

# **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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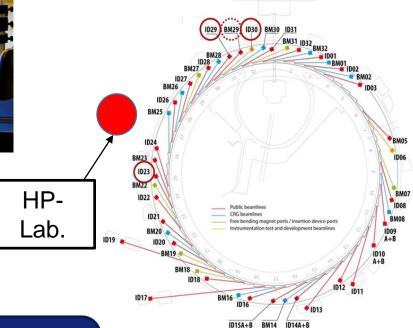


### MEX, HIGH PRESSURE LABORATORY.



#### Diamond Anvil Cell (DAC) sample preparation laboratory

<u>SERVICE</u> to DAC experiments on the ESRF beam lines
 <u>LOAN POOL</u> with all equipment for DAC experiments
 <u>DEVELOPMENT</u> of beam line requested projects...



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

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

•Pressure range : 0.1 Gpa  $\rightarrow$  150 Gpa (1.5 Mbar)

•Temperature range : 5 Kelvin  $\rightarrow$  1300° K



### HP LAB, LOAN POOL EQUIPMENT



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



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

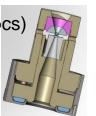






Panoramic DAC (4pcs)

Low temp. DAC (20pcs)





Manual pressure drives

Automatic pressure drives



<u>Beamlines:</u> ✓ID12

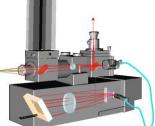
✓ID15B



Laboratory (free access) equipment:

Laser drill, for gasket hole drilling / sample cutting Femto laser, Mbar applications (under construction)

Leica, Mbar exp. microscope



2x Pressure by Ruby Luminescence measuring devices Green laser: 55 GPa max., Blue laser: 150 GPa max.

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✓BM23



### DAC RESERVATION PERIOD, JULY – DECEMBER (SHUTDOWN) 2018.

Beam line	Requestor	User	Date 2018	Number Cells	Gas loading	Culet sizes	Comments	Experiment numb
ID24	Marija Krstulovic	inhouse	03/07 - 12/07	2		1x150*300NANO+1x250NANO		inhouse
ID24	Raffaella Torchio	11110036	06/07 - 12/07	1		1x300NANO		milliouse
ID28/ID15B	Girard/Hanfland	Daniele	04/07 - 10/07	1	Ne	1x150*300		ES-748
ID27	Volodymyr Svitlyk	inhouse	09/07 - 17/07	1	110	600 cryo.		inhouse
ID20	Christoph Sahle	Stella	11/07 - 17/07	2		1x250 + 1x300		ES-774
ID20/ID09	Christoph Sahle	M.Sander/Wilke	11/07 - 17/07	0	He	BX90 user provided		ES-773
ID15A	Valerio Cerantola	Marco	12/07 - 20/07	1	110	1x150*300 (cylinder ringcracked)		TEST
ID27	Volodymyr Svitlyk	B.Wehinger	23/07 - 30/07	2		1x500 +1x300	ext restitive heating 50degrees C.	HC-3691
ID12	Fabrice Wilhelm	IHR	24/07 - 28/07	1	He	600 cryo PP+full	on room to nouning boungrood of	IHR
10.12			2001 2001		110			
						1x250cryo + 1x300cryo +		
ID27	Gaston Garbarino	G.Gergiou	20/08 - 28/08	3	He	1x350*400	Laser+gasloading+cryo	
ID27	Gaston Garbarino	Santoro	11/09-21/09	4		3xcryo + 1xamb.		
						2xcryo perf+600 plus 30or20micron		
ID12	Fabrice Wilhelm	Pokrovsk	12/09-17/09	3		disks		ES-786
	Silvia Boccota		21/09 - 05/10	5		5xNANO	laserheating	ES-865
ID20	Chiara Cavallari	Vittoria	25/09 - 02/10	2		2x Pano-DAC, 500 sapphires		
ID27	Volodymyr Svitlyk	cepatelli	01/10-05/10	3		2*300+1x150*300		CH-5606
ID27	Mohamed Mezouar	Sebastian Vogel	16/10 - 19/10	2	Ne	2x250microns		CH-5609
bm23	Angelika Rosa	Rodriquez	17/10-23/10	6		NPd's+standard		HC-3913
ID18	Valerio Cerantola	Petitgirard	18/10-23/10	1		Mbar 150*300		HC-4029
ID24	Rosa/Marija	Rosa/Marija	24/10 - 30/10	5		5xNANO	laserheating	ES868
BM23/ID27	Rosa/Garbarino	Koemets	31/10-05/11	4	Ne	4x250 sc		ES-866
BM23	Angelika Rosa	Michaela Souliou	08/11-13/11	3		Cryo		HC-3915
DIVIZO	Aligelika Kusa	Wilchaela Soullou	00/11-13/11	3		Ciyo	loaded by pentane-isopentane	10-3915
ID27	Volodymyr	Zakharov	08/11 -11/11	2		2xcryo 500	mixture	CH-5600
ID27	Mezouar	Kamil	11/11-16/11	3		3x Mbar 150*300 to be reimbursed		ES-814
ID20	Christoph Sahle		20/11-06/12	2		2xPano		Inhouse
ID24	Angelika Rosa	Redfern	21/11-26/11	5			external heating + vacuum trolley	ES-791
ID27	G.Garbarino	S.Boccato	23/11-29/11	5		2xMbar+100+250+300	Housing - Facture Holloy	HC-3943
ID15	Ines Collings	0.200000	26/11-02/12	2		2xcryo 500		ES-807
.2.10	inter comingo		20,02,.2					20 001
			2018	166				

Period 03/07/18 – 02/12/18 : <u>7 different beamlines (bridging Groups)</u>, <u>72 DAC's</u> + vacuum chamber equipment Total number of HP-DAC loans:

2017 - 2018 = 166

2016 - 2017 = 172

- 2015 2016 = 177
- 2014 2015 = 143 2013 - 2014 = 131
- 2013 2014 = 13
- 2012 2013 = 93

More DAC preparations experiment dedicated configurations:

- Partially or perforated anvils set-up
- Nano anvils set-up



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### **EXAMPLE SESS ENGINEERING EQUIPMENT**

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

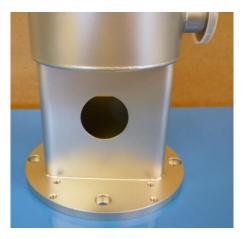
Temperature range: ~4K - 350K Descente Tambient  $\rightarrow$  15 K in ~40min 15 K  $\rightarrow$  Tambient ~1hour Mechanical stability< 1um after 4-5 hours at controlled T Thermal stability< 10<sup>-2</sup> 15 min after regulation

First cryostat was introduced on ID27 in 2010. Today both ID15B and ID28 have there own.



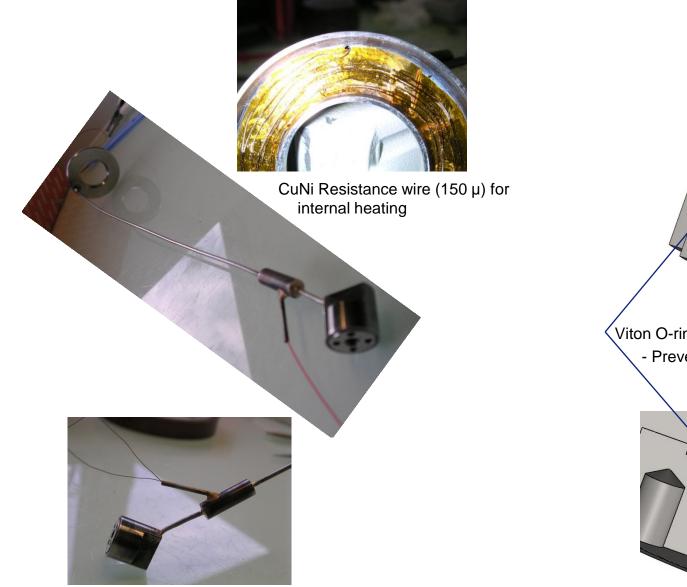




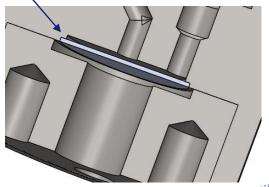




### CONSTANT UPGRADE, 2 EXAMPLES.



Viton O-ring replaced by laser welded disk - Prevent Helium leakage at lowT.





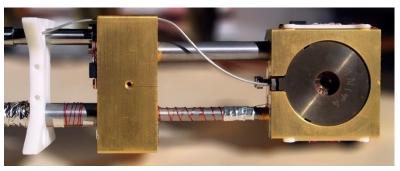
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#### Sample Environment Service, - ID24, BM23, HP-Lab. collaboration.

Cryostat ID24 can be used with HP-lab Cryo-DAC from 2011.

### Chervin DAC dedicated cryostat (BM23)

Temperature range: ~4K - 350K Descente Tambient  $\rightarrow$  15 K in ~40min 15 K  $\rightarrow$  Tambient ~1hour Mechanical stability< 1um after 4-5 hours at controlled T Thermal stability< 10<sup>-2</sup> 15 min after regulation



Cryo-DAC Cell cover + membrane modification for utilization HP-Lab loan pool DAC's





### **EXAMPLE SESS ENGINEERING EQUIPMENT**

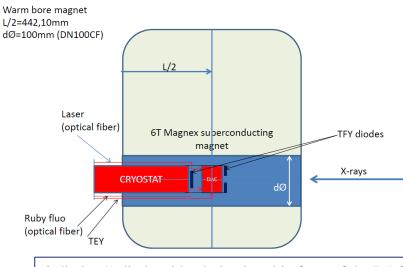
ID12, ID27, HP-Lab. collaboration.

**Specifications of the High Pressure XMCD project on ID12** 

#### Short description:

#### Experimental set-up for XMCD under <u>high pressure, high field</u> (6T) and low temperatures (2-5Kelvin), Energy range 3.5 keV to

15 keV Side view

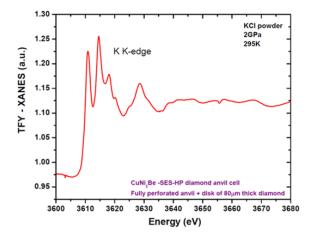


2 diodes (1 diode with a hole placed in front of the DAC and 1 diode placed behind the DAC)

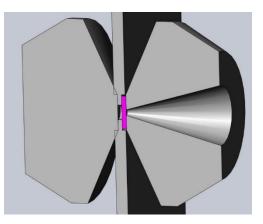
High pressure XANES and XMCD in the tender X-ray energy range <u>F.Wilhelm, G.Garbarino, J.Jacobs, H.Vitoux, R.Steinmann, F.Guillou, A.Snigirev, I.Snigireva, P.Voisin, D.Braithwaite, D.Aoki, J.-P.Brison, I.Kantor, I.Lyatun & A.Rogalev</u>

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#### Project startup ID12 - 2012.



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

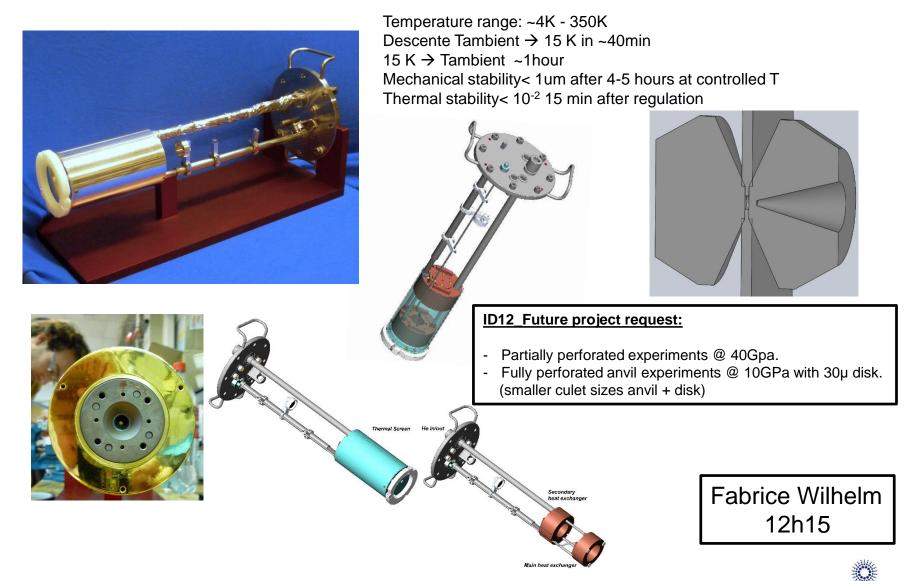


Based on ID12 experience A.Rosa is implementing same principal on ID24/BM23.

The European Synchrotron



#### **EXAMPLE SESS ENGINEERING EQUIPMENT**



The European Synchrotron | ESRF

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Introduction HP-Lab – 2006.

-Pressure range : 0.1GPa - 125 GPa
-Temperature range : 300 K to 450 K.
-Optical access to sample : Working distance ≥ 13mm.
-Accessible electromagnetic spectrum : Visible, X rays.
-Techniques: Diffraction/scattering/spectroscopy
-Materials : Maraging Steels & Tungsten Carbide
-Sizes : Diameter 50mm, Height 32mm

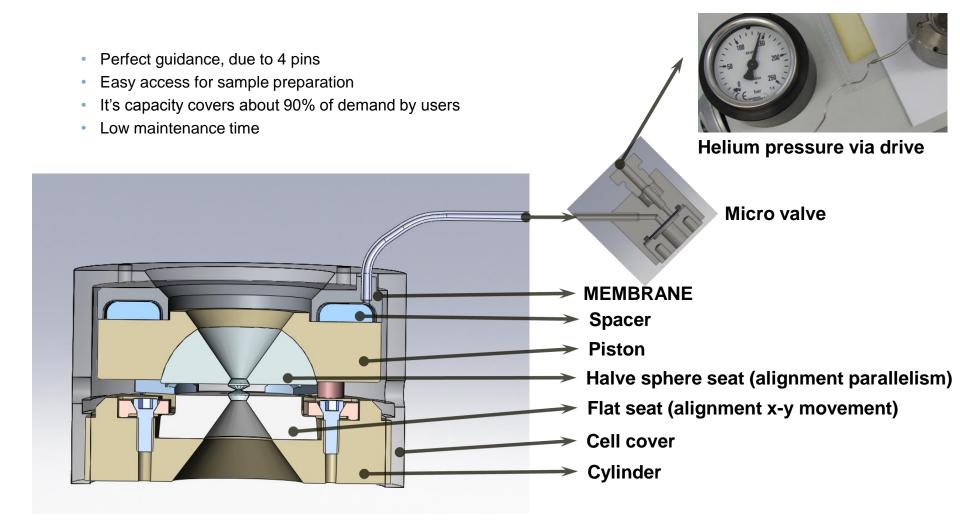








### CRYODUR, MEMBRANE DRIVEN BOEHLER-ALMAX DAC





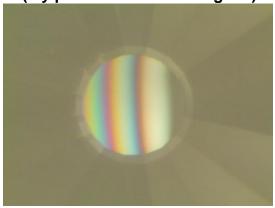
#### BOEHLER-ALMAX 250X300 MICRON (BEVELED)



1st view non aligned anvil



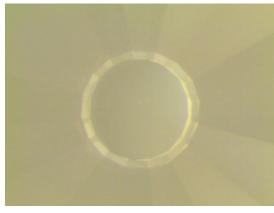
2nd view (x-y position has been aligned)



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



4th view (Fringes are gone, last x-y correction)





-Pressure range : 0.1GPa - 60 GPa

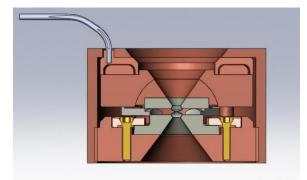
-Temperature range : 5 K to 300 K.

-Optical access to sample : Working distance ≥ 13mm

-Access electromagnetic spectrum : Visible, X rays

-Techniques: Diffraction/scattering/spectroscopy

-Sizes : Diameter 50mm, Height 32mm







#### 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.

GRADE / NUANCE / GRAD	0			
NI06				
CHEMICAL COMPOSITION / COMPOSITION CHIMIQUE / COMPO	SICION QUI	MICA		
Element	Weight %			
Tugsten carbide / Carbure de tungstène / Carburo de Tungsteno	93,9			
Binder / Liant / Ligante	Nickel	6		
Chromium Carbide / Carburo de Cromo	Cr3C2	0,1		
		1.3µm		
Grain / Grain / Grano		1.3	um	
Density / Poids Spécifique / Densidad		1,3 14,90 ± 0,1	um g/cm3	
Density / Poids Spécifique / Densidad Hardness / Dureté / Dureza				
Density / Poids Spécifique / Densidad Hardness / Dureté / Dureza Hardness / Dureté / Dureza Rockwell A		14,90 ± 0,1	g/cm3	
Density / Poids Spécifique / Densidad Hardness / Dureté / Dureza Hardness / Dureté / Dureza Rockwell A Transverse rupture strength / Résistance à la fiexion / Resistencia a F		14,90 ± 0,1 1525 ± 3%	g/cm3 HV10	
Density / Poids Spécifique / Densidad Hardness / Dureté / Dureza Hardness / Dureté / Dureza Rockwell A Transverse rupture strength / Résistance à la fexion / Resistencia a 1 Compressive strength / Résistance à la compression / Resistencia a 1		14,90 ± 0,1 1525 ± 3% 90,5 - 91,5	g/cm3 HV10 HRA	
Densty / Poids Spécifique / Densidad Hardness / Dureté / Dureza Hardness / Dureté / Dureza Rockwell A Transverse rupture strength / Résistance à la fexion / Resistencia a l Compressive strength / Résistance à la compression / Resistencia a l Young's modulus / Module d'elasticité / Modiop de Elasticidad		14,90 ± 0,1 1525 ± 3% 90,5 - 91,5	g/cm3 HV10 HRA MPa MPa Gpa	
Denský / Pokis Spécifique / Densidad Hardness / Dureté / Dureza Hardness / Dureté / Dureza Rockwell A Hardness / Dureté / Dureza Rockwell A Transverse rupture strength / Résistance à la fexion / Resistencia a i Young's modulus / Module d'elastiché / Modiop de Elasticidad Facture toughness / Dureté de rupture / Resistencia a la Rotura Ko	Compresión	14,90 ± 0,1 1525 ± 3% 90,5 - 91,5	g/cm3 HV10 HRA MPa MPa Gpa MPa vm	
Density / Poids Spécifique / Densidad Hardness / Dureté / Dureza Hardness / Dureté / Dureza Rockwell A Transverse rupture strength / Résistance à la fexion / Resistencia a 1 Compressive strength / Résistance à la compression / Resistencia a 1	Compresión	14,90 ± 0,1 1525 ± 3% 90,5 - 91,5	g/cm3 HV10 HRA MPa MPa Gpa	

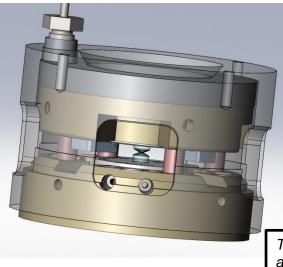
Introduction HP-Lab - 2011.

Typical microsture [2500x] / Microstructure typique [2500x] / Microestructura típica [2500x]



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### **RESISTIVE HEATING HT-DAC + VACUUM CHAMBER**



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

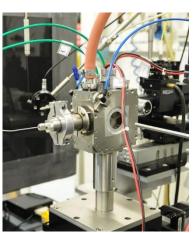
-Pressure range : 0.1GPa - 80 GPa
-Temperature range : <u>300 K to 1200° Celcius.</u>
-Optical access to sample : Working distance ≥ 13mm.
-Accessible electromagnetic spectrum : Visible, X rays.
-Techniques: Diffraction/scattering/spectroscopy
-Materials : Pyrad53NW & HT Tungsten Carbide
-Sizes : Diameter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

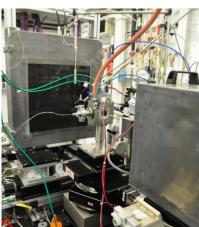
Start-up project – 2010.

*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 Autor(s): A.D.Rosa<sup>\*</sup>, M.Merkulova, G.Garbarino, V.Svytlik, J.Jacobs, O.Mathon, M.Munoz, S.Merkel









### **RESISTIVE HEATING HT-DAC + UPGRADE CONNECTORS**



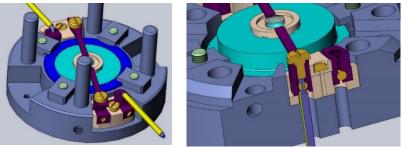


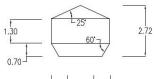
Version 2.



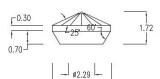
Version 3.

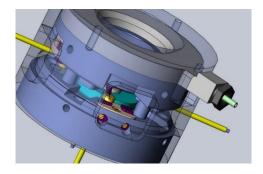












#### Resbond 931 GRAPHITE / GRAPHITE BI-COMPOSANTS

- 100% graphite
- 3000°C. max.
- Good electrical conductivity

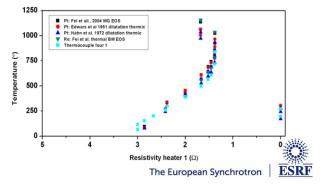


Rapport de stage de fin d'études (trainée report) du DUT Mesures Physiques, Romain Jarnias.

Conclusion: 3<sup>rd</sup> version:

- Easier to prepare
- Reusable after HT experiment

- Future heater upgrades, Zircon or Alumine, Pyrophyllite.



### HEATER RING VACUUM CHAMBER



#### ID28, HP-Lab. collaboration.

Introduction HP-Lab - 2013 (V1) - 2016 (V2)

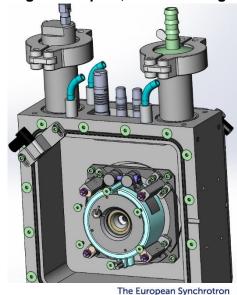
- -Pressure range : n.a
- -Temperature range : <u>Ambient to 600° Celcius.</u>
- -Optical access to sample : Working distance  $\geq$  13mm.
- -Accessible electromagnetic spectrum : Visible, X rays.
- -Techniques: Diffraction/scattering/spectroscopy
- -Materials : Stainless steel, 316L.
- -Sizes :140x154x57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

<u>Critical scattering and incommensurate phase transition in antiferroelectric PbZrO<sub>3</sub> under pressure</u> R.Burkovsky, Y.Bronwald, D.Andronikova, B.Wehinger, M.Krisch, J.Jacobs, D.Gambetti, K.Roleder, A.Majchrowski, A.Filimonov, A.Rudskoy, S.Vakhrushev, A.Tagantsev



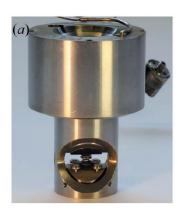


Numerously used also on ID24, BM23 and ID27. Therefor designed adapted, chamber for general-use.



ESRF

### PANORAMIC, MEMBRANE DRIVEN DAC



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  $\geq$  13mm.

-Access electromagnetic spectrum : Visible, X rays.

- -Materials : Maraging Steels & Tungsten Carbide
- -Sizes : Diameter 50mm, Total Height 69.5mm.

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

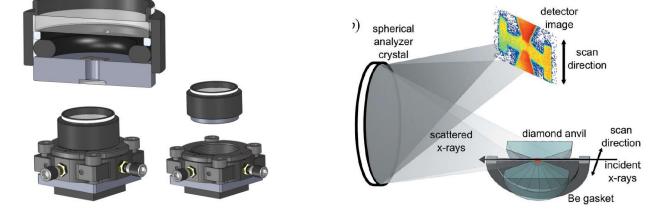
**PROTOCOL for work procedure**: Laser drilling of Beryllium gaskets at ESRF, on HP-Lab Laser drill facility + Be dedicated glovebox.

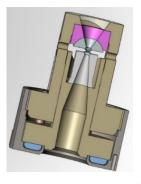




Constrains:

- 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.

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btained with this set-up on the O K-edge and e in SiO<sub>2</sub> quartz at high pressure are presented [V] and finally, section [V] gives a summary and r our new procedure.

#### THEORETICAL BACKGROUND

aman scattering is non-resonant inelastic X-ray from core electrons. The measured quantity experiment (like in all non-resonant inelastic tering experiments) is the double differential cross section<sup>[12]</sup>

$$\frac{\mathrm{d}^{2}\sigma}{\mathrm{d}\Omega\mathrm{d}\omega} = \left(\frac{\mathrm{d}\sigma}{\mathrm{d}\Omega}\right)_{\mathrm{Th}} S(\mathbf{q},\omega). \quad (1$$

)<sub>Th</sub> is the Thomson scattering cross section

$$\left(\frac{\mathrm{d}\sigma}{\mathrm{d}\Omega}\right)_{\mathrm{Th}} = r_{\mathrm{e}}^{2} \frac{\omega_{2}}{\omega_{1}} \left(\epsilon_{1} \cdot \epsilon_{2}\right)$$

(2)

lassical electron radius  $r_e$  and the polarization the incident ( $\epsilon_1$ ) and scattered beam ( $\epsilon_2$ ), and the so-called dynamic structure factor

$$\omega) = \sum_{\mathbf{i},\mathbf{f}} p_{\mathbf{i}} \left| \langle \mathbf{f} | e^{i\mathbf{q} \cdot \mathbf{r}} | \mathbf{i} \rangle \right|^2 \delta(E_{\mathbf{i}} - E_{\mathbf{f}} + \omega). \quad (3)$$

ve equations,  $\omega = \omega_1 - \omega_2$  is the energy transhe sample system during the inelastic scatterwhere  $\omega_1$  is the energy of the incident phoand  $\omega_2$  that of the scattered photons. The is over all final states  $|f\rangle$  and all initial states l by their according probability  $p_i$ . Note that e energy transfer  $\omega$ , a momentum **q** is trans-

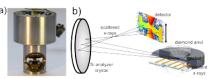
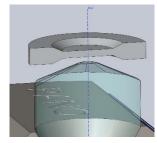


FIG. 1. a) Photograph of the membrane driven panoramic diamond anvil cell. b) Schematic image demonstrating the imaging properties: X-rays scattered at different positions along the beam are focused in a one-to-one fashion by the spherically bent Si analyzer crystal onto different positions on the detector.

spurious signal from the DAC can arise from the valence electrons of the sample environment, either collective valence excitations (dominating at small energy and momentum transfers) or Compton scattering (dominating at larger energy and momentum transfers)<sup>19</sup> Often, the signals originating from these valence electron excitations vary smoothly as a function of energy loss over the range of interest and, if kept small enough, can be subtracted from the measured signal using parameterized functions. However, typical sample sizes in high pressure experiments are few tens of microns compared to milli meters of the diamond anvils or the high-pressure gasket material (e.g. Be, Rh, BN). In more unfortunate cases, the shallow absorption edge of interest coincides with an absorption edge of the diamond anvil cell. This, for example, is the case when measuring the Si L<sub>2.3</sub>-edge (edge onset at 99.2, 99.8 eV in elemental Si and approximately



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-anvil 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 I. The used Be grades and their properties, as specified by the producer (Brush-Wellmann Company).

	Be-grades							
Properties	PF-60	S-65A	S-200F	I-70	S-200E	I-400		
wt. %:								
Be	99	98.5	99.2	99.2	98	94.8		
BeO	0.8	1.0	0.6	0.7	2.0	6.86		
Fe	0.07	0.12	0.07	0.06	0.18	0.15		
Al	0.05	0.06	0.03	0.01	0.16	0.02		
Grain size (µm)			10.3	8.1		3.9		
Density								
theor. (%)		99	99.9	99.4		99.2		
measur. (g/cm <sup>3</sup> )		1.8364		1.8863	1.845	1.882		
Ultim. tensile								
strength (GPa)		0.289			0.275			
trans.			0.413	0.399		0.539		
long.			0.391	0.377		0.630		
Yield tensile								
strength (GPa)		0.206			0.206			
trans.			0.288	0.232		0.419		
long.			0.287	0.229				
Elongation (%)		3			2.3			
trans.			6.4	7.0		8.56		
long.			4	3.9		2.8		



Start-up project - 2017.

#### ID20 request: Design new cell specialized for there current and future needs:

- Compact design allowing secondary probes:

Xray Emission Spectroscopy (XES), Xray Absorption Spectroscopy (XAS), Xray Power Diffraction (XRD), Raman, IR. X-ray Scattering (XRS) trough 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.

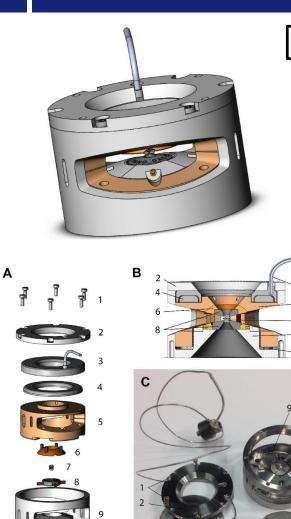




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ESRF

### PANORAMIC DAC, MBX110.



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#### ID20, ID18, ETH-Zurich (S.Petitgirard) and HP-Lab. collaboration.

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.

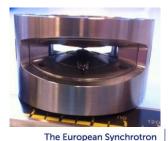
Compatible with most of the standard techniques used:

- 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.



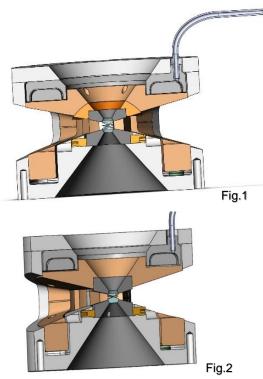




### PANORAMIC DAC, MBX110.

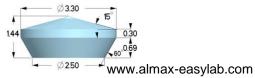
The mBX110 is available in two versions:

- Rotatable piston halve sphere seat (fig 1.)
- Fixed piston (fig.2)



- Fixed piston (possibility) radial opening angles: 85 degrees, Pmax=50Gpa.

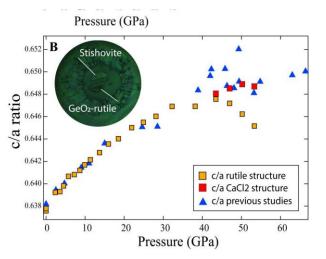
Single crystal diffraction, large angle powder diffraction, Brillouin 25 scattering spectroscopy, and off-axis laser heating.





### MBX110, PRELIMINARY TESTS DONE AT ESRF.

#### Stishovite single crystal data in Helium on ID15B



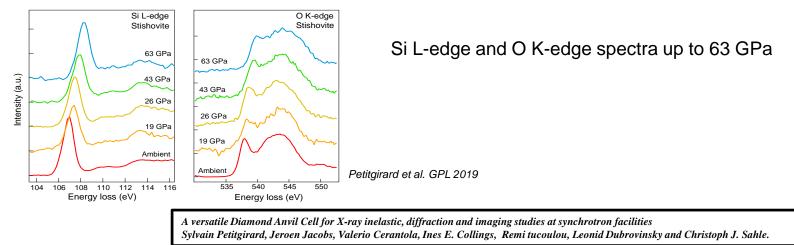
#### Rutile to CaCl2 structural change at ~ 40 GPa

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





# Thank you for your attention.

