Contents

- Introduction ESRF MEx- High Pressure Lab.
- Examples HP Lab. collaboration projects.
- HP-Lab. loan pool equipment.
- Introduction new Panoramic DAC mBX110.
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Diamond Anvil Cell (DAC) sample preparation laboratory
- SERVICE to DAC experiments on the ESRF beam lines
- LOAN POOL with all equipment for DAC experiments
- DEVELOPMENT of beam line requested projects…

Mex, HP laboratory spaces:
Room 21.0.09: Mbar DAC-exp. preparation space, Femto laser.
Room 21.0.11: DAC Gas Loading System
Room 21.0.12: User DAC preparation space
Room 21.0.13: Super-User + DAC reservation preparation space

Contact:
gaston.garbarino@esrf.fr
jeroen.jacobs@esrf.fr

• Pressure range: 0.1 Gpa → 150 Gpa (1.5 Mbar)
• Temperature range: 5 Kelvin → 1300º K
HP LAB, LOAN POOL EQUIPMENT

Beamlines:
✓ ID12
✓ ID15B
✓ ID18
✓ ID20
✓ ID24
✓ ID27
✓ ID28
✓ BM23

General use DAC (20pcs)
Mbar use (6pcs)

Low temp. DAC (20pcs)

Panoramic DAC (4pcs)

High Temperature (resistive) DAC, 8pcs.
Medium Temperature (heater-ring), 4pcs.

Manual pressure drives

Automatic pressure drives

**Laboratory (free access) equipment:**

Laser drill, for gasket hole drilling / sample cutting

Femto laser, Mbar applications (under construction)

Leica, Mbar exp. microscope

Gas Loading System

2x Pressure by Ruby Luminescence measuring devices

Hands on! High-pressure techniques at the ESRF-EBS, 20/06/2019, J.Jacobs
### DAC RESERVATION PERIOD, JULY – DECEMBER (SHUTDOWN) 2018.

<table>
<thead>
<tr>
<th>Beam line</th>
<th>Requestor</th>
<th>User</th>
<th>Date 2018</th>
<th>Number Cells</th>
<th>Gas loading</th>
<th>Culet sizes</th>
<th>Comments</th>
<th>Experiment number</th>
</tr>
</thead>
<tbody>
<tr>
<td>ID24</td>
<td>Marija Krstulovic</td>
<td>inhouse</td>
<td>03/07 - 12/07</td>
<td>2</td>
<td>1x150*300NANO+1x250NANO</td>
<td>inhouse</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID24</td>
<td>Raffaela Torchio</td>
<td>06/07 - 12/07</td>
<td>1</td>
<td>1x300NANO</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID28/ID15B</td>
<td>Girard/Hantlend</td>
<td>Daniele</td>
<td>04/07 - 10/07</td>
<td>1</td>
<td>Ne</td>
<td>1x150*300</td>
<td>ES-748</td>
<td></td>
</tr>
<tr>
<td>ID27</td>
<td>Volodymyr Svityk</td>
<td>inhouse</td>
<td>09/07 - 17/07</td>
<td>1</td>
<td>600 cryo</td>
<td>inhouse</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID20</td>
<td>Christoph Sahle</td>
<td>Stella</td>
<td>11/07 - 17/07</td>
<td>2</td>
<td>1x250 + 1x300</td>
<td>ES-774</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID20/ID09</td>
<td>Christoph Sahle</td>
<td>M.Sander/Wilke</td>
<td>11/07 - 17/07</td>
<td>0</td>
<td>He</td>
<td>BX90 user provided</td>
<td>ES-773</td>
<td></td>
</tr>
<tr>
<td>ID15A</td>
<td>Valerio Cerantola</td>
<td>Marco</td>
<td>12/07 - 20/07</td>
<td>1</td>
<td>1x150*300 (cylinder ringcracked)</td>
<td>TEST</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID27</td>
<td>Volodymyr Svityk</td>
<td>B.Weihinger</td>
<td>23/07 - 30/07</td>
<td>2</td>
<td>1x500 + 1x300</td>
<td>ext restitive heating 50degrees C.</td>
<td>HC-3691</td>
<td></td>
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<tr>
<td>ID12</td>
<td>Fabrice Wilhelm</td>
<td>IHR</td>
<td>24/07 - 28/07</td>
<td>1</td>
<td>He</td>
<td>600 cryo PP+full</td>
<td>IHR</td>
<td></td>
</tr>
<tr>
<td>ID27</td>
<td>Gaston Garbarino</td>
<td>G.Gergiou</td>
<td>20/08 - 28/08</td>
<td>3</td>
<td>He</td>
<td>1x250cryo + 1x300cryo + 1x350*400</td>
<td>Laser+gasloading+cryo</td>
<td></td>
</tr>
<tr>
<td>ID27</td>
<td>Gaston Garbarino</td>
<td>Santoro</td>
<td>11/09-21/09</td>
<td>4</td>
<td>3xcryo + 1xamb.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID12</td>
<td>Fabrice Wilhelm</td>
<td>Pokrovsk</td>
<td>12/09-17/09</td>
<td>3</td>
<td>2xcryo perf+600 plus 30or20micron disks</td>
<td>ES-786</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID20</td>
<td>Chiara Cavallari</td>
<td>Vittoria</td>
<td>25/09 - 02/10</td>
<td>2</td>
<td>5xNANO</td>
<td>laserheating</td>
<td>ES-865</td>
<td></td>
</tr>
<tr>
<td>ID27</td>
<td>Volodymyr Svityk</td>
<td>cepatelli</td>
<td>01/10-05/10</td>
<td>3</td>
<td>2<em>300+1x150</em>300</td>
<td>CH-5606</td>
<td></td>
<td></td>
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<tr>
<td>ID27</td>
<td>Mohamed Mezouar</td>
<td>Sebastian Vogel</td>
<td>16/10 - 19/10</td>
<td>2</td>
<td>2x250microns</td>
<td>CH-5609</td>
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</tr>
<tr>
<td>bm23</td>
<td>Angelika Rosa</td>
<td>Rodriguez</td>
<td>17/10-23/10</td>
<td>6</td>
<td>NPds+standard</td>
<td>HC-3913</td>
<td></td>
<td></td>
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<tr>
<td>ID18</td>
<td>Valerio Cerantola</td>
<td>Petitgirard</td>
<td>18/10-23/10</td>
<td>1</td>
<td>Mbar 150*300</td>
<td>HC-4029</td>
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<tr>
<td>ID24</td>
<td>Rosa/Marija</td>
<td>Rosa/Marija</td>
<td>24/10 - 30/10</td>
<td>5</td>
<td>5xNANO</td>
<td>laserheating</td>
<td>ES868</td>
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<tr>
<td>BM23/ID27</td>
<td>Rosa/Garbarino</td>
<td>Koemets</td>
<td>31/10 - 05/11</td>
<td>4</td>
<td>4x250 sc</td>
<td>ES-866</td>
<td></td>
<td></td>
</tr>
<tr>
<td>BM23</td>
<td>Angelika Rosa</td>
<td>Michaela Souliou</td>
<td>08/11-13/11</td>
<td>3</td>
<td>Cryo</td>
<td>CH-3915</td>
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<tr>
<td>ID27</td>
<td>Volodymyr</td>
<td>Zakharov</td>
<td>08/11 - 11/11</td>
<td>2</td>
<td>2xcryo 500</td>
<td>loaded by pentane-isopentane mixture</td>
<td>CH-5600</td>
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<tr>
<td>ID27</td>
<td>Mezouar</td>
<td>Kamil</td>
<td>11/11-16/11</td>
<td>3</td>
<td>2x Mbar 150*300 to be reimbursed</td>
<td>laser +oxygen +Mbar</td>
<td>ES-814</td>
<td></td>
</tr>
<tr>
<td>ID20</td>
<td>Christoph Sahle</td>
<td>20/11-06/12</td>
<td>2</td>
<td>2xPano</td>
<td>Inhouse</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID24</td>
<td>Angelika Rosa</td>
<td>Redfern</td>
<td>21/11-26/11</td>
<td>5</td>
<td>Ext external heating + vacuum trolley</td>
<td>ES-791</td>
<td></td>
<td></td>
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<tr>
<td>ID27</td>
<td>G.Garbarino</td>
<td>S.Boccati</td>
<td>23/11-23/11</td>
<td>5</td>
<td>2xMbar+100+250+300</td>
<td>HC-3943</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ID15</td>
<td>Ines Collings</td>
<td>26/11-02/12</td>
<td>2</td>
<td>2xcryo 500</td>
<td>ES-807</td>
<td></td>
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</table>

<table>
<thead>
<tr>
<th>Period</th>
<th>03/07/18 – 02/12/18</th>
<th>7 different beamlines (bridging Groups), 72 DAC’s + vacuum chamber equipment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total number of HP-DAC loans:</td>
<td>2017 - 2018 = 166</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2016 - 2017 = 172</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2015 - 2016 = 177</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2014 - 2015 = 143</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2013 - 2014 = 131</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2012 - 2013 = 93</td>
<td></td>
</tr>
</tbody>
</table>

More DAC preparations experiment dedicated configurations:  
- Partially or perforated anvils set-up  
- Nano anvils set-up
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First cryostat was introduced on ID27 in 2010. Today both ID15B and ID28 have their own.

Sample Environment Service, ID27, - HP-Lab., collaboration.

Temperature range: \( \sim 4K - 350K \)
Descente Tambient \( \rightarrow 15 K \) in \( \sim 40 \text{min} \)
\( 15 K \rightarrow \text{Tambient} \) \( \sim 1 \text{hour} \)
Mechanical stability\(< 1 \mu m \) after 4-5 hours at controlled T
Thermal stability\(< 10^{-2} \) 15 min after regulation
CONSTANT UPGRADE, 2 EXAMPLES.

CuNi Resistance wire (150 µ) for internal heating

Viton O-ring replaced by laser welded disk - Prevent Helium leakage at lowT.
Chervin DAC dedicated cryostat (BM23)

Temperature range: ~4K - 350K
Descente Tambient → 15 K in ~40min
15 K → Tambient ~1 hour
Mechanical stability< 1um after 4-5 hours at controlled T
Thermal stability< $10^{-2}$ 15 min after regulation

Cryo-DAC Cell cover + membrane modification for utilization HP-Lab loan pool DAC’s
Specifications of the High Pressure XMCD project on ID12

**Short description:**
Experimental set-up for XMCD under **high pressure, high field (6T) and low temperatures (2-5 Kelvin)**, Energy range 3.5 keV to 15 keV

Diamond window, type Ia, Dia. 550mm, low birefringence, Raman ultra low fluorescence 80, 50 and 30 micron disks used. Pmax. = 2 Gpa ~ 15 Gpa.

High pressure XANES and XMCD in the tender X-ray energy range

Based on ID12 experience A.Rosa is implementing same principal on ID24/BM23.
Temperature range: ~4K - 350K
Descente Tambient $\rightarrow$ 15 K in ~40min
15 K $\rightarrow$ Tambient ~1hour
Mechanical stability < 1µm after 4-5 hours at controlled T
Thermal stability < $10^{-2}$ 15 min after regulation

**ID12 Future project request:**
- Partially perforated experiments @ 40GPa.
- Fully perforated anvil experiments @ 10GPa with 30µ disk.
  (smaller culet sizes anvil + disk)

Fabrice Wilhelm
12h15
Contents

- Introduction ESRF MEx- High Pressure Lab.
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- HP-Lab. loan pool equipment.
- Introduction new Panoramic DAC mBX110.
- Pressure range: 0.1 GPa - 125 GPa
- Temperature range: 300 K to 450 K.
- Optical access to sample: Working distance ≥ 13 mm.
- Accessible electromagnetic spectrum: Visible, X rays.
- Techniques: Diffraction/scattering/spectroscopy
- Materials: Maraging Steels & Tungsten Carbide
- Sizes: Diameter 50 mm, Height 32 mm
- Perfect guidance, due to 4 pins
- Easy access for sample preparation
- It’s capacity covers about 90% of demand by users
- Low maintenance time

Helium pressure via drive

Micro valve

MEMBRANE
Spacer
Piston
Halve sphere seat (alignment parallelism)
Flat seat (alignment x-y movement)
Cell cover
Cylinder
ALIGNING DIAMOND ANVILS.

BOEHLER-ALMAX 250X300 MICRON (BEVELED)

1st view
non aligned anvil

2nd view
(x-y position has been aligned)

3rd view
(Fringes almost suppressed by aligning half sphere)

4th view
(Fringes are gone, last x-y correction)
Pressure range: 0.1 GPa - 60 GPa
Temperature range: 5 K to 300 K.
Optical access to sample: Working distance ≥ 13 mm
Access electromagnetic spectrum: Visible, X rays
Techniques: Diffraction/scattering/spectroscopy
Sizes: Diameter 50 mm, Height 32 mm

Materials:
- CuNi2Be, high thermal conductivity
- Non magnetic Stainless steel (316L)
- Tungsten Carbide WC 93.9 %, 6% Nickel. Ni06.
- Graphite coating, piston & cylinder.
- Gold coating cell cover.

RESISTIVE HEATING HT-DAC + VACUUM CHAMBER

ID27, ID24/BM23, HP-Lab. collaboration.

- Pressure range: 0.1 GPa - 80 GPa
- Temperature range: 300 K to 1200° Celsius
- Optical access to sample: Working distance ≥ 13 mm.
- Accessible electromagnetic spectrum: Visible, X rays.
- Techniques: Diffraction/scattering/spectroscopy
- Materials: Pyrad53NW & HT Tungsten Carbide
- Sizes: Diameter 50 mm, Height 32 mm max
- Diamonds: Extra high, 2.72 mm (standard 1.72 mm)

Start-up project – 2010.

Title: In situ monitoring of phase transformation microstructures at Earth’s mantle pressure and temperature using multi-grain XRD
Author(s): A.D. Rosa*, N. Hilairet, S. Ghosh, G. Garbarino, J. Jacobs, J-P. Perrillat, G. Vaughan and S. Merkel

Applications of amorphous boron composite gaskets for high pressure and temperature diamond anvil cell experiments
Author(s): A.D. Rosa*, M. Merkulova, G. Garbarino, V. Svitylik, J. Jacobs, O. Mathon, M. Munoz, S. Merkel
RESISTIVE HEATING HT-DAC + UPGRADE CONNECTORS

**Version 1.**

**Version 2.**

**Version 3.**

Resbond 931 GRAPHITE / GRAPHITE BI-COMPOSANTS
- 100% graphite
- 3000°C max.
- Good electrical conductivity

Rapport de stage de fin d'études (trainée report) du DUT Mesures Physiques, Romain Jarnias.

Conclusion: 3rd version:
- Easier to prepare
- Reusable after HT experiment
- Future heater upgrades, Zircon or Alumine, Pyrophyllite.
**HEATER RING VACUUM CHAMBER**

**ID28, HP-Lab. collaboration.**

- Pressure range: n.a
- Temperature range: Ambient to 600° Celsius
- Optical access to sample: Working distance ≥ 13mm.
- Accessible electromagnetic spectrum: Visible, X rays.
- Techniques: Diffraction/scattering/spectroscopy
- Materials: Stainless steel, 316L
- Sizes: 140x154x57 (mm.)

*Critical scattering and incommensurate phase transition in antiferroelectric PbZrO$_3$ under pressure*


Numerously used also on ID24, BM23 and ID27. Therefor designed adapted, chamber for general-use.

**Introduction HP-Lab - 2013 (V1) - 2016 (V2)**
Classical Pano-DAC for X-Ray & Raman scattering experiments at ID20

- Pressure range: 0.1GPa - 20 Gpa (Be-gaskets)
- Temperature range: 300 K.
- Optical access to sample: Working distance ≥ 13mm.
- Access electromagnetic spectrum: Visible, X rays.
- Materials: Maraging Steels & Tungsten Carbide
- Sizes: Diameter 50mm, Total Height 69.5mm.

- Opening angles: 80 degrees x 140 degrees
- Used with Beryllium gaskets, in-house laser drilling.

PROTOCOL for work procedure: Laser drilling of Beryllium gaskets at ESRF, on HP-Lab Laser drill facility + Be dedicated glovebox.

Constrains:
- Difficult to gas-load (max.height)
- Laser heating almost impossible
- Single crystal diffraction impossible
Direct tomography imaging for inelastic x-ray scattering experiments at high pressure
Ch.J.Sahle,y, A.D.Rosa, M.Rossi, V.Cerantola, G.Spiekermann, S.Petitgirard, A.Mirone, J.Jacobs, S.Huotari, and M.Moretti.

Beryllium gaskets suitable for pressures up to 10 GPa
J. Macavei and H. Schulz
Institut für Kristallographie der Universität München, Theresienstrasse 41, D-8000 München 2, Federal Republic of Germany
(Received 11 August 1989; accepted for publication 22 April 1990)
A systematic study of the possibility of using beryllium gaskets in diamond-aviol pressure cells was carried out. Six different Be-BeO alloys were tested and treated in different ways in order to obtain Be gaskets suitable for pressures up to 10 GPa. The beryllium quality, the shape of the diamond anvils, the thickness of the gasket, the diameter of the sample chamber, and the depth of the gasket preindentation determine the highest obtainable pressure and its stability.

Be-IP70: Dia.5mm – Indent surface thickness 200micron - best compromise, material hard and brittle.

<table>
<thead>
<tr>
<th>TABLE 1: The used Be grades and their properties, as specified by the producer (Brush-Wellman Company).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Properties</td>
</tr>
<tr>
<td>wt. %</td>
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<tr>
<td>Be</td>
</tr>
<tr>
<td>BeO</td>
</tr>
<tr>
<td>Fe</td>
</tr>
<tr>
<td>Al</td>
</tr>
<tr>
<td>Grain size (μm)</td>
</tr>
<tr>
<td>Density theor. (g/cm³)</td>
</tr>
<tr>
<td>modulus (GPa)</td>
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<tr>
<td>Ultimate tensile strength (GPa)</td>
</tr>
<tr>
<td>trans.</td>
</tr>
<tr>
<td>long.</td>
</tr>
<tr>
<td>Yield tensile strength (GPa)</td>
</tr>
<tr>
<td>trans.</td>
</tr>
<tr>
<td>long.</td>
</tr>
<tr>
<td>Elongation (%)</td>
</tr>
<tr>
<td>trans.</td>
</tr>
<tr>
<td>long.</td>
</tr>
</tbody>
</table>

The European Synchrotron | ESRF
ID20 request: Design new cell specialized for their current and future needs:

- Compact design allowing secondary probes:
  - X-ray Emission Spectroscopy (XES),
  - X-ray Absorption Spectroscopy (XAS),
  - X-ray Power Diffraction (XRD), Raman, IR.
  - X-ray Scattering (XRS) through diamond or side (110° opening)
  - Single crystal diffraction (80° opening)

- Easy alignment of the diamonds
- Gas-loading system compatible

- Laser heating, double sided, compatible (EBS upgrade)
- Combine membrane and screws
- Possibility to use the mini diamonds (Sylvain Petitgirard's design, Dia.1mm, H:0.575mm)
- Allow both “through gasket” and “through diamond” geometry, with satisfactory, solid angle.

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Petitgirard et al. JSR 2018

Start-up project – 2017.
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,
- X-ray emission,
- X-ray Fluorescence,
- X-ray absorption.

An even larger opening of 150 degrees can be manufactured enabling X-ray tomography.

Compatible with most of the standard techniques used:
- single crystal diffraction,
- large angle powder diffraction,
- Brillouin scattering spectroscopy,
- and laser heating.

Fully compatible with various gas-loading systems.

mBX110 combines both the advantages of a membrane and screws to generate high pressure.
PANORAMIC DAC, MBX110.

The mBX110 is available in two versions:
- Rotatable piston half sphere seat (fig 1.)
- Fixed piston (fig.2)

- Fixed piston (possibility) radial opening angles: 85 degrees, \( P_{\text{max}} = 50 \text{Gpa} \).

Single crystal diffraction, large angle powder diffraction, Brillouin 25 scattering spectroscopy, and off-axis laser heating.

www.almx-easylab.com
Stishovite single crystal data in Helium on ID15B

Rutile to CaCl2 structural change at ~ 40 GPa

c/a ratio of the crystal when fitted to the 3 stishovite structure or CaCl2 structure. It shows the changes from rutile to CaCl2 4 structure take place at about 40 GPa in Helium medium.

Insert, picture of the loading 5 with the 2 double polished samples (stishovite and GeO2 powder) cut with the FIB.

Data courtesy of S.Petitgirard

Stishovite as 6-fold coordination reference spectra for XRS at ID20

Si L-edge and O K-edge spectra up to 63 GPa

Petitgirard et al. GPL 2019

A versatile Diamond Anvil Cell for X-ray inelastic, diffraction and imaging studies at synchrotron facilities
Sylvain Petitgirard, Jeroen Jacobs, Valerio Cerantola, Ines E. Collings, Remi Tucoulou, Leonid Dubrovinsky and Christoph J. Sahle.
Thank you for your attention.