreme conditions
10:30	Coffee break
	Lecture round 2 - Chair: Y. Kono
11:00 - 11:30	**J.P. Perrillat, Laboratoire de Géologie, Univ. Lyon1, France** Exploring magmas under pressure using the Paris-Edinburgh press and synchrotron light
11:30 - 12:00	**T. Sheppard, KIT, Karlsruhe, Germany** *In situ* and *operando* hard X-ray tomography from micro- to nanoscale: opportunities and applications in catalysis and materials science
12:00 - 12:30	**C. Sanloup, IMPMC, Sorbonne, Paris, France** Trace elements in silicates/melts at high pressure
12:30 - 13:30	**Lunch**
13:30 - 16:30	Meeting point in the entrance hall of the Central Building HANDS ON!
16:30 - 17:30	**Coffee and beamline visits ID15B/ID06** - (Meeting point in the entrance hall)
	Frontiers lecture - Chair: G. Pokrovski
17:30 - 18:15	**Leonid Dubrovinsky, BGI, Bayreuth, Germany** Inorganic Synthesis and Crystal Chemistry at Multimegabar Pressures
18:15- 18:30	**Discussion**
19:30	**Workshop Dinner in town** **Transfer by Tram – Meeting point in front of the site entrance at 19:00**

Thursday 20th of June

Cryo DAC

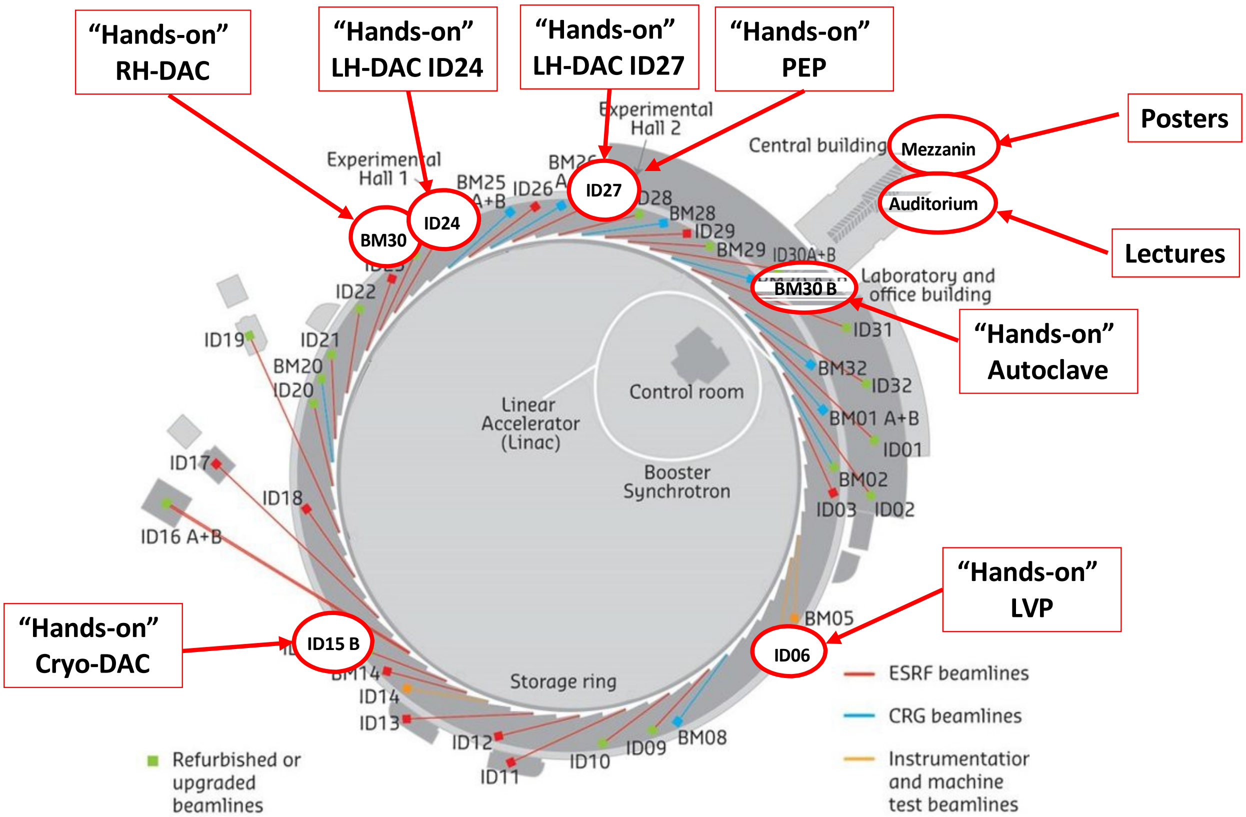
	Lecture round 1 - Chair: L. Dubrovinsky
9:00 - 9:45	**J. Geck, Institute of Solid State Physics, Univ. Dresden, Germany** X-ray scattering at high pressures and low temperatures: Squeezing cool electrons
9:45 - 10:00	**A. Chumakov, ESRF, France** Nuclear resonance scattering at high pressure: status and future
10:00 - 10:30	**I. Sergeev, P01 extreme condition beamline, Desy Synchrotron, Germany** High pressure studies on magnetism and lattice dynamics by Nuclear Resonance Scattering
10:30	Coffee break
	Lecture round 2 - Chair: F. Datchi
11:00 - 11:15	**J. Jacobs, ESRF, France** A versatile Diamond Anvil Cell for X-ray inelastic, diffraction and imaging studies at synchrotron facilities
11:15 - 12:00	**D. Braithwaite, IMAPEC Laboratory, CEA Grenoble, France** Strongly correlated electron systems under high pressure and other extreme conditions
12:00 - 12:15	**M. Hanfland, ESRF, France** The high pressure crystallography beamline ID15B
12:15 - 12:30	**F. Wilhelm, ESRF, France** High pressure activity at the ESRF ID12 beamline
12:30 - 13:30	**Lunch**
13:30 - 16:30	Meeting point in the entrance hall of the Central Building HANDS ON!
16:30 - 17:30	**Coffee and beamline visits ID24-BM23/ ID12** (Meeting point in the entrance hall)
	Frontiers lecture - Chair: H. Reichert
17:30 - 18:15	**Gleb Pokrovski, Geosciences Environnement Toulouse, Univ. Toulouse, France** *In-situ* spectroscopy of sulfur and critical metals in fluid-mineral-melt systems at high temperatures and pressures
18:15 -18:30	H. Reichert: Director of Research, ESRF Impact of the EBS upgrade
18:30 - 18:45	**Discussion**
18:45	**Poster Session + Vodka & Pickles**

Friday 21st of June

RH-DAC / Autoclave

	Lecture round 1 - Chair: L. Truche
9:00 - 9:45	**M. Wilke, GFZ, Univ. Potsdam, Germany** Introduction to resistively heated DAC techniques
9:45 - 10:00	**D. Testemale, Institut Néel – CNRS, CRG FAME - ESRF, France** FAME and FAME-UHD beamlines
10:00 - 10:30	**M. Louvel, Univ. Muenster, Germany** *In-situ* studies of high-temperature fluids and melts (P < 2 kbar) and their application to Geosciences
10:30	Coffee break
	Lecture round 2 - Chair: M. Wilke
11:00 - 11:45	**F. Datchi, IMPMC, Sorbonne, Paris, France** Experimental studies at high P-T in the diamond anvil cell
11:45 - 12:15	**L. Truche, ISTERRE, Univ. Grenoble, France** Chemical controls on the solubility of metal bearing phases in hydrothermal solution
12:15 - 12:30	**Discussion - Closing remarks**
12:30 - 13:30	**Lunch**

"Hands-on" Venue(s)



Group	Location (beamline and hutch)	Teacher
Cro-DAC	ID15	Gaston Garbarino (ESRF)
RH-DAC	BM23	Angelika Rosa (ESRF)
LH-DAC	ID24-S	Raffaella Torchio (ESRF) Guillaume Morard (Isterre)
LH-DAC	ID27 EH2	Mohamed Mezouar (ESRF)
PEP	ID27 EH1	David Sifre (ESRF) Jean Philippe Perrillat (Univ. Lyon)
Autoclave	BM30 B	Denis Testemale (Neel Institute) Elena Bazarkina (Neel Institute)
LVP	ID06	Wilson Crichton (ESRF)

Short descriptions of “Hands–On” groups programmme

(alphabetically order of group names)

- **AUTOCLAVE BM30/Neel Institute (D. Testemale, E. Bazarkina)**

  - Monday: Presentation: general principle of autoclaves, detailed introduction to the setup used. [Location: ESRF]
  - Tuesday: First experiment: sulfur speciation at hydrothermal conditions. Sample preparation, loading, experiment with observation, discussion. [Location: ESRF]
  - Wednesday: Second experiment: measurement of hydrothermal fluids density. Sample preparation, loading, experiment with x-ray absorption measurements. [Location: Néel Institute]
  - Thursday: Visit of the Néel Institute HP equipment: large volume press, x-ray lab source for PE, DAC and autoclaves experiments. [Location: Néel Institute]

- **Cryo-DAC at ID15B (G. Garbarino)**

  Monday
  - Preparation of the diamond anvil cell loading for cryogenic experiments
  - Gasket preparation and drilling
  - Tips and tricks: special features of cryo-DACs (component materials, etc…) and discussion about gasket materials and pressure transmitting medium

  Tuesday
  - Loading of samples in a DAC, special discussion about sample dimensions respect to pressure chamber
  - Tips and tricks: pressure and temperature generation, monitoring and calibration.

  Wednesday
  - Mounting of the DAC on the cryostat, preparation of experiment, (ID15b)
  - Tips and tricks: Beamline requirements for cryogenic studies, data acquisition protocols and strategies, safety aspects about manipulation of cryogenic fluids

  Thursday
  - Run with a real sample. (ID15b)
  - Tips and Tricks: Data analysis, a scientific example

- **LH-DAC at ID24 (R. Torchio and M. Morard)**

  - Monday/Tuesday: Preparation of the diamond anvil cell loading for laser heating experiments: gasket indentation, gasket drilling, cell loading (insulating material, sample, ruby) cell pressurization and pressure measurements
  - Wednesday/Thursday: Description of the laser heating system on ID24. Heating runs and temperature measurements

  Time schedule can slightly vary as a function of the cells preparation advancement.

- **LH-DAC at ID27 EH2 (M. Mezouar and V. Svitlyk)**

- **LVP at ID06 (W. Crichton)**
  During the course of the practical's the users will prepare and mount several 6/8 assemblies and will cover:
  - preparation of octahedra
  - preparation of gaskets & cubes and their mounting
  - thermocouple preparation and cementing
  - sample, capsule, furnace and insulation preparation and mounting
  - assembly
  - running the experiment

  Time will be tight, so several stations will be employed and different jobs run in parallel.

- **PEP at ID27 EH1 (D. Sifre and J.P. Perrillat) Paris-Edinburgh press (PEP) hands on**
  - preparation of PEP assemblies (gaskets, heaters, sample container capsules, etc)
  - discussion about different type of assemblies depending on pressure-temperature range
  - metrology, how to measure pressure and temperature
  - sample, capsule, furnace and insulation preparation and mounting
  - assembly
  - running the experiment

- **RH-DAC at BM23 (A.D. Rosa)**

Monday: (HP-lab)	**HANDS-ON**: Gasket preparation and sample chamber drilling **Tips and tricks:** principle of a DAC, special features of RH-DACs (component materials, etc…)
Tuesday: (HP-lab)	**HANDS-ON:** Loading of fluid and solid samples in a DAC **Tips and tricks:** pressure and temperature generation, monitoring and calibration.
Wednesday**:** (BM23)	**HANDS-ON:** Mounting of the DAC on the beamline, preparation of heater and remote control, Thermocouple calibration run. **Tips and tricks**: Beamline requirements for RH-DAC studies, functionality of a Raman system for pressure monitoring, data acquisition protocols and strategies
Thursday**:** (BM23)	**HANDS-ON:** Run with a real sample. **Tips and Tricks:** Data analysis, a scientific example

Abstracts

(in order of presentation)

Frontiers of High Pressure Research at the European Synchrotron Radiation Facility

S. Pascarelli
European Synchrotron Radiation Facility, 71 avenue des Martyrs, 38000 Grenoble, France

High pressure research at the European Synchrotron Radiation Facility has always played a central role. Today, more than 50 % of the beamlines are engaged in high pressure research, in various fields from earth sciences and physics to chemistry, biology and material science.

Recent years have seen technical breakthroughs in high pressure instrumentation at synchrotrons, with the development of laser heating of the Diamond Anvil Cell (DAC), resistively heated DACs, high pressure cryostats, nano-crystalline diamond anvils or double-stage DACs. These techniques have considerably extended the P-T domain towards higher pressures (~ 700 – 800 GPa, at ambient T) and temperatures (up to ~ 5000-6000 K). In parallel, the development of time resolved techniques in X-ray Absorption Spectroscopy, X-ray Diffraction and X-ray Imaging, in particular using single bunch acquisition, has opened the possibility to use synchrotron radiation to probe dynamically compressed matter generated by a high power laser or a high-energy projectile. These developments have pushed the beamlines toward their limit and triggered new projects.

ESRF-EBS (Extremely Brilliant Source), operational in 2020, will offer significantly higher flux density and higher coherence together with new experimental facilities, leading to important perspectives for extreme matter studies. The Matter at Extremes group is involved in three EBS-related projects: 1. A high flux nano-X-ray diffraction beamline for science at extreme conditions 2. Pushing the limits of nuclear resonant scattering in energy and spatial resolution and 3. a high brilliance EXAFS beamline optimized for time resolved and extreme conditions applications.

In parallel, we are building a platform - the High Power Laser Facility (HPLF) - dedicated to dynamic compression studies to probe matter at pressures and temperatures beyond the static limit of the DAC, and to investigate the dynamic behaviour of materials under high strain rates. The Phase I of this project foresees the coupling of a 100 J, nanosecond, laser to energy dispersive X-ray absorption spectroscopy. In 2018 several milestones were reached, with the design of the new clean room hosting the laser in 2020, the design of the laser beam transport into the experimental hutch and the successful commissioning of its 15J Front End.

In the first part of the presentation I will give an overview of static and dynamic compression activities and recent scientific results obtained at the ESRF. In the second part, I will present our future projects and the unique science opportunities offered by ESRF-EBS.

High pressure: Making it, measuring it, and avoiding pitfalls

S. Klotz

Sorbonne Université, Paris, e-mail : **Stefan.Klotz@upmc.fr**

In this talk I will give an introduction to basic aspects of high pressure techniques and metrology, understandable for participants from various backgrounds. For further reading refs. [1-3] may be useful. Principle keywords of my presentations are:

- Pressure and stress: Basic elements of elasticity theory. Uniaxial, shear and hydrostatic stress, Von-Mises stress.
- High pressure devices: From piston-cylinders to opposed-anvil cells. How strong is a cylinder? Pressure-volume trade-off.
- Measuring pressure: Basic elements of pressure determination from 1 bar to 1 Mbar, accuracy versus precision. Common pressure markers. The Decker and ruby scales.
- Hydrostaticity: Pressure transmitting fluids/media. Solid media: the "Lamé effect.
- Common pitfalls: A collection of reported "anomalies" due to non-hydrostatic or non-homogeneous pressure conditions.

I admit that this program might be challanging for a 45 minutes talk. I encourage the participants to have a double-expresso for breakfast to follow my talk till the end!

References

[1] J.S Loveday (edt.) High-Pressure Physics, CRC Press/Taylor and Francis, 2012.
[1] S. Klotz, Techniques in High Pressure Neutron Scattering, CRC Press/Taylor and Francis, 2013.
[3] J.M. Recio, J.M. Menendez, A.O. de la Roza (edts.), An Introduction to High-Pressure Science and Technology, CRC Press/Taylor & Francis, 2016.

«Diamonds»

DE HANTSETTERS, KOEN

Diamond anvils: types, designs, dimensions, selections, specials…

Synchrotron X-ray experiments for studying structure and properties of liquids and glasses at high-pressure and high-temperature conditions in large volume press

Y. Kono[1]

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Knowledge of pressure-induced structure and physical property changes in liquids and glasses is of great interest in various scientific fields, such as condensed matter physics, geoscience, and materials science. However, due to experimental difficulties, structure and properties of liquids and glasses under high pressure and high temperature conditions have not been well understood in experiments. In the past decades, new developments in high-pressure synchrotron X-ray experiments have advanced the study of liquids and glasses under pressure (cf. reviews in [1]). Here I will introduce experimental studies of structure and properties of liquids and glasses at high pressure and high temperature conditions at the beamline 16-BM-B in the Advanced Photon Source, USA.

The beamline 16-BM-B utilizes white X-ray combined with Paris-Edinburgh large volume press for studying structure and physical properties, such as viscosity and elastic wave velocities, of liquids and glasses at in situ high-pressure and high-temperature conditions [2]. The Paris-Edinburgh press allows the usage of large sample volumes (up to 2 mm in both diameter and height) to high pressures up to 7 GPa and high temperatures to 2000 °C. Structures of liquids and glasses are determined by a multi-angle energy dispersive X-ray diffraction technique. Ultrasonic techniques have been developed to investigate elastic wave velocity of liquids. Falling sphere viscometry, using high-speed X-ray radiography (>1000 frames/s), enables us to investigate a wide range of viscosity, from those of high viscosity silicates melts to low viscosity (<1 mPa s) liquids such as liquid metals or salts. The integration of these multiple techniques has promoted comprehensive studies of structure and physical properties of liquids and glasses at high pressures and high temperatures, making it possible to investigate correlations between structure and physical properties of liquids in situ. In addition, our recent development of double-stage large volume cell opened a new way to investigate structure of oxide glasses under ultrahigh pressure conditions of >100 GPa [3,4].

References

[1] - Y. Kono and C. Sanloup, Magmas Under Pressure: Advances in High-Pressure Experiments on Structure and Properties of Melts (2018).

[2] - Y. Kono, C. Park, C. Kenney-Benson, G. Shen, and Y. Wang, Physics of the Earth and Planetary Interiors **228**, 269-280 (2014).

[3] - Y. Kono, C. Kenney-Benson, D. Ikuta, Y. Shibazaki, Y. Wang, and G. Shen, Proceedings of the National Academy of Sciences **113**, 3436-3441 (2016).

[4] - Y. Kono, Y. Shibazaki, C. Kenney-Benson, Y. Wang, G. Shen, Proceedings of the National Academy of Sciences **115**, 1742-1747 (2018).

«Phase transformations, chemical reactions and melting properties investigated *in situ* using the laser heated diamond anvil cell»

D. Andrault[1] and many co-authors, in particular from ESRF[2]

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[1]European Synchrotron Radiation Facility, Grenoble

An intrinsic problem of cold compression in the diamond anvil cell (DAC) is the development of deviatoric stresses in the sample chamber. Also, most of phase transformations and, a fortiori, chemical reactions do not occur without providing energy to overcome the kinetic barriers. For example, a frequent observation is high-pressure amorphization, which yields to apparition of a metastable material instead of the transformation into a stable assemblage of phases.

For many years now, laser heating (LH) has been a tool of choice to investigate the material properties under high pressures. It can be used to release the deviatoric stresses, overcome the kinetic barriers and for *in situ* measurements of material properties in an extensive range of pressures and temperatures. Limitations are (i) temperatures below 1000 K, when the hot spot is difficult to detect using a classical optical system, (ii) low pressures, when the gasket remains weak (with a pressure limit depending on the type of DAC) and (iii) above 150-200 GPa, when the distance between the two diamond culets is smaller than ~5 µm.

In this presentation, we will present the basic of LH-DAC experiments, emphasizing on details that yield to proper or improper experimental conditions.

We will show that the ESRF beam-lines are exceptional tools to probe the sample properties *in situ* at high pressures and temperatures using the LH-DAC, using different approaches. We will present different examples of the monitoring of phase transformations, chemical reactions, measurements of P-V-T equations of state and melting properties. The *in situ* measurements using X-ray beams are also ideal to optimize the experimental conditions, before other types of LH-DAC experiments can be performed.

High flux nano-XRD beamline for Science under extreme conditions

M. Mezouar, G. Garbarino, V. Svitlyk and S. Bauchau

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We will build a new high pressure X-ray diffraction, fluorescence and imaging beamline to take full advantage of the outstanding performance of the EBS. The proposed beamline upgrade will provide significantly higher photon flux density and higher coherence, especially for photon energies above 20 keV, i.e. the energy range most relevant for diffraction and imaging at extreme conditions. This will enable a new class of nano-XRD, XRI and XRF studies under extreme P-T conditions. The direct impact on studies at extreme conditions is that higher pressure and temperature states which can be generated only in smaller volumes will be finely characterized. Transient processes under extreme will be seen. Submicron sample heterogeneities will also become accessible, at the microsecond time scale, with a deeper understanding of processes such as transport (diffusion, viscosity) or crystallization/melting, under extreme conditions. Breakthroughs can be expected in various scientific areas. Here, the main components of the new instrument and expected performance will be presented.

«The LH-DAC in synchrotrons: general principles and an overview of some important techniques»

N.Guignot[1]

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In this lecture I will first detail some general principles of the Laser-heated diamond anvil cell technique (LH-DAC) and how this applies to the synchrotron beamlines and dictates some design choices. I will address the questions concerning the laser beam shape, laser absorption, temperature (T) gradients and temperature measurement. I will then give an overview of some important techniques used on different synchrotrons and new developments done at the beamline PSICHE of the synchrotron SOLEIL (Fig.1). Among these developments, I believe that the integration of 4-color pyrometers is a very interesting addition, being the only T metrology technique being truly achromatic and providing 2D T mapping during the experiments.

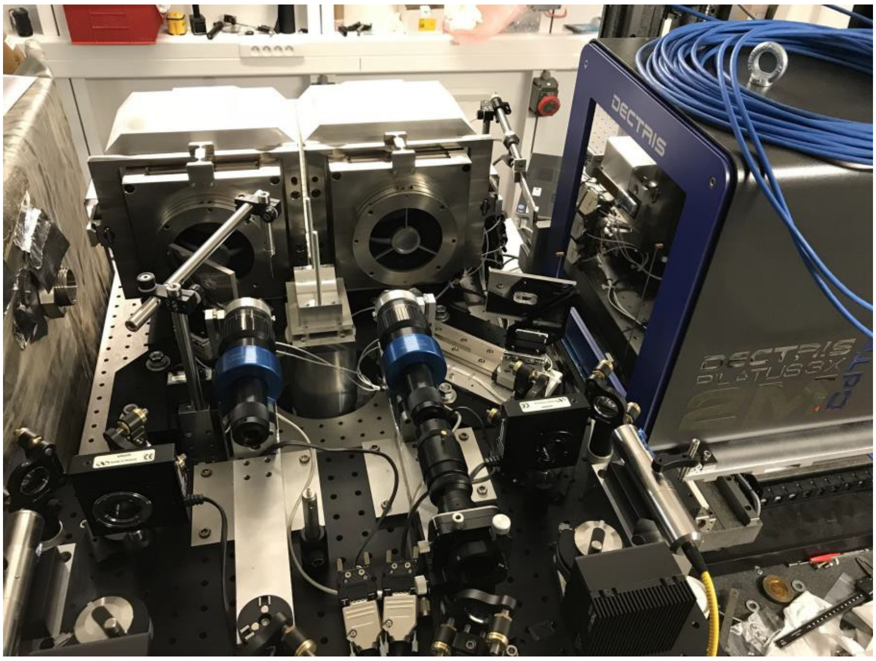

Figure 1: The PSICHE LH-DAC setup, with beam shaping optics and high resolution long working distance Schwarzschild objectives

ID20: non-resonant inelastic X-ray scattering at extreme conditions

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X-ray Raman scattering (XRS) spectroscopy is an emerging technique to study low energy absorption edges using hard X-rays [1,2]. The use of hard X-rays makes this technique inherently suitable for the study of low-Z-element-containing samples inside e.g. high-pressure diamond anvil cells, especially for disordered and complex materials where diffraction experiments are challenging. ID20 hosts a dedicated state-of-the-art instrument for XRS [3].

Here, we will briefly introduce the XRS technique, present new technical developments [4-6], and show the most recent results obtained of samples under extreme conditions [6-7].

References

[1] Schülke, Winfried. Electron dynamics by inelastic X-ray scattering. Vol. 7. Oxford University Press, 2007.

[2] Sahle, Ch. J., A. Mirone, J. Niskanen, J. Inkinen, M. Krisch, and S. Huotari. "Planning, performing and analyzing X-ray Raman scattering experiments." Journal of synchrotron radiation 22, no. 2 (2015): 400-409.

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[4] Sahle, Christoph Johannes, A. D. Rosa, Matteo Rossi, Valerio Cerantola, Georg Spiekermann, Sylvain Petitgirard, Jeroen Jacobs, Simo Huotari, Marco Moretti Sala, and Alessandro Mirone. "Direct tomography imaging for inelastic X-ray scattering experiments at high pressure." Journal of synchrotron radiation 24, no. 1 (2017): 269-275.

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[7] Petitgirard, S., C. J. Sahle, C. Weis, K. Gilmore, G. Spiekermann, J. S. Tse, M. Wilke, C. Cavallari, V. Cerantola, and C. Sternemann. "Magma properties at deep Earth's conditions from electronic structure of silica." (2019).

Phase transitions in laser heated diamond anvil cell: observations from in situ and ex situ analyses

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In my contribution, I will present different methods to establish phase diagrams of geomaterials by coupling X-ray diagnostics and Laser-Heated Diamond Anvil Cell. First, I will present different example to establish phase boundaries for solid-solid and solid-liquid phase transitions. I will also discuss the metrology issue related with laser heating experiments. Finally, I will finally present how analysis of recovered samples from high pressure and high temperature conditions can help to confirm in situ diagnostics, and also to place better constrain on phase diagrams under extreme conditions.

ESRF high pressure laboratory: present and future

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In the last decades, we have witnessed an unprecedented surge in high-pressure research that has greatly improved our fundamental understanding of materials under high compression. The X-ray investigation of matter under extreme conditions has become one of the major activities at the ESRF and other 3rd generation synchrotron sources. The array of techniques, initially restricted to structural measurements using X-Ray diffraction, is now extended and includes many others such as Inelastic X-ray Scattering, Nuclear Inelastic Scattering, X ray absorption and emission spectroscopy, X ray magnetic circular dichroism, X-ray Compton scattering and X-ray magnetic scattering. As a direct consequence, many scientific breakthroughs have been achieved across fields ranging from Earth and planetary sciences to fundamental physics, chemistry, materials research, and extending into biophysics and biochemistry including questions concerning life and biological function under extreme conditions. The very intense and highly focused X-ray beam available at the new EBS-ESRF will be a unique tool for probing microscopic samples at extreme pressures and temperature. In this context, we will present the on-site capabilities available at the High Pressure Laboratory to prepare the most challenging extreme conditions experiments. We will also discuss recent instrumental developments and new scientific results obtained at ESRF beamlines.

Extreme conditions programme at BM23/ID24 after the EBS upgrade

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X-ray absorption spectroscopy is a powerful tool to explore matter under extreme conditions of pressure and temperature and has important applications in various scientific domains such as materials science, Earth and planetary sciences and fundamental physics. It enables monitoring local structural changes and electronic transitions of trace and major elements in different matrixes (solids, fluids and melts) at high density. A large part of BM23/ID24 activity has been devoted to high pressure science including the study of phase transitions from semiconductor to metal, electronic transitions such as changes of the oxidation or spin state as well as the incorporation of trace elements in melts or solids up to conditions of 3000 K and 150 GPa.

The new Extremely Brilliant Source (EBS) will allow us to extend the reachable P/T domain to the conditions prevailing in the Earth's core (T up to 6000 K and P>150 GPa) and to study trace elements at very high dilution levels (few ppm) and at such extreme conditions. Here, I will present the extreme conditions programme at ID24/BM23 after the EBS and provide information about the new capabilities of these instruments (X-ray beam size, energy range, brilliance, scan speed, experimental stations, technical advances of the high pressure and temperature devices) and give an outlook of the new and unique scientific possibilities.

Synthesis, features, and applications of nano-polycrystalline diamond: Toward multi-Mbar pressures in multianvil apparatus

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Multi-anvil apparatus (MA) has advantages in synthesis of novel functional materials under ultrahigh-pressures (>10 GPa, by definition in materials science), because of its large sample volume, small thermal gradient, stable heating, etc., relative to those in competetive diamond anvil cell. We first succeeded in synthesis of pure nano-polycrystalline diamond (NPD) at pressures higher than ~15 GPa and tempratures above 2300K in MA [1], which was found to have peculiar nano-textures with ultrahard nature. By using a large-volume MA operated in a 6000-ton hydraulic ram at the GRC (BOTCHAN-6000), we are able to synthesize NPD rods with dimensions up to 1 cm, which have been used for various applications in high-pressure sciences, as well as for some other scientific and industrial applications.

One of such applications is to use NPD as anvils for higher pressure generation in MA. Pressures available in this apparatus using conventional tungsten carbide anvils have been limited to ~30 GPa except for those with very fine WC particles and less binders, where pressures to 50-60 GPa are reported [2,3]. Efforts have been made to expand the pressure limit by using harder sintered-polycrystalline diamond (SD) anvils, leading to the maximum pressure of ~120 GPa [4]. However, it seems quite difficult to reach pressures higher than this limit due to significant elastic deformation of the SD anvils. The anvil deformation also makes it difficult to perform in situ X-ray observations of the sample under pressure, as SD anvils contain metals such as Co as binders, which significantly absorb X-ray beams.

We have been trying to expand the pressure limit in MA using NPD anvils with two different approaches; one using a triple-stage (6-8-2) anvil configuration and the other with the conventional 6-8 system. The 6-8-2 MA with the third-stage NPD anvils led to pressures to 125 GPa at ~1000K [5]. For the latter 6-8 system with the second-stage NPD anvils, we confirmed pressures to ~90 GPa, which is about 1.5 times higher than the maximum pressure achieved using SD anvils with identical cell assembly (Fig.1). Attempts to further expand the pressure range are currently being pursued, which should lead to pressures of 150-200 GPa using MA with NPD anvils in the near future.

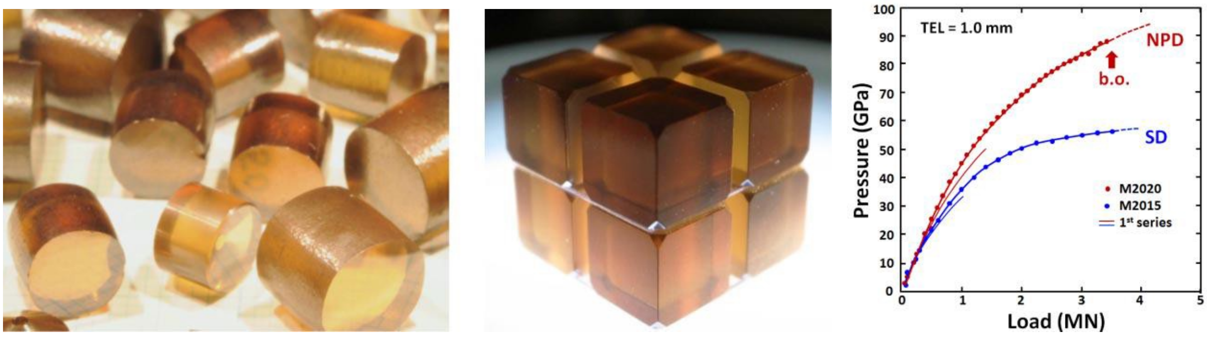

Figure 1: Raw NPD rods (left), NPD anvils for 6-8 MA (middle), and a comparison of pressure generation efficiencies in 6-8 MA with NPD and SD anvils (right, from [5])

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In-situ LVP experiments for investigation of materials deformation under high pressures

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The early 2000's saw a general development of in-situ measurements using synchrotron x-rays and the large volume presses. These occurred a few years after the first beams at third generation synchrotrons. More than twenty years after, developments of experimental setups (including high pressure cells, apparatuses) have been made, a number of lessons have been learned on data interpretation and significance, some tools have been developped or adapated for data analysis.

This is especially true for deformation of materials under high pressures and high temperatures. Fundamental progresses were made in that area, including (but not limited to) the understanding of deformation of minerals in the earth interior. Even though these studies remain demanding today both from the data analysis and the technical point of view, the basic measurements have been facilitated.

I will give an overview of these experiments, from hardwares, to setups, and x-ray data analysis, and illustrate how they can be used to understand mechanical behavior of materials under high pressures. I will include recent developments on these setups using acoustic emission monitoring, and illustrate how these can be used to monitor brittle-like failure of materials. While some challenges have been overcome, others actually remain at the limit of what the technique can offer. I will outline some directions in which upgrades such as the EBS at ESRF can bring more information in the field of material deformation under high pressures.

Recent upgrades to beamline ID06LVP, the ESRF's large-volume press station.

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We highlight recent advances in large-volume operation at ID06LVP and will cover testing and development of new technologies that have allow:

i./ full autonomous sampling of ultrasonic data at diffraction-competitive rates, and its on-going development,

ii./ the extension of 6/8 compression to pressures above 30 GPa, at high temperature and their further combination with ultrasonic studies,

iii./ the extension of deformation to pressures exceeding 16 GPa, at high temperature, and,

iv./ an opposed-anvil setup (Drickamer) for radial diffraction measurements that easily exceeds 30 GPa.

Other ancillary changes to the setup that are required by these measurements will be mentioned. These will be highlighted from examples from user experiments, and on-going collaborations.

Further time will also be allocated to the development of a recently-ordered Pilatus-based detection system.

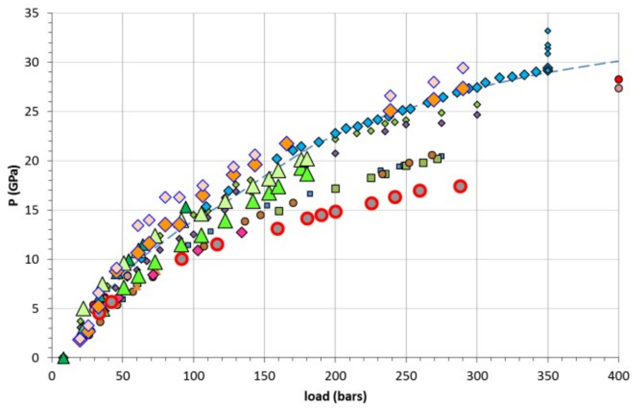

Figure 1: Calibration of higher pressure regimes with a variety of anvil types.

Novel portable Paris-Edinburgh presses for synchrotron time-resolved 3-D micro-imagining under extreme conditions

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Synchrotron X-ray microtomography is a non-destructive 3D imaging/microanalysis method selective to a wide range of properties such as morphology, density, chemical composition, chemical states, structure, and crystallographic perfection with extremely high sensitivity and spatial resolution. To extend this technique to extreme conditions (high-pressure/high-temperature/high stress), we developed two new portable devices based on the Paris-Edinburgh press:

1/ the RoToPEc (Rotating tomography Paris–Edinburgh cell [1]), where two opposed conical anvils are used to pressurize a sample embedded in an X-ray transparent boron epoxy gasket. In our new system, both anvils can rotate independently under load, with no limitation in angle, through two sets of gear reducers and thrust bearings. The accurate and simultaneous rotation of the top and bottom anvils is achieved using stepper motors and optical encoders positioned precisely on the both anvils. The ability to fully rotate the sample chamber under extreme conditions (up to 15 GPa and 2200K), overcomes the usual limited angular aperture of ordinary high pressure set-ups, allowing complete sets of tomographic projections to be acquired, in both full-field imaging (where a large (approx. 2x2 mm^2) monochromatic (or pink) x-ray beam is used to collect 2D radiographs) or micro-diffraction modes (scanning with a pencil beam of FWHM 3 x 3 μm^2 at several projection angles). Additionally, independent and controlled rotation of each anvil enables operation in shearing (one anvil rotates while the other is stationary) or deformation modes (both anvils rotate in opposite directions) under high P,T conditions. Hence, our portable device can operate in four different modes: (i) tomography, (ii) shearing, (iii) deformation or (iv) combination of (iii) or (ii) and (i). Our portable device has been easily and successfully adapted to various multi-modal synchrotron experimental set-up at beamlines ID27 (**ESRF**), PSICHE (**SOLEIL**), and I12 (**DIAMOND**).

2/ the UtoPEc (Ultra-fast Tomography Paris-Edinburgh cell) is a new Paris-Edinburgh press optimised for high speed tomography (0.5 seconds per full tomogram) at high pressures and temperatures (up to 15 GPa and 1500 K). This press has been developed at PSICHE beamline (**SOLEIL**) and is compatible with performing tomography on millimetre-sized samples. Rotary couplings allow continuous rotation of the press for fast tomography time series. At the PSICHE beam line, a spatial resolution of a few microns can be obtained for a full 2k x 2k reconstruction in 0.5 seconds. *In situ* tomography can also be combined with diffraction to provide measurements of pressure, temperature, phase transitions or composition.

The potential of our new presses for *in situ* synchrotron experiments will be illustrated by preliminary results [1-3] recently obtained from these facilities on many scientific cases. To conclude, we will present the new scientific opportunities our portable devices allow for studies of phase transition, density, crystallization and deformation under extreme PT conditions.

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« Exploring magmas under pressure using the Paris-Edinburgh press and synchrotron light »

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Magmas, which are generated by the melting of rocks in the deep interiors of the Earth and other planets, are involved not only in the present-day volcanism but also in 4.5 billion years evolution of the Earth's and planetary interiors. Understanding the generation, transportation and eruption of magmas requires the knowledge of some fundamental structural and physical properties of melts, like elasticity, density and viscosity. In this presentation, I will review recent advances in studying silicate liquids of geophysical importance using the large volume Paris-Edinburgh press at 3rd generation synchrotron facilities. After a brief account on the structural properties of silicate melts under pressure, I will describe the available techniques for density and elasticity measurements for non-crystalline materials. This will include X-ray diffraction and absorption methods, X-ray imaging volume measurements, and the ultrasonic technique. I will also introduce the use of X-ray radiography to estimate viscosity of melts from falling sphere experiments; with applications to lunar magmas. Finally, I will present the last developments in X-ray tomography under extreme conditions for studying melt migration processes.

“In situ and operando hard X-ray tomography from micro- to nanoscale: opportunities and applications in catalysis and materials science”

Thomas Sheppard

The study of heterogeneous catalysts and functional materials while performing specific chemical functions (in situ) and with simultaneous collection of product information (operando), are key concepts in modern chemistry research. At the same time, the penetrating power of hard X-rays allows for the imaging of interior structural features in a non-invasive manner and with a variety of contrast modes through application of tomography. Here we will explore recent developments in sample environments and experimental infrastructure for collecting tomographic data of catalysts at work, from the micrometer scale (STXM, XRD tomography) to the nanometer scale (X-ray ptychographic computed tomography). Experimental possibilities with the upcoming EBS upgrade will be highlighted particularly for coherent and high energy imaging, but also including applications under high pressure and temperature regimes.

« Trace elements in silicates/melts at high pressure »

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How trace elements are incorporated in silicate melts at depth, and how that may change with pressure?
To answer these questions, results obtained on a few key trace elements using x-ray absorption spectroscopy (XAS) and x-ray diffraction (XRD) techniques under high P-T conditions generated with a Paris-Edinburgh press will be presented [1-4]. Results will be compared with resistive-heating diamond-anvil cell studies. The final geological goal, *i.e.* how retention mechanisms relate to element partitioning between two co-existing phases will be discussed on the basis of in situ x-ray fluorescence (XRF) experiments.
The lecture will include the following aspects:
- Assembly designs
- Applications in Earth sciences
- Pros and Cons of XAS *vs* XRD
- Challenges and new scientific opportunities.

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Inorganic Synthesis and Crystal Chemistry at Multimegabar Pressures

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The impact of high-pressure studies on fundamental physics and chemistry, and especially on the Earth and planetary sciences, has been enormous. Modern science and technology rely on the fundamental knowledge of matter that is provided by crystallographic studies. The most reliable information about crystal structures and their response to changes in pressure and temperature is obtained from single-crystal diffraction experiments. Advances in diamond anvil cell (DAC) techniques and double-stage DACs, as well as in modern X-ray facilities have increased the accessible pressure range for structural research up to multimegabar range. We have developed a methodology to perform single-crystal X-ray diffraction experiments in double-side laser-heated DACs. Our results demonstrated that the solution of crystal structures, their refinement, and accurate determination of thermal equations of state of elemental materials, oxides, carbides, borides, carbonates, and silicates from single-crystal diffraction data are possible above 200 GPa at temperatures of thousands of degrees. These resulted in findings of novel compounds with unusual compositions, crystal chemistry, and physical properties. We illustrate application of new methodology for simultaneous high-pressure and high-temperature single crystal diffraction studies using examples of investigations of chemical and phase relations in the Fe-O system, transition metals carbonates, silicates, and nitrides.

«X-ray scattering at high pressures and low temperatures: Squeezing cool electrons»

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This lecture will focus on x-ray scattering experiments at cryogenic conditions as a function of external pressure. After a first motivation for such experiments, technical aspects and realisations of the corresponding sample environments for synchrotron and laboratory instruments will be discussed.
It will then be described how non-resonant and resonant x-ray scattering experiments can be used to clarify the many-body behaviour of interacting electron systems. In such systems one cannot -as it is often done- treat the electrons as independent entities. Rather the behaviour of a given electron depends in a non-trivial way on other electrons and, on top of that, may also be influenced by the lattice. Under these circumstances pronounced many-body physics can emerge and result in what is called a collective electronic quantum state. Famous representative phenomena are (unconventional) superconductivity, spatial charge and orbital order, long-range magnetic order or spin-liquids. Indeed, collective electronic quantum states of this kind constitute central unsolved puzzles of today's condensed matter physics. Here experiments as a function of hydrostatic pressure at low temperature can help to solve them: Non-resonant x-ray diffraction can be used to study the lattice structure at a given pressure and temperature, while resonant x-ray scattering provides a means to investigate spatial electronic order and collective electronic excitations at the same conditions. This will be illustrated in particular by recent research on charge density wave [1,2] and spin-liquid systems [3].

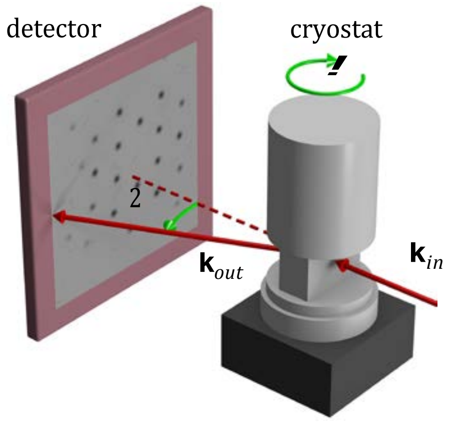

Figure 1: Schematic experimental setup for low temperature x-ray diffraction as a function of external pressure. The is sample inside a diamond anvil cell, which is situated within the cryostat.

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Nuclear resonance scattering at high pressure: status and future

Aleksandr Chumakov

European Synchrotron Radiation Facility

For high-pressure studies, nuclear resonance scattering offers hyperfine spectroscopy with high spatial resolution and phonon spectroscopy with high energy resolution. Hyperfine spectroscopy provides data on magnetic state, oxidation degree, and oxygen coordination, whereas phonon spectroscopy allows for accessing dynamic and thermodynamic properties, in particular, elastic moduli and sound velocity at extreme conditions.

At present, Nuclear Resonance beamline ID18 at ESRF serves users with the beam size of about 10 microns, and the energy resolution of about 0.5 meV. In frames of the EBS Upgrade Programme of the ESRF, we expect to improve both parameters by about an order of magnitude, namely, to come to the beam size of about 200 nm and the energy resolution of about 50 – 100 □eV.

The talk provides a short overview of the new instrumentation and corresponding scientific perspectives.

High pressure studies on magnetism and lattice dynamics by Nuclear Resonance Scattering

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Nuclear Resonance Scattering rapidly developed with the advent of third generation synchrotron radiation sources to a method covering a field of the hyperfine and phonon spectroscopy. High pressure applications are one of the domains where the technique is extremely powerful. The electronic, magnetic and vibrational properties of the solid state at high pressures can be studied by nuclear forward and inelastic scattering. Forthcoming upgrade of ESRF and other synchrotron sources to the extremely brilliant sources will extend the method in several directions. The application of the technique to study magnetic and lattice properties of compounds under high pressures will be presented in the talk with possible improvement of the results after upgrade.

The first part of the talk will be devoted to the study of the magnetism in Ni metal [1] and NiO [2] under applied pressure up to 260-280 GPa. The hyperfine magnetic splitting was observed up to the highest pressure which confirms that the compounds stay magnetic. Thus, the applied pressure above 300 GPa is required in order to identify critical pressure where magnetism is suppressed in Ni and NiO. This step in the experimental setup might be possible after improvement of the synchrotron source.

Lattice dynamics in the iron based superconductors under high pressure will be presented in the second part of the talk. The general scaling on Fe-As distance was found for the phonon modes in *L*FeAsO [3]. On the other hand, the significant change of the lattice dynamics across the tetragonal to collapsed tetragonal isostructural phase transition was found in RFe_2As_2. The explanation of this change can be related to the suppression of the magnetism and transition to the non-magnetic state in the collapsed phase. The investigation of the lattice dynamics in such compounds can gain significantly by improvement of the monochromator energy resolution which is expected after upgrade of the synchrotron source and nuclear resonance beamline.

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« A versatile Diamond Anvil Cell for X-ray inelastic, diffraction and imaging studies at synchrotron facilities»

Sylvain Petitgirard [a], Jeroen Jacobs [b], Valerio Cerantola [b], Chiara Cavallari [b], Leonid Dubrovinsky [a] and Christoph J. Sahle [b]

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We present a new Diamond Anvil Cell design –hereafter called mBX110- that combines both the advantages of a membrane and screws to generate high pressure. It enables studies at large-scale facilities for most of synchrotron X-ray techniques and set-ups with the possibility to remotely control the pressure and the ease of use of the screws in the laboratory. It is fully compatible with various gas-loading systems as well as high/low temperature environments in the lab or at large scale facilities. The mBX110 posses an opening angle of 85 degrees suitable for single crystal diffraction and a large side opening of 110 degrees which can be used for X-ray inelastic techniques such as X-ray Raman scattering spectroscopy but also for X-ray emission, X-ray Fluorescence or X-ray absorption. An even larger opening of 150 degrees can be manufactured enabling X-ray tomography.

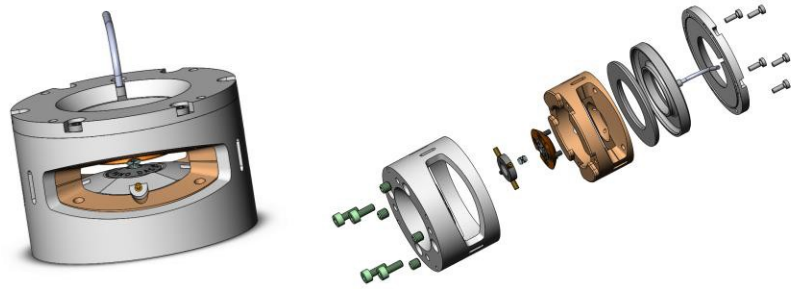

Figure 1: New Diamond Anvil Cell, mBX110, assembled and exploded view.

«Strongly correlated electron systems under high pressure and other extreme conditions»

Daniel Braithwaite

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Strongly correlated electron systems harbour some of the most fascinating properties of matter, such as strongly renormalized energy scales, and unusual electronic and magnetic properties. Among these the most striking is perhaps unconventional superconductivity, often co-existing with antiferromagnetic or ferromagnetic order, where the pairing mechanism is based on magnetic excitations rather than the usual electron-phonon interaction. High pressure is an extremely important parameter for the study of these systems as it can be used to modify the microscopic interactions and easily explore the different ground states of the rich and complex phase diagrams of these systems. Especially it can be used to tune a system exactly to the instability between different phases where many fascinating properties occur. The study of these systems often requires the association of 2 other extreme conditions, which are very low temperatures and high magnetic fields. These are challenging conditions for xray studies. I will show mainly the complementary measurement techniques under high pressure that have revealed the physics of these systems, and which may be useful for many of you even in very different subjects. I will also show some examples where synchrotron studies have brought valuable contributions to strongly correlated electrons and discuss future potential prospects

« The high pressure crystallography beamline ID15B »

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ID09A [1] was a state of the art high pressure diffraction beamline at the ESRF, carrying out monochromatic diffraction experiments with large area detectors. Powder and single crystal diffraction experiments could be performed at high pressures in diamond anvil cells, permitting accurate determination of crystallographic properties of the investigated samples. After more than 20 years of successful operation, ID09A has been closed in November 2015. It has been replaced by a new and vastly improved beamline, ID15B, which started operation in November 2016.

On ID15 two beamlines with a canted straight section have been constructed. The first one (ID15A) is for materials chemistry and engineering applications, the second one (ID15B) for monochromatic high pressure diffraction with large area detectors, replacing ID09A. Due to canting the two beamlines can be operated independently.

X-ray source for ID15B is the U20 in vacuum undulator from ID09A. The monochromator is a horizontally diffracting nitrogen cooled Si (111) single bounce Bragg monochromator. ID15B operates at a fixed angle with an energy of 30 keV. Experience with ID09A has shown, that 30 keV is well matched for high pressure diffraction experiments in DACs. Two transfocators with 200 μm diameter linear (1-D) beryllium compound refractive lenses for vertical and horizontal focusing, respectively, provide a highly variable and very clean beam with a minimum spot size on the sample of approximately 7 x 7 μm^2. The flux is comparable to ID09A and will increase by a factor 20 after the EBS upgrade. The experimental setup is located on an extremely stable granite table. Data are collected with the MAR555 flat panel detector, which will be replaced by an Eiger 2, 9M, CdTe detector after the upgrade. A X-ray camera for high resolution X-ray transmission microscopy can be installed behind the optical table about 5m from the sample (see figure).

ID15B offers similar possibilities for data collection as ID09A, powder and single crystal diffraction with high resolution well into the megabar pressure range, with, if requested, variable temperatures from a few to several hundred Kelvin.

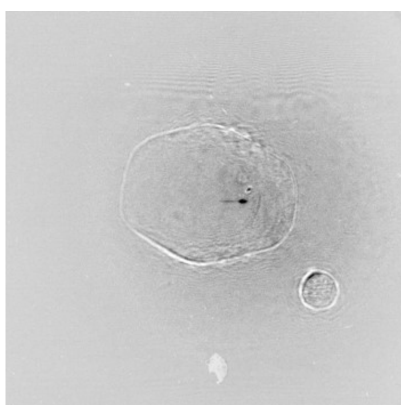

Figure: High resolution X-ray transmission image of a Sulphur crystal in a diamond anvil cell.

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«High pressure activity at the ESRF ID12 beamline»

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The scientific activity of the ESRF ID12 beamline is the investigation of the electronic and magnetic properties of materials exploiting the polarization dependence of the x-ray absorption near edge spectroscopy (XANES) in wide X-ray energy range (from 2 to 15 keV) [1]. X-ray magnetic circular dichroism (XMCD), that is the difference of XANES spectra taken with right and left circular polarization, is particularly interesting and allows to determine separately spin and orbital magnetic moments of the absorbing atoms in para-, ferri- or ferromagnetic systems. The combination of those spectroscopies with recent advances in high pressure technology has offered unique possibilities in understanding the physics of materials under pressure. Instrumental developments [2] at the ID12 beamline made now possible to study under pressure the XANES of light elements having absorption edges at photon energies below 4 keV, e.g. chlorine, sulphur and even phosphorus [3]. Further, XMCD experiments could be performed at high pressure in combination with low temperatures down to 2.7K and high magnetic field up to 8T.[4]. Selected examples showing the possibilities of the high pressure setups available at the beamline ID12 will be presented.

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In-situ spectroscopy of sulfur and critical metals in fluid-mineral-melt systems at high temperatures and pressures

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Our understanding of geological processes in the Earth's interior involving fluids and magmas, such as chemical element cycling and fractionation, magma generation and degassing, volcanic activity and ore deposit formation, to name a few, all require knowledge of metal and sulfur solubility, partitioning, and chemical speciation at depth. This knowledge generally comes from analyses of products brought to the Earth's surface and cooled down or quenched in laboratory experiments, and thus lacks direct data at elevated temperatures (*T*) and pressures (*P*).

In this contribution, I overview recent advances of in-situ spectroscopic approaches such as Raman spectroscopy and X-ray absorption spectroscopy (both XANES and EXAFS) for studies of sulfur speciation and partitioning in fluid-melt systems and the effect of sulfur on the solubility and transport of critical metals (gold and platinum) by geological fluids and formation of their economic resources. These spectroscopic data, combined with complementary thermodynamic and molecular modeling methods, reveal the formation, both in fluids and melts together with traditional sulfate and sulfide, of previously overlooked S chemical forms, the trisulfur and disulfur radical ions $S_3^{\bullet-}$ and $S_2^{\bullet-}$. These particular S species are stable at elevated *T-P* but cannot be preserved in quenched products due to their extremely fast breakdown to sulfate, sulfide and/or molecular sulfur on cooling. The radical ions have specific properties that distinguish them from traditional sulfur forms. The radical ions partition 10 to 1000 times more than sulfate and sulfide from silicate melts into the volatile aqueous phase, thereby enhancing sulfur degassing and transfer during magma generation in subduction zones. Furthermore, these species have an exceptionally high affinity for binding "chalcophile" metals such as Au, Pt or Mo in the fluid phase and thus greatly enhance these metals transfer across the lithosphere, from magmas to hydrothermal ore deposition sites.

These findings highlight the necessity of using in-situ spectroscopic approaches for studying high *T-P* "fugitive" fluid and melt phases inaccessible to direct observation or sampling; they thus open large perspectives for probing these "extreme" geological *milieux* using cutting-edge synchrotron techniques. Among these perspectives are, for example, the use of high-resolution XAS (using crystal analyzer spectrometers) for quantifying redox and structural state of critical trace metals in complex fluids and their host minerals; the design of novel diamond-anvil cells that would enable direct XANES measurements at the S K-edge of the identities and amounts of different S species in fluids, melts and minerals at high *T-P*; and the rapidly growing progress in development of laser-heated diamond-anvil cells that will open the door to a host of both laboratory and synchrotron in-situ studies of fluid-mineral-melt systems far beyond the *T-P* conditions of the lithosphere.

Introduction to resistively heated DAC techniques

Max Wilke

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This lecture will give an introduction to resistively heated diamond anvil cells (DAC) with illustration by several applications. On one hand side the Basset-type DAC, also known as the hydrothermal DAC, will be introduced. This cell is optimized for experiments using aqueous fluids as pressure medium or as sample. The sample chamber serves as an isochoric container and high P-T conditions (< 2 GPa, < 800°C) are achieved by resistive heating and very precise temperature measurements (±0.1°C). P is usually determined from the EOS of the fluid. This cell is generally used for studying element speciation in fluids and solid-liquid equilibration experiments. On the other hand resistively DACs are used with solid pressure media to study properties of solids at very high pressure at elevated temperatures. In this case, either the complete DAC is resistively heated, by a heater put around the anvils or by a gasket heater. To avoid oxidation of metal parts and the diamond anvils the cell is set into a vacuum chamber. These cells may reach up to 1300°C and have been used to determine phase properties, phase relations, crystallization kinetics and sample deformation. Temperature is often measured on the outer side of the diamond, the pressure is measured by pressure markers using XRD or spectroscopy. The performance of both cell types is illustrated by several examples.

FAME and FAME-UHD beamlines

D. Testemale

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A rapid overview of FAME and FAME-UHD X-ray spectroscopy beamlines will be given, and their capabilities for high-pressure research. In particular, the autoclaves that have been developed and are routinely used will be presented through scientific examples. Finally, the new opportunities related to the EBS upgrade will be discussed.

In-situ studies of high-temperature fluids and melts (P < 2 kbar) and their application to Geosciences

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The hydrothermal autoclave enables pressure and temperature conditions relevant to metamorphic and magmatic-hydrothermal crustal processes (200-900 °C and 300-1500 bars).

In this presentation, I will review how the hydrothermal autoclave can be combined with different spectroscopic technics (Raman, X-ray absorption) to study the physico-chemical properties of fluids and melts, with dedicated examples on the aqueous complexation of Rare Earth elements in mineralizing fluids and density of H_2O-CO_2-NaCl mixtures. I will also present new developments that are currently under way to enable the in-situ study of volcanic degassing.

Experimental studies at high P-T in the diamond anvil cell

Frédéric Datchi

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Experimental studies at high pressures and high temperatures in a diamond anvil cell (DAC) have seen a large growth in the last ~20 years thanks to numerous technical developments and their availability at an increasing number of laboratories such as synchrotron radiation facilities. The simultaneous generation of high pressure and high temperature allows addressing long-standing scientific issues such as the determination of the phase diagram, thermodynamic and dynamic properties of components of planetary interiors, bond and chemical stability at very high density, and have made possible the discoveries of novel phenomena such as molecular-to-superionic, insulator-to-metal and molecular-to-polymeric transitions in simple molecular solids and fluids.

This lecture will cover both technical and scientific topics related to experimental studies at high temperature in the diamond anvil cell. This includes:

- An overview of the DAC techniques based on resistive heating (RH-DAC)
- Pressure and temperature metrology in the RH-DAC
- Going beyond the temperature limit of the RH-DAC: induction and laser heating
- Making in-situ measurements at high P-T: examples of some x-ray and spectroscopic setups

I will illustrate the above topics with some examples of applications in the domains of fundamental and planetary physics, including melting line measurements, phase diagrams and chemical stability of simple molecules, structure of dense fluids and liquid-liquid transitions. Finally, I will discuss some prospective high P-T studies which should be made possible with the advent of the Extreme Brilliant Source of the ESRF.

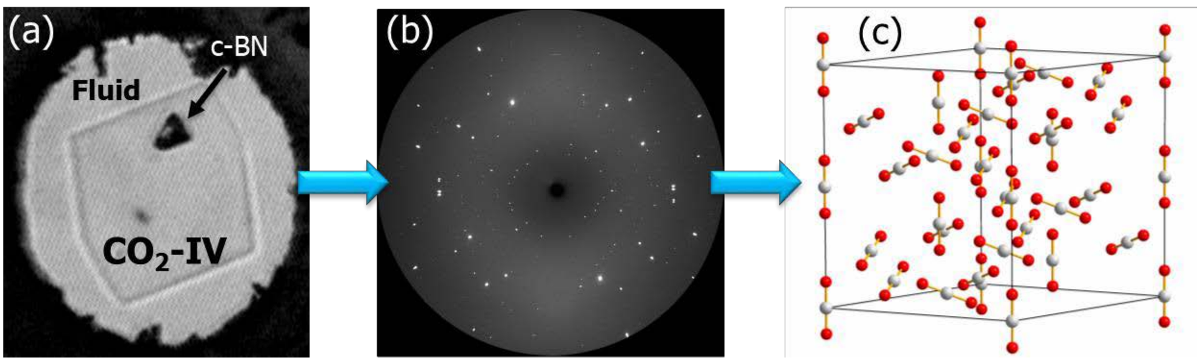


Figure 1: Structure of the high P-T solid phase IV of carbon dioxide. (a) Single crystal of phase IV grown in equilibrium with the fluid at 830 K, 12 GPa, as seen through the diamond anvils. (b) X-ray diffraction image measured at ESRF (c) Representation of the unit cell comprising 24 CO_2 molecules (C in white, O in red). After F. Datchi et al, Phys. Rev. Lett. 103 (2009)

Chemical controls on the solubility of metal bearing phases in hydrothermal solution

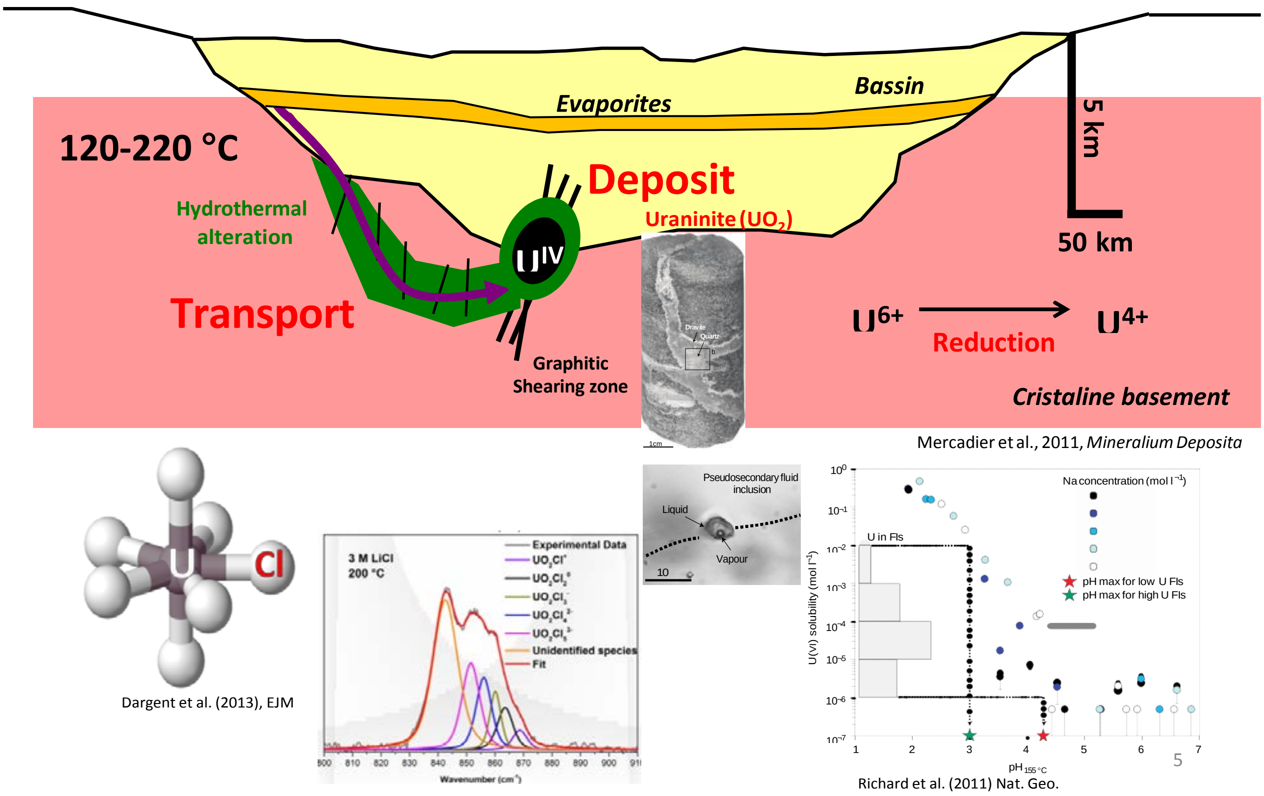
L. Truche

ISTerre, University Grenoble Alpes, CNRS

In this lecture, I will address the following topics:

- How do we form an ore deposits?
- Chemical composition of hydrothermal fluids,
- Water properties at elevated T-P,
- Metal complexation and mineral solubility in aqueous solution,
- How to study metal speciation in hydrothermal fluids?

Genetic model for unconformity U ore deposits



List of Posters

Poster Number	Name	Laboratory	Document title
1	BARANNIKOV Aleksandr	Immanuel Kant Baltic Federal University	X-ray microscopy implementation in High Pressure research
2	BRAZHNIKOVA Anastasia	Institute of Geology and Mineralogy	Study of K2O-B2O3-GeO2 glasses at pressure up to 9 GPa
3	CELESTE Anna	Synchrotron Soleil	Pressure response of the metal-organic framework MIL-101 filled with metal nanoparticles
4	CHAKRABORTI Amrita	CNRS CEA UMR 7642	Ex-situ and in-situ experiments on Paris Edinburgh Press
5	CHEN Qi	Sorbonne Universite - IMPMC	Partitioning of heavy noble gases between planetary reservoirs at high pressure
6	DE ANGELIS Francesco	ESRF	Effect of the fcc-hcp martensitic transition on the compression behaviour of solid krypton up to 65 Gpa from EXAFS and XRD
7	FARSANG Stefan	University of Cambridge	Carbonate mineral solubility in deep Earth
8	FIGUEIREDO SOARES RODRIGUES Joao Elias	CSIC - ICMM	First-principles calculations and Raman scattering evidences for local symmetry lowering in rhombohedral ilmenite: temperature- and pressure-dependent studies
9	GARBARINO Gaston et al.	ESRF Grenoble	High Pressure Technology Network
10	GARBARINO Gaston et al.	ESRF Grenoble	Extreme conditions science at ESRF
11	GARBARINO Gaston et al.	ESRF Grenoble	Pressure-temperature phase diagram on strongly correlated electron systems
12	GIL ALABARSE Frederico	Elettra Sincrotrone Trieste	Synthesis of new conducting polymer based nano-composite performed at Xpress – the high pressure diffraction beamline of the Elettra Sincrotrone Trieste
13	GORDEEVA Alisa	Stockholm University	Hydrous zinc hydroxide, Zn5(OH)10x2H2O, obtained in hydrothermal environments at gigapascal pressures – an intermediate toward cubic ZnO?
14	GUTIERREZ CANO Vanessa	Universidad de Cantabria	Raman Study of LaGdO3 under High Pressure
15	JARA Enrique	Universidad de Cantabria	Structural correlations in Cs2CuCl4: pressure dependence of electronic and vibrational structures
16	JONANE Inga	University of Latvia	High pressure effect on the local structure of a-MoO3 and a-HxMoO3
17	KRYSIAK Sonia	AGH University of Science and Technology	Influence of Cd2+ ions on properties of non-heme iron in bacterial reaction centers of type II
18	LARANJEIRA Jorge Diogo	Universidade de Aveiro	3D C60 polymers with ordered binary-alloy type structures
19	LASKAR Clément	CNRS UMR 5563	The impact of sulfur on the transfer of platinoids by geological fluids
20	MARTIN SANCHEZ Camino	Universidad de Cantabria	Monodisperse gold nanorod for high-pressure refractive index sensing
21	MARTIROSYAN Naira	GFZ German Research Centre for Geosciences	Pressure induced phase transitions in Ca-carbonate and effect of cationic substitution on the phase behavior
22	NIKIFOROVA Yulia	Institute of Crystallography (RAS)	Equation of state, structural and magnetic transitions in a novel multiferroic Ba3TaFe3Si2O14 at high pressures.
23	NUNEZ Julius Andrew	Institut Neel - CNRS UPR 2940	High Pressure and High Temperature synthesis of new Magnesium-based Hydrides
24	PENNACCHIONI Lea	GFZ German Research Centre for Geosciences	Brillouin spectroscopy studies on Calcium Carbonate
25	POREBA Tomasz	Paul Scherrer Institute (PSI)	Pressure-induced polymerization and electrical conductivity in a polyiodide
26	PORTNIAGIN Aresenii	Institute of Chemistry of FEBRAS	Spark Plasma Sintering Technology to obtain Functional Ceramics for Various Applications
27	ROMANENKO Aleksandr	Sobolev Inst. of Geology & Mineralogy SB RAS	High-pressure synthesis and study of single-crystal K-cymrite
28	ROSA Angelika	ESRF Grenoble	Extreme conditions programme at BM23/ID24 after the EBS upgrade
29	ROSA Angelika	ESRF Grenoble	Local structural changes at silicate melts at extreme conditions
30	SADOVYI Bohdan SADOVYI Petro	Polish Academy of Sciences	Nature of solid-solid phase transition induced by high pressure and temperature in GaN experimentally studied by Ga K-edge EXAFS and supported by DFT calculations
31	SEARS Jennifer	DESY	High Pressure Resonant X-ray Diffraction Set up at Beamline P09
32	SEMERIKOVA Anna	Sobolev Inst. of Geology & Mineralogy SB RAS	Cubic platinum hydride synthesis at high-pressure high-temperature conditions
33	SHAKHOV Fedor	Ioffe Institute	Diamonds synthesized at HPHT without metal catalyst
34	SIEBER Melanie	GFZ German Research Centre for Geosciences	Carbonate melting at 200-300 km and partition coefficients for trace elements into carbonate melt
35	STARCHIKOV Sergey	Shubnikov Institute of Crystallography (RAS)	Structural, magnetic and electronic properties of the langasite type multiferroic Ba3NbFe3Si2O14 observed by X-ray diffraction and synchrotron Mössbauer source spectroscopy at high pressures
36	TORCHIO Raffaella	ESRF Grenoble	High Power Laser Facility at the ESRF
37	TORCHIO Raffaella	ESRF Grenoble	Melting and local structure of liquid 3d metals under extreme conditions of P and T.
38	YAO Yi	Karlsruhe Institute of Technology (KIT)	Raman scattering study of lattice and magnetic excitations in CrAs
39	ZADOIA Anastasiia	University of Lille	Towards new oxo-centered architectures using HP-HT technique.

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